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Thermal performance modeling and optimization of bio- and geo-based materials using a multi-scale approach : contribution to the valorization of a wide range of agricultural co-products

Séverine Rosa Latapie

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Doctorat de l'Université de Toulouse

préparé à l'Université Toulouse III - Paul Sabatier

Modélisation et optimisation des performances thermiques des matériaux bio et géosourcés par approche multi-échelle : apport à la valorisation d'une large gamme de co-produits agricoles.

Thèse présentée et soutenue, le 7 novembre 2024 par

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Membres invités

M. Vincent SABATHIER, UTTOP

A mes parents,
A Michaël,
A Bastien et Raphaël,

« Je suis de ceux qui pensent que la science est d'une grande beauté. Un scientifique dans son laboratoire est non seulement un technicien : il est aussi un enfant placé devant des phénomènes naturels qui l'impressionnent comme des contes de fées ».

Marie Skłodowska Curie

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Résumé

Construire durablement devient un enjeu stratégique face aux défis climatiques et énergétiques actuels. Les matériaux bio et géosourcés constituent des alternatives intéressantes car ils peuvent contribuer à la fois au confort intérieur et à l'efficacité du bâti. Disponibles localement, ils peuvent permettre l'émergence d'économies locales et vertueuses, respectueuses de l'environnement. Cependant, les matières premières utilisées (terre crue, paille, bois, ...) et certaines techniques de construction (pisé, bauge, torchis, ...) sont connues depuis des siècles mais sont encore peu étudiées dans la littérature.

Pourtant, la terre crue est un matériau de construction exemplaire sur bien des aspects : disponibilité, recyclabilité, faible impact environnemental, performances mécaniques et hydriques reconnues. L'incorporation de granulats végétaux à une matrice argileuse (terre allégée) permet d'atteindre de bonnes performances thermiques, grâce à une importante porosité multi-échelle. En effet, l'espace poral qui sert à la circulation de nutriments pendant la phase de croissance, se remplit d'air lorsque la plante n'est plus en terre. Ceci explique les capacités isolantes remarquables des co-produits agricoles. Cependant leur diversité, ajoutée à celle des sols utilisés en tant que matrice liante, conduit à des performances très variables selon les formulations et la nature des constituants. Le même constat peut être fait lorsque les granulats végétaux sont incorporés dans une matrice minérale (chaux, métakaolin, ...). On parle alors de bétons végétaux, utilisés en isolation répartie et principalement mis en œuvre en remplissage d'une structure porteuse en bois. Malgré des performances hygrothermiques intéressantes, la pluralité des formulations et le manque de recul sur le comportement de ces éco-matériaux freinent significativement leur utilisation à grande échelle. Bien que le béton de chanvre soit le plus étudié ces dernières années, il apparaît que d'autres co-produits agricoles que la chènevotte sont exploitables et plus largement disponibles selon le territoire considéré.

Dans ce contexte, ces travaux de recherche visent à proposer des méthodes de prédiction du comportement thermique de granulats en vrac, de bétons végétaux ou de terres allégées selon trois axes de travail :

- Compréhension des phénomènes physico-chimiques complexes qui s'opèrent selon les sollicitations extérieures (variations de température et d'humidité relative de l'air ambiant).
- Modélisation du comportement thermique des matériaux en considérant la variabilité de la ressource.
- Méthodologie d'optimisation de la formulation selon l'usage.

Afin de pouvoir considérer la nature spécifique et la complexité -tant sur le plan microstructural qu'à l'échelle macroscopique- des matériaux étudiés, un travail interdisciplinaire s'impose. Ainsi, la méthodologie développée dans cette thèse s'appuie notamment sur les domaines des sciences physiques et de la chimie en plus de celui du génie civil. Elle utilise des outils d'homogénéisation analytiques par champs moyens et des méthodes inverses communément utilisés dans d'autres disciplines scientifiques. Des

modèles de prédiction de la conductivité thermique sont développés à partir des phénomènes physico-chimiques qui se produisent sous les contraintes d'usage (température et humidité relative). Une attention particulière est portée sur la cohérence de ces modèles vis-à-vis de la réelle morphologie des matériaux étudiés. Par ailleurs, la prise en compte de la variabilité de la ressource est un point fondamental pour contribuer au développement de ces éco-matériaux, dont l'utilisation est favorisée par l'entrée en vigueur d'une nouvelle Règlementation Environnementale (RE 2020) depuis le 1^{er} janvier 2022 en France. Ainsi, ces travaux s'articulent à chaque échelle du matériau pour tenter de lever différents verrous identifiés dans la littérature.

D'abord, l'échelle moléculaire est explorée. On note que granulats végétaux sont considérés dans la littérature comme un granulats inertes. Or, son origine biologique lui confère des affinités particulières avec l'eau. Ce caractère hydrophile mène à une réflexion argumentée afin de proposer une nouvelle explication au phénomène d'hystérésis basé sur un couplage hydro-mécanique à l'échelle moléculaire. Cette compréhension des mécanismes de fixation de l'eau dans l'espace poral des particules végétales est fondamentale pour une prise en compte pertinente à l'échelle particulaire, sous sollicitation hydrique.

Ensuite, l'étude de l'échelle particulaire s'avère stratégique. La littérature souligne la diversité des propriétés d'une chènevotte à l'autre notamment, due à des conditions météorologiques, des lieux, des méthodes ou encore des périodes de récolte différentes. A la recherche d'un point commun pour contourner la difficulté liée à la variabilité, les microstructures de différents co-produits agricoles sont analysées. On note que la composition chimique du squelette solide est très semblable pour tous les granulats lignocellulosiques, principalement constitués de cellulose. La variabilité intrinsèque des granulats est ainsi corrélée à leur porosité intra-particulaire, donnée accessible expérimentalement grâce à des travaux récents. En considérant la réelle morphologie de chaque type de particule végétale, des processus d'homogénéisation successifs donnent accès à la conductivité thermique particulaire selon les sollicitations extérieures (température et humidité relative).

Grâce à la connaissance des propriétés thermiques à l'échelle particulaire, une dernière étape d'homogénéisation permet d'accéder à l'échelle matériau. Pour valider les modèles prédictifs développés, une campagne expérimentale est menée sur des panneaux isolants à base de moelle de tournesol. Un très bon accord entre valeurs expérimentales et valeurs modélisées permet d'asseoir la pertinence des méthodes utilisées. Par ailleurs, dans le cadre de ces travaux expérimentaux, une technique de mise en œuvre innovante est mise au point. En effet, pour minimiser leur impact environnemental, les panneaux isolants sont fabriqués sans aucun liant ni procédé énergivore.

Enfin, à partir des résultats obtenus aux différentes échelles, la variabilité de la conductivité thermique d'un béton végétal ou d'une terre allégée peut être anticipée avant même l'étape de fabrication et sur toute la gamme d'usage. L'optimisation de la formulation devient une réalité et différentes pistes sont explorées en ce sens (changement de liant, de granulats, orientation des granulats...). En complément, l'amorce d'une analyse multicritère est proposée pour une réflexion et une optimisation plus globale sur ces matériaux, au-delà de l'aspect thermique.

Ces travaux de thèse proposent des méthodes inédites de prédiction du comportement thermique des matériaux bio et géosourcés, s'appuyant sur la réelle morphologie de composites incorporant des co-produits agricoles. Ils ouvrent la voie vers une utilisation massive des granulats végétaux dans les matériaux de construction bio et géosourcés. L'approche multi-échelle et multi-physique menée démontre l'intérêt d'un travail collaboratif et interdisciplinaire afin de mieux appréhender le comportement complexe de ces éco-matériaux. L'accessibilité des données d'entrée de la modélisation est un point fondamental. Elle permet une applicabilité rapide de cette contribution, au plus près des besoins des acteurs d'une économie locale de la construction. Elle répond également à l'urgence climatique et environnementale à laquelle nos sociétés sont confrontées. Dans le contexte actuel, ces travaux peuvent aider de manière significative à la transition écologique du secteur du bâtiment qui se doit d'être à la fois rapide, efficace et pérenne.

Mots-clés

Matériaux de construction bio-sourcés, Modélisation multi-échelle, Propriétés thermiques, Granulat végétal, Matériaux de construction géo-sourcés, Microstructure.

Abstract

Sustainable construction is becoming a strategic issue in today's climate and energy challenges. Bio- and geosourced materials are interesting alternatives, as they can contribute to both indoor comfort and building efficiency. They are locally available, enabling the emergence of virtuous local economies that respect the environment. However, the resources used in building materials (raw earth, straw, wood, etc.) and certain construction processes (rammed earth, cob, wattle and daub, etc.) have been known for centuries but remain little studied in the literature.

In many respects, raw earth is an exemplary building material: availability, recyclability, low environmental impact, recognized mechanical and hydric performance. The incorporation of plant aggregates into a clay matrix (lightened earth) ensures good thermal performance, thanks to high multi-scale porosity. In fact, the pore spaces, which circulate nutrients during the growth phase of plants, fill with air when the plant is no longer in the ground. However, the diversity of these by-products, combined with the variability of soils used as binder matrices, leads to highly variable performance depending on the formulations and nature of the constituents. The same observation can be made when plant aggregates are incorporated into a mineral matrix (lime, metakaolin, etc.). Plant-based concretes are used for distributed insulation and are mainly used to fill load-bearing wooden structures. Despite interesting hygrothermal performances, their widespread use is significantly slow down by the wide range of formulations and the lack of feedback concerning the behavior of these eco-materials. Whereas hemp concrete has been the most widely studied material in recent years, it appears that agricultural by-products other than hempcrete can be exploited and may be more widely available, depending on the given region.

In this context, the aim of this research work is to propose methods for predicting thermal behavior of bulk aggregates, plant-based concretes and lightweight earth along three lines:

- Understanding the complex physico-chemical phenomena involved under external stresses (variations in temperature and relative humidity of the ambient air).
- Modeling the thermal behavior of materials taking into account resource variability.
- Methodology for optimizing formulation according to use.

In order to consider the specific nature and complexity - both at microstructural and macroscopic levels - of the materials studied, interdisciplinary work is essential. The methodology developed in this thesis therefore combines the fields of physics and chemistry with that of civil engineering. It uses mean-field analytical homogenization tools and inverse methods commonly used in other scientific disciplines. Thermal conductivity prediction models are developed on the basis of physico-chemical phenomena occurring under usage conditions (temperature and relative humidity). Particular attention is paid to ensuring that models are consistent with the actual morphology of the materials studied.

In addition, taking into account the variability of the resource is a key factor in the development of these eco-materials. Their use is encouraged by the entry into force of new

Energy Regulations (RE 2020) on January 1, 2022 in France. Thus, this work is structured around each scale of the material in an attempt to overcome the various obstacles identified in the literature.

First, the molecular scale is explored. The literature considers plant aggregates to be inert. However, its biological origin gives it a particular affinity with water. This hydrophilic character suggests a new explanation for the hysteresis phenomenon, based on hydro-mechanical coupling at the molecular level. Understanding the mechanisms of water binding within the pore space of plant particles is crucial for considering these effects at the particle scale under water sollicitation.

Secondly, the particle scale is of strategic importance. The literature highlights the diversity of properties from one hemp shiv to another, due in particular to different meteorological and geographic conditions, methods and harvesting periods. In search of a common ground to overcome the difficulty of variability, the microstructures of different agricultural co-products are analyzed. The chemical composition of the solid skeleton is very similar for all lignocellulosic aggregates, mainly made up of cellulose. The intrinsic variability of the aggregates is thus correlated to their intra-particle porosity. This parameter is experimentally accessible thanks to recent work. By considering the actual morphology of each type of plant particle, successive homogenization processes provide access to particle thermal conductivity according to external loadings (temperature and relative humidity).

Knowledge of thermal properties at the particle scale enables a final homogenization step to access the material scale. To validate the developed predictive models, an experimental campaign is carried out on sunflower pith insulating panels. A very good agreement between experimental and modelled values confirms the relevance of the proposed methods. In addition, an innovative implementation technique is developed during the experimental work. To minimize environmental impact, insulating panels are manufactured without binders or energy-consuming processes.

Finally, based on the results obtained at different scales, the variability of the thermal conductivity of a plant-based concrete or lightweight earth can be anticipated even before the manufacturing stage and over the entire range of uses. Formulation optimization is becoming a reality, and various avenues are explored in this direction (change of binder, aggregate, orientation of aggregates, etc.). In addition, a multi-criteria analysis is proposed beyond the thermal aspect for a more global approach to the optimization of these materials.

This thesis proposes new ways of predicting the thermal behavior of bio- and geo-based materials, exploiting the real morphology of composites incorporating agricultural co-products. This work paves the way for the widespread use of plant aggregates in bio- and geo-based building materials.

The multi-scale and multi-physics approach demonstrates the value of collaborative and interdisciplinary work to better understand the complex behavior of these eco-materials. The accessibility of modeling input data is a fundamental point. It enables this contribution to be applied rapidly, as close as possible to the needs of local construction industry players. It also addresses the urgent climatic and environmental issues facing our societies. In the current

context, this work can make a significant contribution to the ecological transition of the building sector, which needs to be rapid, efficient and sustainable.

Keywords

Bio-based building materials, Multiscale modeling, Thermal properties, Bio-aggregate, Geo-based building materials, Microstructure.

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Chapitre 1. Introduction, enjeux et méthodologie

1.1 Contexte global et enjeu climatique

Un des enjeux majeurs du XXI^{ème} siècle est sans conteste celui du réchauffement climatique. L'effet de serre « naturel » est pourtant bien celui qui a permis à notre planète Terre de voir la vie se développer [1], [2]. La biodiversité de notre planète bleue est le fruit d'une combinaison complexe de phénomènes physico-chimiques en un point précis de l'Univers. Alors que la communauté scientifique s'interroge encore sur les mécanismes intrinsèques de l'étoile, communément appelée Soleil, qui nous permet d'exister [3], [4], [5], force est de constater qu'« aucun autre vaisseau spatial » connu ne saurait aujourd'hui accueillir la vie au sens où nous, êtres humains, nous la définissons [6]. Pourtant, l'effet de serre additionnel d'origine anthropique [7] menace de manière tangible notre existence ainsi que l'ensemble des écosystèmes terrestres [8], [9], [10]. Limiter le réchauffement climatique et ses conséquences est devenu un enjeu crucial. La littérature, dans de nombreux domaines, dresse les problématiques qui pourraient émerger face à ce bouleversement inédit de notre planète. Le danger pèse de manière significative sur la pérennité des espèces animales et végétales, et ce de manière multifactorielle [11], [12]. Des travaux récents posent notamment la question de la capacité d'adaptation de l'espèce humaine exposée à des températures extrêmes et à l'apparition de nouveaux agents pathogènes [13]. Par ailleurs, la vulnérabilité de la biodiversité pourrait être à l'origine de maladies chroniques, dégradant de manière notable la qualité de vie des jeunes générations [14]. Les conséquences sanitaires et sociétales du changement climatique sont significativement inquiétantes : manque d'eau potable, insécurité alimentaire, risque accru de conflits [15], [16], [17]. Nos sociétés sont aujourd'hui dans une ère charnière : il convient d'appréhender l'enjeu climatique sur le plan politique, socio-économique et scientifique pour actionner tous les leviers d'action à notre disposition [18], [19].

Les nouvelles générations semblent particulièrement sensibles aux questions environnementales, soucieuses de s'engager pour agir face au changement climatique annoncé [20], [21]. Le rôle des sciences, que ce soit au niveau de l'enseignement ou de la recherche, prend alors tout son sens dans l'accompagnement de la transition éco-responsable de notre société [22], [23]. En réponse aux actuels défis environnementaux et au besoin d'innovation qui en découle, produire des connaissances et lever les verrous scientifiques persistants doivent devenir des priorités pour limiter le réchauffement climatique.

Notons que le secteur de la construction est l'un des plus impactant sur le plan environnemental [24]. Il représente à lui seul 36 % de la consommation énergétique mondiale et 40 % à l'échelle européenne [25], [26]. Par ailleurs, il contribue majoritairement aux émissions de gaz à effet de serre (GES), responsable d'environ 40 % des émissions globales de dioxyde de carbone [27]. Cette situation résulte notamment d'une utilisation massive de matériaux dits « conventionnels ». Les matériaux de construction bio-sourcés (utilisant des ressources végétales issues de l'agriculture, de la sylviculture ou du milieu naturel) et géo-

sourcés (à base de terre crue) constituent une alternative intéressante pour diminuer l’empreinte carbone du bâti tout en offrant des performances comparables aux techniques constructives actuelles, en particulier sur le plan thermique [28], [29], [30]. Toutefois, leur utilisation reste limitée eu égard à différents freins comme la diversité de la ressource, le manque de structuration de filières, la complexité du comportement des matériaux, le caractère chronophage et coûteux de leur caractérisation ou, de manière plus surprenante, l’image péjorative qu’ils peuvent renvoyer [31], [32], [33].

1.2 Contexte politique et réglementaire

La prise de conscience collective du réchauffement climatique et de ses conséquences est assez récente [34]. Aussi, les stratégies politiques internationales ne commencent à se coordonner sur ce sujet que depuis le début des années 90. La Commission Mondiale sur l’Environnement et le Développement (CMED, 1983-1987) de l’Organisation des Nations Unies (ONU) publie un rapport intitulé « Our common future » en 1987. Aussi appelé « Rapport de Brundtland », ce dernier marque un tournant décisif de l’Histoire en introduisant la notion de « développement durable » [35], [36]. Selon ce texte fondateur, il est défini comme « un développement qui répond aux besoins du présent sans compromettre la capacité des générations futures de répondre aux leurs » [37]. En 1992, le sommet de la Terre de Rio, pose les jalons des engagements internationaux en faveur du développement durable à travers notamment la Convention-Cadre des Nations Unies sur le Changement Climatique (CCNUCC). Il faut attendre la 3^e conférence des parties (COP 3), organisée par les Nations Unies en 1997, pour fixer des objectifs chiffrés au niveau international : 38 pays s’engagent par le protocole de Kyoto à réduire de 5,2 % en moyenne l’émission des GES pour l’année 2012 par rapport au niveau de 1990. En 2012, la COP 18 prolonge et révisé ces objectifs avec une réduction de 18% entre 2013 et 2020 (toujours par rapport à 1990) [38]. La COP 21 ratifie l’Accord de Paris en 2015 avec une cible nouvelle : « maintenir l’augmentation de la température mondiale en dessous de 2°C et poursuivre les efforts pour la limiter à 1,5°C par rapport aux niveaux préindustriels » [39] [40]. La Déclaration de Chaillot de mars 2024 vient concrétiser la mise en route opérationnelle de cette collaboration internationale [41]. Au niveau européen, la déclinaison des accords internationaux se traduit notamment par l’adoption du « Paquet Energie-Climat 2030 ». L’Union Européenne s’engage ainsi à réduire d’au moins 55 % l’émission des GES pour 2030 (par rapport à 1990) et à atteindre la neutralité climatique d’ici 2050 [42]. Il s’agit de compenser la quantité de GES émise dans l’atmosphère par des puits naturels (espaces végétalisés, bocages, phytoplancton, etc.) [43] ou le recours à de nouvelles technologies (captage du dioxyde de carbone, transformation des GES, ...). Cet objectif devient aujourd’hui un véritable enjeu politique [44] mais cette transition passe nécessairement par l’apport de nouvelles connaissances scientifiques et le développement de technologies innovantes [45].

Les politiques environnementales se déclinent de l’échelle internationale à l’échelle nationale : elles prennent alors tout leur sens en termes de mise en application. Une des articulations au niveau du territoire français, qui découle des engagements internationaux, est la RE 2020 (Règlementation Environnementale). Elle est entrée en vigueur sur le territoire national français en janvier 2022 et se distingue notablement des réglementations thermiques (RT 2005, RT 2012) antérieures [46]. La nouveauté réside dans les valeurs seuils fixées par la RE 2020 : elles favorisent le confort d’été ainsi que le faible impact environnemental du bâti.

Désormais, de nouveaux indicateurs permettent d'apprécier les performances du projet de construction sur le plan énergétique. Cette nouvelle dynamique réglementaire impose que les matériaux de construction soient à la fois performants sur le plan hygrothermique et à faible impact environnemental. Les matériaux bio et géo-sourcés répondent à ces critères ; ils sont donc très prometteurs dans le développement des solutions constructives de demain [47]. La démarche d'Analyse de Cycle de Vie (ACV) imposée par la RE 2020 est un levier pour promouvoir leur utilisation à grande échelle [48]. Toutefois, bien que la terre soit utilisée dans la construction depuis des millénaires [49], les techniques de construction en terre crue ne sont pas entièrement couvertes par des règles professionnelles [50]. On recense actuellement un guide de bonnes pratiques de construction en terre crue couvrant notamment les techniques de bauge, d'enduit en terre ou encore de terre allégée [51]. Des règles professionnelles spécifiques aux enduits sur supports de terre crue ne sont disponibles que depuis 2020 [52]. Les matériaux bio-sourcés, souffrent également d'un manque de textes réglementaires. Des Règles professionnelles de Construction en Paille (RFCP éditées en 2012 et révisées en 2018) [53] et un guide de bonnes pratiques de la construction en chanvre (édité en 2008 et révisé en 2023) [54] sont les seuls documents réglementaires référencés à ce jour. Compte tenu du cadre assurantiel en France, un projet de construction est dit de « technique courante » - et donc facilement assurable- lorsqu'il se réfère à des solutions techniques normalisées (Normes Françaises Document Technique Unifié, Règles Professionnelles acceptées par la Commission Prévention Produit, Eurocodes) ou couvertes par une Appréciation Technique d'Expérimentation (ATEX) [55]. A défaut, le projet entre dans la catégorie de « technique non courante ». L'assurabilité demande alors une analyse de risques ce qui peut générer un surcoût pour le projet et être un frein à l'utilisation des matériaux bio et géo-sourcés.

1.3 Contexte scientifique

Les travaux de recherche concernant les matériaux de construction bio et géo-sourcés émergent globalement depuis le début des années 2000. Le bois et la chènevotte (co-produit de la culture du chanvre) sont les ressources les plus étudiées à ce jour. La technique de construction en pisé (coffrage de terre crue) fait également l'objet de nombreux travaux récents [56]. Différents projets encouragent actuellement la production de connaissances scientifiques, l'objectif est de contribuer à lever les freins techniques, logistiques et réglementaires des solutions constructives à base de terre crue ou de granulats végétaux. Il s'agit, par exemple, du Projet National Terre Crue [57], du Projet Bauges Porteuses [58] ou encore du projet LOCABATI (Apport à la connaissance des performances réelles - hygrothermiques, acoustiques et de comportement au feu- des matériaux de construction low-tech locaux et bio-sourcés par approche multi-échelles). En parallèle, des groupes de travail s'articulent autour de ces thématiques de recherche grâce au Groupement de Recherche, initialement axé sur les matériaux bio-sourcés (GDR Matériaux Bio-Sourcés lancé en 2020). Ce dernier évolue pour intégrer les matériaux géo-sourcés à partir de 2025 (GDR Matériaux Bio et Géo-Sourcés). Des comités techniques émergent également ces dernières années au niveau de la RILEM (Réunion Internationale des Laboratoires et Experts des Matériaux, systèmes de construction et ouvrages) sur la thématique des éco-matériaux [59], [60].

Concernant les matériaux à base de terre crue, la littérature, souligne que la terre la plus adaptée pour la construction est celle issue des premières couches du sous-sol, dite « terre minérale » [61]. Ainsi les terres d'excavation représentent un potentiel important et leur disponibilité directe sur le chantier permet de minimiser notamment les coûts de transport et, de surcroît, l'impact environnemental qui en découle. Elles constituent une solution constructive pertinente pour les pays industrialisés qui doivent gérer de grandes quantités de terre lors de la réalisation d'ouvrages, notamment pour la réalisation des fondations de bâtiments [62] ou d'infrastructures de transport [63], [64]. Elles ont également toute leur place dans un mode constructif peu coûteux pour des territoires défavorisés, favorisant l'émergence d'économies circulaires de la construction [33]. Dans une logique de gestion et de valorisation des déchets, les fines de lavage des granulats calcaires utilisés dans l'industrie du béton sont également utilisables [65]. La ressource pour construire en terre est donc aujourd'hui abondante. La terre crue peut être utilisée en tant que matériau structurel en utilisant les techniques relatives au pisé, à l'adobe ou encore à la brique compactée (BTC) [66], [67]. L'ajout de granulats végétaux (on parle alors de terre allégée) dégrade les résistances mécaniques du matériau terre tout en améliorant sa ductilité et diminuant sa conductivité thermique, lui conférant plutôt une fonction d'isolation répartie [68]. En complément, grâce à la bonne inertie du matériau terre, les consommations énergétiques du bâti peuvent être réduites, notamment lors des épisodes de forte chaleur [69]. Les parois en terre crue participent donc au confort mais également à la qualité de l'air intérieur des constructions. Elles offrent des propriétés remarquables d'isolation acoustique et de régulation hygrique [70].

Concernant les matériaux bio-sourcés, les travaux de la littérature mettent en évidence un intérêt marqué pour la chènevotte. Le chanvre est utilisé notamment dans l'industrie textile et plus récemment dans le bâtiment sous forme de panneaux isolants (laine de chanvre) [71], [72]. La chènevotte, en tant que granulat végétal, peut être utilisée en l'état pour de l'isolation en vrac ou incorporée à une matrice liante cimentaire pour fabriquer un béton de chanvre [73], [74]. Toutefois, le chanvre est loin d'être largement disponible sur tous les territoires [75]. Aussi, de plus en plus de travaux se concentrent sur des ressources exploitables localement comme les co-produits issus de la culture du lin, du tournesol, du maïs ou encore du riz [76], [77], [78], [79], [80]. Le potentiel de plantes sauvages telles que le roseau ou le miscanthus est également évalué dans des travaux récents [81], [82]. Il convient de différencier les granulats végétaux des fibres végétales, dont la granulométrie et les propriétés sont différentes [83], [84]. Les granulats végétaux révèlent des capacités d'isolation thermique [71] tandis que les fibres tendent à améliorer les performances mécaniques des matériaux [85], [86]. Par ailleurs, des réactions délétères sont rapportées dans certains cas : les constituants des granulats végétaux (dits lignocellulosiques en référence à leur composition en lignine et cellulose) peuvent retarder la prise des matrices hydrauliques lors de la fabrication des bétons végétaux et impacter défavorablement leurs propriétés mécaniques [26], [79]. Dans tous les cas, leur compressibilité élevée limite, à ce jour, leur utilisation en tant qu'éléments porteurs [87]. Ils sont plutôt utilisés en remplissage d'une structure (bois par exemple) pour assurer une fonction d'isolation répartie [25] mais ils pourraient également s'avérer efficaces dans le contreventement face au risque sismique [88]. A ce jour, les travaux de la littérature se concentrent principalement sur les aspects expérimentaux et notamment sur la détermination de la conductivité thermique ou encore de la capacité tampon hygrique

[25], [78], [89]. Quelques références de la littérature donnent des éléments sur les propriétés acoustiques des composites à base de granulats végétaux, en soulignant des performances prometteuses sur ce point [90], [91], [92]. Le comportement au feu des matériaux bio et géo-sourcés est également un sujet émergent de la littérature [93], [94], [95]. Par ailleurs, de nombreux auteurs travaillent à la compréhension et à la modélisation des transferts d'air, d'eau et d'humidité qui ont lieu naturellement dans ces matériaux sous les sollicitations d'usage [96], [97], [98], [99], [100], [101], [102]. Le but est de mieux prendre en compte cette contribution dans l'analyse des performances énergétiques à l'échelle du bâti voire à l'échelle urbaine [103] [104]. Quelques travaux émergent également sur la modélisation du comportement thermique ou mécanique des matériaux bio-sourcés, se concentrant uniquement sur la chènevotte [105], [106], [107]. Enfin, il est intéressant de remarquer que la valeur expérimentale de conductivité thermique, à l'échelle d'un granulat végétal, n'est disponible dans la littérature que pour ce type de granulat. Cela s'explique probablement par la complexité de la technique à mettre en œuvre, la thermographie infra-rouge, pour accéder à cette valeur microstructurale [108].

Les propriétés hygro-thermiques et le faible impact environnemental des matériaux bio et géo-sourcés sont aujourd'hui reconnus. Cependant, une meilleure connaissance de ces matériaux s'impose pour conduire à une normalisation des techniques de construction [109]. Le monde scientifique s'empare de cette problématique, probablement convaincu de leur fort potentiel. Ainsi, la dynamique des publications sur le sujet est très favorable ces dernières années [56]. Cela permet d'espérer des avancées significatives concernant ces éco-matériaux dans un futur proche.

1.4 Identification des verrous scientifiques

Aujourd'hui, de nombreuses interrogations subsistent sur les matériaux bio et géo-sourcés. Parmi les différents verrous qui restent à lever pour contribuer à leur développement et ainsi favoriser une utilisation plus large, les points suivants peuvent être mis en avant :

- **Nature spécifique des granulats végétaux.** La nature ligno-cellulosique des granulats exige une réflexion basée sur différents domaines comme la biologie la physique du vivant ou encore la chimie. L'impact de la dégradation naturelle au champ (rouissage avant récolte), du procédé de défibrage et de pré-traitement ou encore la réactivité avec les matrices liantes hydrauliques sont encore mal appréhendés [111], [112].
- **Variété et variabilité des granulats exploitables.** Le grand nombre de granulats végétaux exploitables (chènevotte, lin, écorce ou moelle de tournesol, balle de riz, spathes de maïs, ...) ajouté à la diversité de chaque espèce démultiplie la variabilité potentielle de la ressource et donc des matériaux de construction qui en résulte [113]. Cela complexifie l'étude des propriétés d'usage et le processus d'optimisation des formulations notamment.
- **Anisotropie multi-échelle.** La microstructure tubulaire de la plupart des granulats induit une anisotropie à très petite échelle [114]. L'orientation préférentielle des granulats, lors de la mise en œuvre, induit également une anisotropie à l'échelle

macroscopique [88], [115], [116]. Le caractère anisotrope se traduit ainsi à différentes échelles et s'étend, logiquement, à toutes les propriétés des matériaux bio-sourcés, compliquant ainsi le processus de caractérisation et de prédiction de leur comportement [117].

- **Couplage de phénomènes complexes.** A cause notamment de leur porosité multi-échelle [118], [119], les phénomènes physiques qui s'opèrent dans ces matériaux doivent être décrits de manière différente, i.e. de manière adaptée à l'échelle considérée [120]. De plus, des couplages s'opèrent à la fois entre les échelles et entre les phénomènes physiques, dans les matériaux étudiés. Les observations faites sous les sollicitations d'usage illustrent le niveau de complexité à traiter. Les sollicitations extérieures ont des conséquences différentes selon l'échelle considérée : gonflement et rétrécissement local à l'échelle des particules [96], [121], transfert hygrothermique à l'échelle de la paroi [122]). Le lien entre les différentes échelles est encore mal connu.
- **Durabilité difficile à appréhender.** Les données sur le vieillissement des matériaux bio-sourcés sont quasi-inexistantes dans la littérature. En effet, leur étude étant relativement récente à ce jour, les protocoles de vieillissement normalisés font défaut [123], [124], [125]. Il est donc aujourd'hui très difficile de comprendre et de prédire comment évoluent les propriétés d'usage de ces matériaux. Notons également que très peu d'études traitent de la recyclabilité de ces matériaux notamment lorsqu'ils sont adjuvés.
- **Filières et acteurs en devenir.** La structuration des filières est encore au stade embryonnaire, excepté pour le chanvre dont l'économie se développe maintenant depuis plusieurs décennies [126]. Pourtant l'articulation des différents acteurs, de la récolte à la mise en œuvre chantier, est essentielle pour contribuer au développement de ces éco-matériaux [77], [125].

Les travaux présentés ici se concentrent sur la prédiction du comportement thermique des matériaux bio et géo-sourcés en considérant, en particulier, les verrous liés à la nature des granulats végétaux, leur variabilité, l'anisotropie multi-échelle et le couplage des phénomènes physiques à différentes échelles.

Cette thèse s'intéresse en particulier à l'utilisation, pour un usage d'isolation, de granulats végétaux en vrac, incorporés dans des matrices liantes cimentaires (pour fabriquer des bétons végétaux) ou dans une matrice à base de terre crue (pour obtenir des terres allégées). Les performances thermiques des matériaux bio et géo-sourcés s'expliquent par un réseau poreux complexe et multi-échelle. Ainsi l'étude se focalise notamment sur la porosité particulaire (au sein même du granulats végétal), la porosité interparticulaire (entre les granulats végétaux) et celle à l'interface entre le liant et le granulats ou encore au sein même de la matrice liante [110].

En toute rigueur, le transfert de chaleur au sein d'un matériau résulte de trois phénomènes : conduction, convection et rayonnement [127]. Le transfert thermique par

conduction est le seul considéré dans cette thèse, en première intention. En effet, le transfert thermique par convection est négligeable dans le type de matériaux considéré ici [90]. Le transfert par rayonnement n'est pas considéré dans la littérature concernant les bétons végétaux [25]. Toutefois, il pourrait être non négligeable pour des matériaux à faible masse volumique comme les laines végétales [90].

1.5 Méthodologie de thèse

Dans ce contexte, ces travaux de recherche visent à contribuer au développement des matériaux de construction bio et géo-sourcés en s'articulant autour de trois axes principaux de travail qui sont récapitulés dans la Figure 1.

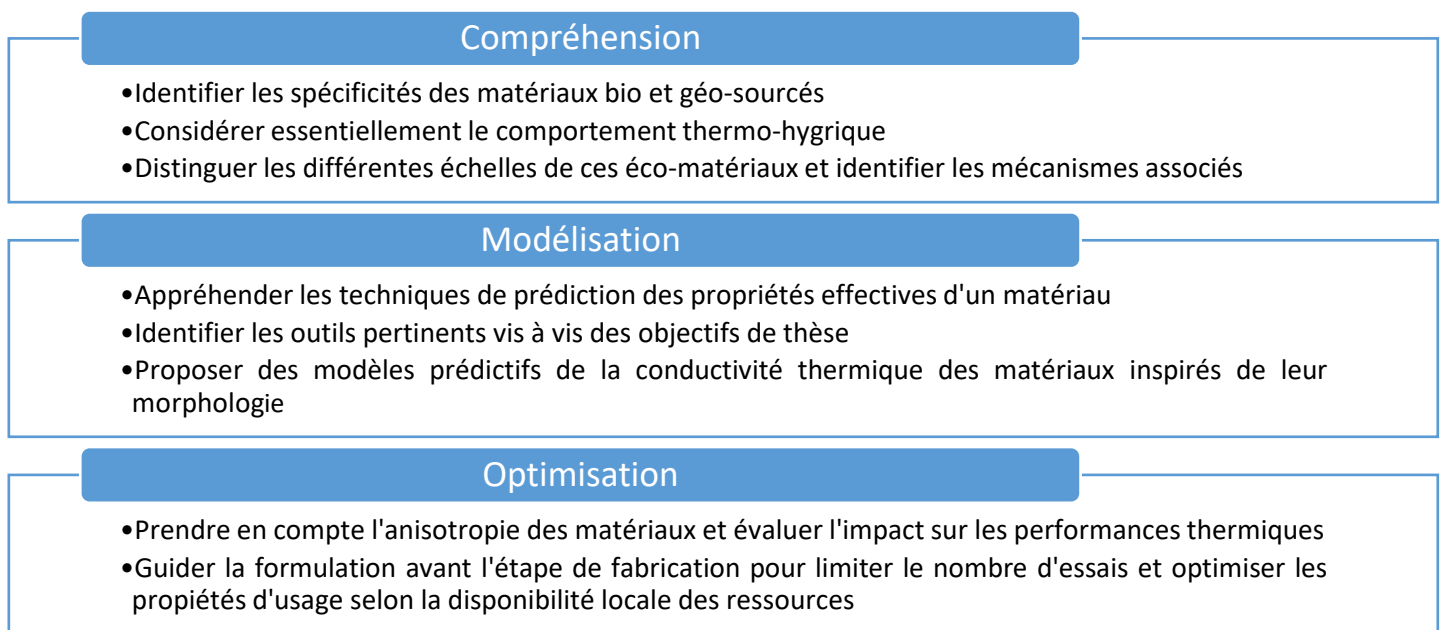


Figure 1. Axes de travail des travaux menés dans cette thèse.

En outre, pour contribuer à lever les verrous identifiés précédemment (section 1.1.4), les points suivants ont fait l'objet d'une attention particulière et ont permis d'orienter les travaux de recherche :

- **Réalisation d'une étude bibliométrique** pour identifier les thématiques peu explorées dans le domaine des matériaux bio et géo-sourcés et proposer de nouveaux « angles d'attaques » pour progresser dans la connaissance de ces matériaux.
- **Considération de la nature organique des granulats végétaux** pour envisager de nouvelles pistes dans la compréhension de phénomènes encore mal connus comme l'hystérésis par exemple. Cela impose de mobiliser des connaissances en chimie et de considérer la microstructure des granulats végétaux dès l'échelle moléculaire.
- **Développement de modèles de prédiction du comportement thermique** et étude de leur pertinence sur toute la gamme d'usage (température et humidité relative) par

comparaison à des valeurs expérimentales (issues de ces travaux de thèse et de la littérature). Le choix des techniques de modélisation est motivé par un usage à la fois simple (temps de calcul limité) et pertinent (adéquation avec le matériau réel). De plus, l'approche multi-échelle, qui guide ces travaux, nécessite de pouvoir passer relativement facilement d'une échelle à l'autre. L'homogénéisation par champs moyens est ainsi retenue, bien que la connaissance des propriétés de chaque phase - condition nécessaire à l'utilisation de cette technique- ne soit pas toujours connue. Ceci constitue une difficulté majeure dans ces travaux. Une fois ces points délicats traités, les méthodes de modélisation sont validées et utilisées comme outil d'optimisation de la formulation des éco-matériaux (béton végétal ou terre allégée). Un travail préalable et approfondi sur les données d'entrée pour la modélisation permet ensuite de considérer la diversité de la ressource mais également les effets d'anisotropie, enjeux stratégiques des éco-matériaux considérés dans cette thèse.

Enfin, ces travaux sont menés dans un souci de cohérence, de l'échelle la plus fine - l'échelle moléculaire- à l'échelle macroscopique – celle du matériau. Les valeurs modélisées sont systématiquement comparées aux valeurs expérimentales, dès lors qu'elles sont disponibles dans la littérature. L'objectif est de pouvoir apprécier de manière objective la pertinence des travaux menés dans l'état actuel des connaissances. En effet, les méthodes de mesure de la conductivité thermique questionnent encore à ce jour les expérimentateurs [78], [128], [129].

La méthodologie multi-échelle développée dans cette thèse et les objectifs ciblés à chacune des étapes sont résumés dans la Figure 2.

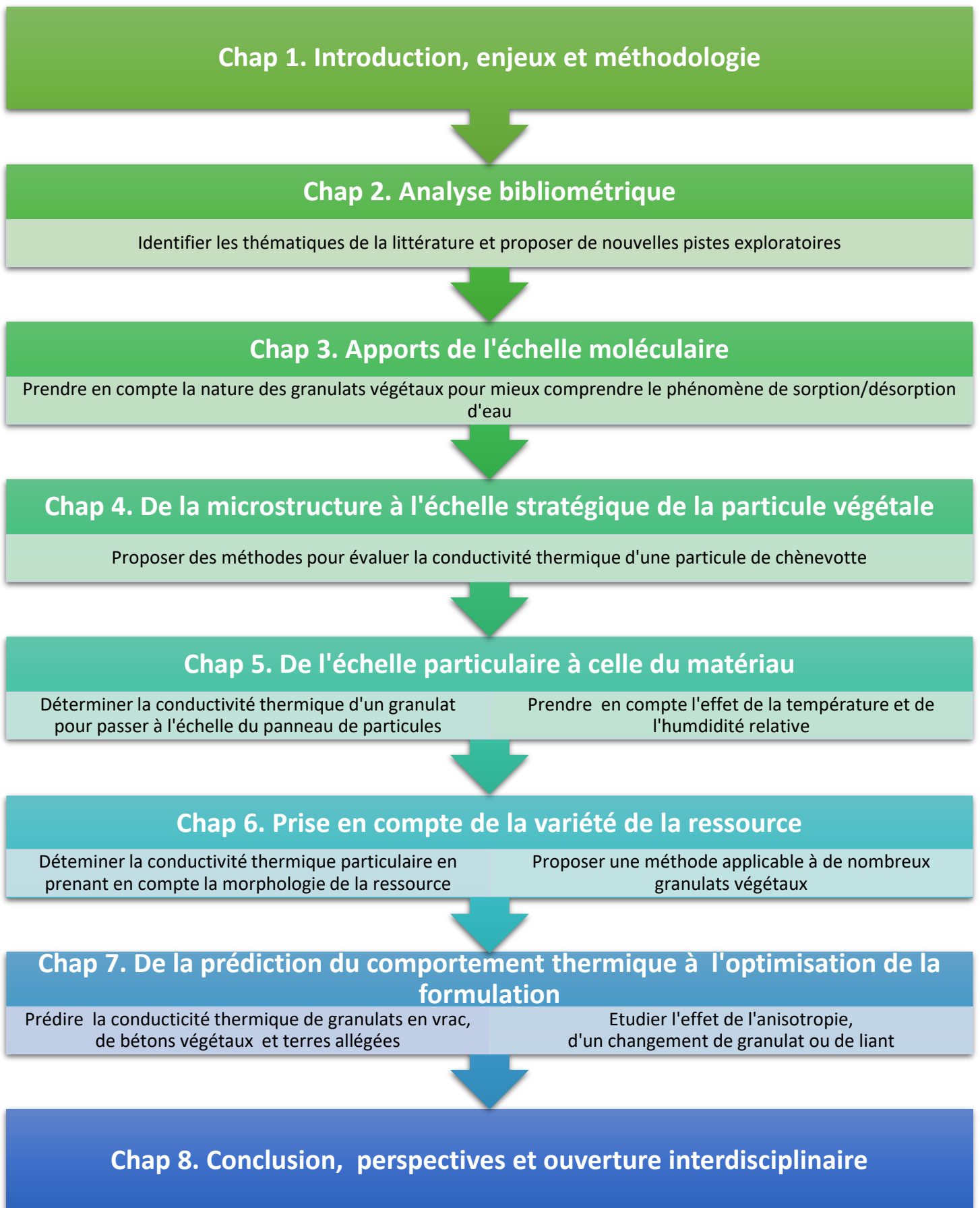


Figure 2. Récapitulatif de la démarche globale de la thèse et objectifs fixés à chaque échelle considérée

1.6 Structure du manuscrit

Ce manuscrit de thèse est composé de huit chapitres, construits principalement autour d'articles publiés ou soumis. L'articulation des chapitres permet de rendre compte de la démarche globale adoptée -à la fois multi-physique et multi-échelle- et de mettre en valeur les différents résultats obtenus.

Ce premier chapitre sert d'introduction : il développe le contexte global de la thèse sous différents angles. Il permet en outre d'explicitier la stratégie mise en place pour traiter les différents verrous identifiés dans la littérature concernant les matériaux de construction bio et géo-sourcés. Le cadre de l'étude est explicité et le choix des outils de modélisation est argumenté.

Le deuxième chapitre est une analyse bibliométrique sur les matériaux de construction bio et géo-sourcés. Elle donne un aperçu global sur de nombreux travaux menés jusqu'à présent dans le domaine de la thèse. Elle ne saurait être exhaustive mais livre de précieuses informations notamment sur les thématiques de recherche encore peu abordées dans la littérature. Cette étude met en avant l'intérêt de considérer davantage les phénomènes physiques, d'explorer la microstructure et de recourir à la modélisation pour contribuer au développement de ces éco-matériaux.

Dans le troisième chapitre, les domaines du génie civil, de la physique appliquée et de la chimie sont exploités afin d'ouvrir de nouvelles perspectives. Une explication complémentaire au phénomène d'hystérésis concernant les matériaux bio-sourcés, est proposée. Elle s'appuie sur une réflexion argumentée, basée sur des résultats récents dans le domaine de la simulation moléculaire. Ainsi, dès l'échelle moléculaire, un couplage hygro-mécanique est envisagé. Ce couplage pourrait expliquer les observations macroscopiques faites au niveau des matériaux bio-sourcés soumis à des sollicitations hygriques.

Le quatrième chapitre se positionne à l'échelle inédite de la particule de chènevotte. Deux méthodes semi-expérimentales de détermination de la conductivité thermique particulaire sont mises en évidence. Elles nécessitent notamment la connaissance de la porosité intra-particulaire ou la fraction volumique des granulats végétaux dans un mélange en vrac. Elles utilisent des résultats connus d'homogénéisation par champs moyens. La comparaison aux rares valeurs disponibles dans la littérature démontre la pertinence de considérer cette nouvelle échelle. Ces travaux ouvrent la voie pour une application élargie à d'autres co-produits agricoles.

Le cinquième chapitre montre comment, à partir de la connaissance de la conductivité thermique particulaire, il est désormais possible de prédire la conductivité thermique à l'échelle matériau. D'abord, les travaux s'orientent vers des sous-produits agricoles largement disponibles dans le sud-Ouest de la France. Ainsi, des panneaux d'isolants à base de moelle de tournesol sont fabriqués avec un procédé innovant, ne nécessitant ni liant ni procédé énergivore. La conductivité thermique est mesurée sous différentes conditions de température et d'humidité relative. En parallèle, une triple homogénéisation analytique est

réalisée afin de passer de l'échelle particulière à celle du panneau. La conductivité thermique est ainsi modélisée en prenant en compte sa variabilité sous les contraintes d'usage (température et humidité relative). La comparaison des valeurs modélisées aux valeurs expérimentales montre une bonne cohérence, soulignant l'intérêt de la méthode développée. Ensuite, la prise en compte d'une phase liante dans la modélisation, permet de prédire la conductivité thermique de composites à base de terre crue et de granulats végétaux. Ces travaux mettent en évidence que, pour offrir des résultats de modélisation pertinents, il est nécessaire de porter une attention particulière sur l'accessibilité des données d'entrée ainsi que sur l'adéquation entre matériau modèle et morphologie réelle du matériau géo-sourcé.

Une application élargie des modèles développés précédemment est proposée dans le sixième chapitre. L'objectif est de considérer la variabilité -et donc la spécificité- de la ressource disponible localement, tout en proposant une méthode « universelle » de prédiction de la conductivité thermique en vrac. Dix sous-produits agricoles sont considérés grâce aux travaux expérimentaux menés récemment dans la thèse de Ratsimbazafy [26]. Des points communs sont identifiés notamment au niveau du squelette solide, de la macrostructure et de la microstructure des granulats végétaux. Deux méthodes de prédiction de la conductivité thermique en vrac sont proposées, distinguant deux types de morphologie particulière. Dans le but de transposer et de rendre facilement applicables les résultats de cette étude à l'échelle chantier, une comparaison est menée, en termes de résistance thermique équivalente, pour des parois constituées de différents sous-produits agricoles.

Le septième chapitre s'intéresse à la possibilité offerte, grâce aux résultats précédents, de prédire la conductivité thermique des matériaux bio et géo-sourcés pour de multiples formulations (terres allégées ou bétons végétaux). Cette démarche vise à limiter le nombre d'essai en amont de la fabrication et à optimiser la formulation en fonction de l'usage visé. Dans un premier temps, une discussion est menée sur la variabilité de la conductivité thermique de l'isolation en vrac (cas de la chènevotte) selon le degré de compactage et sous l'effet d'un épisode de forte chaleur. Elle pose la problématique d'une caractérisation systématique de la ressource sous des conditions « normales » à 23 °C compte-tenu des conditions climatiques futures. Elle souligne également l'impact de la température sur le confort d'été en termes d'effusivité et de diffusivité. Dans un second temps, la prédiction et l'optimisation de la conductivité thermique de bétons végétaux et de terres allégées est proposée en prenant en compte des critères variés comme la nature du sous-produit agricole utilisé, sa proportion volumique, sa forme ou encore l'orientation préférentielle des particules induite par la mise en œuvre. Les prémices d'une optimisation multicritère complètent cette contribution.

Enfin, le dernier chapitre aborde les perspectives envisagées dans la continuité de ces travaux. Elles concernent notamment le passage à l'échelle paroi puis à l'échelle du bâtiment. A court terme, de nouvelles études interdisciplinaires devraient permettre de considérer la variabilité de la ressource, grâce à une approche probabiliste. A moyen terme, des mesures expérimentales ciblées à l'échelle particulière devraient permettre d'asseoir, voire d'affiner, la pertinence des méthodes proposées dans cette thèse. A plus long terme, des outils de modélisation seront développés afin de mieux comprendre et prédire également le

comportement hygrique voire mécanique des matériaux bio et géo-sourcés, en vue de contribuer significativement à leur utilisation à plus large échelle.

1.7 Références

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Chapitre 2. Analyse bibliométrique

Préambule

Pour identifier de nouvelles pistes exploratoires et repousser les limites des connaissances scientifiques dans une thématique de recherche donnée, il est intéressant de pouvoir mener une analyse sur un grand nombre de publications. Cela aide à valider ou réfuter des hypothèses existantes et à en formuler de nouvelles. Par ailleurs, identifier les verrous scientifiques est essentiel pour donner de nouvelles directions dans les travaux à mener. Souvent, l'analyse bibliographique permet cette identification, rassemblant l'état de l'art et les références principales. Toutefois, dans le but de donner un nouvel éclairage sur les matériaux de construction bio et géo-sourcés, ce n'est pas une analyse bibliographique mais bibliométrique qui est proposée dans ces travaux. En effet, la littérature est relativement fournie en analyses bibliographiques, notamment dans les manuscrits de thèses portant sur le sujet des matériaux de construction bio et géo-sourcés. L'intérêt est ici de donner de nouveaux indicateurs qui ne peuvent être le fruit d'un travail de bibliographie. Ainsi, il s'agit de quantifier l'évolution dans le temps des publications ainsi que leur typologie, tout en mettant en évidence les principaux pays, auteurs et journaux contributeurs. Cette analyse se base sur une étude statistique, appuyée ensuite par un logiciel de visualisation de réseaux bibliométriques (VOSviewer). Ce dernier permet d'aller plus loin en révélant les collaborations entre auteurs, les travaux fondateurs ainsi que les thématiques les plus abordées dans la littérature à ce jour. L'étude cible en outre des sous-thématiques des matériaux bio et géo-sourcés. Il s'agit de techniques de construction en terre crue (comme par exemple le pisé, l'adobe, torchis ou encore la terre allégée) d'une part et de différentes matières premières des matériaux bio-sourcés d'autre part (en particulier le chanvre, le bois, le bambou, la paille, le riz, le lin ou encore le tournesol). Enfin, une comparaison aux thématiques traitées dans le domaine plus large des matériaux de construction composite, permet une analyse qualitative. Étudié depuis de très nombreuses années, ce domaine de recherche livre des pistes inexplorées au niveau des matériaux bio et géo-sourcés.

Cette analyse bibliométrique permet de fournir des données quantitatives pour orienter la recherche sur les matériaux de construction bio et géo-sourcés. Le travail interdisciplinaire, faisant appel notamment aux sciences dures comme la physique ou la chimie, pourrait ainsi ouvrir de nouvelles perspectives. Considérer davantage la microstructure des matériaux et leur variabilité intrinsèque constituent également des éléments pertinents pour des travaux futurs. Enfin, le recours à la modélisation devrait permettre de contribuer davantage au développement de ces éco-matériaux.

Article A. Bibliometric Analysis of Bio- and Earth-Based Building Materials: Current and Future Trends

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Abstract: The energy and environmental transition in the building sector requires the development and use of low-impact materials. Despite the growing interest in bio-based and earth-based building materials, their widespread adoption is still limited due to a lack of hindsight, as their study is relatively recent. This study aims to contribute to the development of these materials by providing an extensive overview of key contributors (authors, countries, journals) in these fields. Then, the keywords of the corresponding publications were analyzed to reveal the main topics covered to date. First, a broad scale is presented, followed by a focus on sub-categories, specifically raw materials for bio-based building materials and implementation techniques for earth-based ones. Finally, a comparative analysis, with the themes covered by composite construction materials as a whole, completes the study. Using statistical analysis coupled with bibliometric network visualization software, this study provides clear, quantifiable, and objective insights into current trends. Furthermore, it facilitates the identification of new, promising research perspectives and highlights the importance of interdisciplinary collaboration. Physics, modeling, durability and microstructure studies emerge as relevant levers for advancing the future development of these eco-friendly building materials.

Keywords: bibliometric; bio-based building material; earth-based building material

1. Introduction

The 21st century is the one of global warming awareness. The risks for humanity are major: an increase in natural disasters and their consequences (high mortality, major destruction of buildings and historic sites, etc.), loss of biodiversity, food insecurity, societal conflicts or negative impacts on health (pollution-related pathologies, anxiety, access to drinking water, more pathogens, etc.) [1–4]. As the construction sector is one of the most environmentally impacting [5,6], research into low-impact building materials has been multiplying in recent years. This especially applies to bio-based and earth-based building materials.

Bio-based materials provide performances that contribute to both energy efficiency and building comfort by incorporating agricultural by-products (hemp shiv, sunflower pith or bark, flax shiv, etc.) or wild plants (reed, bamboo, etc.) [7–9]. In the literature, recent reviews of their thermo-hydric performances and applications [10], their fire behaviors [11], the alternative binders used [12], but also the economic and political obstacles to their development [13] are available. No overall analysis has been carried out on this emerging field of research in terms of bibliometric analysis.

Earth-based materials have been used in construction for thousands of years, but their study is relatively new [14]. Their potential is known: low gray energy, low carbon dioxide emissions and recyclability (if not stabilized) [15,16]. Moreover, the hygroscopic capacity of clay-based material ensures indoor hygrometric equilibrium [17]. However, while this eco-material is being used with innovative techniques such as 3D printing [18], it is still difficult to understand its behavior. This presents difficulties of characterization [19] and reproducibility [20]. Review articles examine the environmental benefits of earth construction [15], the effect of incorporating plant fibers or aggregates [14,21], or characterization and production techniques [22,23]. The social, political, and economic aspects of using raw earth in construction have also been studied [24,25]. However, there is neither a comprehensive study nor a bibliometric analysis of the research carried out to date on this ancestral material.

The choice of treating both bio- and geo-based building materials in the same bibliometric study is a conscious one. These materials share a common history. Although they have only recently been studied, ancestral construction techniques demonstrate the relevance of using plant fibers in earth-based matrices [26–28]. This is due to the complementary nature of the two materials. Earth is a structural material that passively regulates ambient humidity, thus contributing to the building's interior comfort [22]. Plant-based aggregates also have the ability to moderate humidity variations in the indoor environment, and no deleterious effects are observed when plant aggregates are incorporated into a clay base, contrary to observations made for certain combinations of aggregates and cementitious matrices [9]. Consequently, the combined effects of the two materials offer great passive humidity regulation capacities [29]. In addition, incorporating plant aggregates can improve the thermal performance of an earth material [30–32] and limit its drying shrinkage [33]. A growing number of research projects are exploiting this potential by combining the use of plant-based aggregates and raw earth [34–36].

In this light, the purpose of this study is to present a bibliometric overview of the field concerning bio- and earth-based building materials in order to give an overall view of each one. Data concerning these two areas have been processed using two methods: a statistical analysis and the design of visualization maps. The idea is to map each discipline. The results are intended to be used by anyone starting research in one or both of these disciplines. The results are also intended to serve as a reference for experienced researchers in order to identify a new working focus. Several avenues are proposed in this study, but they cannot be exhaustive.

Based on 1778 publications concerning bio-based building materials and 1605 concerning earth-based building materials, this study provides, therefore, a general overview of these emerging research fields. The bibliographic data of these publications were collected from the Web of Science database. This database was chosen because its content is multi-disciplinary [37], enabling a wide range of publications to be taken into account. However, the choice of database may introduce biases [37,38]. However, the large number of publications considered may limit these biases. Again, this study simply sheds new light on general trends in the field of bio- and geo-based building materials and can not represent an exhaustive study.

A preliminary statistical analysis provides an overview of current research activities. A comparison with a field that emerged a few years earlier, composite materials, allowing a comparison of current dynamics and giving an indication of possible future prospects. Then, a more in-depth analysis of the research data was carried out using VOSviewer software. This recent visualization tool can relate and illustrate bibliometric networks [39–42]. Exploring the literature—particularly, the links between authors, themes, and most cited articles—has highlighted several significant points in the fields, as mentioned above. This bibliometric analysis thus underscores major research lines of the scientific community to date. It also identifies grey areas, i.e., topics that have not yet been addressed in order to propose new perspectives of work and help the development of bio- and geo-based materials. Likewise, a more targeted study is carried out on the raw materials for bio-based building materials and on implementation techniques for geo-based building materials.

2. Materials and Methods

2.1. Database: Web of Science

Particular attention has been paid to the selection of publications and corresponding data. The aim was to establish a sufficiently large dataset for conducting a relevant statistical study. Thus, all publication years from 1900 (database limit) to 2022 were systematically considered. It was also essential to base the study on relevant publications, so retractions were not taken into account. Table 1 summarizes the criteria used to select the publications considered in this review for the primary statistical analysis. They constitute the original database.

Table 1. Web of Science search criteria to extract the initial database.

Topic	Year Published	Number of Publications
“Bio-based building materials”	1900–2022	1778
“Earth-based building materials” or “geo-based building materials”	1900–2022	1605
“Composite materials”	1900–2022	416 745

From this database, a systematic statistical analysis was carried out on bio- and geo-based building materials. This analysis highlights several aspects, including the number of publications per year, the typology of these publications, the countries/authors/journals with the highest publication rates, and the main topics studied. It is worth noting that a publication may at this stage be reported more than once, for example, if several authors have collaborated, or if it covers more than one topic. However, this gives an overview of each discipline considered in this study. The case of composite materials has been approached in a different way, as it provides a basis for comparison.

2.2. Bibliometrics Analysis Software: VOSviewer

VOSviewer [43] is a computer program created in 2010 with the aim of facilitating bibliometric studies and addressing the increasing volume of data [44]. Versions have evolved up to the 1.6.19 one used in this study. The program’s algorithm, developed in Java script, is configured to extract bibliometric data from the considered database, enabling the creation of bibliometric network visualization maps. The larger the map element, the more it is cited in the analyzed database. Elements can represent publications, researchers, or research topics, and they are connected pairwise. The strength of the connection is indicated by the width of the line. The links available from a considered database are the following:

- Co-authorship: Refers to publications co-authored by two authors, organizations or countries.
- Co-occurrence: Describes publications in which two keywords appear together.
- Citation: Involves one element (document, source, author, organization, country) citing another one.
- Bibliometric coupling: Measures the number of common references between two documents, sources, authors, organizations or countries.
- Co-citation: Quantifies the number of times items (references, sources, authors) are cited together in the same document.

The database considered should not be too large to allow an effective visualization of the map. It is one of the software’s limitations. The analysis of the co-authorship link reveals the primary contributors in a field. The advantage of visualization maps is that they allow us to understand the collaborations between various authors, in addition to their individual contributions. To provide a temporal dimension to the visualization, a time scale can be added. Moreover, the choice of highlighted links is essential for a pertinent analysis. First, the main contributors and their cooperation concerning bio- and geo-based building materials is analyzed with co-authorship links study. Then, in order to bring out the founding works as well as the themes covered in each field, co-citation and co-occurrence links are considered. It

enables us to identify themes still neglected or not covered at all and complements the simple comparison with composite materials.

In addition, the visualization maps highlight clusters of elements (depending on the chosen analysis). These clusters require subsequent analysis and interpretation. A wide range of map combinations can be generated from a given database. In this study, the results that seemed most relevant are presented, aiming to provide an overview of the current state in each discipline and suggest new research directions. Keyword analysis is carried out for each category, as well as for sub-categories.

2.3. Work Methodology

To ensure representativeness, a sufficient amount of data is needed, necessitating careful selection in advance. It is also important to ensure that the major works in the green building sector are included. The Web of Science was chosen as the literature database for publication extraction due to its compatibility with VOSviewer software and its recognized relevance for the specific review conducted in this study [37,45]. Data selection played an essential role in this study. In order to retain only relevant publications related to the construction sector, filters within the Web of Science platform were applied. Two distinct working databases, respectively noted “Base BBM-0” and “Base GBM-0”, for bio- and geo-based materials were thus established. Subsequently, the extracted database underwent further processing using spreadsheet software to conduct the statistical study. To create visualization maps, the refined database was imported into the VOSviewer software. From the “Base BBM-0” and “Base GBM-0” databases, the Web of Science platform was used to select publications concerning “sub-categories”. These sub-categories specifically focused on raw materials for bio-based materials and processing techniques for geo-based materials. To summarize the adopted methodology, a visual representation is presented in the form of a flow chart (Figure 1).

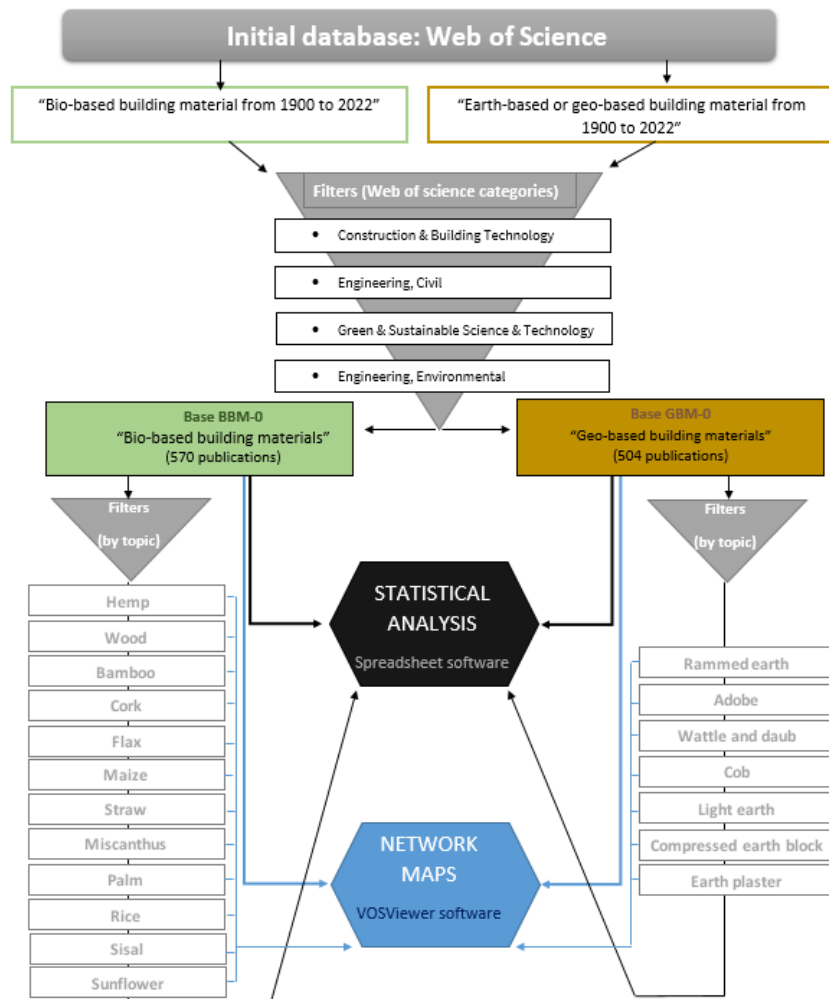


Figure 1. Flow chart of the methodology applied in the study.

To complete the analysis, the same filters (categories) as those described in Figure 1 were applied to the Web of Science publication data corresponding to “Composite building materials”. Only the year 2022 was retained, corresponding to 518 articles, a number compatible with the capabilities of the VOSviewer software.

3. Results

3.1. Preliminary Statistical Analysis

3.1.1. Bio-Based Building Materials

- Increasing number of scientific publications

A small number of research projects were carried out in the late 1990s on bio-based building materials. The field really emerged in the late 2000s (Figure 2).

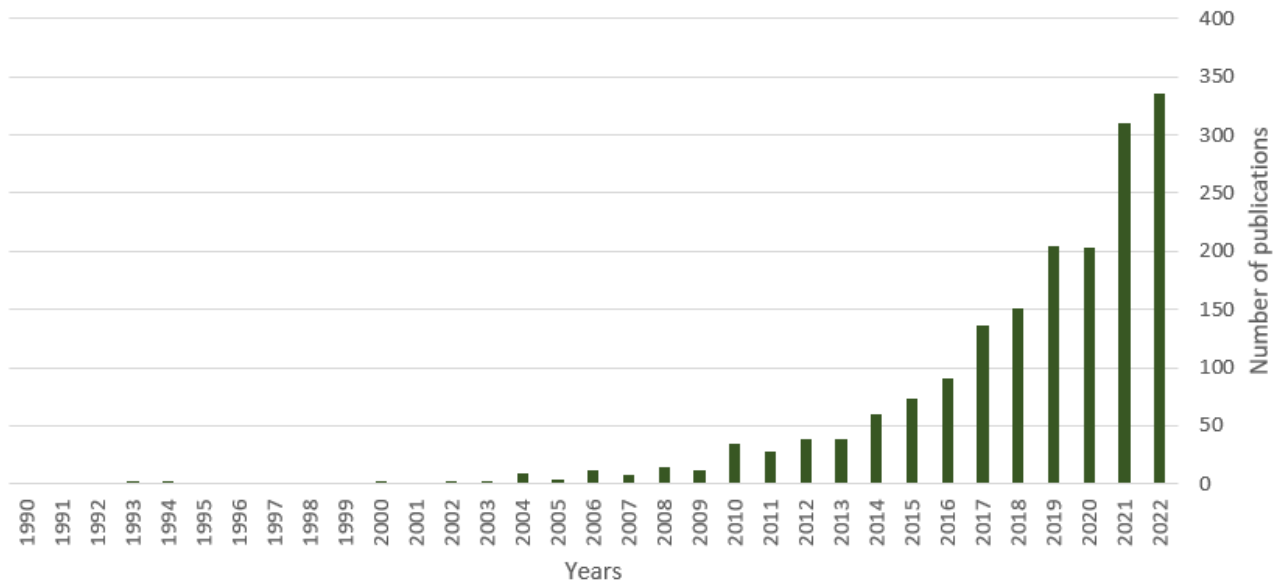


Figure 2. Number of publications concerning bio-based materials between 1990 and 2022 according to Web of Science.

The environmental impact of the construction industry was raised in the literature as early as the 1990s [46–49]. Bio-based building materials, known for their low environmental impact, became the focus of an increasing number of research projects in the following years. The academic community has embraced environmental issues to propose alternative solutions to conventional building materials [50,51]. Reflecting this global awareness, eco-friendly materials have been the subject of a growing number of publications since the 2010s. In 2020, a temporary slowdown in research activity was observed, likely attributable to the global crisis caused by the Covid virus.

However, the last few years, 2021 and 2022, have shown a notable resurgence in research activity within the field of bio-based building materials.

Furthermore, the publication typology reveals a strong tendency to write original articles (Figure 3). Although research activities in this field are relatively recent, review articles represent a relatively large proportion of the publications. In similar proportions, proceedings account for almost 15% of total publications, with a small percentage of book chapters and “other types” of publications including data papers, meeting abstracts and editorial material.

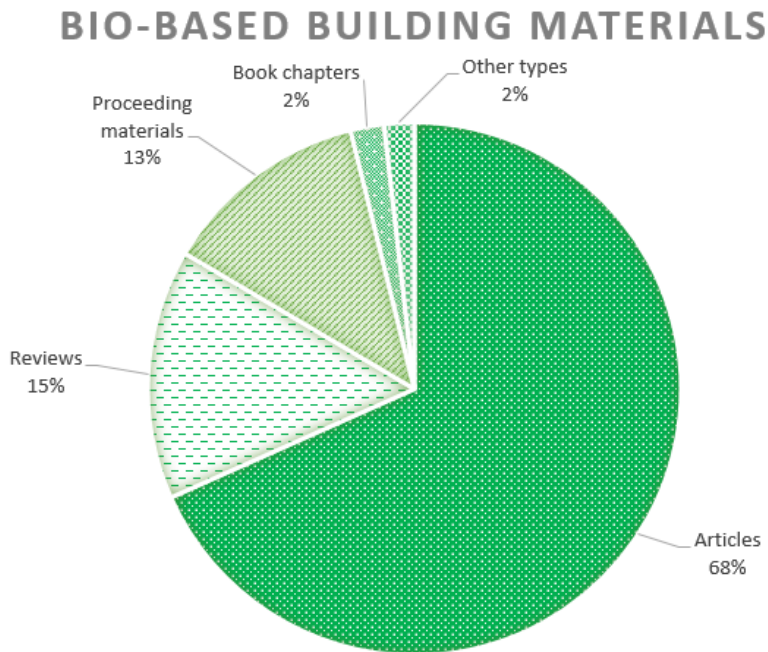


Figure 3. Typology of publications on bio-based building materials between 1990 and 2022 according to Web of Science data.

- Geographic and editorial dynamics

Societal and political issues may have diverged, leading to significant disparities among countries [46,52,53]. Looking closely at the countries publishing the most papers on bio-based building materials, it is interesting to note that European countries are well represented, but they are not the only ones. China, France and the United States are the countries most involved in the academic dynamic in the bio-based building materials sector (Table 2). Regarding authors, a significant portion of publications on the subject of bio-based building materials have been contributed by authors in France and more broadly across Europe (Table 3).

Table 2. Top ten countries of publication concerning bio-based building materials to date.

Countries	Articles
China	309
France	241
USA	225
Italy	147
Germany	139
England	107
India	100
Spain	91
Canada	77
Netherlands	62

Table 3. Most contributive authors in the field of bio-based building materials to date.

Author	Affiliation	Articles
Collet Florence	Université de Rennes 1, Laboratoire de Génie Civil et Génie Mécanique	18
Lanos Christophe	Université de Rennes 1, Laboratoire de Génie Civil et Génie Mécanique	15
Blanchet Pierre	Department of Wood and Forest Sciences, Laval University, Québec, QC G1V0A6, Canada	15
Magniont Camille	Université de Toulouse, UPS, INSA, LMDC (Laboratoire Matériaux et Durabilité des Constructions), Toulouse Cedex 04, France	14
Habert Guillaume	Chair of Sustainable Construction, Eidgenössische Technische Hochschule (ETH) Zurich, Stefano Francini Platz 5, CH-8093 Zurich, Switzerland	13
Pretot Sylvie	Université de Rennes 1, Laboratoire de Génie Civil et Génie Mécanique	12
Lawrence Mike	BRE Centre for Innovative Construction Materials, Department of Architecture and Civil Engineering, University of Bath, BA2 7AY, United Kingdom	11
Langlet Thierry	Laboratoire des Technologies Innovantes (LTI), University of Picardie Jules Verne, Avenue des Facultés—Le Bailly, 80 025 Amiens Cedex 1, France	10
Maalouf Chadi	University of Reims Champagne-Ardennes, GRESPI Laboratory (EA 4694), Reims 51100, France	9

Finally, *Construction and Building Materials* is the journal that publishes the most on bio-based building materials. The other leading journals in the discipline have very similar publication frequencies, between 20 and 40 publications per year (Table 4).

Table 4. Top ten journals publishing articles about bio-based building materials.

Journal	Articles
Construction and building materials	79
Polymers	38
Journal of cleaner production	37
Materials	34
Energy and buildings	32
Journal of building engineering	27
Sustainability	26
Building and environment	25
Industrial crops and products	20
Material today proceedings	20

- Main topics covered

Another valuable aspect of this preliminary statistical study is the list and proportion of topics considered in bio-based building material publications (Figure 4). This demonstrates that fields such as engineering, materials science and chemistry seem to be widely explored, whereas areas like physics, polymer science or environmental sciences (ecology) receive comparatively less attention.

BIO-BASED BUILDING MATERIALS

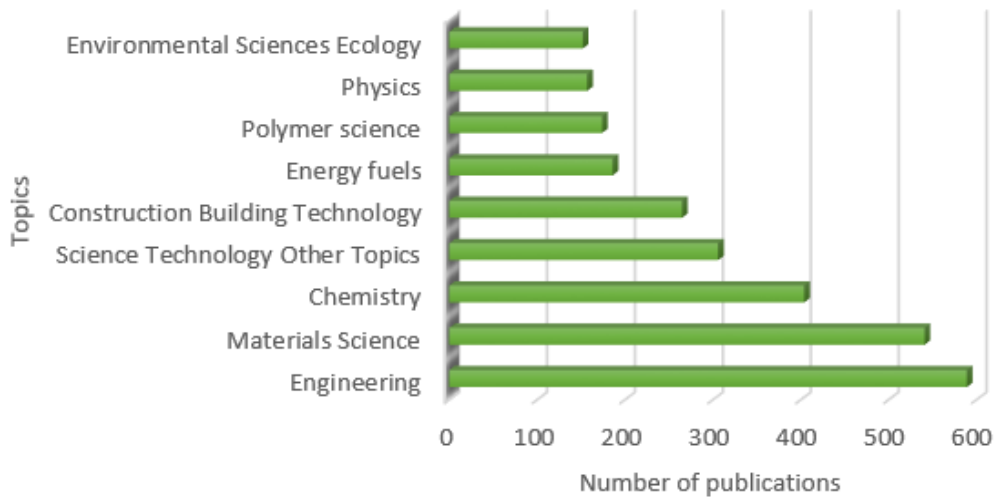


Figure 4. Main topics studied in publications concerning bio-based building materials between 1990 and 2022 according to Web of Science data.

- Focus on raw materials

An additional analysis of the raw materials used as bio-based building materials was carried out (Figure 5).

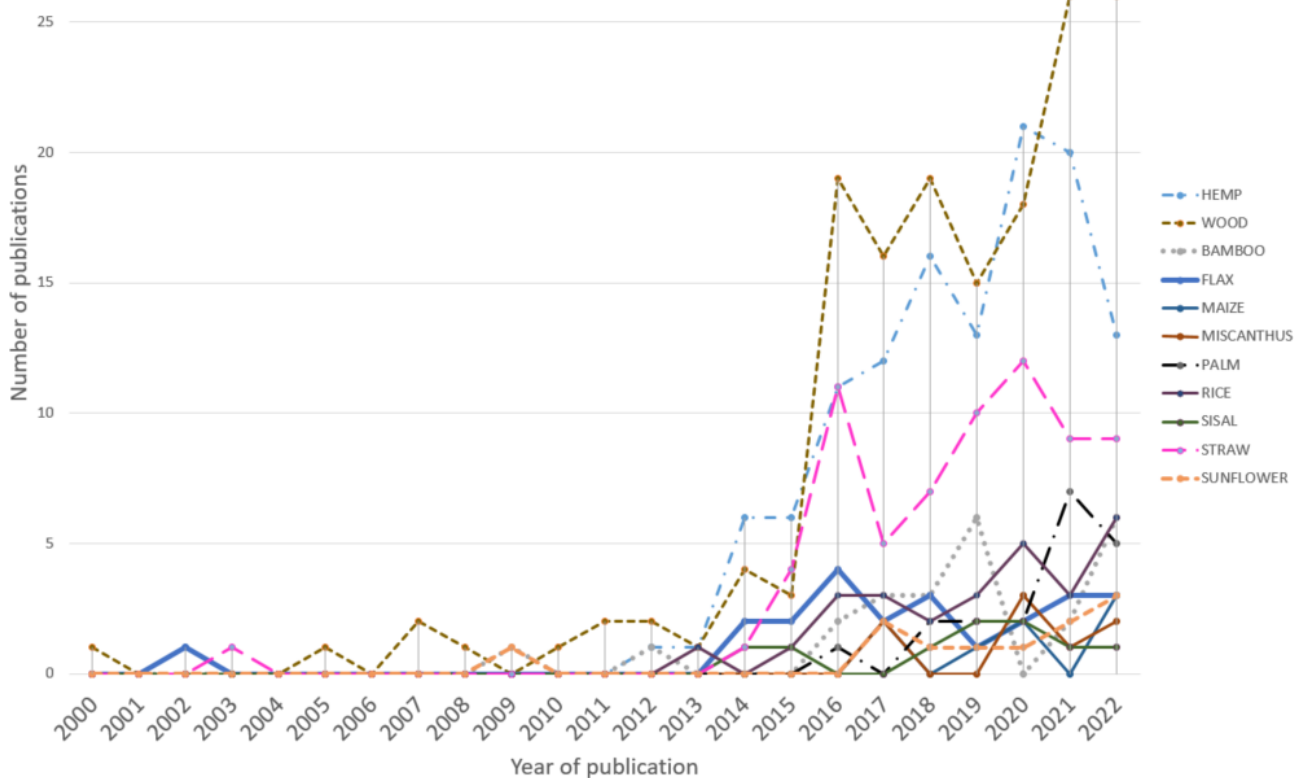


Figure 5. Number of publications for the main raw materials identified in bio-based building materials over the years.

Not surprisingly, wood has been the most widely published topic for almost 20 years. It is followed by hemp, which is also widely studied, well ahead of all other plant-based aggregates. The prevalence of hemp is attributed to its rapid growth in many climates [54]. Additionally, there is promising research on other raw materials from wild plants (such as bamboo) or agricultural by-products (like maize, sunflower, etc.) [55,56]. Since 2015, there has been a growing interest and diversification in the study of raw materials. Particularly, straw stands out with a relatively high number of publications. For other raw materials, it is difficult at this stage to differentiate them in terms of publication dynamics. It should be pointed out that, for the sake of clarity in the graphical representation, raw materials with fewer than five publications in the Web of Science database have been omitted. This group includes jute, reed, cork, mussel or oyster shell, barley, rapeseed and sheep wool. Nevertheless, the extensive range of resources studied is probably an opportunity to use local resources minimizing the environmental footprint of building materials [57]. However, a significant challenge arises in ensuring equivalent performance based on the local resource [58]. Overcoming this scientific bottleneck is crucial for advancing the development of bio-based building materials.

3.1.2. Earth-Based Building Materials

- Increasing number of scientific publications

Research related to raw earth building materials emerged in the 1990s (Figure 6). The number of annual publications has significantly increased since 2005, with dynamics in the following years showing a relatively similar trend to that of bio-based building materials.

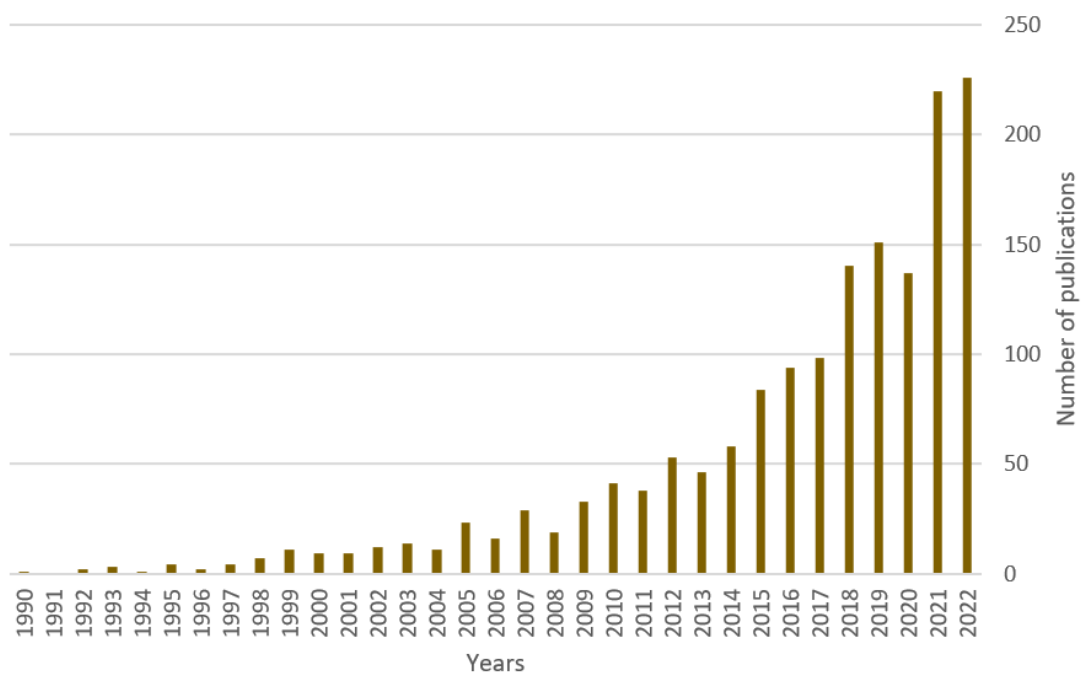


Figure 6. Number of publications concerning earth-based materials between 1990 and 2022, according to Web of Science data.

However, it can be observed that the field of materials using earth as a raw material faces challenges in terms of publication output. In recent years, approximately 30% fewer publications have been published on geo-based building materials than on bio-based ones. It

is certainly a multi-factorial phenomenon that combines a biased representation of the material, a lack of incentive policies and sustainability concerns [24,59,60].

The number of publications remained particularly limited in 2020. This was probably due to the slowdown in research activities during the Covid crisis [61]. However, in subsequent years, publications have been on the rise.

The types of publications also logically show a significant proportion of original articles, such as for bio-based building materials (Figure 7). Review articles represent a small proportion, approximately half of what was observed for bio-based materials. In contrast, the proportion of proceeding materials was much higher, roughly twice as high as that for bio-based materials.

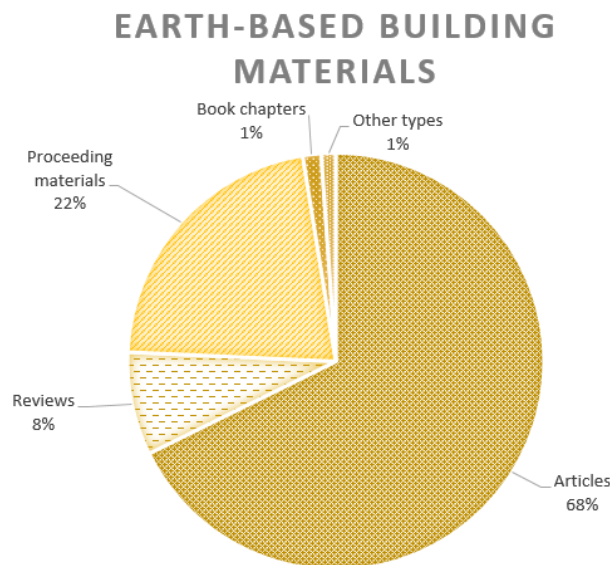


Figure 7. Typology of publications on geo-based building materials between 1990 and 2022 according to Web of Science data.

- Geographic and editorial dynamics

Despite a large proportion of the population living in earth-based dwellings [62] and the recognized advantages of this material in construction [25], research activities on this subject are concentrated in only a few countries (Table 5). Over 50% of publications come from the following countries: the United States of America, China, France and Italy.

Table 5. Top ten countries of publication concerning earth-based building materials to date.

Countries	Articles
USA	309
China	291
France	145
Italy	113
Germany	89
England	86
Spain	82
India	80
Portugal	64
Australia	55

The majority of authors in the field of raw earth building materials are Europeans. French authors are particularly active in this domain (Table 6).

Table 6. Most contributive authors in the field of earth-based building materials to date.

Author	Affiliation	Articles
Aubert Jean-Emmanuel	Université de Toulouse, UPS, INSA, LMDC (Laboratoire Matériaux et Durabilité des Constructions), Toulouse Cedex 04, France	13
Faria Paulina	CERIS-ICIST and Civil Engineering Department, NOVA University of Lisbon (FCT NOVA), Campus da Caparica, 2829-516 Caparica, Portugal	12
Laborel-Preneron Aurélie	Université de Toulouse, UPS, INSA, LMDC (Laboratoire Matériaux et Durabilité des Constructions), Toulouse Cedex 04, France	11
Magniont Camille	Université de Toulouse, UPS, INSA, LMDC (Laboratoire Matériaux et Durabilité des Constructions), Toulouse Cedex 04, France	9
Humberto Varum	CONSTRUCT-LESE, Department of Civil Engineering, Faculdade de Engenharia da Universidade do Porto, Portugal	8
Niroumand Hamed	Department of Civil Engineering, Faculty of Engineering, Buein Zahra Technical University (IKIU-BZ), Qazvin 34149, Iran	8
Pinto Joao Fransisco	Natural Works, Rua Ricardo Esp. Santo N5 CVD, 1200-790, Lisboa, Portugal	8
Beckett Christopher	Institute of Infrastructure and Environment, School of Engineering, The University of Edinburgh, Edinburgh, UK	7
Zai, M.F.M	University of Reims Champagne-Ardennes, GRESPI Laboratory (EA 4694), Reims 51100, France	6
Hamard Erwan	LUNAM University, IFSTTAR, MAST, GPEN, F-44344 Bouguenais, France	6

Similarly to bio-based building materials, the leading journal for geo-based building materials is *Construction and Building Materials*. It contributes approximately three to four times more than most other journals in the field (Table 7).

Table 7. Top ten journals publishing in the field of earth-based building materials to date.

Journal	Articles
Construction and building materials	63
Sustainability	26
Proceeding of SPIE	23
Journal of building engineering	22
International journal of architectural heritage	18
Journal of cleaner production	18
Inorganic chemistry	17
AIP Conference Proceedings	15
Accounts of chemical research	14
Materials today proceedings	14

- Main topics covered

Interestingly, the main themes covered by the publications closely align with those in bio-based materials. The most represented themes are engineering, materials science and chemistry, while physics and environmental sciences (ecology) remain under-treated (Fig. 8).

EARTH-BASED BUILDING MATERIALS

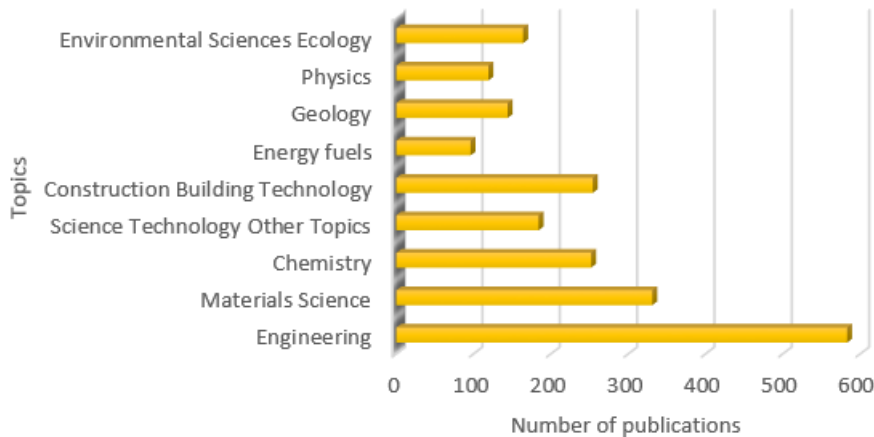


Figure 8. Main topics studied in publications concerning earth-based materials between 1990 and 2022 according to Web of Science data.

This preliminary statistical analysis provides an overview of the research activities carried out on bio- and geo-based building materials since the emergence of these fields. In particular, it highlights the countries and authors driving this domain. Clearly, Europe emerges as a key driver of the ecological transition, supported by these eco-friendly materials. This leadership can be attributed to contemporary history and environmental concerns.

Concern about global warming, its origins and consequences dates back to the 1990s. In response, the United Nations Framework Convention on Climate Change (UNFCCC) was adopted during the 1992 Earth Summit in Rio de Janeiro [63,64]. Negotiations conducted within this context led in 1997 to the Kyoto Protocol [65,66]. The objective was a 5% reduction in greenhouse gas emissions compared to 1990 levels by 2005. The European Union undertook an 8% reduction in its first commitment period (2008–2012). On 31 May 2002, the Kyoto Protocol was ratified by both the EU and its member states. Europe's commitment was reiterated in 2005 with new objectives, while the United States and China made no secret of their reluctance to ratify the protocol [67–69].

Europe's strong political engagement in the 2000s is certainly at the origin of its leadership in researching environmentally friendly materials [70,71]. The early 2000s coincided with the very beginning of research activities in bio- and geo-based building materials. Since then, Europe has clearly supported innovation and research for a greener and more sustainable society. One example is the EU's Horizon 2020 framework program, with an overall budget of approximately EUR 70,000 million over seven years (2014–2020) [72]. Another example is the Interreg POCTEFA scheme [73,74] that financed the trans-Pyrenean SAVASCO project, which aims to establish a cross-border value-added chain for corn and sunflower stalks for the construction industry [75,76]. Another example is the CobBauge project funded under the INTERREG VA France (Channel)/England cross-border cooperation program [77,78], promoting raw earth construction.

Today, Europe is aiming for carbon neutrality by 2050 and making it a top priority through the Green Deal initiative [79]. To reach this objective, continued support for research, particularly in the field of eco-construction, will be crucial.

- Focus on implementation techniques

An additional analysis of the implementation techniques and associated studies was carried out concerning geo-based building materials (Figure 9).

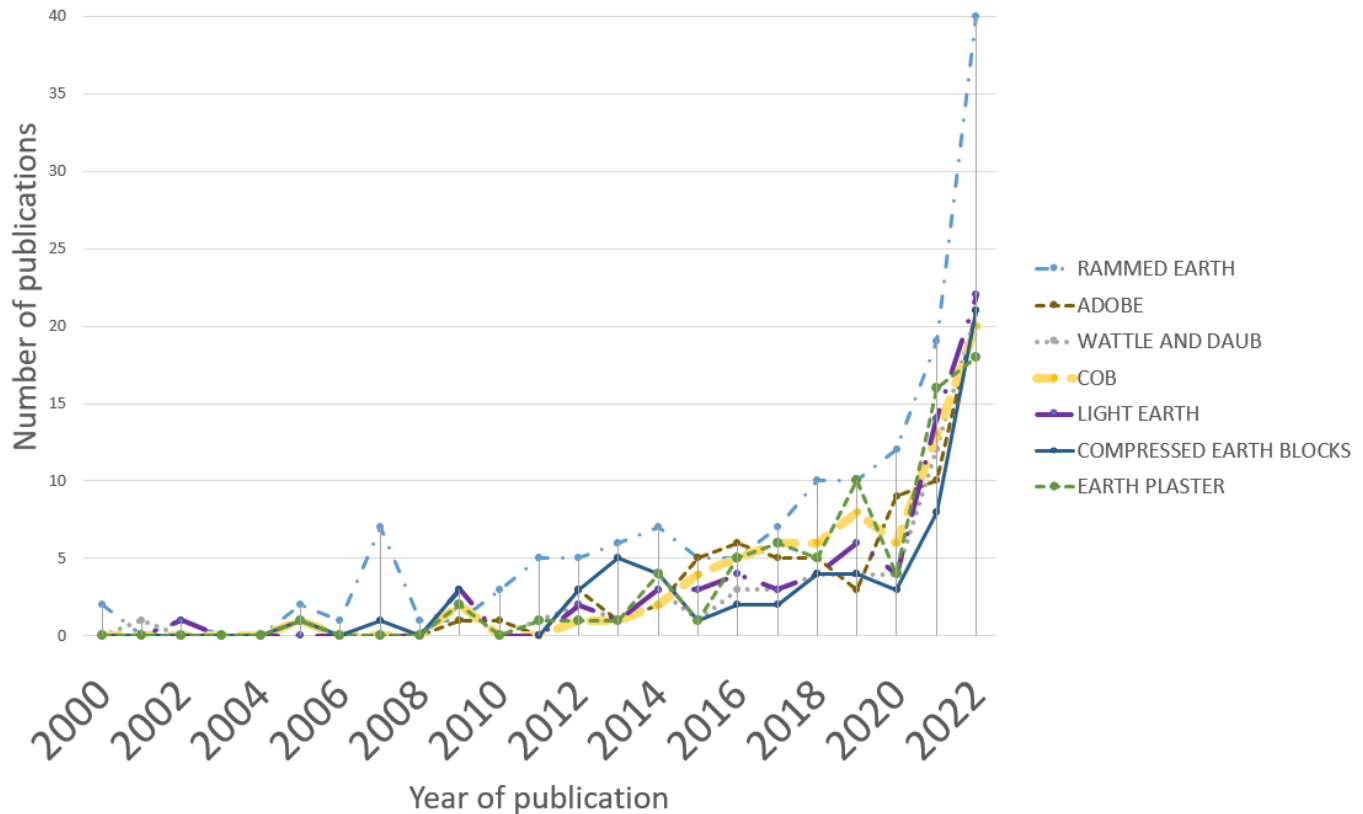


Figure 9. Number of publications for the main implementation techniques of geo-based building materials over the years.

While raw earth stands as an ancestral material, our study reveals its recent emergence as a subject of study. According to our study, research into various implementation techniques primarily commenced in the 2010s, with a notable surge in activity for rammed earth a few years earlier. It is worth noting that, since then, all techniques have followed a very similar trend, with the exception of rammed earth, which exhibited nearly double the scholarly attention in 2022. This growing interest can be attributed to architects valuing its aesthetic appeal or its potential for prefabrication, aligning with contemporary demands [80–82].

3.2. Comparative Study with Composite Building Materials

To further enhance the analysis, a comparison with activities carried out in the field of composite materials has been included. This section provides an overview of research areas in bio- and geo-based building materials that could contribute to their development by identifying those that have been promising for composite materials.

3.2.1. Evolution in the Number of Publications on Composite Materials

It seemed interesting to compare the evolutionary dynamics of bio- and geo-sourced materials with a research field that emerged a few decades earlier, in the 1960s—composite materials. To take a global view and appreciate the research dynamics in these materials, the

statistical study was not limited to composite materials used in construction. All sectors have been considered here (Figure 10).

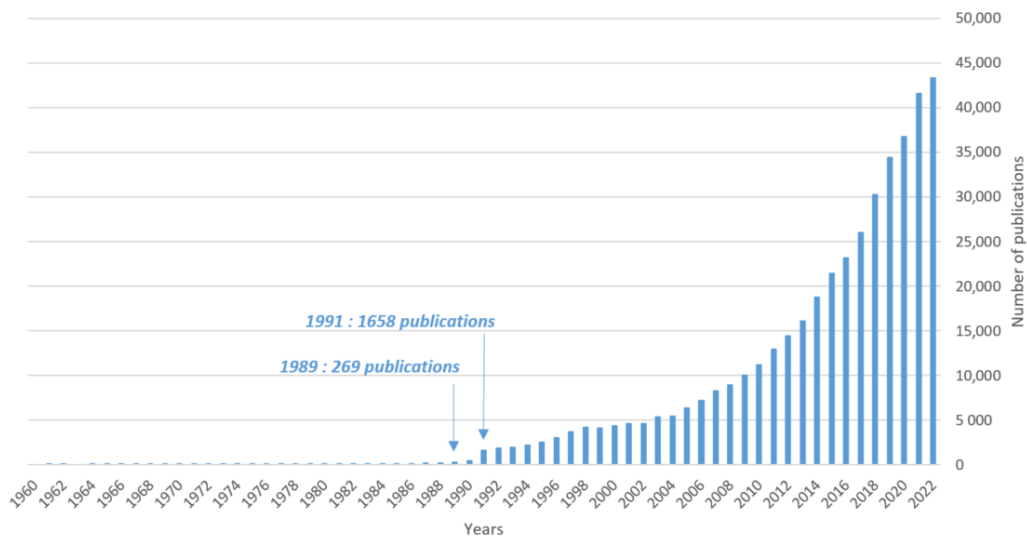


Figure 10. Number of publications concerning composite materials between 1960 and 2022 according to Web of Science data.

Obviously, the number of publications is extremely high, providing an overall perspective. What is striking is the number of publications, which increased more than six-fold between 1989 and 1991. It is legitimate to question the reasons for this major breakthrough in composite materials and related work in those years.

The 1990s marked the beginning of the computer age. Since then, computer networks have been driving progress in science and engineering [83]. One possible explanation for the inflection point in publication growth is the development of computer technology. Before the 2000s, numerous publications incorporating computerized tools were noticed [84–87].

3.2.2. Emerging Themes and Tools for New Developments

In order to assess the above hypothesis, the evolution of the number of publications related to composite materials dealing with modeling, which requires the use of computer tools, was observed around the year 2000 (Figure 11).

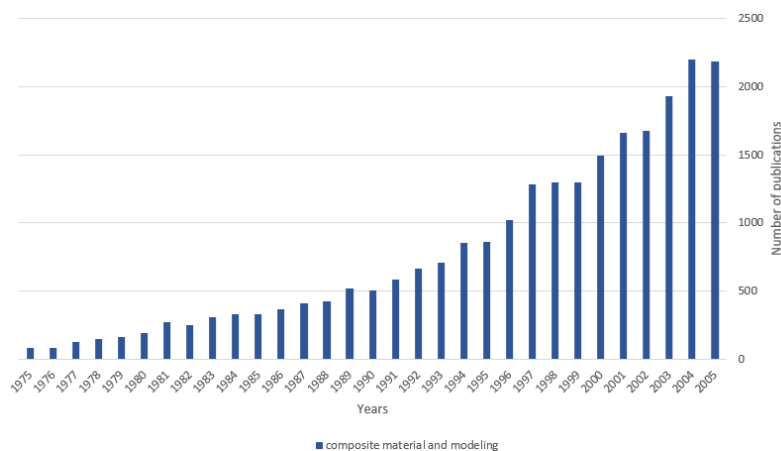
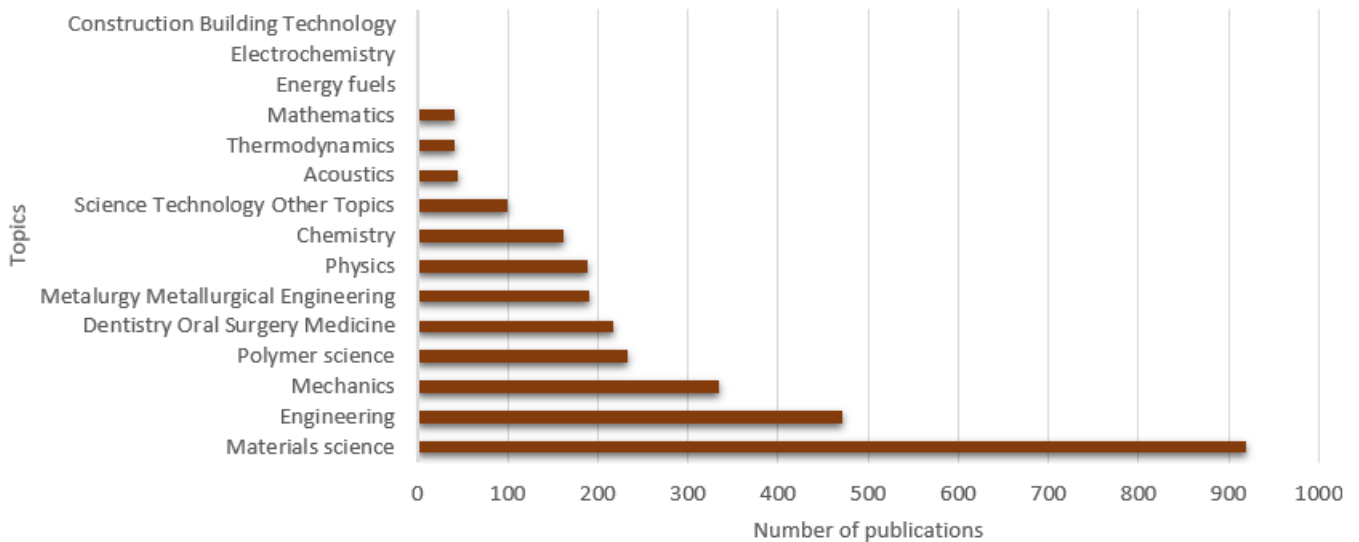


Figure 11. Number of publications concerning modeling and composite materials in the 2000s according to Web of Science data.

Between 1990 and 2000, the number of research projects focused on modeling composite materials tripled. The use of IT tools was undoubtedly a major lever in the development of these emerging materials.

The exploration of new and promising research themes could also explain the rapid growth in the number of publications. A comparative study of the themes studied before and after 1990 were carried out (Figure 12).

COMPOSITE MATERIALS before 1990 (a)



COMPOSITE MATERIALS after 1990 (b)

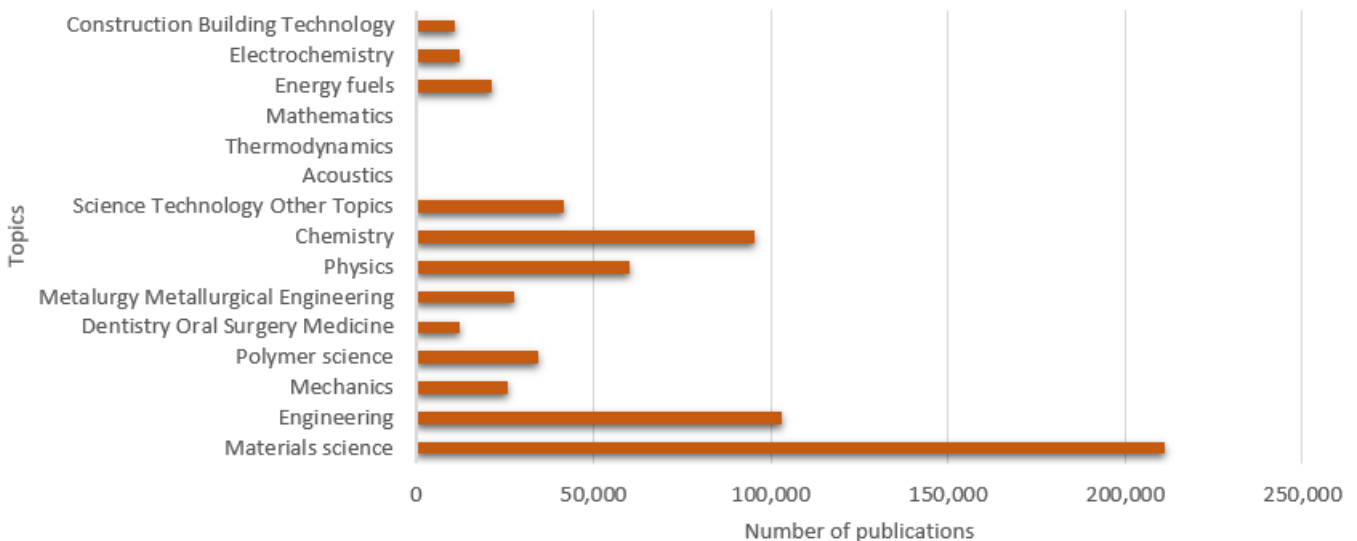


Figure 12. Main research areas studied in the field of composite materials from 1960 to 1990 (a) and from 1990 to 2022 (b) according to Web of Science data.

This comparison reveals that the fields emerging after the 90s were the hard sciences, particularly chemistry and physics. They have respectively risen from 8th and 7th place to 3rd and 4th place in the list of main themes covered. One of the mainsprings for the development of composite materials is probably a better understanding of their physico-chemical behavior.

3.2.3. Eco-Materials: What about Modeling and the Hard Sciences?

The subject of modeling in bio- and geo-based building materials seems to be in its infancy, as indicated by the number of publications over the years (Figure 13).



Figure 13. Number of publications concerning modeling and bio-based or geo-based building materials according to Web of Science data.

The number of publications related to modeling in the two research areas is increasing. Cumulatively, it represents 7% of the total number of publications for bio-based building materials and 15% for geo-based ones. By comparison, modeling is involved in 6% of publications on composite materials. However, if the focus is limited to construction-related composites: from the 19,459 publications reported by Web of Science, 5166 deal with modeling, accounting for over 26%. This suggests that modeling is still underexplored with regard to bio- and geo-based building materials.

Secondly, the percentage of works exploring physics and chemistry has been determined as a proportion of the total number of publications for each research area (Figure 14).

Chemistry's contribution to bio-based building materials and composites is very similar. It is slightly less represented for raw earth building materials. However, the notable disparity lies in the field of physics. Physics appears to be relatively underexplored in the field of eco-materials, whereas it plays a significant role in more than a third of publications in the composite domain.

In conclusion, it appears that physics and modeling tools are probably the levers to be considered for improving knowledge of bio- and geo-sourced building materials and contributing to their development.

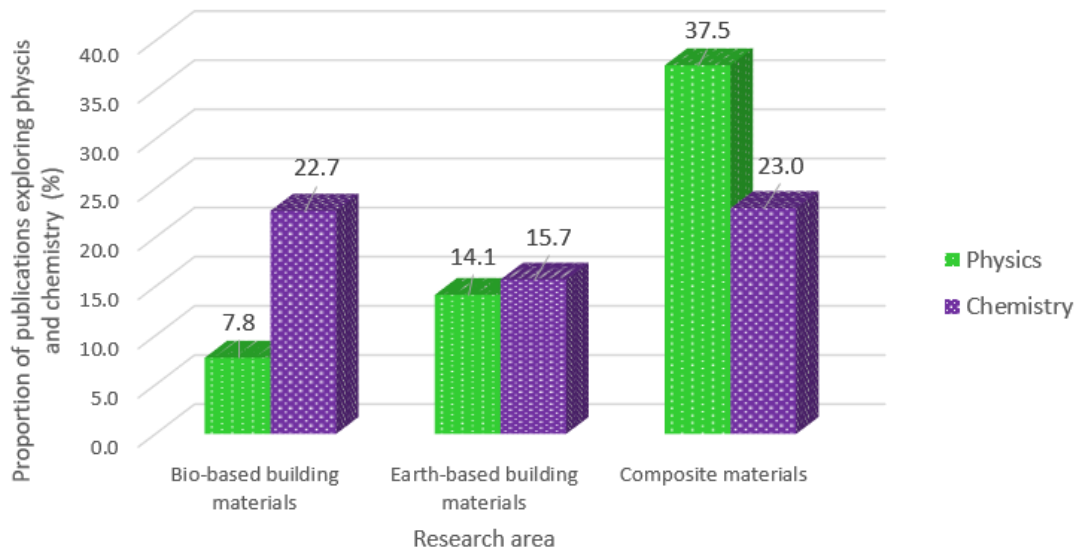


Figure 14. Proportion of publications, from 1900 to 2022, dealing with physics or chemistry in different types of materials according to Web of Science data.

3.3. Bibliometric Networks

3.3.1. Bio-Based Building Materials

- Main authors and their collaborations

The analysis of the co-authorship link reveals the main contributors in the field of bio-based building materials (Figure 15). The advantage of visualization maps is to enable an appreciation of the collaborations between the different authors, in addition to their individual contribution. To provide a temporal dimension to the visualization, a time scale can be added.

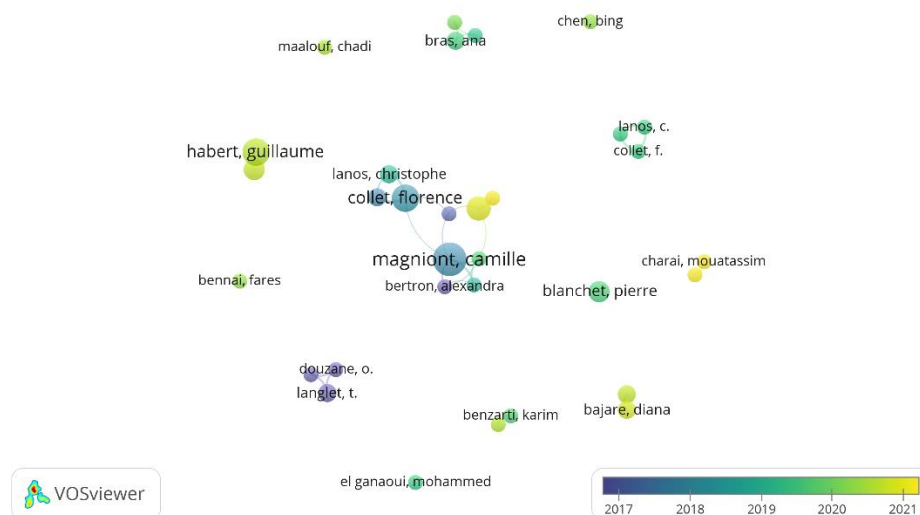


Figure 15. Co-authorship networks of authors (with minimum of five publications) concerning bio-based building materials, with time scale over the last few years.

This map highlights the main authors identified in the previous study. Collaboration between major authors appears to be generally low, especially if they have different affiliations. The

only notable exception is the recent collaboration between colleagues from the University of Toulouse and Rennes. Moreover, according to these results, it appears that international cooperation is currently limited.

- Founding works

An interesting feature is the possibility of identifying the founding works of the discipline. The analysis of reference co-citation links reveals the most widely cited and associated works (Figure 16). For better readability of references and their importance, the visualization density map has been preferred to the visualization network map.

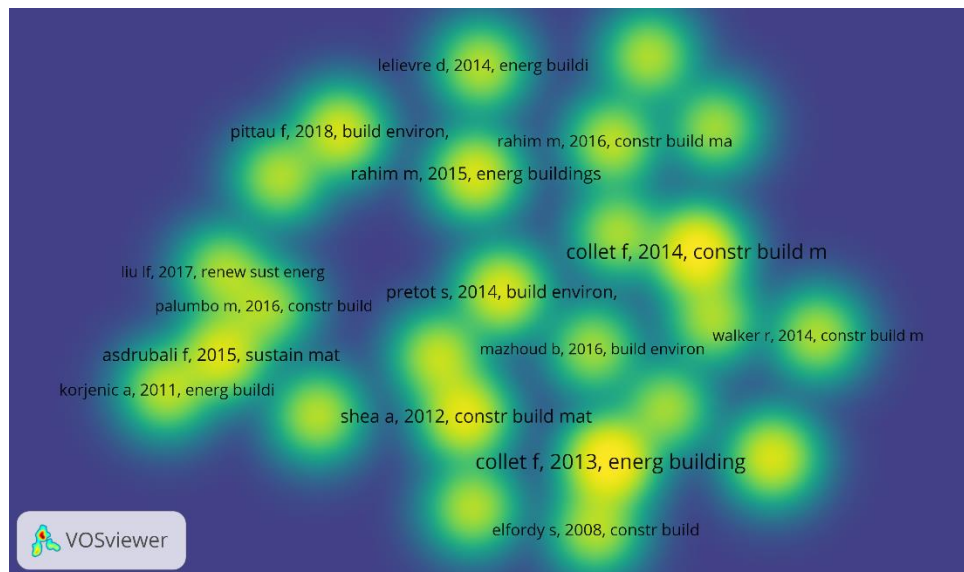


Figure 16. Founding works concerning bio-based building materials [88–102]

This approach can be very useful for targeting the main works to be consulted when beginning a research work on bio-based building materials. It can be pointed out that these publications are globally concentrated around the year 2015.

Key themes in the literature

Analysis of the co-occurrence of keywords in the 570 selected publications (cf. section 2.2) reveals four clusters, representing the four main themes explored in the literature (Figure 17).

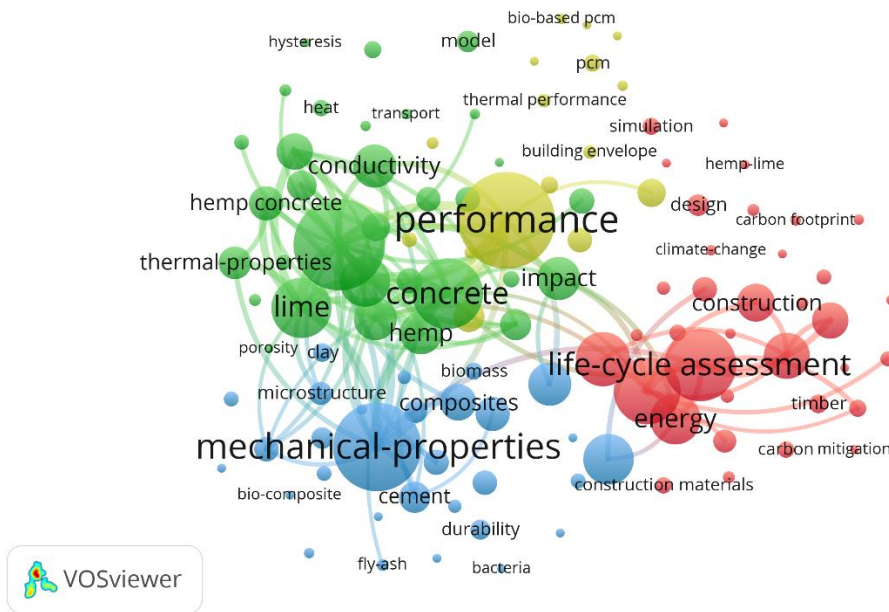


Figure 17. Co-occurrence networks of keywords concerning bio-based building materials.

The analysis of the clusters generated by the VOSviewer software allows their identification (Table 8). However, it is worth mentioning that this is not always easy, as some themes are shared by several clusters, such as mechanical properties. Therefore, the general theme assigned to each cluster is an interpretation based on the articles identified in each cluster.

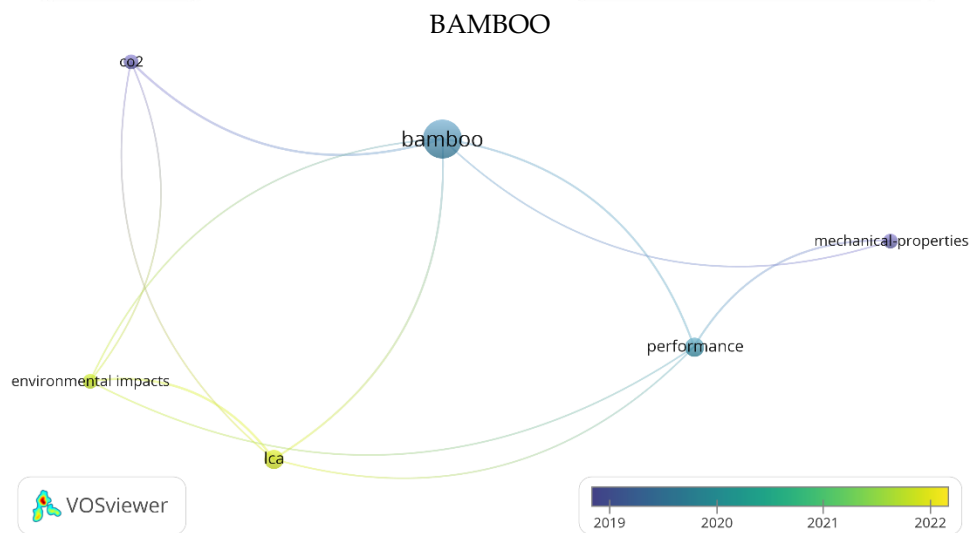
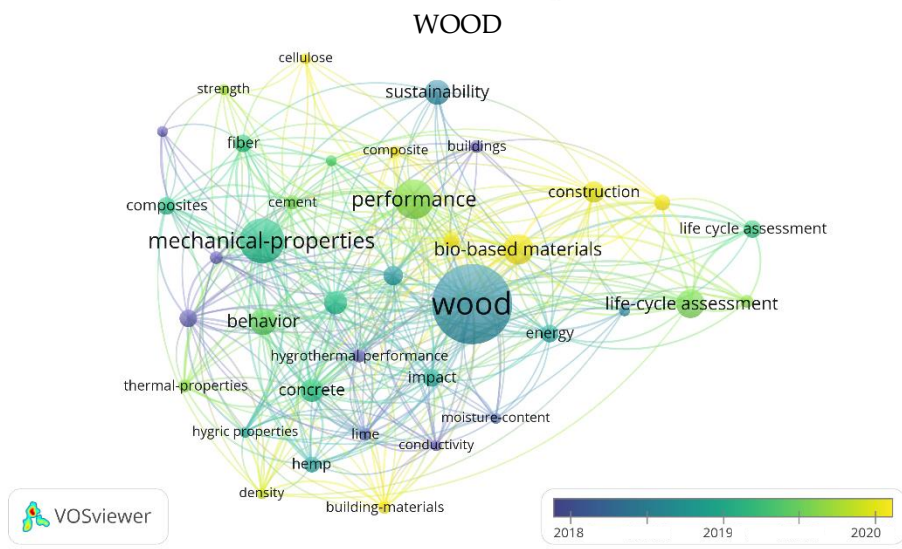
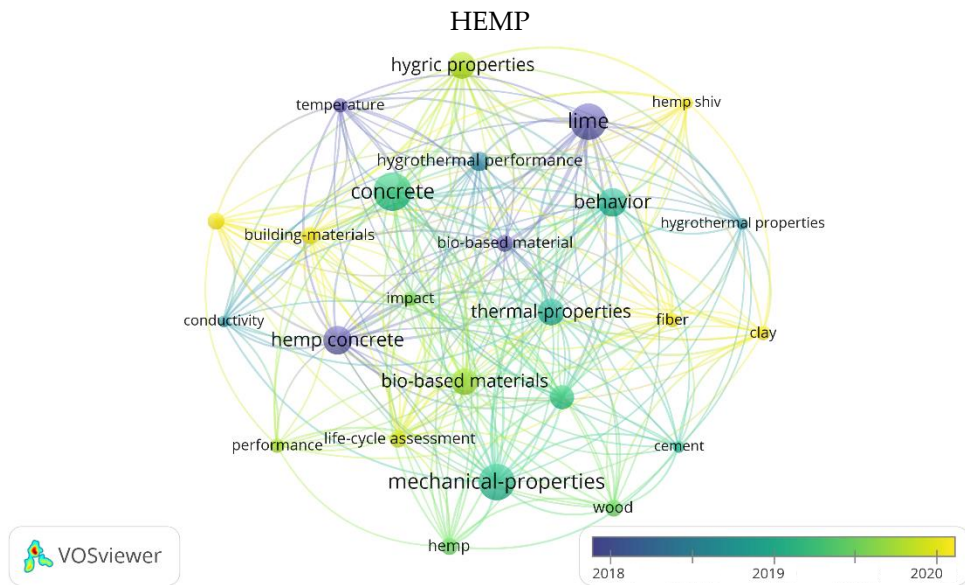
Cluster	Associated Color	General Theme
1	Red	Environmental impact
2	Green	Thermo-hydric behavior
3	Blue	Mechanical properties
4	Yellow	Building-scale performance

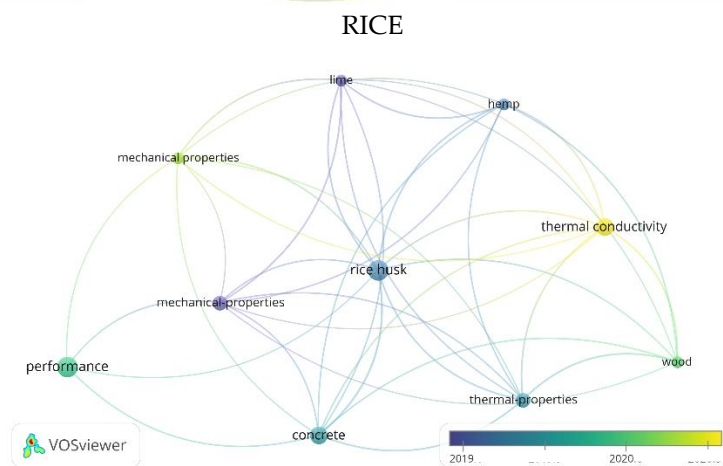
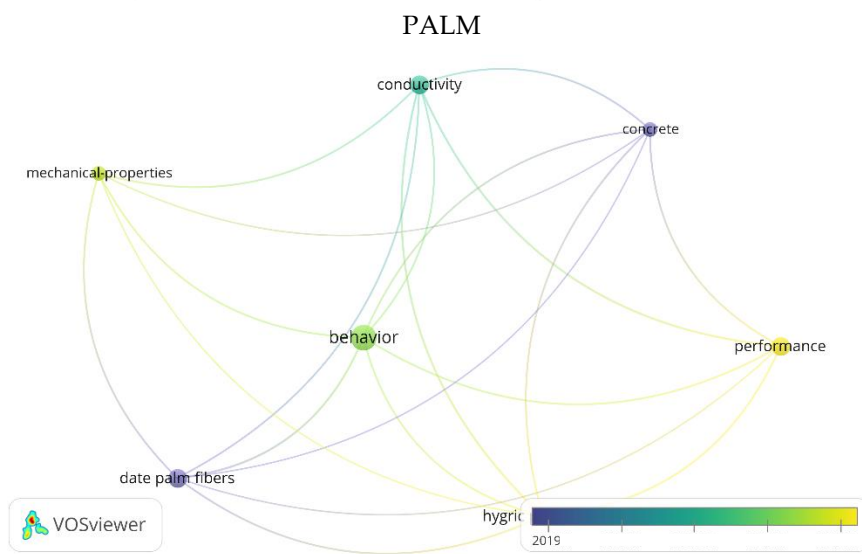
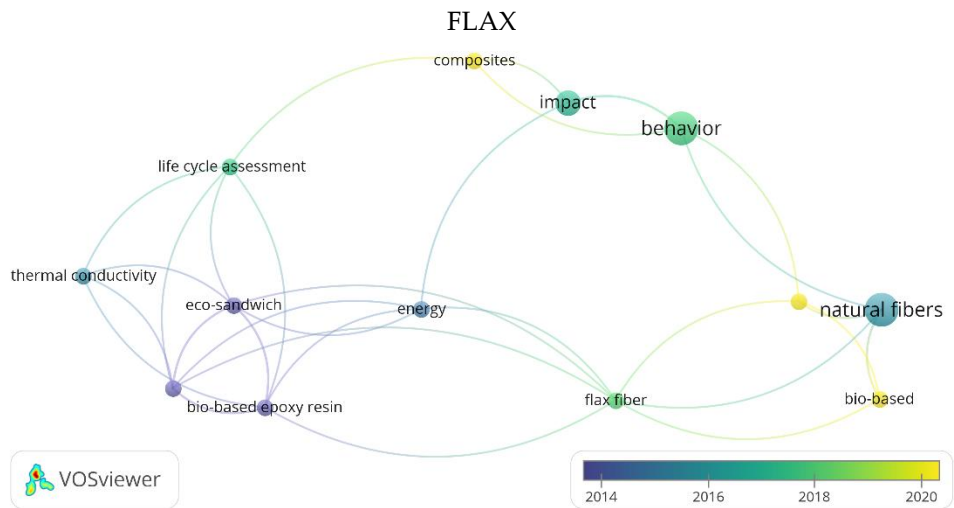
Table 8. Main areas of research according to cluster analysis.

The complexity of bio-sourced materials may explain the interweaving of the different themes. Nonetheless, these research topics enable one to identify the current areas of research. Interestingly, none of the clusters directly refers to the hard sciences like physics, and the concept of “model” appears only modestly in the cluster related to thermo-hydric behavior.

- Focus on main raw materials

In order to provide a closer insight into the contributions made to date on the main raw materials identified in Section 3.1.1, an analysis of keyword co-occurrence was carried out within the relevant publications. The visualization includes a time scale to emphasize trends over recent years. For the sake of relevance, this section focuses exclusively on primary resources with more than 10 publications each: hemp, wood, bamboo, flax, palm, rice, straw, sunflower (Figure 18).





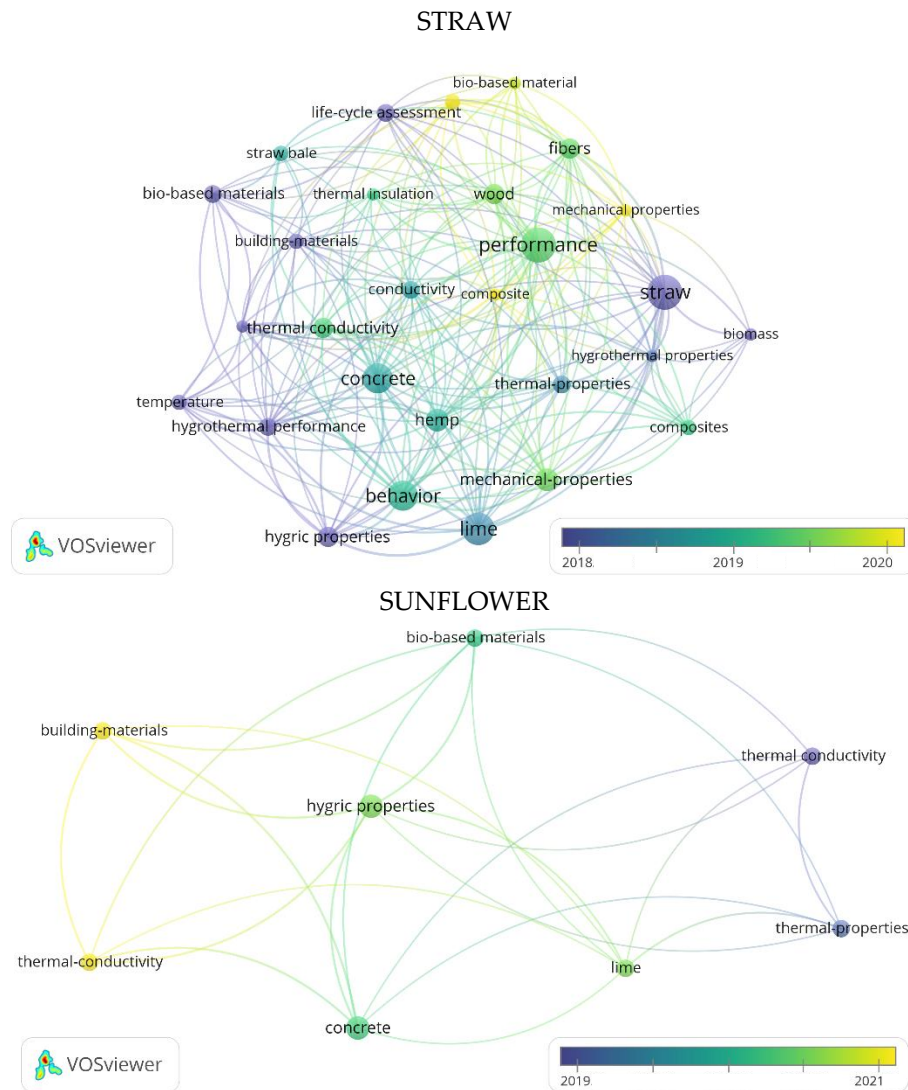


Figure 18. Co-occurrence networks of keywords concerning publications about the most widely studied raw materials in the literature.

These maps highlight the keywords corresponding to the topics mainly studied for each raw material. The notion of life cycle assessment appears to be one of the most discussed subjects in the literature concerning the raw materials considered. However, this subject does not emerge for some of them, thereby highlighting new areas of work. This multi-criteria analysis enables us to objectively demonstrate the benefit of using bio-based materials compared to conventional ones [7]. Notably, the concepts of performance (thermal, hygrothermal, mechanical) are well represented, in line with their function in construction. Wood, hemp and straw naturally yield a richer array of keywords due to their extensive research focus. Wood, used as a structural element, is highlighted by the keywords “timber” and “mechanical properties”. Hemp shiv, often added to a binding matrix (lime, cement) for hemp concrete, is highlighted by the map. An earth-based binder matrix is also possible, which explains the keyword “clay”. Straw, used for insulating wood structures or as a substitute for hemp in plant-based concrete, offers properties very similar to those of hemp. This explains the great similarities between the maps for straw and hemp.

All of the topics covered in wood, hemp and straw are inspirational sources for continuing to develop our knowledge of other raw materials applicable to bio-based building materials. These include the following topics:

- “Cellulose”: linked to the chemical composition of the resource, particularly relevant to wood as a primary chemical compound.
- “Moisture content”: in relation to water adsorption by the material and potential pathologies arising under such conditions.
- “Sustainability “: referring to the evolution of a material's properties under various stresses, including temperature and relative humidity variations over its lifetime.
- “Energy”: related to the overall energy efficiency of the building, i.e., covering a more global scale than that of the material or the wall.

It should not be overlooked that the development of bio-based building materials also requires taking into account the diversity of resources, which probably has an impact on their use properties [103,104]. Lastly, the use of local raw materials necessitates the establishment of effective harvesting and recovery procedures [76].

3.3.2. Geo-Based Building Materials

- Main authors and their collaborations

The main contributors in the geo-based building materials sector and their collaborations are presented (Figure 19).

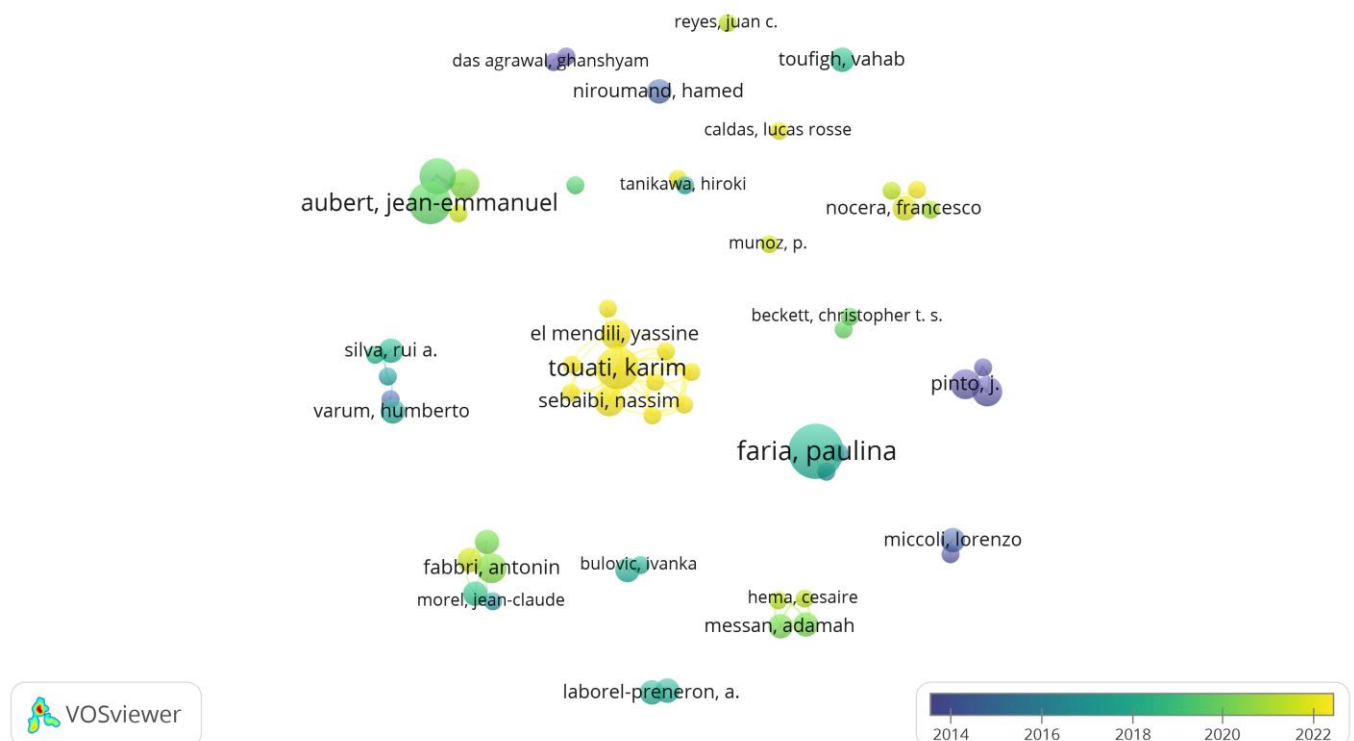


Figure 19. Co-authorship networks of authors (with minimum of three publications) concerning geo-based building materials, with time scale over the last few years.

According to this map, collaboration between authors appears even more limited than that for bio-based building materials. The few collaborations that are visualized mostly involve authors from the same affiliation. The time scale shows that the number of collaborations have been multiplying very recently. No significant European or international collaborations are to be reported, since they involve authors from the same country and often from the same affiliation.

- Founding works

The most frequently cited research works date from the 1990s to 2000s (Figure 20). These works are considered foundational in the field.

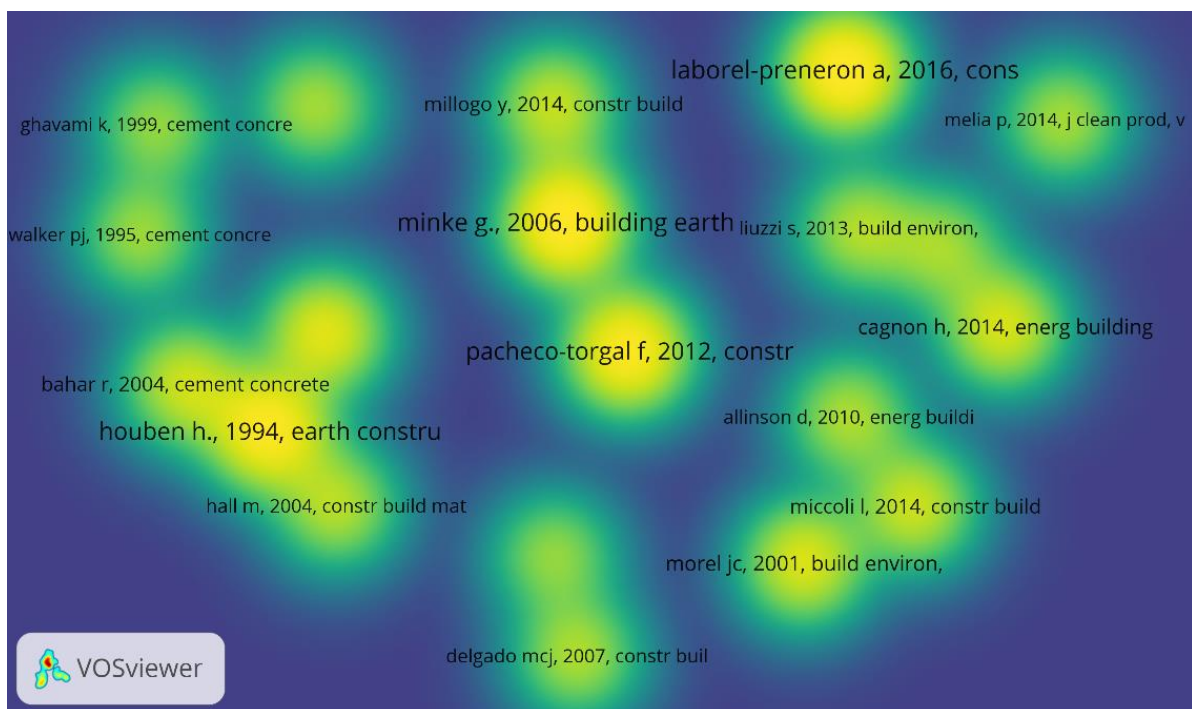


Figure 20. Founding works concerning geo-based building materials [14,15,51,105–117]

Similarly to bio-based materials, this list of works can help identify fundamental results in the field of raw earth building materials research.

- Co-authorship links

Analysis of the co-occurrence of keywords in the 504 selected publications (cf. Section 2.2) reveals four clusters, i.e., four main themes explored in the literature (Figure 21 and Table 9).

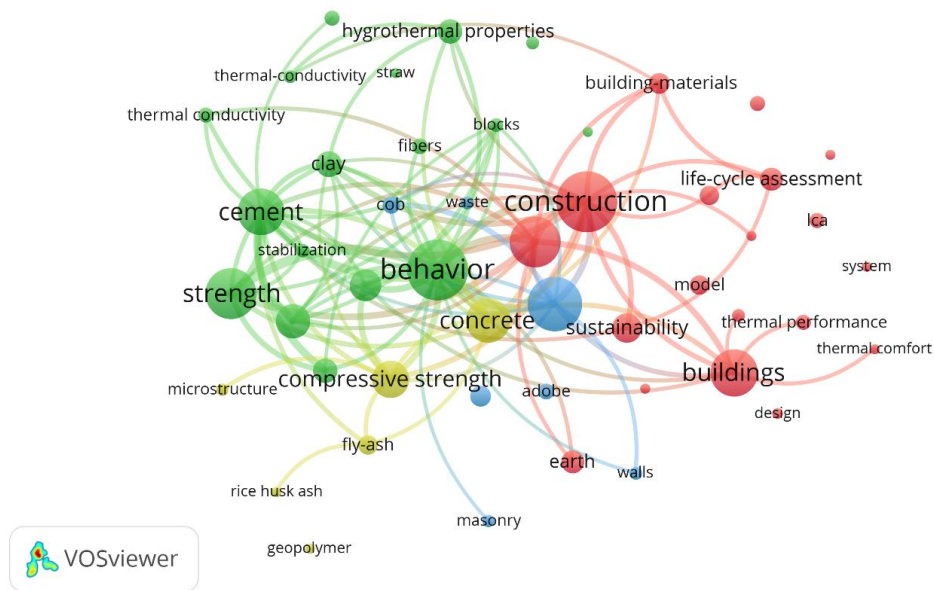


Figure 21. Co-occurrence networks of keywords concerning geo-based building materials.

Table 9. Cluster analysis (in decreasing order: n°1 corresponds to the greatest number of publications).

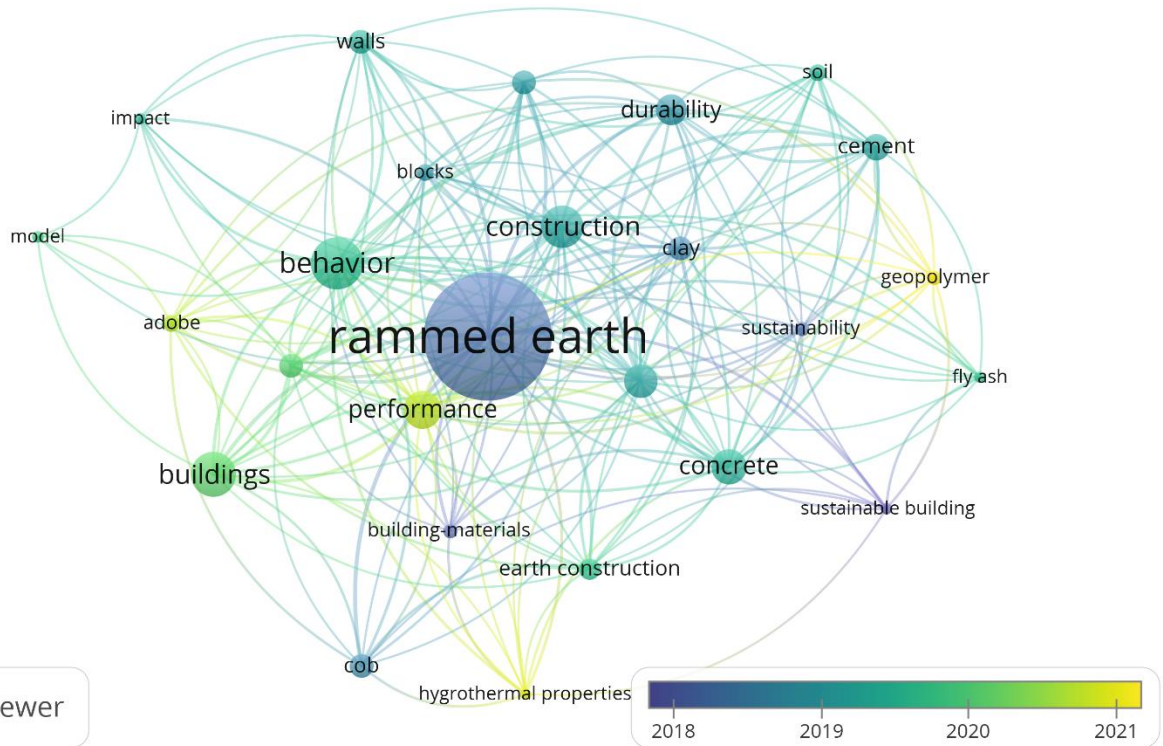
Cluster	Associated Color	General Theme
1	●	Environmental impact
2	●	Thermo-hydric behavior
3	●	Construction techniques
4	●	Mechanical behavior

The keywords mainly associated with the research work do not highlight strong themes related to the hard sciences. It is reasonable to conclude that physics in particular is a little-explored field. Moreover, modeling, which is identified in the “environmental impact” cluster, is clearly very little covered.

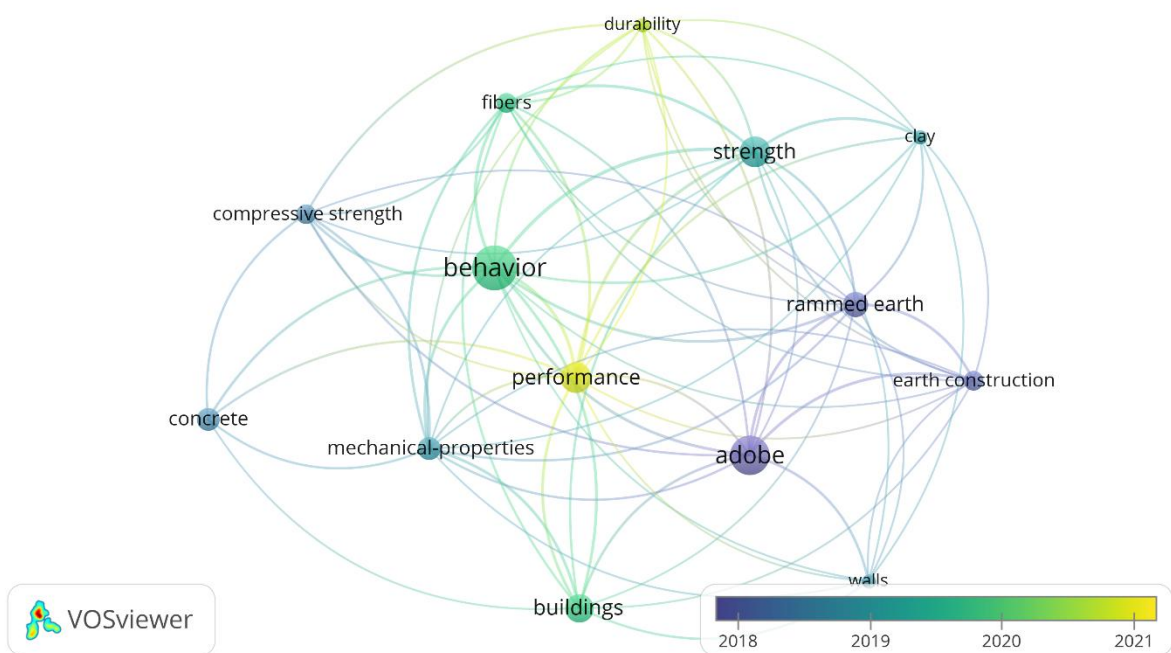
- Focus on implementation techniques

In order to provide a closer insight into the contributions concerning implementation in Section 3.1.2, an analysis of keyword co-occurrence was carried out on the publications relating to them. The time scale is added to the visualizations to underline the trend over the last few years. All techniques are included, as they have been studied in over 10 publications (Figure 22).

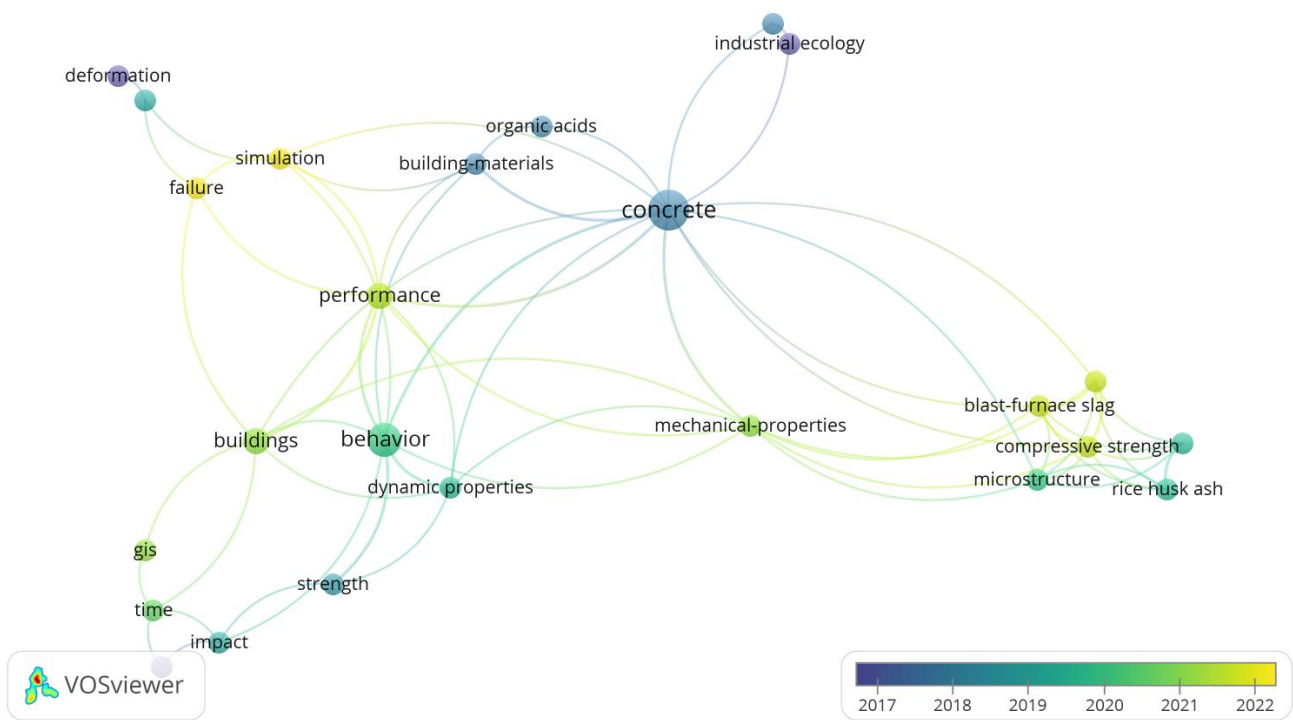
RAMMED EARTH



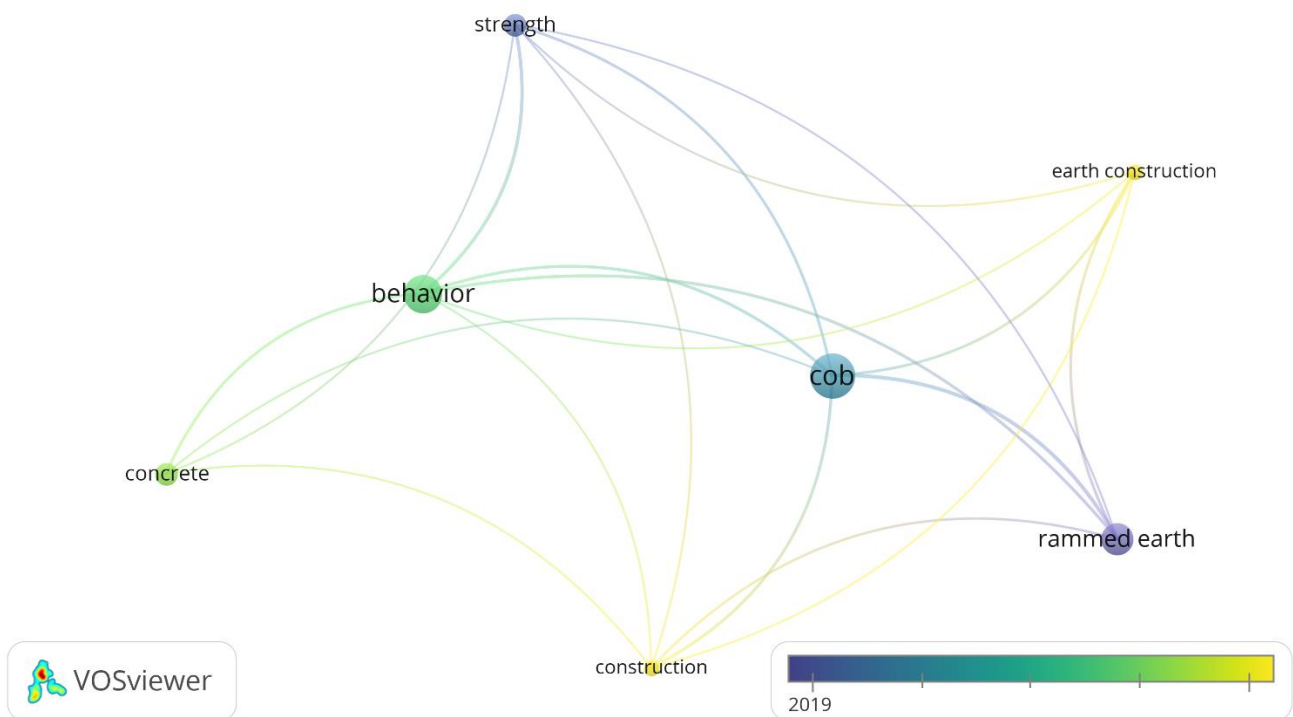
ADOBE



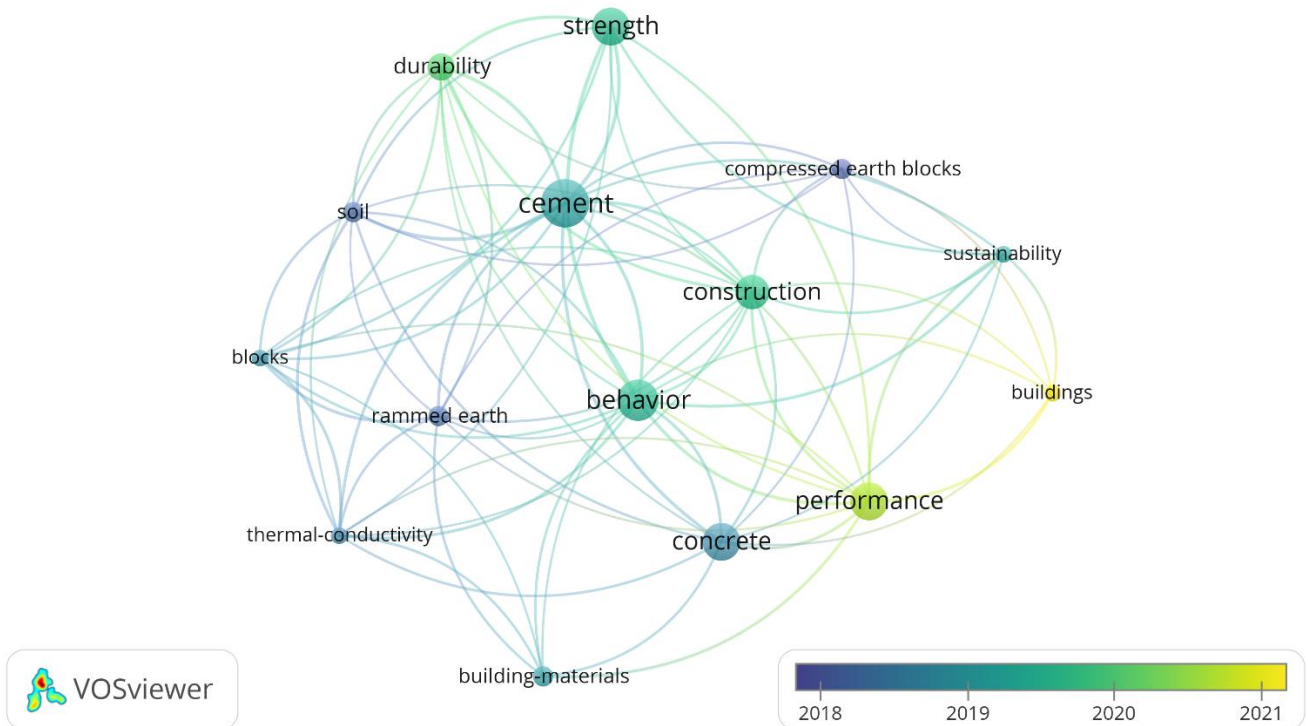
WATTLE AND DAUB



COB



COMPRESSED EARTH BRICKS



EARTH PLASTER

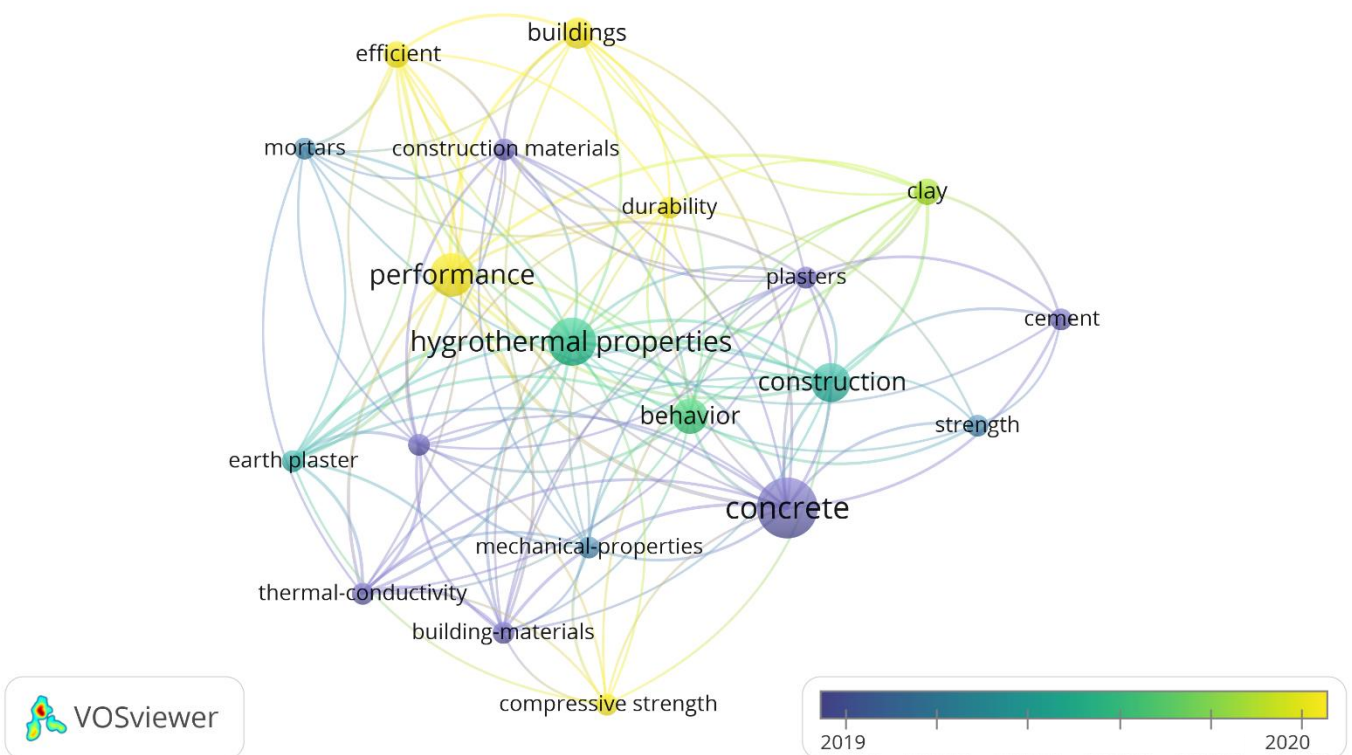


Figure 22. Co-occurrence networks of keywords concerning publications about the most widely studied raw materials in the literature.

In load-bearing techniques such as rammed earth, adobe, cob and compressed earth blocks, terminology related to mechanical properties is logically prevalent. For wattle and daub, the subjects span a broader spectrum, ranging from microstructure to mechanical properties and organic acids. The hygrothermal properties of plasters are clearly targeted by the studies carried out to date. It is interesting to note that the term “cement” appears on many cards. This may appear surprising, but it certainly stems from the systematic comparison of earth materials with conventional ones. The emergence of the terms “fibers” and “rice husk” demonstrates the close link between bio- and geo-based building materials. Identified areas for further exploration to address certain limitations likely include:

- Variability of the raw resource [118,119]: raw earth is a mixture of clay, silt, sand, gravel and pebbles of varying natures and proportions, depending on where it was extracted [120].
- Pre-conceived ideas: earth can suffer from a bad image for economic, political and social reasons [24,60]
- Traditional craftsmanship: as earth is a material used for millennia, the implementation techniques are often the result of real know-how sometimes difficult to reconcile with current techniques and constraints [60,121].
- Life cycle assessment: as earth is a low-embodied carbon construction material [24], life cycle assessment methodology may adequately demonstrate its potential in place of conventional materials.

3.3.3. Potential emerging themes

The visualization maps in the sections focusing on bio- and geo-based building materials enable one to appreciate the most frequently addressed themes and, consequently, those that are less explored. To further enhance the analysis, a co-occurrence map of key words corresponding to articles on composite building materials in 2022 has been generated (see Section 2.2). By comparison, this allows the detection of different themes that could potentially contribute to the development of eco-materials (Figure 23).

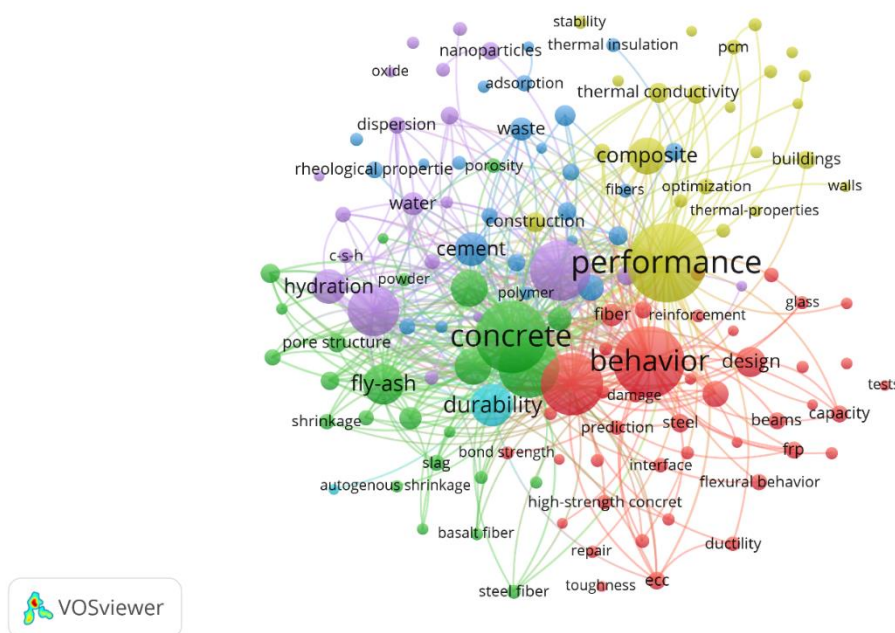


Figure 23. Co-occurrence networks of keywords concerning composite building materials (2022).

Logically, topics similar to eco-materials can be found, such as mechanical behavior and performance. Performance is linked in an interesting way to optimization, which was not identified in the case of bio- and geo-based building materials. This suggests it as a first avenue of study.

In the context of composite building materials, it is worth noting that the study of behavior is linked to modeling with the notion of machine learning. This link was not represented in previous maps, making it another avenue of exploration.

Additionally, emerging themes of interest include durability and microstructure. Sustainability is linked to all of the other clusters, demonstrating its importance in research themes, potentially serving as a third avenue of exploration. Microstructure is widely explored, even down to the nanometric scale according to the themes identified, such as nanoparticles, nanocomposites, nanosheets and graphene. Given its significance in composite materials, it would probably be wise to explore microstructure at a similar scale in bio- and geo-based building materials.

4. Conclusion

In conclusion, a statistical analysis using Web of Science data provided an overview of the disciplines considered: bio- and geo-based building materials. It highlights, in particular, the growing dynamism of research with the increasing number of publications in recent years. The main contributing authors, sources and countries are clearly identified. In terms of volume, the main contributing countries today are China, France and the United States. The authors who contribute the most tend to be European.

In addition, this study relied on a bibliometric network visualization software application. It confirmed the main contributors and also allowed us to appreciate their contribution. Overall, collaborations on a European and global scale remain very limited. A complementary analysis made it possible to identify the major works in each discipline, providing a significant asset for gaining a more academic perspective.

Finally, the combined use of visualization maps and statistical analysis confirmed areas to explore in order to gain visibility in the field of bio- and geo-based building materials. A table summarizes the elements that are particularly useful when starting research work in these areas. It also indicates the main work perspectives proposed following this in-depth analysis (Table 10).

Table 10. Main conclusions of the study.

Topic	Major Authors	Main Subcategories Studied	Research Perspectives
Bio-based building materials	Collet F. Lanos C. Blanchet P. Magniont C. Habert G. Pretot S. Lawrence Mike Langlet T. Maalouf C.	Hemp Wood Straw	Modeling Physics Microstructure Chemical composition Sustainability Energy Resource diversity Structuring the local sector
Earth-based building materials	Aubert J.E. Faria P. Laborel-Préneron A. Magniont C. Humberto V. Niroumand H. Pinto J.F Beckett C. Zai M.F.M Hamard E. Zai	Rammed earth	Modeling Physics Microstructure Variability of the earth Traditional craftsmanship to modern techniques Pre-conceived ideas on earth

Lastly, recent developments in the IT field offer an additional opportunity. Quantum computers, with increased computing capacities, may be able to address the current challenges of computing time in modeling [122]. Artificial intelligence, notably with digital twins or neural networks, also holds a promising future in advancing scientific knowledge, especially in civil engineering [123–128]. Seizing upon these new tools and the themes highlighted in this article can contribute even further to the development of bio- and geo-sourced materials.

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Chapitre 3. Apports de l'échelle moléculaire

Préambule

Une des caractéristiques remarquables des matériaux de construction bio-sourcés est leur capacité à réguler de manière passive l'humidité de l'ambiance intérieure du bâti. En effet, lorsque la proportion d'eau à l'état de vapeur augmente dans l'air (i.e. l'humidité relative), ces matériaux fixent naturellement de l'eau par un mécanisme de sorption. Ils sont capables de désorber les molécules d'eau lorsque l'humidité relative de l'ambiance diminue, de manière spontanée et sans aucun apport d'énergie. Ce phénomène est mis en évidence par de nombreux travaux expérimentaux dans lesquels la teneur en eau dans le matériau est mesurée en fonction de l'humidité relative, et ce à une température donnée. Ainsi, le graphe obtenu, appelé « isotherme de sorption », montre l'évolution de l'état sec à l'état saturé en eau (i.e. lorsque la pression de vapeur saturante est atteinte et que l'eau se liquéfie dans le matériau). A l'inverse, lorsque la courbe rend compte du processus inverse, on parle d'« isotherme de désorption ». Dans le cas des matériaux de construction bio-sourcés, les deux courbes de ne se superposent pas. Ce phénomène d'hystérèse est encore mal compris.

Ce chapitre explore notamment le domaine de la chimie, en présentant la possibilité de liaison hydrogène entre le granulat végétal -en considérant sa composition chimique riche en cellulose- et les molécules d'eau contenues dans l'air ambiant. Ainsi, un nouveau mode de fixation de l'eau est proposé. Cet article est le fruit d'une réflexion profonde et argumentée. Elle s'appuie sur des résultats récents obtenus par simulation moléculaire, expliquant le phénomène d'hystérèse sur des polymères nanoporeux. L'hypothèse d'une transposition aux matériaux bio-sourcés permet d'expliquer de nombreux résultats de la littérature, comme le gonflement des granulats végétaux par sorption d'eau, l'existence de courbes primaires de sorption ou encore la dépendance en température des phénomènes de sorption et désorption. C'est en fait un mécanisme de couplage hydromécanique qui est ainsi envisagé. Il vient compléter les pistes fournies à ce jour dans la littérature pour expliquer le phénomène d'hystérésis observé sur les matériaux de construction bio-sourcés.

Article B. Microstructure of Bio-Based Building Materials: New Insights into the Hysteresis Phenomenon and Its Consequences

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Abstract: Considering the current energy environment, both efficient and environmentally friendly solutions have to be developed for building construction. Bio-based building materials offer new perspectives through their insulating and natural humidity regulation capacities. Nevertheless, these materials are as complex as they are promising, and grey areas still remain regarding their behavior. Their water sorption and desorption curves recorded in experimental work demonstrate a hysteresis phenomenon and, although plausible hypotheses have been formulated in the literature, there is currently no consensus on its causes. Furthermore, it is important to emphasize that no reference considers the hydrophilic nature of the resource. Yet this is a specificity of raw material coming from the plant world. In this context, this paper explores the microstructure and chemical composition of plant aggregates to propose a new explanation for the hysteresis. It is based on recent work demonstrating the existence of differentiated hydrogen bonds between the water sorption and desorption phase in cellulose. Obviously, hysteresis also has an origin at the molecular scale. Lastly, the hypothesis put forward here is supported by the swelling of bio-based materials that has been observed at high relative humidity and this study aims to identify a link between the mechanical of bio-based materials and their hygroscopic behavior. A swelling/shrinking is macroscopically observed. Combining the fields of chemistry, physics, and civil engineering allowed to demonstrate it comes from a molecular-scale hydromechanical coupling. This is a major breakthrough in the understanding of bio-based composites.

Keywords: Bio-based building material; hysteresis; swelling; microstructure; cellulose

1. Introduction

The study of bio-based materials is quite recent and works on these promising materials have increased over the past twenty years. This is probably due to their multiple advantages: local availability of resources, recovery of agricultural waste, low grey energy, limited environmental impact, and improvement of energy efficiency or even indoor air quality. Today, given the energy and climate upset facing humanity, large-scale development of these materials has become essential. Therefore, more and more authors are focusing on the experimental characterization of these materials, considering different binders and/or vegetal aggregates [1]–[6]. Those experimental data are fundamental. However, the literature is still limited concerning reflection on the phenomena underlying the macroscopic observables. So, this study aims to focus on the phenomenon of hysteresis. Although it is widely observed experimentally [7]–[11], its causes are still poorly understood. This article provides a new consideration of the microscopic origin of the phenomenon at the molecular level.

First, some specific characteristics of bio-based materials are recalled. In particular, experimental observations and recent results concerning these materials are highlighted. It is also stressed that, to date, there is no consensus on how hysteresis in bio-based materials can be understood and described. Then, the hydrogen bond is briefly defined in order to facilitate understanding of a recently conducted study on sorption-swelling coupling in nanoporous materials [12].

The results of this study are then applied to bio-based building materials. The interest and relevance of considering the hysteresis phenomenon differently are highlighted: first at the microscopic molecular scale so that the plant aggregate's specific nature can be considered, then by exploring, one by one, the macroscopic consequences found in the literature. They are explained with the new theory put forward. This article thus proposes a new explanation, supported by numerous arguments, of hysteresis and its consequences.

2. Focus on bio-based building materials and their specificities

2.1 Microstructure

Bio-based building materials are made of agricultural by-products mixed with a binder (lime-based, metakaolin-based, clay, etc.). This leads to a microstructure that is both complex and variable, depending on the formulation, how the plant was grown, or even manufacturing [1], [13]. A multi-scale porosity is also underscored by several studies [2]–[4], [9]. Bio-based and conventional materials have comparable intrinsic binder porosity due to the arrangement of hydrates and trapped air. However, the final porosity values for bio-based materials also include the additional contributions of inter particulate and intra particulate porosity, and the porosity at the binder-aggregate interface (Fig 1).

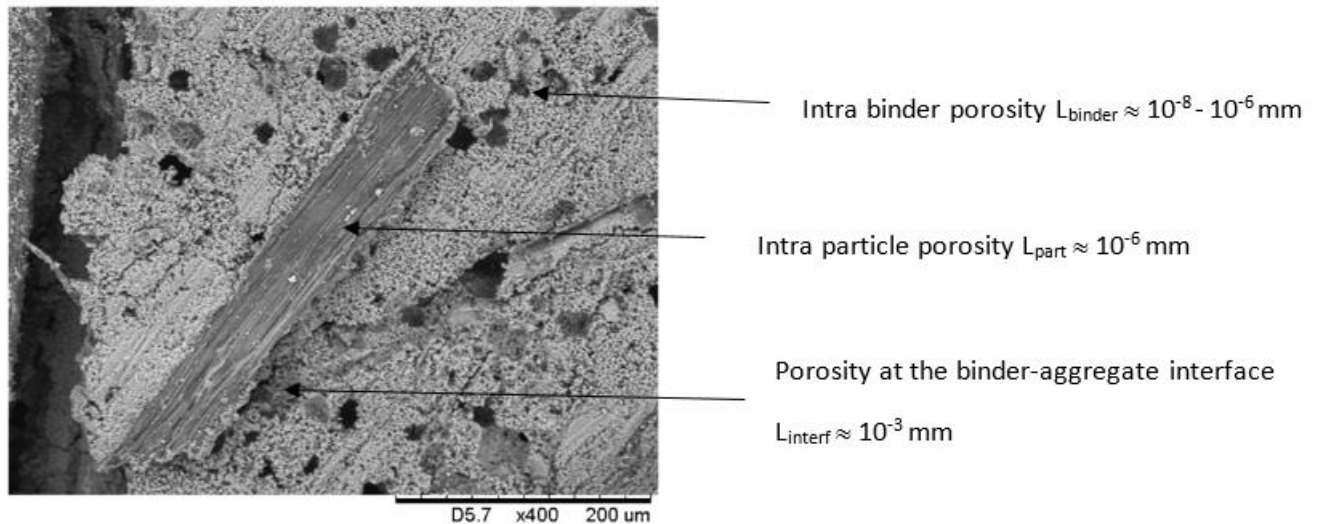


Fig 1. Multi-scale porosity of hemp concrete observed with SEM and L_i , order of magnitude [4].

Thus, this large, interconnected and complex porosity allows the mass transfer of water within the microstructure.

A large porosity can be observed more closely at the intra particulate scale (Fig 2).

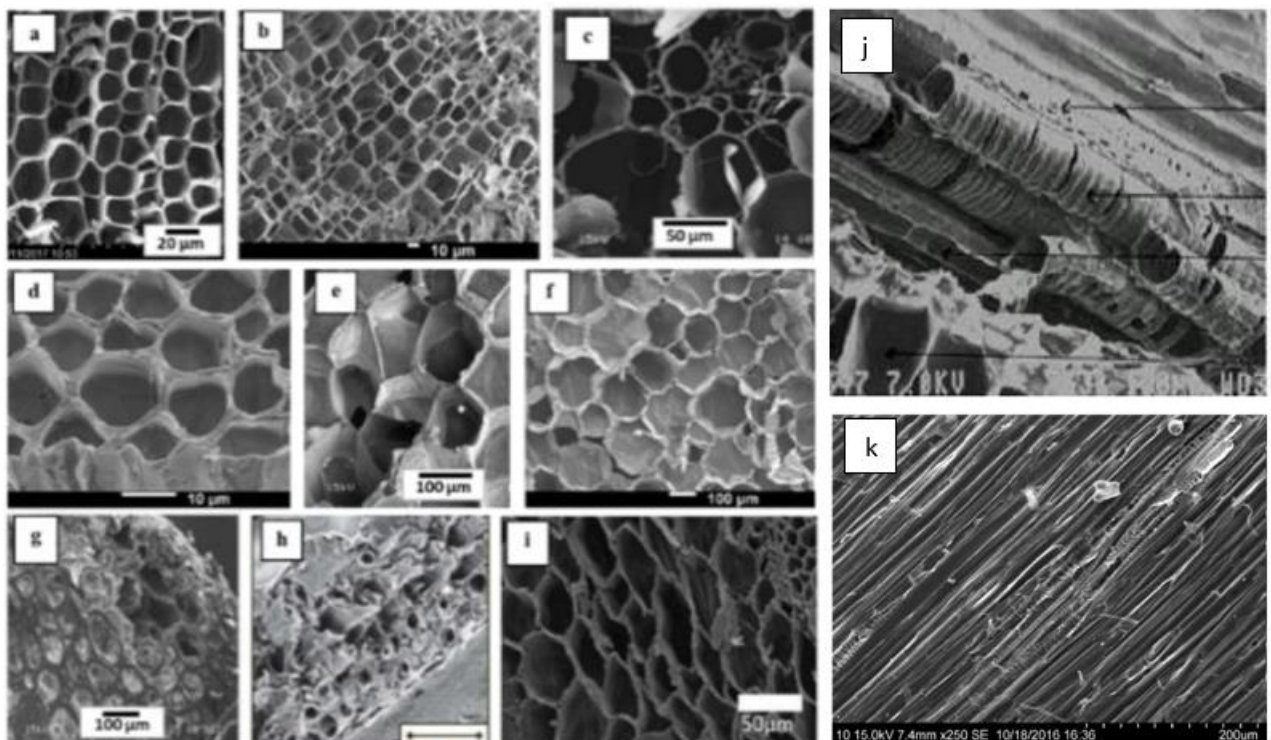


Fig 2. SEM cross-sectional images of (a) hemp shiv, (b) flax shiv, (c) barley straw, (d) wheat straw, (e) sunflower pith, (f) rape straw, (g) corn cob, (h) rice husk, (i) miscanthus stem [7], longitudinal (j) and transversal (k) images of hemp shiv [8] [9].

These tubules allow the sap to circulate when the plant is growing. After drying, they fill with air, which causes intra-particle porosity, explaining, in particular, the insulating capacity of bio-based building materials. It is useful to note that a tubular morphology exists in a wide range of plant aggregates.

2.2 Hygroscopic properties

Hygroscopic material is able to fix and store water depending on moisture conditions. This capacity is governed by the pore size distribution [14]. According to many studies, both binder and plant particles are hygroscopic materials [3], [9], [13], [15]–[17]. This means that they are able to adsorb excess water from the environment (in the form of vapor that condenses under certain conditions) and desorb this water when the environment is drier. This natural humidity regulation capacity is a major asset as it contributes to healthier indoor conditions. Sorption effects are widely described in the literature by Van der Waals interactions between water and the pore walls. The type of binding that causes poly-molecular adsorption and capillary condensation are not specified. Thus, three different mechanisms schematically describe how water is adsorbed in the pore network of bio-based building materials. (Fig 3) [18]–[22].

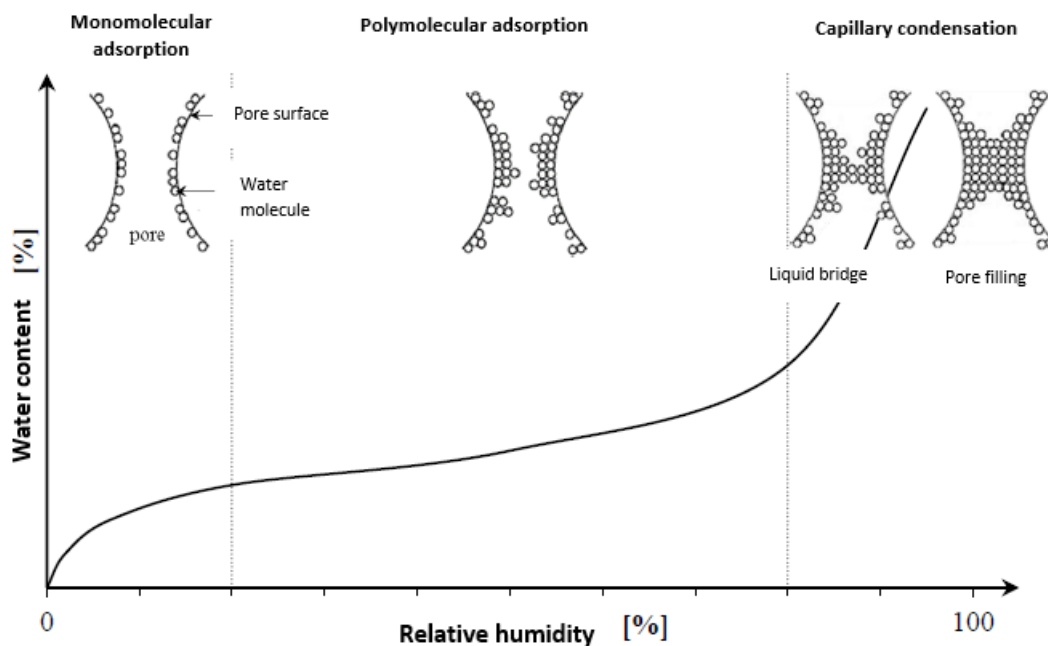


Fig 3. Modes of water fixation in a pore [22].

Up to now, the nature of the solid skeleton surrounding the pores has not been differentiated. The same fixing modes are suggested for both the organic and the mineral parts of bio-based materials.

2.3 Chemical composition

Bio-aggregates are mainly composed of cellulose, hemicellulose, and lignin [23]–[26]. Because cellulose is the major component [27], [28], it is interesting to take a closer look at its microstructure. This bio-polymer is organized into chains of glucose. They are linked together by hydrogen bonds (interchain or intermolecular bonds) [12], [29]. The cellulose chains are organized in the form of microfibrils, the arrangement of which determines the deformation capacity of the plant particle [30] (Fig.4).

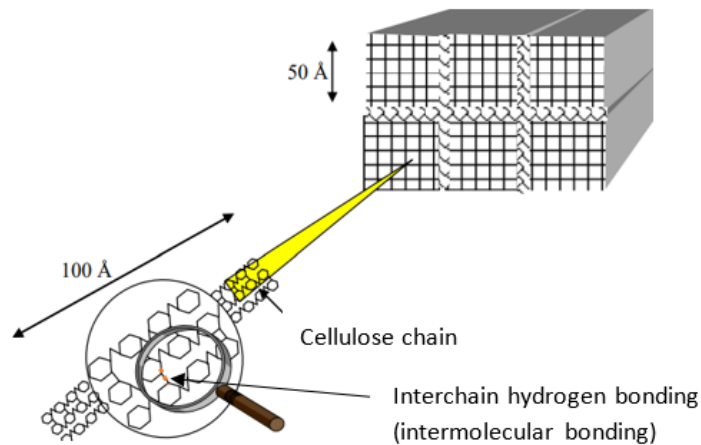


Fig 4. Cellulose microfibrils and inter-molecular hydrogen bonding [30]

In reality, cellulose chains are not always perfectly aligned (crystalline part) and amorphous regions are present, especially in natural cellulose [31] (Fig 5). Some authors indicate that cellulose is mainly crystalline in agro-resources [32], while dosages on hemp shiv have shown very low crystallinity levels [33]. However, the sources agree that amorphous and crystalline zones coexist within plant walls. The proportion of each part is variable and depends on the harvesting zone, the method of extracting the fibers from the plant, and the treatments applied [34].

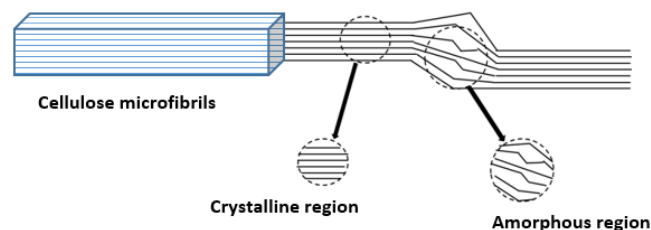


Fig 5. Semi-crystallinity of cellulose, adapted from [35]

Cellulose chains have both intra- and inter-molecular hydrogen bonds. The hydroxyl groups on the surface of the chains are more accessible to create hydrogen bonds with other molecules than those located inside (Fig 6).

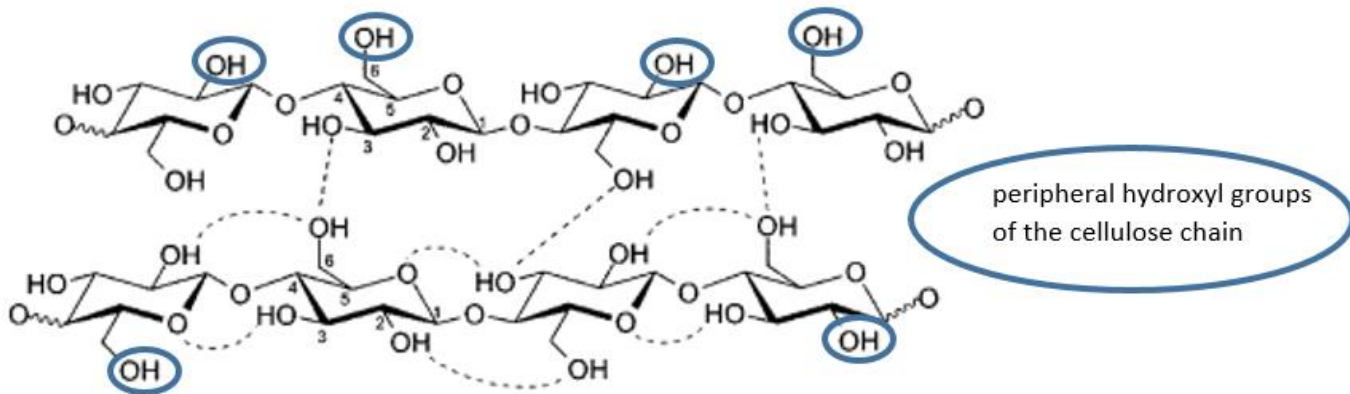


Fig 6. Intra- and inter-hydrogen bonds in cellulose (dotted lines) , adapted from [36].

Thus, the reactivity of cellulose, i.e. its ability to create hydrogen bonds with its environment (with another cellulose molecule or a different molecule), is facilitated by its semi-crystallinity. This means that the steric hindrance is weaker in amorphous portions; space between cellulose chains increases the possibilities of hydrogen bonds with other potential molecules. Consequently, amorphous cellulose is a particularly favorable site for the creation of hydrogen bonds with its environment [37]. This is not the case for mineral or clay binders, as they do not contain cellulose [5], [38]–[40].

2.4 Swelling and shrinkage

Cracking due to plastic shrinkage of composites is well known and is explained by mechanical stresses during drying [41], [42]. Furthermore, other swelling and shrinkage phenomena are reported in the literature, whether at a young age or after a period of accelerated aging [9], [21], [43]. This is observed at the particulate scale under increasing or decreasing relative humidity conditions (Fig.7). The result is a significant change in the aggregate interface, which alters its porosity. This modification of microstructure in bio-materials is simply attributed to the presence of plant particles by the authors and has not been explained to date.

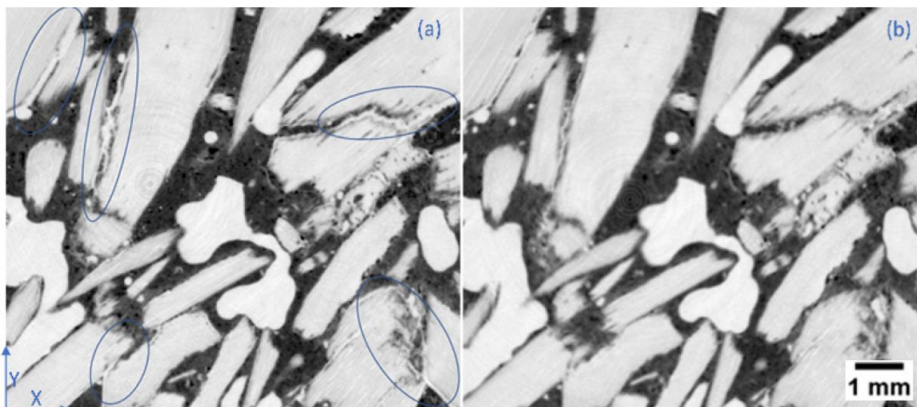


Fig 7. X-ray tomography images of hemp concrete showing the swelling phenomenon between dry and wet states ((a) 0% RH (b) 85% RH) [9].

Swelling and shrinkage impact the porosity of bio-materials [21], [44], [45] and can therefore have effects on their thermal, hydric, and mechanical properties in the more or less long term.

2.5 Functional properties with age

According to recent studies, thermal and hygric properties of bio-based materials change with age [6], [46]. Thus, whether aging is accelerated or not, several authors show that age reduces the adsorption and desorption rate and the water vapor permeability. However, some authors indicate an increase with age for thermal conductivity while others show the opposite [6], [9].

2.6 Temperature effects

The thermal conductivity of bio-based materials is temperature dependent [47]. Moreover, several studies have demonstrated the temperature dependence of sorption curves [11], [17], [48], [49]. Thus, the effects of temperature on the sorption process have been promisingly modeled but no explanation of this phenomenon has been proposed so far.

2.7 Local kinetic sorption

Recent work has highlighted the relevance of considering local sorption kinetics in bio-based materials, especially when coupled with hysteresis [50], [51]. This was motivated by the observation that these materials take a long time to stabilize when the environment changes (more or less humid). The consideration of local kinetics is very relevant to describe the transformation of water from vapor to liquid (and vice versa). This has been experimentally validated. Thus, the fixation of liquid water in hemp concrete would be conditioned by a very slow diffusion of water molecules.

2.8 Sorption hysteresis

Sorption hysteresis observed on bio-based materials is widely reported in the literature [7], [11], [14], [21], [26], [50], [52]–[57]. At a given relative humidity, the eco-material does not have the same water content (after stabilization), depending on the sorption or desorption phase (Fig 8).

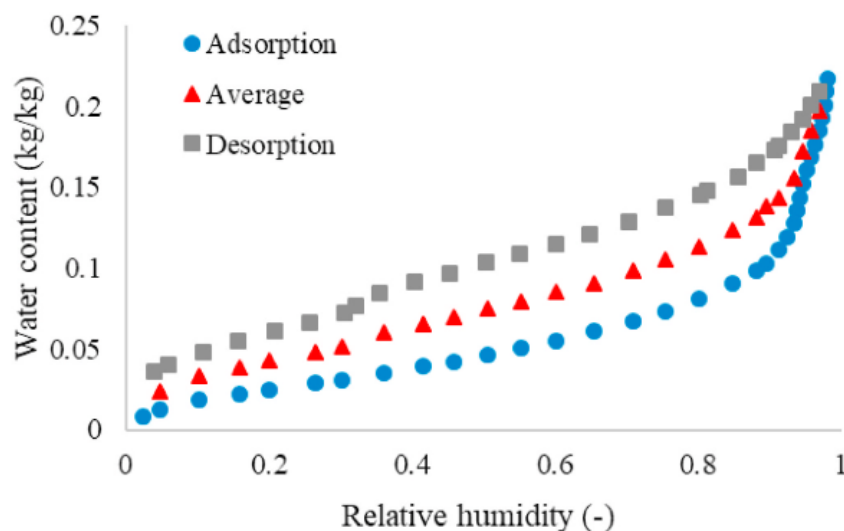


Fig 8. Hysteresis in the case of hemp concrete [7]

This fundamental point must be considered to assess the hygrothermal behavior of bio-based walls. Thus, the following items have been experimentally demonstrated:

- The sorption mechanism is reversible since the original state is obtained at a dry state [56]
- Hysteresis is more pronounced for plant-based concrete materials than for aggregates [24], [54], [56], [58]
- Aging reduces the rate of adsorption and desorption for hemp concrete [59]
- Temperature can influence hysteresis [11] [60], or not [61]
- Swelling of the plant particles or fibers during hysteresis is irreversible [43], [60]
- Hysteresis increases while crystallinity decreases [60]
- Water content is always higher in the desorption than in the adsorption phase for the same relative humidity.

Moreover, the hysteresis phenomenon can be observed on binders alone, whether they are mineral or geo-sourced [8], [14], [62]–[64]. It can also be demonstrated on plant fibers or aggregates (Fig.9) used both in the textile and the building industries [22], [60].

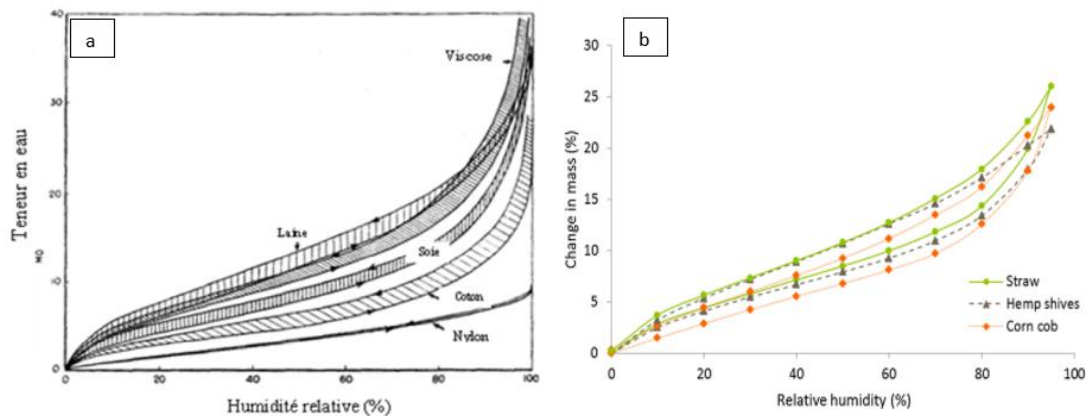


Fig 9. Hysteresis for a wide range of plant fibers and aggregates used in the textile industry (a) [60] or in the building industry (b) [10].

The offset between the sorption and desorption curves is commonly explained by an ink-bottle effect (Fig 10), capillary condensation and/or the contact angle difference [18], [21], [54], [56], [64], [65]. No distinction is made according to the nature of the material where hysteresis is observed.

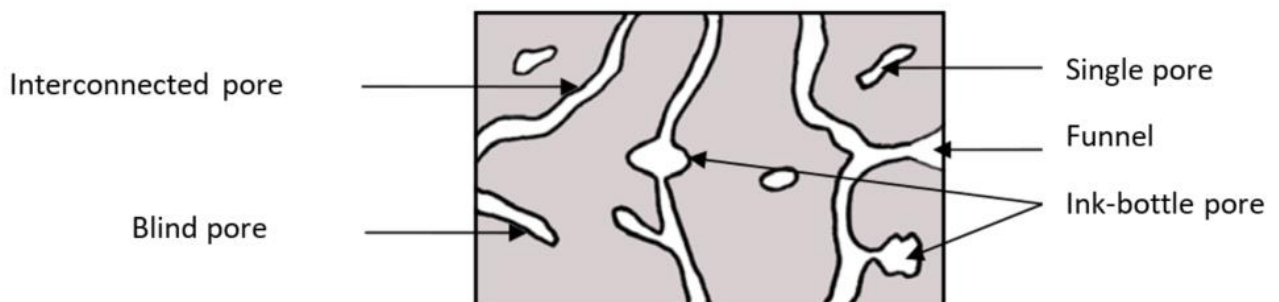


Fig 10. Schematic diagram of the porous network [18].

To date, the explanations remain open since there is no real consensus on this subject [53], [66].

3. From hydrogen bonding to hysteresis

3.1 Hydrogen bonding

Hydrogen and Van der Waals bonds are modelled by dipole-dipole interactions [67], [68]. Although they both result from electrostatic interaction, hydrogen bonds have a higher binding energy due to the strong polarization of the hydroxyl groups. On the other hand, a covalent bond results from the pooling of valence electrons between two atoms. It involves a chemical reaction that is not reversible. Consequently, the binding energies differ since they result from different phenomena (Table 1). Finally, the greater the binding energy, the more difficult it is to break the bond. Thus, hydrogen bonds can break or change form easily at ambient temperature (300 K) due to their low bonding energy [67]. Nevertheless, for the same type of bond, the binding energy may differ according to the nature of the atoms and molecules involved.

Table 1. Order of size of different bonds [67]

Type of bonding	Bonding energy [kJ.mol ⁻¹]
Covalent	≈ 100
Hydrogen	≈ 10
Van der Waals	≈ 1

It should be noted that Van der Waals interactions, mentioned to explain the first step of water fixation in bio-based materials (cf. section 2.2), are about 10 times weaker than the hydrogen bonds. Moreover, it is well known that water, a polar molecule, forms hydrogen bonds [69]. It establishes hydrogen bonds rather than Van der Waals bonds as far as possible. The two interactions are different and should not be confused [68].

In addition, cellulose is a host polymer concerning hydrogen bonding, due to its hydroxyl groups [70]. Recent work highlights the potential of hydrogen bonding in porous molecular materials [71]: they condition the spatial organization of the microstructure [72].

3.2 Microscopic understanding of hysteresis

A recent study has demonstrated a molecular-scale coupling mechanism to explain the sorption hysteresis and swelling of polymers such as cellulose [12]. The authors explain that three types of hydrogen bonds coexist within cellulose:

- Inter molecular hydrogen bonds between water molecules (HB^{WW})
- Inter molecular hydrogen bond between water molecules and cellulose (HB^{CW})
- Inter chain Hydrogen bonds in cellulose (HB^{CC})

The sequence of creation/breakage of these hydrogen bonds is conditioned by the phase observed: sorption or desorption.

Water is first physisorbed (through HB^{CW}) in the sorption phase (to the cellulose chains. The cellulose swells and the number of HB^{CC} decreases with increasing water content. Consequently, the intermolecular bonds in cellulose (HB^{CC}) break: more and more hydroxyl groups are accessible to form HB^{CW} . Simultaneously, the number of HB^{WW} increases, reflecting the formation of water clusters within the polymer pore space (Fig 11).

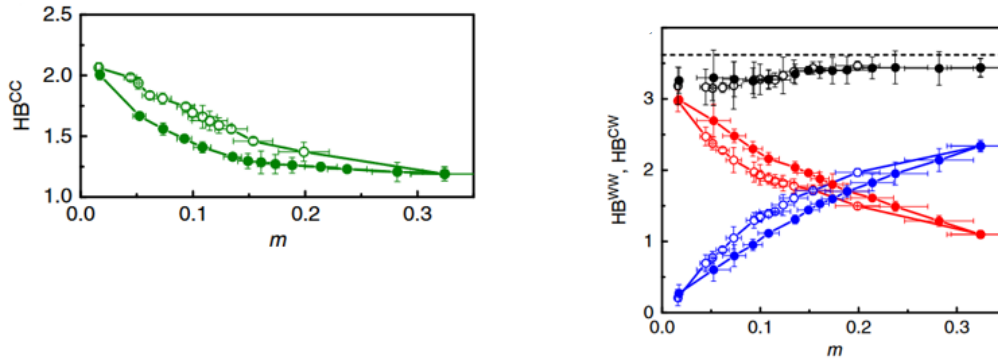


Fig 11. Number of HB^{CC} (on the left) / HB^{WW} and HB^{CW} versus moisture content (m) from Chen et al. [12]

In the desorption phase, lower energy bonds break first. As HB^{CW} is stronger than HB^{WW} , water molecules are first removed from pore water clusters. Thus, there are more HB^{CW} in the desorption than in the sorption phase for the same relative humidity since more "host sites" are accessible in the polymer. This microscopic phenomenon leads to an observable hysteresis in the sorption-induced swelling of cellulose (Fig 12).

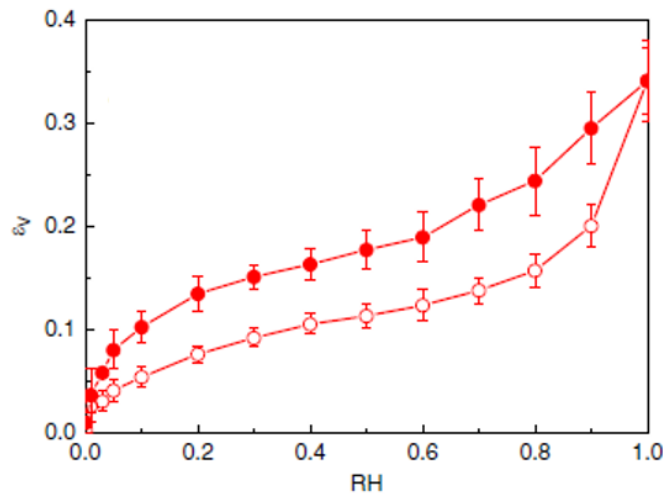


Fig 12. Hysteresis in sorption-induced swelling, case of cellulose [12]

The existence of differentiated hydrogen bonds between the water sorption and desorption phases in amorphous cellulose has been clearly demonstrated. It causes macroscopic hysteresis within the polymer. This leads us to consider these results while analyzing the behavior of bio-based materials.

In addition, Chen et al. point out that the moisture content first increases rapidly (low relative humidity) and then less rapidly (higher relative humidity). This inflection point in the

adsorption isotherm can be explained by an initial rapid and easy adsorption of water molecules into the initially available host sites of cellulose. Subsequently, the cellulose chains open up and more water molecules are adsorbed but the process is slower. Finally, the authors indicate that the pore size prevents the phenomenon of capillary hysteresis in nanoporous media (with pores smaller than 2 nm). Thus, in cellulose, where the pores are smaller than 1 nm, capillary hysteresis cannot take place.

4. Discussion: new insights into hysteresis in bio-sourced materials

4.1 A necessary new approach

The explanations for hysteresis provided in the literature are open to criticism. First, SEM images show that the plant aggregates have a tubular morphology. No pronounced narrowing or widening is visible. The hypothesis of ink-bottle shaped pores is not justified at this scale. It is more relevant at the scale of the material, due to a multi-scale complex and tortuous porosity. However, in the case of plant aggregates, the pore size is measured in tens of micrometers [73], so capillary hysteresis is founded.

Furthermore, covalent bonds cannot explain the sorption mechanism, since sorption is reversible. In contrast, it seems appropriate to regard weaker bonds as Van der Waals or hydrogen bonds to explain water sorption and desorption.

In addition, whether it is through the ink-bottle effect, the capillary condensation and/or the contact angle difference, the hypotheses mentioned assume that the condensed state of the water is reached. However, capillary condensation occurs from 80% RH (cf. Fig 3). The sorption and desorption curves do not overlap, even at low relative humidities. Moreover, while the smallest pores can contain liquid water, they probably cannot account for the entire shift between the curves at low humidity values. Finally, no assumption made so far can explain other observables such as swelling, sorption kinetics and temperature effects.

4.2 Hysteresis: from the aggregate scale to the material one

i) A new water fixation mechanism

It is now possible to differentiate the modes of water fixation between the pore network of the plant aggregate and that of the material (vegetal concrete or lightened earth). This means that the plant aggregate's lignocellulosic nature is considered. Nevertheless, as most sorption sites for hydrogen bonds have been found in the hemicellulose followed by cellulose and lignin [74], this study does not consider the nature of the polymers in the plant aggregate. Only the scale of the plant particle and the material will be differentiated. A new water fixation mechanism is thus proposed (Fig 13).

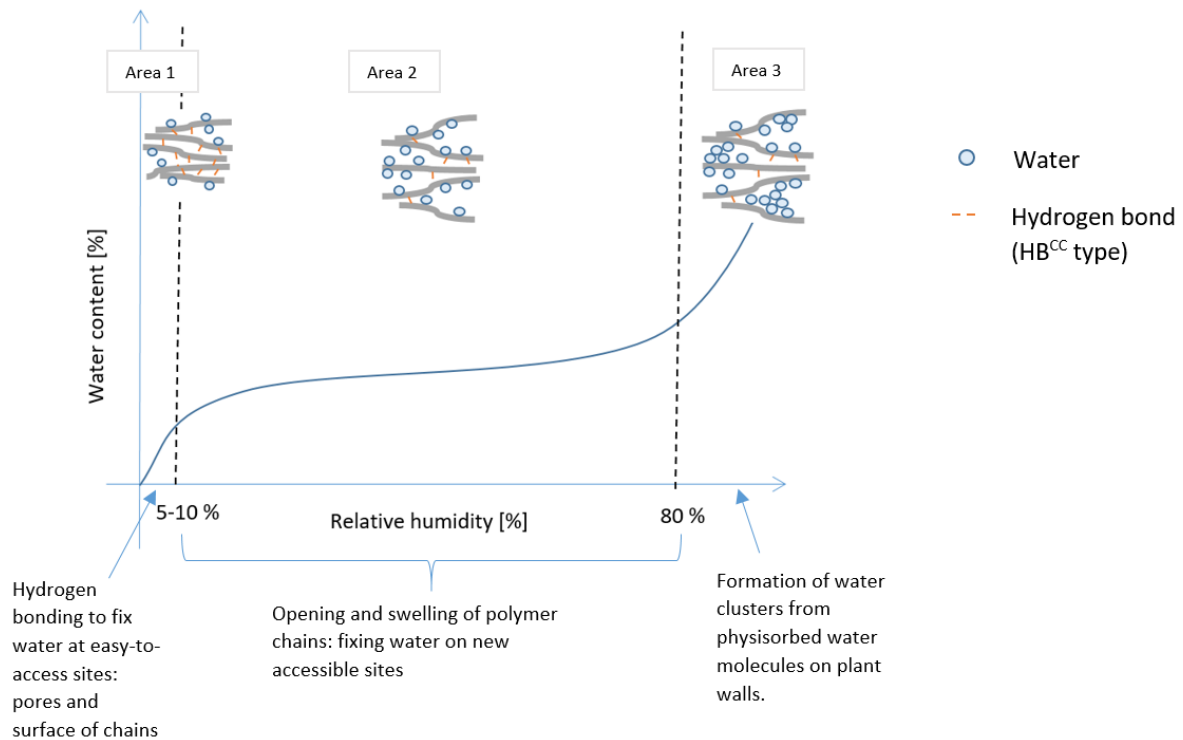


Fig 13. Water fixation mechanism in bio-based materials at the molecular scale in plant particle.

The bonds involved in the process are likely to be hydrogen bonds, although Van Der Waals bonds (induced dipole) may occur at the margin. Only hydrogen bonds are considered here. Thus, different regions of the curve and the corresponding slopes show the existence of 3 processes with different kinetics:

- Area 1: Water fixation on a pore surface is relatively fast because the host sites are easily accessible: HB^{CW} bonds form on polymer surface chains or in pores of an amorphous region then HB^{WW} form easily until the initial pores are filled.
- Area 2: Polymer chains open up, freeing new host sites to create HB^{CW} bonds. In parallel, HB^{CC} bonds break.
- Area 3: At high relative humidity, hydrogen bonds mostly form between water molecules because many host sites are occupied on polymer chains. This leads to water clusters in the new pore spaces created by the swelling of the polymer chains. Because host sites are very accessible, the associated kinetics is quite fast, as in area 1.

ii) A new description of hysteresis

Furthermore, from the results presented in section 2.2, it is assumed that, when plant aggregate is subjected to an increase in relative humidity, more and more new "host sites" become available to create HB^{CW} bonds. Those bonds are not broken at the same relative humidity level during the desorption phase. This is assumed to be the only cause of hysteresis at the particulate scale up to 80 % RH. First, this phenomenon has been proven, and, second, the microstructure of the plant aggregates is inconsistent with the other assumptions made in the literature so far.

In addition, the richer the plant aggregate is in amorphous polymers (cellulose, hemicellulose, and lignin), the likelier it is that the mechanism will occur.

In contrast, at the material level, the ink-bottle effect, capillary condensation and/or the contact angle difference probably coexist - especially at high humidity when water is mostly liquid. Consequently, a new description of hysteresis in bio-based building material can be proposed (Fig 14).

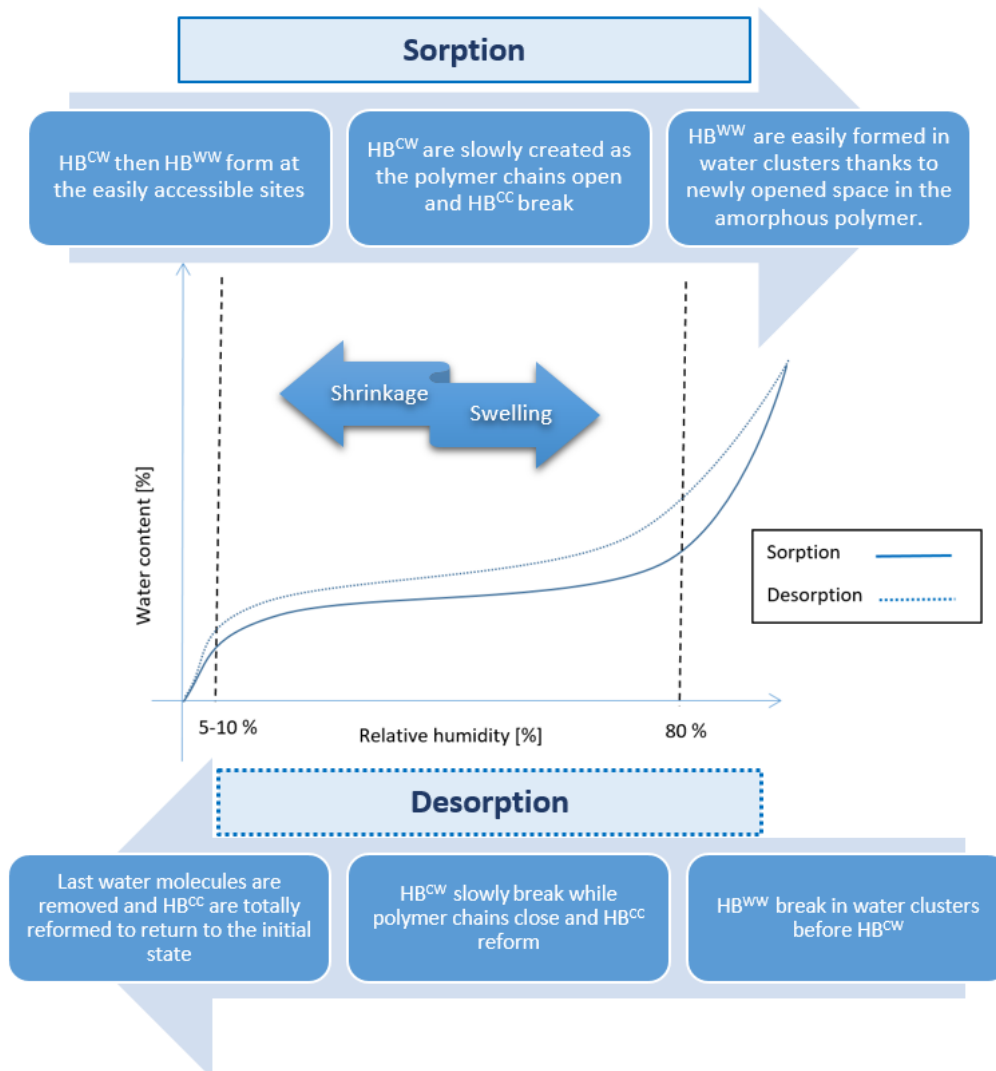


Fig 14. Hysteresis mechanism in bio-based building materials explained thanks to differentiated hydrogen bonds in plant particle.

4.3 Experimental measurements

i) Protocol

Sunflower pith panels manufactured without binder were dried in an oven (under 60 °C) until stabilization of mass to ensure no water adsorbed remains in the aggregate. The dimensions of the particleboards were measured with a caliper at the dry state. Particleboards were then placed in an enclosure under 80% of relative humidity until stabilization. The dimensions of particleboards were measured again with a caliper at this humid state. This allowed to compare the volumes of the panels in their dry and wet states.

ii) Dimension comparison

The results of the dry and wet measurements are presented in the table 2.

Table 2. Comparison of particleboard dimensions (without binder) between dry and wet conditions

	Volume V of particle board (cm ³)			
	Panel 1	Panel 2	Panel 3	Panel 4
Dry state	3123.1	2983.8	2433.93	2607.23
80%	3347.1	3264.1	2578.7	2707.3
Volume increase ΔV (%)	6.7	8.6	5.6	3.7

Both observation and experimental measurements show significant swelling of sunflower pith panels. From dry to wet state, the swelling observed is on average 6%. As these particleboards are made up entirely of particles, the swelling observed is clearly a process that takes place in the sunflower pith. It is the macroscopic demonstration of the microscopic hydro-mechanical coupling within the aggregate. It is worth noticing that sunflower pith, as bio-aggregates, is mainly composed of cellulose (cf. section 2.3). The experimental measurements support the theory defended in this study.

4.4 Macroscopic effects

The theory put forward in the previous section is supported by a wide range of macroscopic observations. Thus, it is possible to connect and explain all these points - raised in section 2 - through sorption-swelling coupling (Table 3).

Table 3. Macroscopic observations of sorption-swelling coupling that causes hysteresis in bio-based building materials.

Observation	Origin	Explanation
Sorption mechanism is reversible.	sorption- swelling coupling at molecular scale in vegetal aggregate	Hydrogen bonds form and break easily, even at ambient temperature, due to their low binding energy.
Hysteresis is more pronounced for plant-based concrete materials than for aggregates.		Because of additional origins of hysteresis in material than in aggregate. Effects are cumulative.
Aging reduces the rate of adsorption and desorption.		Residual water masks "host sites": they are no longer accessible, as inhibited by the first sorption phase.
Swelling of the plant particles or fibers during hysteresis is irreversible.		Returning to a dry state allows the last physisorbed water to be extracted. The intercellulosic chains seem to return to their original state. In any case, there is no (or negligible) macroscopic manifestation of swelling.
Hysteresis increases while crystallinity decreases		The more amorphous the cellulose is, the more important is sorption-swelling coupling. Interchain bonds cannot open in crystalline regions due to high stability.
Swelling and shrinkage is observed at a young age or after a period of accelerated aging.		Swelling and shrinkage are possible as soon as HB ^{CW} replaces HB ^{CC} . This potential decreases with age (inhibited sites) but remains possible given the large number of host sites in the plant aggregate.
Temperature dependence of sorption curves.		Hydrogen bonding is temperature dependent.
Relevance of considering local sorption kinetics in bio-based materials, especially when coupled with hysteresis.		The opening/closing of the cellulose chains is probably a rather slow process that needs a kinetic factor to be taken into account, both in sorption and desorption phases.
Swelling is observed between dry state and 80 % RH		The opening of the cellulose occurs from 5-10 % to 80 % RH (cf. Fig 13)
Water content is always higher in desorption than in sorption phase for the same relative humidity		More water molecules are physisorbed during desorption because they do not have the same chemical potential.

All these elements allow us to establish the relevance of taking this recently demonstrated sorption-swelling coupling effect into account. It improves our understanding and description of hysteresis and its consequences in bio-based building materials.

In view of all the elements discussed above, a new multi-scale schematization of bio-based materials' behavior is proposed (Fig 15).

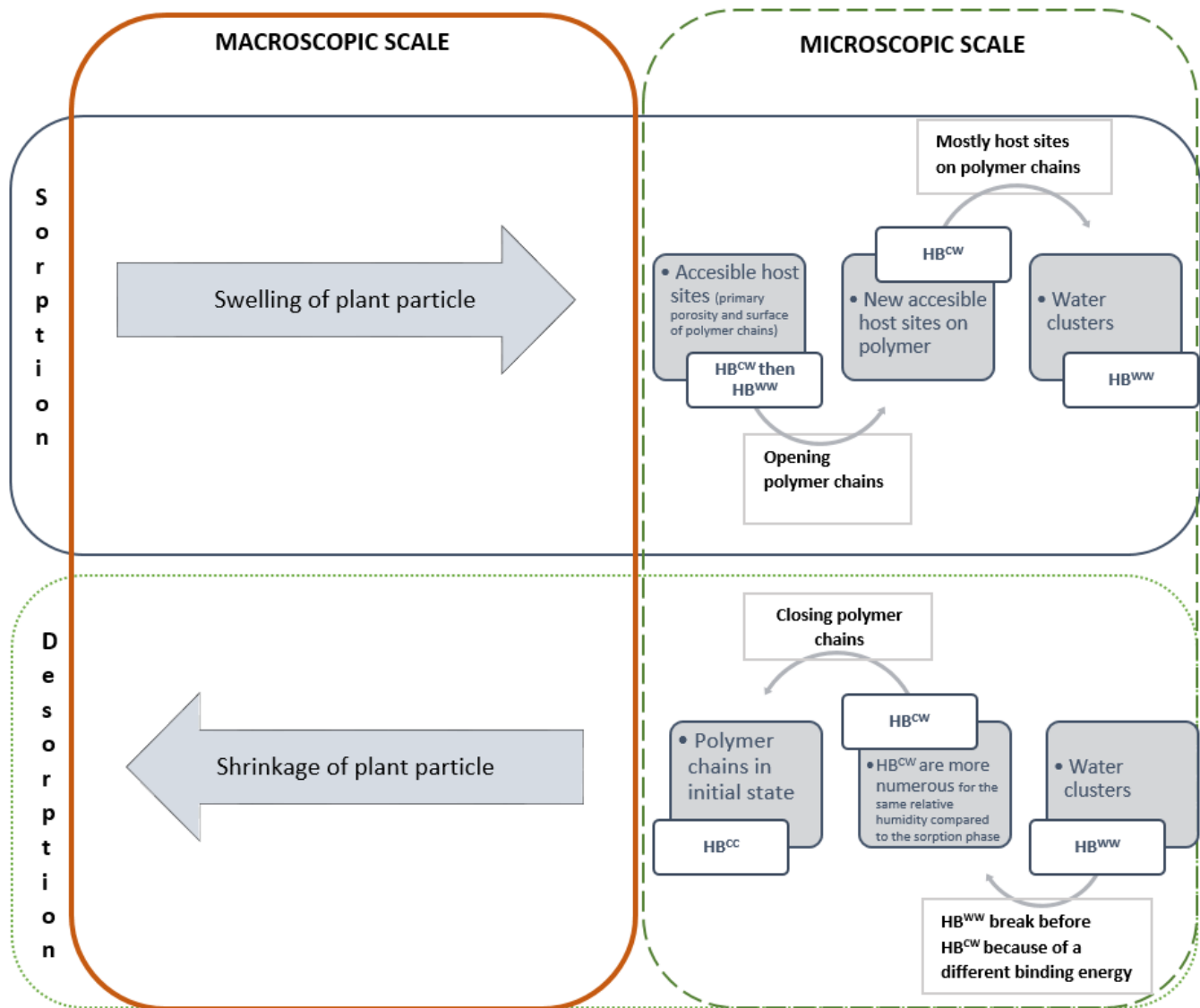


Fig 15. Sorption-Desorption of water: summary diagram of the coupling reversible process at the plant particle scale that causes hysteresis in bio-based building material.

This makes the link between molecular-scale phenomena their consequences at the macroscopic scale. It thus highlights the hydro-mechanical multi-scale coupling of these materials.

5. Conclusion

This study demonstrates the need to reconsider the description of water sorption in bio-based building materials. The explanations put forward in the literature are not to be totally discarded but are not sufficient. Indeed, recent work has shown how water adsorbed at the cellulose scale can induce both hysteresis and swelling of cellulose chains. The lignocellulosic nature of the plant aggregates incorporated should therefore be taken into account.

Thus, a new description of sorption hysteresis in the case of bio-based materials is proposed. It is based on the existence of hydrogen bonds that are differentiated between the water sorption and desorption phases in cellulose. These bonds are stronger than the Van der Waals bonds commonly reported in the literature until now.

It is thus possible to better understand the hysteresis phenomenon widely observed in the literature for bio-based materials. A new scale of porosity, that has been ignored so far, is to be considered: nanoporosity (in the amorphous polymers constituting the plant aggregate). This molecular-scale hydro-mechanical coupling explains many observations at the macroscopic scale. We matched this new consideration with a set of macroscopic observables widely reported in the literature. The results have also been confirmed by our own experimental measurements. This lends support to the new hypothesis suggested and shows how it is relevant.

Finally, this study opens up new perspectives:

(i) A better understanding of macroscopic swelling makes it possible to anticipate and to predict. It is important to leave a corresponding gap in the wall to avoid any disorder. In addition, swelling effects impact the porosity of the material and therefore probably affect its mechanical properties and durability.

(ii) It would be interesting to investigate whether, as in the case of hysteresis in electromagnetism, the area of the hysteresis curve gives additional information but, here, on the sorption/desorption phenomenon.

This article is the fruit of in-depth and well-argued reflection. It is based on experimental results as well as the collection and analysis of results coming from different scientific fields. It leads to propose a new theory to understand the hysteresis mechanism observed in bio-sourced materials. The experiment carried out on aggregate panels demonstrated that the theories put forward in literature are not sufficient to explain what is happening within the material. The ink-bottle effect is not very probable given the microstructural morphology of plant aggregates (cf. section 4.1). A new theory considering the microscopic scale is necessary to describe hysteresis. The hydro-mechanical coupling taking place within cellulose chains has been exploited to propose a new vision. New schemes (cf. figures 13, 14 and 15) of the water sorption and desorption phases within bio-sourced materials are provided. To date, plant aggregates was considered inert with respect to water absorption, in the same way as conventional materials. This work therefore represents a significant advance in the field of bio-sourced materials, since for the first time the lignocellulosic nature of the aggregate is taken into account. This means that new specific physico-chemical phenomena need to be considered.

To conclude, this work combines the fields of chemistry, civil engineering and applied physics in order to open new perspectives. This enabled to remove some grey areas in the hysteresis phenomenon which remains a key factor when considering bio-based building materials. Our work underlines the interest of conducting interdisciplinary studies to understand the full complexity of bio-based materials.

Supplementary Materials: Not applicable

Author Contributions: Conceptualization, S.R.L.; methodology, S.R.L.; software, S.R.L.; validation, S.R.L, V.S and A.A.C; formal analysis, S.R.L; investigation, S.R.L; resources, S.R.L.; data curation, S.R.L; writing—original draft preparation, S.R.L; writing—review and editing, S.R.L; visualization, S.R.L.; supervision, A.A.C; project administration, S.R.L; funding acquisition, S.R.L. All authors have read and agreed to the published version of the manuscript.

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Chapitre 4. De la microstructure à l'échelle stratégique de la particule végétale

Préambule

Mieux comprendre le mécanisme de fixation de l'eau dans un granulat végétal - et particulièrement à l'échelle de la microstructure- est fondamental car la contribution de l'eau dans la conduction thermique est vingt fois plus importante que celle de l'air (conductivité thermique de $0,6 \text{ W.m}^{-1}\text{K}^{-1}$ et de $0,026 \text{ W.m}^{-1}\text{K}^{-1}$, respectivement pour l'eau et l'air sec à $20 \text{ }^\circ\text{C}$). La sorption de l'eau est donc nécessairement une donnée à prendre en compte pour appréhender et prédire de manière pertinente la conductivité thermique d'un matériau de construction bio-sourcé sous les contraintes d'usage.

Toutefois, dans l'optique de modéliser le comportement thermique des matériaux bio-sourcés, un autre facteur semble essentiel. En effet, que l'on considère l'isolation en vrac, l'isolation répartie d'un béton végétal ou d'une terre allégée, l'élément clé est le granulat végétal. C'est principalement ce constituant qui est à l'origine des performances thermiques du matériau, du fait de sa grande porosité intrinsèque (également appelée porosité intra particulaire). Il s'avère que la mesure de la conductivité thermique à l'échelle d'un seul granulat végétal est difficile à mettre en œuvre. Une seule référence de la littérature évalue expérimentalement la conductivité thermique particulière de la chènevotte grâce à une technique de thermographie infrarouge. Il est possible de trouver d'autres valeurs de la conductivité thermique particulière de la chènevotte, déterminées par méthode inverse ou numérique. Notons que dans une grande majorité des cas, le comportement thermique du granulat seul est considéré isotrope (une seule valeur de conductivité thermique). Dans d'autres références, l'anisotropie induite par les pores tubulaires de la chènevotte (dans lesquels circulent la sève pendant la croissance de la plante et qui se remplissent d'air après récolte) est prise en compte : deux valeurs de conductivités thermiques sont alors disponibles (une composante normale et une composante tangentielle). Trop souvent, les conditions de température et d'humidité relative - associées aux valeurs de conductivité thermique fournies- ne sont pas précisées par les auteurs. Dans la mesure où ces contraintes extérieures influencent, a minima, le phénomène de sorption et donc le comportement thermique du matériau, il est difficile à ce stade d'envisager de développer des modèles prédictifs à partir de ces données d'entrée.

Dans ce contexte, ce chapitre propose deux méthodes semi-empiriques de détermination de la conductivité thermique de la chènevotte. La première est basée sur une méthode inverse, à partir de la mesure de la conductivité thermique sur de la chènevotte en vrac. La seconde se place à l'échelle du granulat, le décrivant comme une matrice solide dans laquelle baignent des inclusions (méthode dite « théorique » dans l'article). Cette dernière nécessite toutefois d'avoir accès à la porosité intra particulaire du granulat, ce qui est une donnée relativement accessible grâce aux travaux récents de H. Ratsimbazafy [10]. Toutes deux utilisent des résultats d'homogénéisation connus et les exploitent en faisant l'hypothèse d'un granulat au comportement isotrope d'une part et anisotrope d'autre part. Les résultats

obtenus sont comparés à quelques rares références de la littérature dans le but d'apprécier la pertinence des méthodes proposés. Enfin, les valeurs maximales et minimales de porosité particulière de la chènevotte sont prises en compte pour donner une plage de la conductivité thermique particulière de ce granulat. Ceci est un premier pas dans la prise en compte de la variabilité de la ressource.

Article C. Multiscale Modelling of Bio-composites: Towards Prediction of Their Thermal Conductivity Based on Adequate Knowledge of Their Constituents

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Abstract. To face up to the present energy crisis and the climate challenge, incorporating plant aggregates into reactive or clayey mineral matrices has proved to be an ecological and effective way to improve the thermal performance of buildings. It also helps to reduce both the grey energy involved and the consumption of fossil or non-renewable materials. However, the characterization and optimization of bio-based composites is relatively time-consuming, which may be a hindrance to their development. Our objective is to efficiently predict the thermal conductivity of a hemp particle as this is a fundamental input value for predicting thermal behaviour at the material scale. We found few reliable data on this topic in the literature. This is a major challenge that needs to be addressed as it can lead to erroneous results even when the prediction model is relevant and well developed. An additional difficulty arises from the fact that plant aggregate shows high variability. This paper opens new perspectives by suggesting new procedures for determining the thermal conductivity of hemp shiv using two approaches and considering, in particular, the variability of the raw material. Since existing methods for measuring thermal conductivity do not provide access to the particulate value, an experimental and a theoretical method are proposed to determine it. Both are based on analytical equations obtained by homogenization and both are shown to lead to consistent results. Comparison with the few data available in the literature also highlights a possible overestimation of the values found in previous works.

Keywords: Multiscale modelling, plant aggregates, thermal conductivity

1. Introduction

To address environmental issues, the building sector has to innovate and reduce its energy consumption and greenhouse gas emissions by developing new materials that are environmentally friendly while ensuring the comfort of users. Using plant aggregates is a way to deal with this challenge, as they are identified as agricultural, renewable and locally-available by-products. The use of plant aggregate for construction limits both transport emissions and fossil energy consumption. Plant-based aggregates also allow large quantities of carbon dioxide to be stored in the materials throughout the building's life-cycle thereby reducing its carbon impact [1] and presenting an additional environmental asset. Last but not least, plant particles possess high insulating and hygroscopic capacities. These multiple factors should lead the building industry to massive use of such raw materials. However, the substantial variety of formulations (nature and dosage of aggregates and binder) induces an equally wide range of thermal performance. Therefore, the characterization and optimization of those types of building materials can be costly and time consuming.

In consequence, several models have been proposed to predict thermal properties of bio-based materials. However, the particulate thermal conductivity remains unmeasurable by current techniques while they are needed as model inputs. In addition, while a homogenization model is currently applied by several authors [2]–[5], considering the particle as an assembly of composite spheres could be questioned, in particular in view of the actual particle geometry. A more faithful representation of microstructure should be investigated. It is also worth noting that hemp shiv is the only plant aggregate with values of particle thermal conductivity available in the literature [3], [6], [7]. The methods by which these values are determined are not always explicitly stated by the authors. Finally, the hygroscopic nature of composites enables ambient hydric variations to be attenuated. However, it makes forecasting their behaviour a more complex task.

Our work is aimed at developing a new tool to guide and optimize bio-based material formulations even before the manufacturing step. This paper presents two different methods for determining the particulate thermal conductivity of dry hemp shiv. In order to validate the suggested procedure and also to limit the raw material variability, only one plant aggregate was chosen for this study: commercial hemp shiv. In addition, to avoid having to consider the effect of water absorption on thermal conductivity, this research was carried out on dry aggregates. Thus, the first method requires measurement of the thermal conductivity of dry particles in bulk, while the second one is more theoretical, being based on the chemical composition and microstructure of the particles. Both methods are founded on analytical equations obtained by homogenization. A protocol to access both thermal conductivity and volume fractions of particles in bulk is therefore established in the following. Indeed, this data is required in the models used. The shape and the microstructure of the particles as well as the anisotropy of the material and its effect on the thermal behaviour are also taken into account. Finally, the values obtained for the thermal conductivity of a hemp shiv particle were compared with those found in the literature. Our models predicted results in reasonable agreement with both isotropic and anisotropic considerations. However, this study may indicate an overestimation of the values determined by the self-consistent scheme [3]. This is discussed in this paper.

2. Materials

2.1 Choice of the plant aggregate

Adapted to temperate climates, hemp is a fast-growing plant. Peripheral fibres of the plant are used to manufacture textiles and rope. The central part of the stalk, the porous pith, is crushed to obtain hemp shiv, which has been used for several years now as a building material in hemp concrete. This plant aggregate was selected for our study on different criteria. Firstly, the hemp particle is the one that has been most studied in the literature and for which comparative values of particulate thermal conductivity are available, either in scalar form (isotropic considerations) or as a tensor (anisotropic considerations).

Moreover, the hemp particle has high variability [8], [9]. As it is an agricultural by-product, its intrinsic properties depend on the geographical location and method of production, but also on the climate and the harvesting period. This leads to significant irregularity in the raw material extracted. Thus, variability can be observed in the particulate porosity, the density or the chemical composition of the solid skeleton. It is worth noting that the retting process used to separate fibres has an impact on the chemical composition of the aggregates obtained. In particular, the proportion of crystalline phase in the cellulose of hemp fibres depends on the degree of retting. This degree of crystallinity is known to impact the mechanical properties [5]. A possible effect on thermal conductivity will be highlighted in this paper. Finally, in order to limit intrinsic variability (shape factor, size, density, chemical composition) as far as possible, we chose commercial hemp shiv. To assess the consistency of the proposed models, this aggregate is taken as a reference in this study.

2.2 Physical specificities of hemp shiv

Shape of the hemp particle. The hemp shiv comes in the form of straw. The commercial product chosen for the study was dusted and calibrated (Fig 1). Macroscopically, it can be considered as a kind of elongated parallelepiped or even a cylinder. The shape parameters are available in the thesis of Ratsimbazafy [10]. Finally, hemp shiv particles are assumed to be cylindrical in line with the geometrical values of the aggregate studied and according to the literature [11], [12].



Fig 1. Hemp shiv particle used for this study (size ranging from 2 to 14 mm)

Microstructure of the hemp particle. Many capillaries are present in plant aggregates because they allow sap to circulate when the plant is growing. After drying, this structure naturally fills with air, which causes intra-particle porosity. From SEM observations [6], [7], hemp shiv reveals a tubular microstructure, which can be thus assimilated to a cylinder. In consequence, the solid skeleton of the hemp particle is made of cylindrical pores embedded in a continuous matrix.

Porosity. Hemp shiv is employed in construction for its insulating properties. This insulating capacity is a consequence of the high porosity present both inside the particle (intra-particle porosity) and between the particles themselves in the bulk paste (inter-particle porosity). The particle porosity of our samples was $83.5 \pm 4.8 \%$ [10]. The inter-particle porosity depends on the degree of compaction of the bulk mix.

Density. The density of the particles used in our study was experimentally estimated at $\rho_{\text{part}} = 248 \text{ kg.m}^{-3}$. This result is consistent regarding the high particle porosity and the density of the solid skeleton constituents [10]. Note that the density of the skeleton of hemp shiv and of cellulose in lignocellulosic biomass are both close to the order of 1540 kg.m^{-3} [10], [13].

2.3 Chemical characteristics of hemp shiv

Chemical composition also influences the properties of plant aggregates and cellulose is well known to be the major component of bio-aggregates. The hemp shiv used in our study was composed of more than 55% cellulose. The chemical components of this plant aggregate are listed in Table 1. These values will be useful to justify the assumptions made at the particle level in the homogenization process.

Table 1. Proportions of the constituents of the hemp shiv used expressed in % of their dry mass according Ratsimbazafy [10].

	Cellulose	Hemicellulose	Lignin	Minerals
Dosage (expressed in % of their dry mass)	55.5 ± 0.2	18.9 ± 0.1	16.4 ± 0.4	1.9 ± 0.0

3. Experimental and theoretical methods

3.1 Measurement of the thermal conductivity

Thermal conductivity was measured with the hot wire test since it is very suitable for measuring thermal conductivities ranging from 0.02 to $5 \text{ W.m}^{-1}\text{K}^{-1}$. Values around 0.051 - $0.070 \text{ W.m}^{-1}\text{K}^{-1}$ have been reported in the literature for hemp in bulk [10]. Prior testing, the aggregates were dried in an oven at 60°C until a constant weight was achieved and then cooled in a desiccator to ambient temperature. Measurements were made in a temperature-controlled space at 20°C . The samples (described in section 2.2) were weighed again after a sequence of measurements to ensure that water adsorption was negligible.

A Neotim-FP2C hot wire apparatus was used for this study. Since this transient method only allows local measurements, thermal conductivity measurements were repeated 5 times. The samples were stirred after each test because the hot wire caused local heating. A first round

of measurements was carried out on a bulk mixture. Then, the aggregates were hand-compacted by 30% of the original volume in order to investigate the effect of compaction on the value of particle thermal conductivity.

3.2 Volume fraction measurement

One of the difficulties of our protocol was to know the volume fraction of the aggregates in the bulk in which the thermal conductivity measurement was carried out. This was because the granular arrangement was different for each bulk mix. To overcome this difficulty, and based on the recommendations of the RILEM TC 236-BBM [14], particles were weighed and placed in a container for thermal conductivity measurement and, at the same time, the total volume occupied by the mixture was marked. After removing the shiv from the container, it was filled with water to the level of the mark. Finally, the container was weighted and the volume of water, i.e. of bulk aggregates, was calculated.

The volume fraction of aggregates in the bulk arrangement, f_g , was calculated as follows:

$$f_g = \frac{V_g}{V_{tot}} \quad (1)$$

where V_{tot} is the total volume of bulk and V_g that of aggregates.

To gain access to this value experimentally, the previous equation was transformed into:

$$f_g = \frac{M_g}{\rho_{part} \cdot V_{tot}} \quad (2)$$

where M_g is the mass of aggregates.

3.3 Choice of homogenization schemes

Since it is currently impossible to experimentally measure the thermal conductivity of a single particle, homogenization techniques were used to overcome this difficulty. Note that a self-consistent scheme is generally used in the literature both at the bulk scale [3] and at the composite scale [15]. It is based on an assembly of spherical spheres of various sizes.

Another method was chosen to take the real morphology of the homogenized material into account. Here, we develop the theoretical basis of the techniques chosen for the study. For the heterogeneous material considered, i) the particles in bulk and ii) a single particle, were reduced to an equivalent homogeneous material. A representative volume element Ω (RVE) of the heterogeneous material had to be considered, with $\delta\Omega$ its outer boundary and n the outward unit normal $\delta\Omega$. The RVE was the minimum volume to be considered as representative of the macroscopic behaviour of the heterogeneous medium. Then homogenization required three steps:

Representation, which consisted of describing the material at the microscopic scale (shape, orientation of inclusions in the matrix).

Localization, which allowed the physical relationships to be expressed at the local scale of the material as a function of the different phases considered. Relations between the quantities at the microscopic scale and those defined at the macroscopic scale were defined.

Homogenization, which finally gave the behaviour of the equivalent homogeneous material thanks to averaging operations on the solutions of the previous step.

Thus, for our study, we considered two RVEs (Fig 2. and Fig 3) to work on the thermal conductivity tensor of a hemp shiv particle.

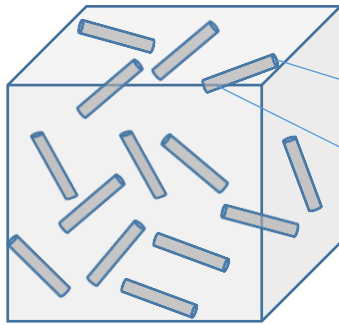


Fig 2. RVE for the particles in bulk



Fig 3. RVE at the particle scale (5-10mm)

The RVE considered for particles in bulk comprised two phases: a matrix of air and hemp cylindrical particles as randomly arranged inclusions. The RVE considered at the particle scale was also composed of a two-phase material containing solid matrix and elongated pore-inclusions. In both cases, the matrix was continuous and the interfaces were assumed to be perfect. Thus, we considered two scales: the macroscopic one (bulk or particle) and the microscopic one, at the scale of heterogeneities (inclusions). The fictitious homogeneous medium had the same geometry as the RVE and, subjected to the same stresses, was assumed to have an identical global response (Fig 4).

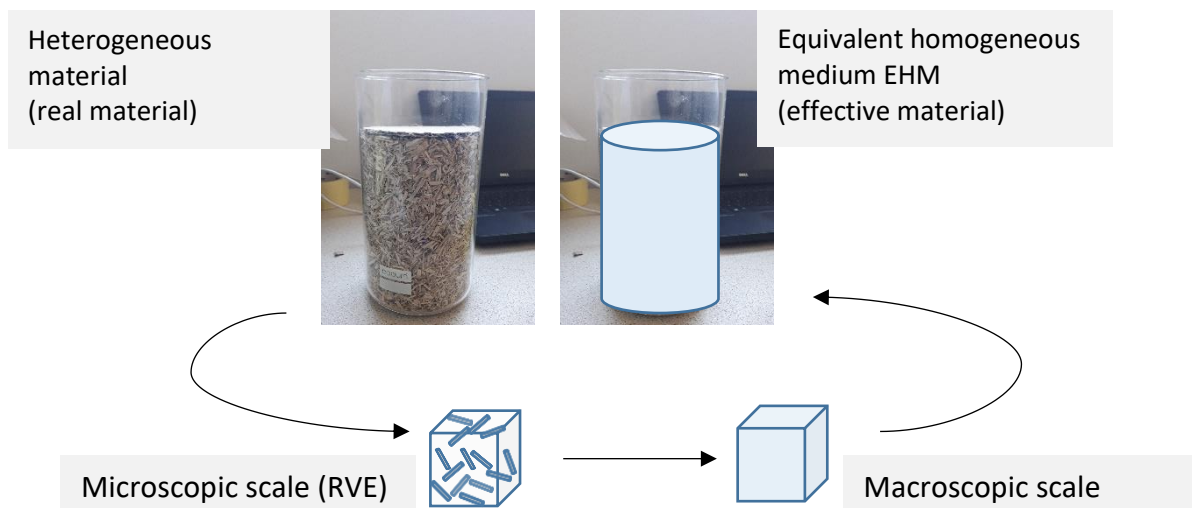


Fig 4. Homogenization process applied to hemp shiv in bulk

In order to provide uniformity in the notations, the following were adopted for this paper: index 0 to for the matrix, index i for the inclusions.

If the heterogeneous material is a matrix-type composite composed of several inclusions, according to classical theory of Eshelby, this homogenized thermal conductivity tensor is given by:

$$\Lambda^{hom} = \sum_{i=0}^n f_i \Lambda_i \mathbb{A}_i = \Lambda_0 + \sum_{i=1}^n f_i (\Lambda_i - \Lambda_0) \mathbb{A}_i \quad (3)$$

where Λ_0 is the thermal conductivity tensor of the matrix, f_i is the volume fraction of the inclusion i , Λ_i its thermal conductivity tensor and \mathbb{A}_i its second order concentration tensor. In this study, only two phases were considered: the matrix (the air or the dry particle) and only one type of inclusions (respectively the hemp shiv particles or the air in the pores).

3.4 Estimation of the effective thermal conductivity of a particle by inverse method: isotropic consideration

Since the plant aggregates made up a relatively large volume fraction in bulk, the Mori Tanaka approach appeared appropriate to consider the interactions between the inclusions [16]. This homogenization scheme considers heterogeneous medium as an isotropic reinforced matrix (air). Its inclusions behave as isolated inclusions in an infinite matrix. Initially, these inclusions (hemp shiv particles) were considered as spherical. In this case, the thermal conductivity tensor was expressed as:

$$\Lambda_{iso}^{hom} = \Lambda_0 + f_i \cdot (\Lambda_i - \Lambda_0) : \mathbb{A}_i \quad \text{with } \mathbb{A}_i = (\mathbb{I} + \mathbb{S} \Lambda_0)^{-1} (\Lambda_i - \Lambda_0)^{-1} \quad (4)$$

where \mathbb{S} is the Eshelby tensor, which depends on the shape of the inclusions and the properties of the matrix.

In the case of a spherical inclusion, the Eshelby tensor is given by:

$$S_{ij} = \begin{cases} 0 & \text{if } i \neq j \\ 1 & \text{if } i = j \end{cases} \quad (5)$$

This gives the following analytical formula for the expression of the homogenized thermal conductivity tensor:

$$\Lambda_{iso}^{hom} = \Lambda_0 + \frac{3f_i (\Lambda_i - \Lambda_0) \Lambda_0}{(1 - f_i)\Lambda_i + (2 + f_i)\Lambda_0} \quad (6)$$

with $\Lambda_{iso}^{hom} = \lambda_{iso}^{hom} \mathbb{I}$

where \mathbb{I} is the second order unit tensor.

The scalar thermal conductivity λ_{iso}^{hom} can be deduced directly from this formula since the distribution is isotropic. The analytical expression obtained is then inverted to lead to the following result:

$$\lambda_i = \frac{\lambda_0 (2 - 3f_i) - 2 \lambda_{hom}}{\lambda_{hom} - \lambda_{hom} (1 + 3f_i)} \quad (7)$$

where λ_0 is the thermal conductivity of the air ($0.026 \text{ W}\cdot\text{m}^{-1}\text{K}^{-1}$ at 20°C), f_i the volume fraction of the hemp particles, and λ_{hom} the measured thermal conductivity of the bulk.

The relation (6) enabled us to determine the thermal conductivity for a single particle of hemp shiv, considering spherical pores and the solid skeleton as the matrix. Expression (7) was calculated to determine the thermal conductivity for a single particle of hemp shiv by an inverse method.

3.5 Estimation of the effective thermal conductivity of a particle by inverse method: anisotropic consideration

Because of the oriented thermal conduction of the samples studied, a transversely isotropic thermal conductivity tensor $\Lambda_{\text{ani}}^{\text{hom}}$ is considered here. According Nguyen [6], the thermal conductivity tensor at the particle scale, $\Lambda_{\text{ani}}^{\text{hom}}$, is given by:

$$\Lambda_{\text{ani}}^{\text{hom}} = \begin{pmatrix} \lambda_N^{\text{hp}} & 0 & 0 \\ 0 & \lambda_T^{\text{hp}} & 0 \\ 0 & 0 & \lambda_T^{\text{hp}} \end{pmatrix} \quad (8)$$

The components are detailed in the work of Nguyen [6]. For the specific case of dry particles used here and according to the results of the author, a value of 1.57 was taken for the anisotropy parameter. This coefficient characterizes the ratio between the longitudinal and transverse conductivities of the particle expressed as:

$$\gamma_{\text{dry}}^{\text{hp}} = \frac{\lambda_N^{\text{hp}}}{\lambda_T^{\text{hp}}} = 1.57 \quad (9)$$

This method is more advanced than the one presented in section 3.4. as it takes the anisotropy of the particle into account, which is more in agreement with experimental results of the literature [17]. It made it possible to give the values of the components of the tensor $\Lambda_{\text{ani}}^{\text{hom}}$ at the particle scale by applying an inverse method.

3.6 A new method to estimate the effective thermal conductivity of a particle based on its chemical composition

By considering randomly oriented, transversely elongated or isotropic inclusions, the thermal conductivity tensor of hemp shiv particles can be obtained [7]. The conductivity tensor and its components are expressed as:

$$\Lambda_{\text{cyl}}^{\text{hom}} = \begin{bmatrix} \lambda_N^{\text{hom}} & 0 & 0 \\ 0 & \lambda_T^{\text{hom}} & 0 \\ 0 & 0 & \lambda_T^{\text{hom}} \end{bmatrix} \quad (10)$$

with :

$$\lambda_N^{\text{hom}} = f_i \lambda_0 + (1 - f_i) \lambda_i \quad (11)$$

$$\lambda_T^{\text{hom}} = \left(\frac{f_i}{\lambda_i + \lambda_0} + \frac{1 - f_i}{2 \lambda_0} \right)^{-1} - \lambda_0 \quad (12)$$

where λ_0 is the thermal conductivity of the solid skeleton, f_i the volume fraction of air (particular porosity), and λ_{hom} the homogenized thermal conductivity of the hemp particle.

In this part, we are concerned with the thermal conductivity of the particulate solid skeleton since it is not referenced in the literature and is difficult to measure. First, we need to focus on the microscopic description of heat transfer modes in solids. In this context, conductive energy transfer occurs mainly through electronic or vibrational excitations. In the case of a dielectric (insulator), as the electrons are strongly bound to the nucleus, thermal energy is only transmitted by vibration of the crystal lattice. These vibratory motions are generally described by elastic waves and associated with quasi-particles called phonons. Thermal conductivity is therefore governed by the mean free path of phonons [18]. The more ordered the atomic lattice (crystalline), the higher the conductivity. According to section 2.3, the solid skeleton of hemp shiv is mainly composed of cellulose, hemicellulose and lignin. However, while the rate of crystallinity observed in cellulose is about 80% [19], hemicellulose and lignin are essentially amorphous polymers [20]. In short, heat energy may be assumed to be transmitted only by cellulose. In the first instance, we considered that the solid skeleton was made of 100% cellulose. We were thus able to apply the formula that relates density and thermal conductivity as a function of temperature [21] for cellulose fibres (CF) from the formula for cellulose nano-fibres (CNF), respectively λ_{CF} and λ_{CNF} :

$$\lambda_{CNF} = (0.0787 + 2.73 \cdot 10^{-3} T - 7.6749 \cdot 10^{-6} T^2 + 8.4637 \cdot 10^{-9} T^3) \left(\frac{\rho_{sample}}{\rho_0} \right)^2 \quad (13)$$

$$\lambda_{CF} = 1.065 \lambda_{CNF} \quad (14)$$

where T is the temperature (in Kelvin) of the sample, $\rho_{sample} = 1543 \text{ kg.m}^{-3}$ its density (average density of cellulose [10]) and $\rho_0 = 1340 \text{ kg.m}^{-3}$ (reference value).

Following through on the strong assumption described earlier, the value of λ_0 will be taken to be equal to λ_{CF} . Thus, we theoretically recovered the value of the thermal conductivity of the particles, using the assumptions of, firstly, spherical pores and, secondly, cylindrical pores.

4. Results and discussion

4.1 Hemp shiv considered as a spherical inclusion

The results of the thermal conductivity (as a scalar value λ_{iso}^{hp}) for a single particle of hemp shiv and under the assumption of a spherical inclusions are shown in Table 2.

Table 2. Values of our spherical inclusion models compared to the literature

λ_{iso}^{hp} (W.m ⁻¹ .K ⁻¹)	Reference	Working methods
0.085 ± 0.014	Experimental method	Dry particles in bulk, temperature-controlled environment (relation 7)
0.0953	Theoretical method	From the chemical composition and particulate porosity (relation 6)
0.102	Cérézo [3]	Particles in bulk, other precautions not specified
0.1	Pham [22]	Not specified
0.110	Dartois [23]	Iterative micromechanical modelling

The relative difference between our theoretical and experimental values is 11 %, which indicates that they are consistent. Although, the limited number of references made comparison difficult, we can see that the relative difference between the average literature value of $0.104 \text{ W.m}^{-1}\text{.K}^{-1}$ and our values is 9% for the theoretical value and 23 % for the experimental one. This larger discrepancy may have several origins: if the bulk particle measurement by Cérézo [3] was conducted on non-dry particles, the adsorbed water increased the value of the particulate thermal conductivity and the measurement method could also have a significant effect on the measured values [24]. In addition, the origin of the particle and the variability this implies is likely to have had a significant impact on the value and will be discussed in section 4.5.

Nevertheless, whether they result from the theoretical or the experimental method, our mean values are systematically below those of the literature. It should be noted that Pham [22] concludes that homogenization models systematically overestimate the thermal conductivity of hemp concrete. However, these models use the thermal conductivity value of a single particle, as seen in Section 3. It may therefore be possible to suggest that overestimation comes from a non-significant value. Thus, according to our results, the value of $0.1 \text{ W.m}^{-1}\text{.K}^{-1}$ for a single dry particle of hemp shiv is probably above the real value.

4.2 Hemp shiv considered as a cylindrical inclusion

The thermal conductivity tensor for a single particle of hemp shiv, under the assumption of cylindrical inclusions, is composed of two types of values: a normal component and a tangential component as detailed in Fig. 5. The transversal conductivity λ_T^{hp} is perpendicular to the plane of the hemp particle and is assumed to be invariant under rotation by angle θ , according to the cylindrical shape. The normal conductivity λ_N^{hp} is oriented parallel to the direction of the tubular pores.

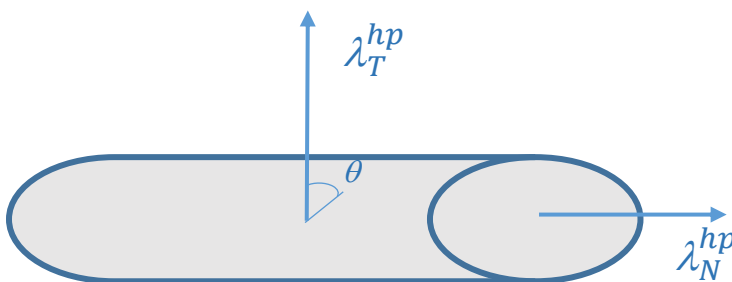


Fig 5. Components of the conductivity tensor in the case of the cylindrical inclusion hypothesis

Our results are summarized and compared with the literature in Table 3.

Table 3. Values of our cylindrical inclusion models compared to the literature

λ_T^{hp} (W.m ⁻¹ .K ⁻¹)	λ_N^{hp} (W.m ⁻¹ .K ⁻¹)	Reference	Working methods
0.067 ± 0.009	0.105 ± 0.013	Experimental method	Dry particles in bulk, temperature-controlled environment
0.0805	0.122	Theoretical method	From the chemical composition and particulate porosity
0.095	0.112	Dartois [23]	Iterative micromechanical modelling
0.088	0.122	Tran-Le[7]	Homogenization from a calibrated value for the conductivity of the solid phase

Transversal component. The relative difference between our theoretical and experimental values is 17 %, which indicates they are fairly consistent. Furthermore, the relative difference between the average literature value of 0.915 W.m⁻¹.K⁻¹ and our values is 14% for the theoretical value and 37 % for the experimental one. Our theoretical value is therefore rather in line with the results of the literature, while our experimental value is clearly different. This discrepancy, which is not significant for the other values, could be explained by a combination of several factors: impact of the measurement method, value of the anisotropy coefficient of our sample different from the one considered, overestimation of the literature values. Another bias is discussed in section 4.4.

Normal component. The relative difference between our theoretical and experimental values is 14 %, which indicates again that they are fairly consistent. Furthermore, the difference between the average literature value of 0.117 W.m⁻¹.K⁻¹ and our values is 4% for the theoretical value and 19 % for the experimental one. Our theoretical value is therefore in very good agreement with the results of the literature, while our experimental value is slightly lower.

4.3 Effects of compaction

Thermal conductivity measurements on bulk particles were repeated using loose or compacted samples by a maximum of 30% of the initial volume. The particle thermal conductivity values deduced from these measurements by the inverse method are given in Table 4.

Table 4. Comparison of values with and without compaction

Case studied	λ^{hp} (W.m ⁻¹ .K ⁻¹) from loose particles	λ^{hp} (W.m ⁻¹ .K ⁻¹) from compacted particles	Relative Difference
Isotropic	0.083 ± 0.008	0.086 ± 0.014	3%
Transversal	0.067 ± 0.007	0.066 ± 0.009	2%
Normal	0.105 ± 0.010	0.104 ± 0.015	2%

The difference between the particle thermal conductivity values with and without compaction is very small. Furthermore, no general tendency could be identified for any case. We can therefore conclude that the particulate thermal conductivity may not be impacted by the compaction of the aggregates. It is deduced that the solid skeleton is not altered by compaction forces. This is in agreement with the study by Antlauf [21], which demonstrates that no significant irreversible changes occur for a cellulose structure up to a pressure of about 0.9 GPa. What is true for an exclusively cellulosic skeleton could therefore be extended to a solid skeleton consisting mainly of cellulose, hemicellulose and lignin, under the conditions of our study.

4.4 Back to the solid skeleton hypothesis

A general trend emerges from the comparison of our experimental values with the literature references and with our theoretical results. Our experimental results are consistently lower. A first explanation could be that an offset was induced by the measurement method. However, to date, it is difficult to quantify this discrepancy [24]. Another possible explanation is the fact that the solid skeleton was considered to be composed exclusively of cellulose. This polymer, which is semi-crystalline, participates significantly in heat conduction, much more than an amorphous solid [25]. However, hemicellulose and lignin, which are the two other main components of the plant particle skeleton, are amorphous. In other words, we have overestimated the thermal conductivity of the solid skeleton and, by extension, that of the plant aggregate with this assumption. It would be useful to have access to the thermal conductivity values of the other constituents. This would allow the results of our theoretical method to be refined. Nevertheless, our value for the thermal conductivity of the solid skeleton is consistent with the only estimated value we found in the literature (Table 5).

Table 5. Comparison of thermal conductivity values of the solid skeleton

λ_s^{hp} (W.m ⁻¹ .K ⁻¹)	Reference	Working methods
0.6109	Theoretical method	From the results of Stelte [20]
0.5755	Nguyen [6]	Inverse analysis

4.5 Variability of particle thermal conductivity

The key strength of the theoretical method is that it quantifies the influence of certain intrinsic parameters of hemp shiv on its particulate thermal conductivity. Thus, the available data [10] on the variability of the cellulose density and the variability of the intra-particle porosity enabled us to set two bounds for theoretical values (Table 6).

Table 6. Extreme values considered for the calculation of the theoretical bounds

Variable parameter	Maximum	Minimum
Density of cellulose ($\text{kg}\cdot\text{m}^{-3}$)	1450	1630
Particulate porosity (%)	88.30	78.30

These maximum and minimum values obtained were compared with our own values and with those of the literature. The results for the different cases studied in this paper (isotropic and anisotropic) are presented in Figs. 6, 7 and 8.

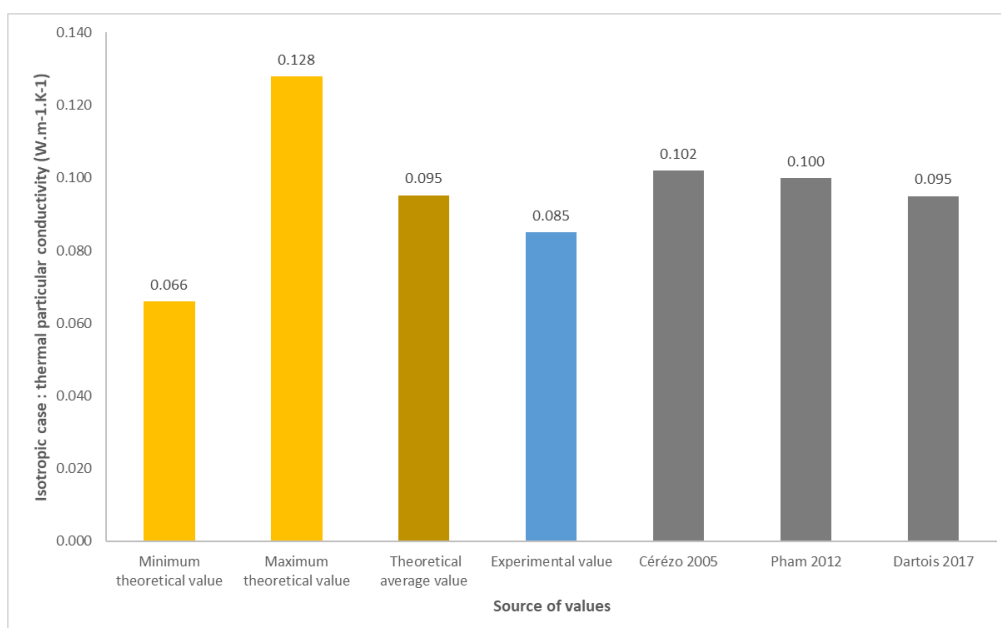


Fig 6. Isotropic case: extreme theoretical values compared to our results and those of the literature [3], [22], [23].

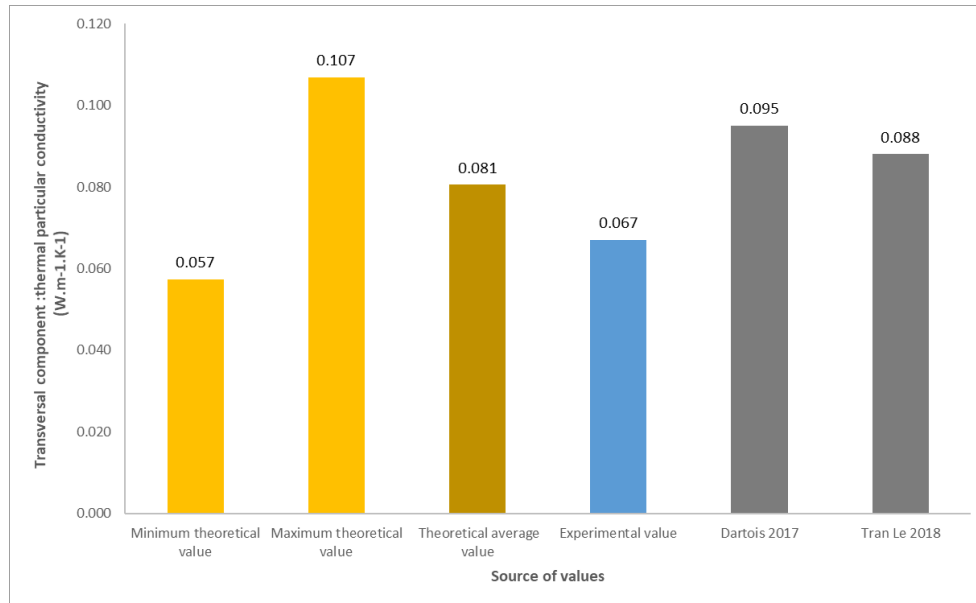


Fig 7. Transversal component: extreme theoretical values compared to our results and those of the literature [7], [23].

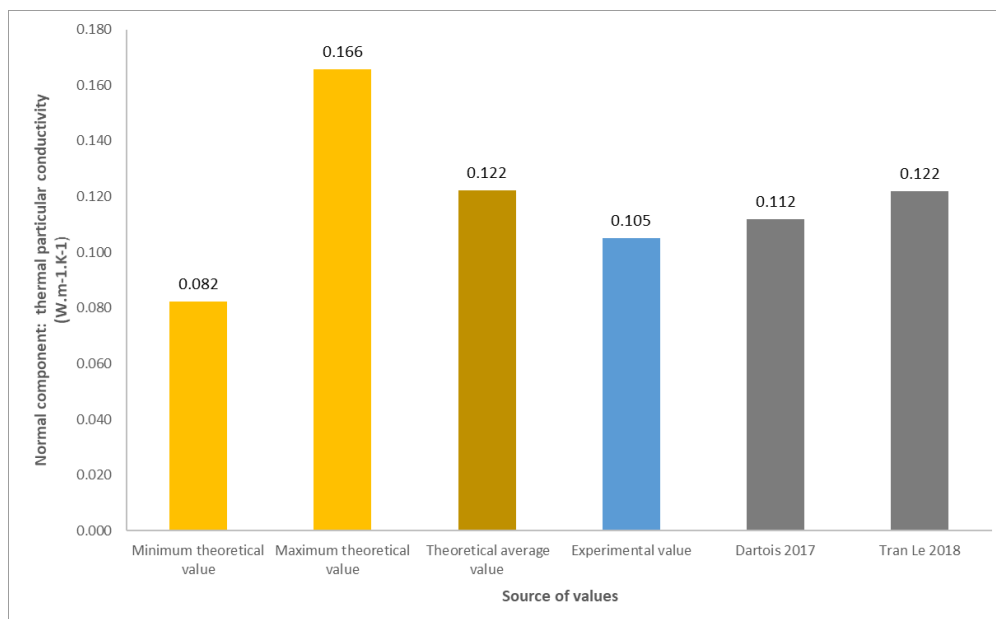


Fig 8. Normal component: extreme theoretical values compared to our results and those of the literature [7], [23].

It is worth noting that the theoretical bounds allow us to include, on the one hand, our experimental value and, on the other hand, those from the literature. Thus, we were able to define a range of particle thermal conductivities for hemp shiv, in both the isotropic and anisotropic cases. This range takes account of the main parameters (particle porosity and density of the solid skeleton), which depend on the very varied conditions of the plant

cultivation (growth, place and time of harvesting) and on the nature of the aggregate (cellulosic composition).

Finally, by calculating the ratio of the extreme theoretical thermal conductivities obtained in the anisotropic case, we recover an anisotropy factor consistent with the literature (Table 7).

Table 7. Anisotropy parameter γ^{hp} of hemp shive particle

γ^{hp}	Reference
1.51	Experimental method
1.55	Theoretical method (with maximum values)
1.44	Theoretical method (with minimum values)
1.57	Nguyen [6]
1.18	Dartois [23]
1.53	Tran-Le [7]

Our results are consistent with those of the literature and support a particle anisotropy factor of around 1.5 for hemp shiv particles. This value is important because it determines the anisotropy at the particle scale, but also impacts the anisotropy at the material and wall scale for composites made from hemp shiv. The application process of these materials (sprayed or compacted) leads to a preferential orientation of the particles and thus to anisotropy in the thermal behaviour of the wall.

5. Conclusion

In this study, a better knowledge and a better definition of the thermal properties of hemp-based building materials were being sought. We demonstrated that, in order to reliably predict their behaviour, a deeper understanding of the intrinsic properties of the plant aggregate is required. The development of methods to access local and microstructure-specific values is also essential.

Thus, homogenization techniques have been applied to determine the conductivity tensor at the scale of the dry hemp shiv particle from its microstructural characteristics (morphology of the particle and properties of the constituents). The isotropic (which can be seen as a simplified study) and anisotropic cases have been systematically treated. First, an experimental method was proposed. Unlike some values in the literature for which we did not have the test conditions, the protocol here has been described in detail, and the conditions controlled and explained. The aim of this was to be able to propose a reproducible method. Nevertheless, the values obtained experimentally were below the values of the literature. We therefore suspected an overestimation of the latter, since we were unable to assess the methods of determination.

Furthermore, a new method for estimating the particulate thermal conductivity has been developed. Using the results of a recent study [21] the hypothesis of a solid skeleton consisting exclusively of cellulose has been proposed. This assumption has been discussed and could lead to an overestimation of thermal conductivity values. Nevertheless, the strong point of this method is to be able to quantify the impact of the variability of the resource, coming from the plant world, on the particulate thermal conductivity. Our study also shows that the impact of compaction on the particle thermal conductivity value can be negligible. In addition, it demonstrates that the anisotropy parameter at the particle scale, considering heat transfer, is around 1.5. This determining factor has to be taken into account at the scale of the particle used as a bulk insulator or incorporated in a binding matrix, as the insulating layer at the wall scale will also be thermally anisotropic as a result.

Finally, the proposed methods were found to be consistent with the results of the literature. This highlights their relevance. Given the lack of data on the thermal conductivity of hemp shiv particles, this study provides new ways of assessing this value, which is impossible to measure directly with current measurement techniques. The experimental method used here is relatively easy to implement although it uses complex micromechanical modelling results. This inverse method therefore deserves to be applied to other aggregates from agricultural co-products. The theoretical method has also demonstrated its applicability by providing the range of values within which our theoretical, experimental and literature values fall. The same models should be applied first to other types of hemp shiv to evaluate the impact of the variability of this plant-aggregate. It could then be extended to other raw materials provided that a minimum of data is available on their microstructure. Further work will therefore focus on extending these methods to other aggregates. It will also be useful to consider the impact of adsorbed water on thermal conductivity at the particle level. This study provides thus new perspectives for determining the particulate thermal conductivity value, which is essential for predicting the insulating properties of bio-based materials.

6. References

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Chapitre 5. De l'échelle particulaire à celle du matériau

Préambule

Dans le but de promouvoir l'utilisation de co-produits agricoles locaux et de participer ainsi à l'émergence d'économies locale de la construction, les travaux de prédiction du comportement thermique s'orientent dans ce chapitre vers les résidus de la culture du tournesol. Cette plante est largement cultivée dans le Sud-Ouest de la France et, de manière plus globale, dans de nombreux territoires à travers le monde.

Dans l'article qui suit, un procédé innovant de fabrication de panneaux isolants à base de moelle de tournesol est présenté. Ce dernier ne nécessite ni liant ni processus énergivore. Les panneaux sont stabilisés sous différentes ambiances de température et d'humidité relative avant de mesurer leur conductivité thermique. En parallèle, un modèle prédictif de la conductivité thermique est déployé sur toute la gamme d'usage. Grâce à une meilleure compréhension du phénomène de sorption (cf. Chap 3) et du lien fait entre performance thermique et morphologie particulaire (cf. Chap 4), le modèle proposé s'appuie sur la réelle microstructure du matériau. Cette modélisation se base sur une triple homogénéisation. D'abord, la sorption de l'eau est traitée à l'échelle de l'espace poral de la moelle de tournesol. Ensuite, la conductivité thermique particulaire est évaluée en prenant en compte ce « pore humide ». Enfin, l'échelle matériau - et la conductivité thermique correspondante - est étudiée en considérant le panneau de particules comme une matrice d'air dans laquelle baignent des inclusions isotropes (en référence à la quasi-sphéricité du granulat considéré ici). En outre, en se référant à plusieurs travaux de la littérature, la température influence la conductivité thermique de l'air, de l'eau mais également celle du squelette solide de la moelle de tournesol. Ces paramètres sont pris en compte dans le processus d'homogénéisation. Enfin, comme dans le chapitre précédent, la plage de porosité intra particulaire est utilisée pour proposer une plage de valeurs prédites par la modélisation.

Article D. From aggregate to particleboard: A new multi-scale model approach to thermal conductivity in bio-based materials

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Keywords

Multiscale modelling, sunflower pith, thermal conductivity, bio-based building materials

Abstract

Bio-based materials have both insulating and eco-friendly properties, so their use is becoming of major interest in the current context of energy and greenhouse gas reduction. This study focuses on the development of 100% bio-based insulating panels. To minimize their environmental impact, particleboards were made from a locally available aggregate and were manufactured with a low energy process, without any binder. The thermal conductivity of the panels was predicted by means of an analytical model developed during the study, which was based only on easily accessible input data. This approach enabled the variability of the resource to be taken into account. This study demonstrates that it is possible to avoid time-consuming characterization steps and to propose a material whose thermal performance can be anticipated, over the entire temperature and relative humidity range of use, even before the manufacturing step and despite the variability of the resource.

1. Introduction

Energy and environmental issues have been a central concern since the start of the 21st century. All sectors must be involved and need to make a sustainable transition to eco-responsible practices. Since the construction sector produces almost 40% of global CO₂ emissions [1], it is a strategic lever for limiting climate change and global warming. Moreover, the general policy strategy set out by the European Union (EU), the European Green Deal, clearly states the objective of "Building and renovating in an energy and resource efficient way" [2]. The objective of the EU member states is to become climate neutral by 2050. In addition, the sixth assessment report of the Intergovernmental Panel on Climate Change [3] also reflects that establishing environmentally friendly practices is of world-wide interest. In this context, developing sustainable materials is a necessity, and bio-based materials can clearly and undoubtedly meet the challenges of tomorrow [4], [5].

The use of agricultural by-products in building materials is relatively recent. Their strength lies in their high insulating and hygroscopic powers [4]. However, although the raw materials are abundant, locally available and renewable, their development still encounters obstacles: the characterization of these materials is relatively costly and time-consuming (long-term stabilization of materials in enclosures) [6]. In addition, the lack of standard procedures or common approaches for characterizing such porous materials leads to arbitrary decisions and thus to a large diversity of results in the literature [6]–[9]. Several factors are holding back the development of bio-sourced materials. In addition to a lack of knowledge and experience of their performance [10], [11], there is probably also a lack of political will and construction-related habits [10]. The geographical location, the age of the plant, the climate, the soil conditions, the variety, the spatial location within the plant, and the processing method applied to obtain the bio-aggregates are all factors capable of causing intrinsic variability of the resource. The variability poses significant challenges in predicting the thermal conductivity of bio-based materials and ensuring consistent performance across different sites with varying locally available resources. Therefore, there is a need to establish a method for predicting the thermal conductivity of bio-based materials to guarantee consistent performance across different sites depending on the availability of the raw material. The present study deals with these important issues.

To promote a circular economy [12], sunflower crop residues were considered, as they are agricultural by-products available in the south-western part of France (where the study was conducted). A new, low-energy manufacturing process using no binder was developed for the production of 100% bio-based insulating panels in order to minimize the environmental impact of the material. Most particle boards described in the literature [13]–[15] are manufactured with a binder (synthetic one as urea formaldehyde or natural ones such as lignin or proteins) to ensure that they can withstand a minimum mechanical stress or show a minimum of composite cohesion. One article in the literature [7] reports a particle board made of sunflower bark and flax shiv, manufactured without any binder but at a high-temperature (190°C). It is therefore an energy-intensive manufacturing process. Furthermore, this type of process can affect the microstructure of the raw material and thus its insulating capacities. Sunflower pith was chosen for the manufacture of the insulating panel investigated here because it has shown good hygrothermal performance in previous studies [16], [17].

Some authors have sought to predict the thermal conductivity of bio-sourced materials by applying a self-consistent scheme, considering the material as concentric spheres [18], [19]. However, this is not representative of the real microstructure. A self-consistent cylindrical model has been used for vegetal wools to approximate the real microstructure [20] but the input data is not easily accessible. Another study uses a micro-scale approach but this is based on X-ray tomography images [21], a costly characterization technique. Furthermore, none of these approaches consider the inherent variability of the resource. Consequently, the model developed in the present study aims to be representative of the material's microstructure while requiring only easily accessible input data. The objective is to propose a relevant and easy-to-apply model that can take the variability of locally available aggregates into account. It is worth noting that current techniques remain incapable of measuring the thermal conductivity of a single particle while local measurements can be carried out but require complex and expensive equipment [22], [23]. A few authors have tried to access the particulate thermal conductivity of hemp shiv by an inverse method [24] or by iterative micromechanical modelling [25] but this remains marginal. The work presented here therefore focused on insulation capacities at a scale that is not frequently used, that of the aggregate. In order to take the temperature effects and the hygroscopic capacity of bio-based material into account, it proposes a prediction of the insulating behaviour of the panels over their entire range of use, in terms of temperatures and relative humidities. A multi-scale [26] model based on Eshelby's theory was chosen as the best way to meet these objectives since it seemed necessary to consider the insulation capacities at the aggregate scale to predict the thermal conductivity of bio-based building materials.

First, the choices – concerning aggregates, manufacturing method, measurement method and modelling – made to carry out this study are clearly explained. The characteristics of the sunflower pith are extensively analysed to explain the input data of the predictive model. Then, the thermal conductivity of a single particle is presented and discussed and the variability of the resource is taken into account from this particulate scale. Finally, the experimental results are compared with modelled ones for the thermal conductivity of the pith particleboards. Thus, the ability of the model to expand from the microstructural to the material scale is highlighted.

2. Materials and methods

2.1 Aggregates

2.1.1 Selection criteria

Sunflower is a crop that is widespread in the south of France but this plant is also commonly cultivated throughout the world [27]–[29]. Consequently, by-products of this crop were chosen to extend the scope of the present work. The pith came from the plant stem, which remains on the ground after harvesting. The pith and the bark separate more or less spontaneously depending on the effect of climatic conditions but a separation process is generally used to obtain samples of each aggregate [30]. This step determines the degree of purity of the particleboards obtained (Figure 1).



Figure 1. Particleboards made of sunflower pith (no binder)

2.1.2 Microscopic and macroscopic shape

On a macroscopic scale, sunflower pith is quite spherical with a variable diameter (about 4 to 10 mm)[31], [32]. The sample used in this study was consistent with the literature (Figure 2). It is useful to note that it was free of impurities (no traces of bark). This sample came from the same cooperative as the sunflower pith characterized in a recent study [31]. However, it was from a different harvest and a different process for separating the bark and pith may have been used.



Figure 2. Sunflower pith used to manufacture particleboards.

On a microscopic scale, the Scanning Electron Microscopy images showed a honeycomb structure visible in two perpendicular sections [32]–[35]. The microstructure of this aggregate could therefore be described as a solid skeleton having numerous porosities (air-filled cells) with a shape close to that of a sphere.

2.1.3 Physical and chemical specificities of sunflower pith

i) Intra-particulate porosity

The alveoli of the sunflower pith are so numerous that the intra-particle porosity of this aggregate is extremely high. It is probably variable from one pith to another but always exceeds 90% (Table 1). For comparison, the intra- particulate porosities of hemp shiv and sunflower bark are about 50 % and 60 %, respectively [36].

Table 1. Intra-particulate porosity of sunflower pith according several studies.

Intra-particulate porosity (%)	References	Measurement technique
96.4 ± 7.8	[31]	from bulk and particulate density
95.8	[36]	acoustic absorption
96	[6]	acoustic measurements

Because the samples were different (harvesting time and method, variety of plant), the particle porosity of the sunflower pith showed a certain variability. To take account of this, the values considered in this study are taken from [31] as the average value is consistent with other references [6],[36]. The maximum and minimum limits were used to give a range of output values of the model. For the rest of this study, porosity is assumed to be between 87 % and 99 % (considering the minimum and the maximum values). The particulate porosity, as input, was impacted by the variability of the resource and more data would be required to define a range of particle porosity for sunflower pith. This would allow the variability of the resource to be taken into account at this level, as the immobile air in the cells provides insulating capacities. In this sense, the variation in particle porosity was probably the most important factor affecting the thermal conductivity of the aggregate and therefore the board. Nevertheless, given the available data, the particulate porosity was assumed to be between 88.6 % and 99.5 % in this study.

ii) Chemical composition

Sunflower pith is mainly made of cellulose (40-50%), hemicellulose (20 %), minerals (15%) and lignin (5%) [31], [37]. Like other lignocellulosic aggregates, the density of its solid skeleton is very close to that of cellulose [38]. The skeleton density of sunflower pith is between 1461 and 1623 kg.m⁻³ (average of 1536 kg.m⁻³) while the average density of cellulose in lignocellulosic materials is 1543 kg.m⁻³ [31].

2.1.4 Adsorbed water

Plant aggregates have the capacity to adsorb and desorb water depending on the ambient humidity. This is an advantage as it allows passive moisture regulation and thus improves comfort and indoor air quality [4]. Nevertheless, thermal conductivity is affected by this phenomenon. The thermal conductivity of water is more than twenty times that of air ($\lambda_{\text{air}} = 0.026 \text{ W.m}^{-1}.\text{K}^{-1}$ at 20 °C). The increase of thermal conductivity with moisture, and accordingly with the water content, has been demonstrated in bio-based materials by several authors [19] [39] [40]. Sunflower pith has a particular affinity with water [41]; it is able to absorb much more water than other plant aggregates [42]. With regard to adsorption, recent work shows that sunflower pith is also capable of fixing more water than aggregates such as hemp shiv, sunflower bark or flax shiv [31].

The model used in this study required knowledge of the volume fraction of water contained in the sunflower pith according to the ambient relative humidity. Thus, water adsorption isotherms were converted into water volume fraction versus relative humidity (Figure 3).

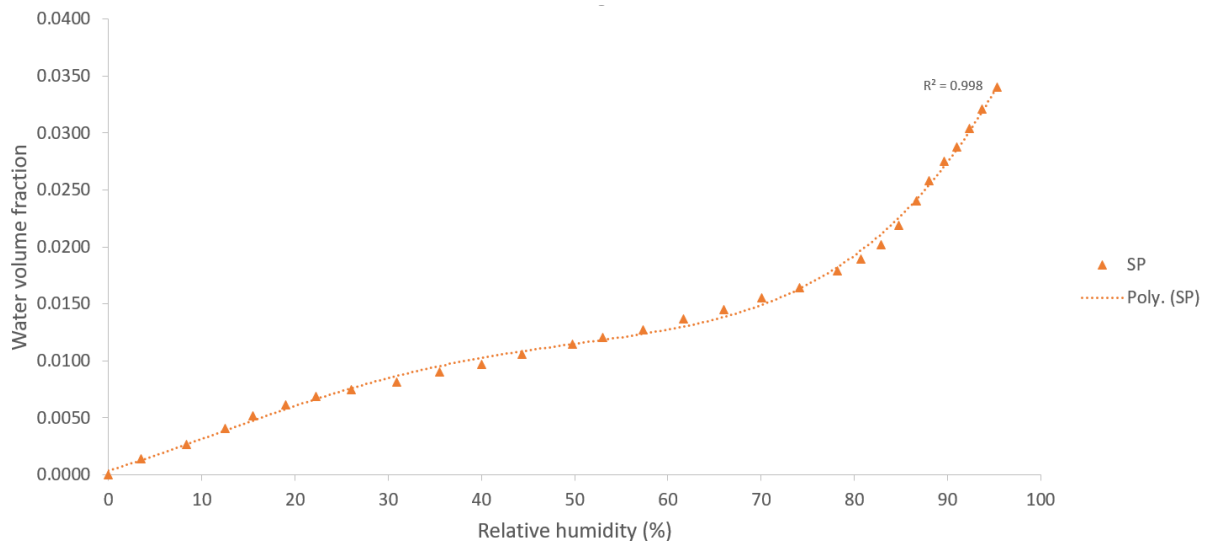


Figure 3. Water volume fraction in sunflower pith (SP) depending on relative humidity from experimental results (SP- loose fill insulation) of Ratsimbazafy [31] and polynomial regression (Poly.)

The experimental curve was modelled by a 4th degree polynomial function. As the coefficient of determination (R^2) was close to 1, an extrapolation of the water volume fraction adsorbed was made for 100% relative humidity, experimental values not being available up to this extreme value. These data allowed the adsorbed water phase to be considered over the entire relative humidity range in the predictive model of thermal conductivity.

2.2 Particleboards

2.2.1 Manufacturing criteria

In this study, a manufacturing method with two priority targets was developed. The manufacturing process had to be easy-to-implement and low-energy while the manufacturing protocol was innovative since it used neither a binder nor a high-temperature process.

2.2.2 Manufacturing steps

The manufacturing procedure applied was adapted from the protocol developed by Ratsimbazafy [31]. The successive steps of the protocol developed in this study are detailed below and the main steps are illustrated in Figure 4.

1. Weigh the aggregates in accordance with the mass ratio of a plant-based concrete formulation;
2. Place the weighed aggregates in a water-permeable net;
3. Completely immerse the net containing the aggregates in a tank filled with water. Keep the entire sample immersed during only 15 seconds (because of its higher water absorption capacity);
4. Shake the net containing the wet aggregate 50 times from bottom to top to remove excess water;

5. Open the net and mix the wet aggregate by hand to homogenize the mixture;
6. Apply mould release oil to the grid and the mould (20x20x20 cm³) to be used on the vibratory plate;
7. Pour a quantity of wet aggregate corresponding to about one third of the mould volume into the mould;
8. Manually scarify the top surface after compaction to facilitate good adhesion with the next layer;
9. Repeat steps 7 and 8 once, making sure that the mould is filled and compacted so that the plunger butts up against the mould;
10. Then, place the sample in a press to finish removing the excess water. Compact to the desired thickness. Stop compaction when no more water comes out of the mould.
11. Wait about 5 minutes and unmount carefully;
12. Leave to dry for 48 hours in the ambient environment;
13. Place the particleboards in an oven at 60°C until a constant weight is achieved.

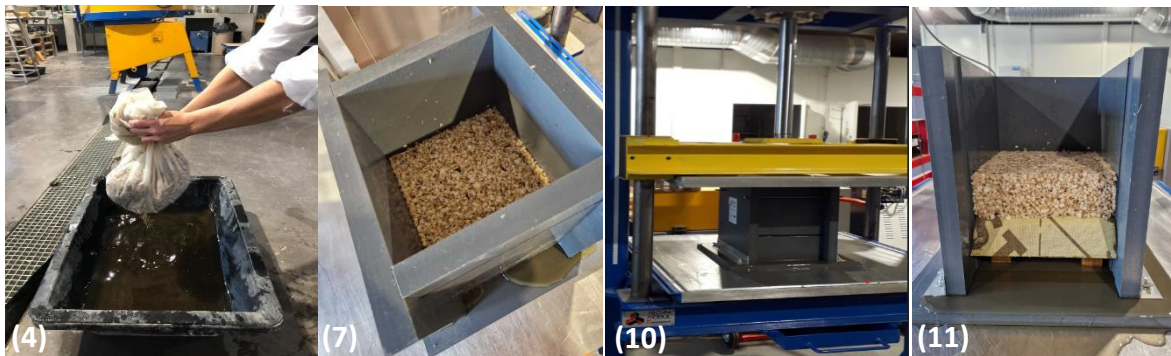


Figure 4. Main manufacturing steps (the number corresponds to the step)

After drying, the sunflower pith turned brown, whereas it had its original colour when removed from the mould (Figure 5).

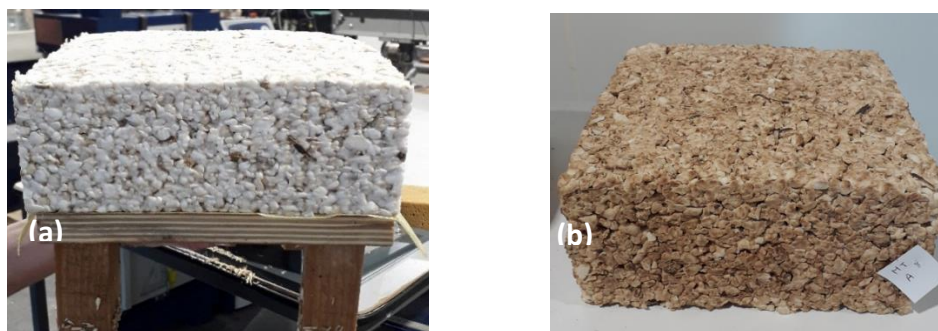


Figure 5. Particleboard after manufacturing process (a) and after drying (b) - Density $\rho = 35 \text{ kg.m}^{-3}$

Particleboards made of sunflower pith proved to be very promising. Pith panels were easy to transport and handle and their excellent cohesion allowed several conductivity measurements

at different temperatures and relative humidities. As the manufacturing process did not use any binder, the plant aggregates had to be able to ensure material cohesion.

2.2.3 Volume fraction measurement

The volume fraction, f_a , of aggregates was required for the models. It was defined as follows:

$$f_g = \frac{V_a}{V_p} \quad (4)$$

where V_p is the total volume of the particle board and V_a that of aggregates.

In order to evaluate this value for the particleboards manufactured, the definition of density was used to modify (4) to:

$$f_g = \frac{\rho_p}{\rho_a} \quad (5)$$

where ρ_p and ρ_a are the density of particleboard and the density of aggregate (kg.m⁻³), respectively.

The particulate densities of selected aggregates are available in the work of Ratsimbazafy [31] as mentioned in section 2.1.4. The density of each particleboard was obtained from the ratio of the mass of the material to its volume (measured with a digital calliper). Analogously, for particles in bulk, the volume fraction was defined as the ratio between the density of the bulk ρ_{bulk} and the density of aggregate ρ_a .

2.3 Thermal conductivity

2.3.1 Stabilization of samples

The particleboards were first dried in an oven at 60°C and then subjected to different relative humidities (30%, 60% and 80%) in a climatic enclosure until they reached a constant mass. They were systematically stabilized at 20°C to carry out the measurement campaign because measurements were made in a temperature-controlled space at 20°C. The samples were weighed again after a sequence of measurements to ensure that any water adsorption was negligible (i.e. mass variation lower than 2%). Finally, particleboards were stabilized at room temperature (23 ± 1°C) and relative humidity of 50 ± 3 %.

2.1.2 Measurement protocol

For thermal conductivity measurement [43], several criteria were considered in this study to determine the most appropriate among the various methods that exist. First, the measurement method had to be suitable for low-thermal-conductivity materials. In addition, on-site availability was essential due to the difficulty of transporting fragile samples. (Tests were carried out on hemp shiv and sunflower bark panels before being discontinued due to damage to the materials). As a result, a Neotim-FP2C hot wire apparatus was chosen for this work. This transient method has the extra advantage of limiting water migration during measurement [19]. Although the relevance of the method is open to discussion in the direction of compaction, it was decided to carry out measurements in two directions (Figure 6). The measurements were repeated at least 5 times, at different locations, for each sample and for each configuration.

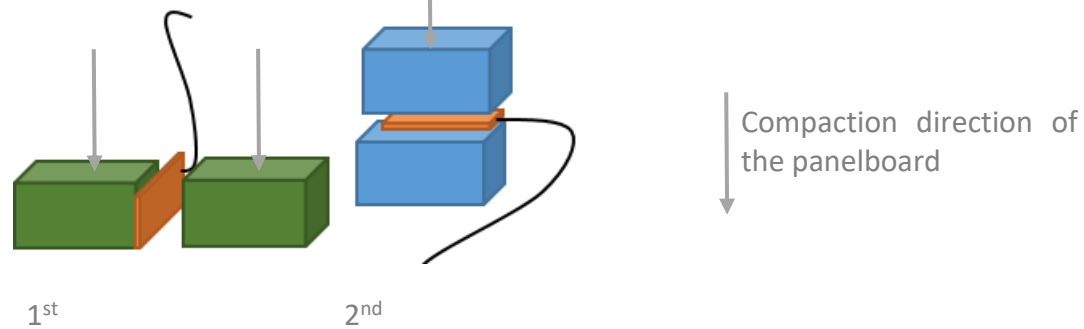


Figure 6. The two configurations for thermal conductivity measurements applied to the panelboards.

The panelboards were held together by a clamp or weighted during the measurement, to limit the air gap around the sensor and avoid distortions in the results. The thermal conductivities given are the average of at least five values and show a coefficient of variation of less than 5%.

2.1.3 Solid skeleton hypothesis

Since it is not possible to measure the thermal conductivity of the particulate solid skeleton with current techniques, assumptions had to be made. No data on the thermal conductivity of hemicellulose and lignin can be found in the literature but a recent work on cellulose fibres was exploited in [44]. It evaluates the dependence on density and temperature of the thermal conductivity of cellulose fibres and deduces a polynomial function that was used in the present work. Furthermore, as cellulose is a polymer formed of glucose with different spatial organizations, it has a semi-crystalline structure [45]. This highly ordered atomic arrangement in crystalline parts is much more efficient than an amorphous one to conduct the heat energy [46]. Since hemicellulose and lignin are amorphous components [47], the thermal conductivity of the skeleton was assimilated to that of cellulose only. Thus, the thermal conductivity of the solid skeleton was evaluated thanks to recent work on cellulose fibres [44]. It depends on the density of the cellulose and the temperature. An average value of $0.57 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ was used in this study. This is largely consistent with the value of $0.58 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ that was determined on hemp shiv with an inverse method [48].

2.4 Homogenization schemes

2.4.1 Choice of homogenization scheme

Although the self-consistent model is commonly used in the literature, there are some limitations to and inconsistencies in its application, which are discussed here. The Mori-Tanaka model, chosen in this study, is presented as it appears more relevant in the modelling of bio-based materials. This model will be applied in an iterative process (section 2.3.3).

It is worth noting that the self-consistent scheme is widely used in the literature to predict the thermal conductivity of bio-based materials [8] [10] [37] [41] [50]. In this scheme, the heterogeneous medium is described as an assembly of concentric spheres of different sizes, which is rather far removed from both the real microstructure and the macroscopic shape of sunflower pith as mentioned in section 2.1.2. More specifically, when the multi-phasic aspect of the bio-materials is considered, this scheme leads to a certain inconsistency in the position of the different phases as illustrated in Figure 7. Water is not only adsorbed on the binder surface. It is actually adsorbed much more within the pore space, which is described by the

sorption isotherms and the associated water binding mechanisms [18], [51]. The sorption curves obtained for the sunflower pith clearly indicate that the water binds to the pith (cf. section 2.1.4) and not to the binder.

In order to propose more realistic scheme, we looked into an equivalent inclusion method in Eshelby's sense [26] first developed for metals and widely applied for heterogeneous materials, such as composites, to gain access to the effective properties [52]–[54]. The Mori Tanaka scheme [55] was then applied in order to consider the interactions between the inclusions. One phase (the matrix) completely surrounds each element of the other phase (the inclusions). The inclusions were assimilated to spheres in this study. This is consistent with the microstructure and macroscopic form of sunflower pith as described in section 2.12. More complex micromechanical models can be developed but, here, we investigate whether a simplified model can be sufficient. In addition, this approach allows us to described the heterogeneous medium in a more realistic way than in the work of Somé [49] (Figure 7). Consequently, the water phase was considered at the pore scale in our model since this is where it is fixed in the plant particle by physical adsorption [56].

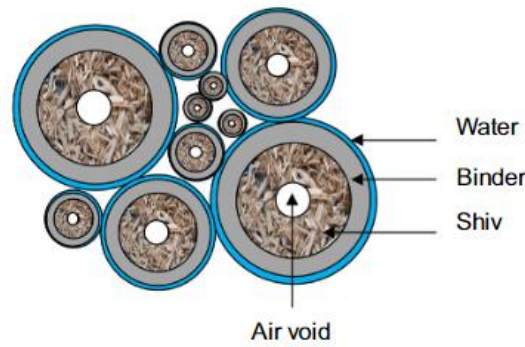


Figure 7. Heterogeneous medium (hemp concrete) considered as an assembly of spheres according to the self-consistent scheme [49].

2.3.2 Basic homogenization model

According to the classical theory of Eshelby, a heterogeneous material can be systematically described as a matrix-type composite (index 0) comprising several inclusions (index i). In these conditions, its homogenized thermal conductivity tensor Λ^{hom} is given by:

$$\Lambda^{hom} = \sum_{i=0}^n f_i \Lambda_i \mathbf{A}_i = \Lambda_0 + \sum_{i=1}^n f_i (\Lambda_i - \Lambda_0) \cdot \mathbf{A}_i \quad (6)$$

where Λ_0 is the thermal conductivity tensor of the matrix, f_i the volume fraction of the inclusion i , \mathbf{A}_i its second order concentration tensor and Λ_i its thermal conductivity tensor. Because only one family of inclusions was considered in each step of homogenization, the homogenized thermal conductivity tensor can be simplified as follows:

$$\Lambda^{hom} = \Lambda_0 + f_i \cdot (\Lambda_i - \Lambda_0) : \mathbf{A}_i \quad \text{with } \mathbf{A}_i = (\mathbb{I} + \mathbb{S} \Lambda_0)^{-1} (\Lambda_i - \Lambda_0) \quad (7)$$
 where \mathbb{S} is the Eshelby tensor whose value is defined relatively simply when a spherical inclusion is assumed:

$$S_{ij} = \begin{cases} 0 & \text{if } i \neq j \\ 1 & \text{if } i = j \end{cases} \quad (8)$$

This results in an analytical formula for the expression of the homogenized thermal conductivity scalar, λ^{hom} , as defined below:

$$\Lambda^{hom} = \lambda^{hom} \mathbb{I} \quad (9)$$

where the analytical formula for λ^{hom} comes from the homogenization scheme of Mori Tanaka [55] .

$$\lambda^{\text{hom}} = \lambda_0 + \frac{3 f_i (\lambda_i - \lambda_0) \lambda_0}{(1 - f_i) \lambda_i + (2 + f_i) \lambda_0} \quad (10)$$

with λ_0 the thermal conductivity of the matrix, λ_i that of the inclusion, and f_i the volume fraction of the inclusions.

Thanks to the formula (10), a link is provided between the microstructural characteristics and the macroscopic behavior law for conductive heat transfer.

2.3.3 Triple homogenization method

In this study, an iterative multi-scale approach was developed for the material by performing a triple homogenization, from the pore scale to the material one. Each homogenization step is based on the principle described in the previous section. The pore scale considered the water adsorbed. The particle scale was strategic, both to obtain the necessary input data for the material scale and to consider the intrinsic properties of the plant aggregate. In this iterative process, the equivalent medium of the k-th homogenization becomes the inclusion in homogenization k+1. These steps are summarized in Figure 8.

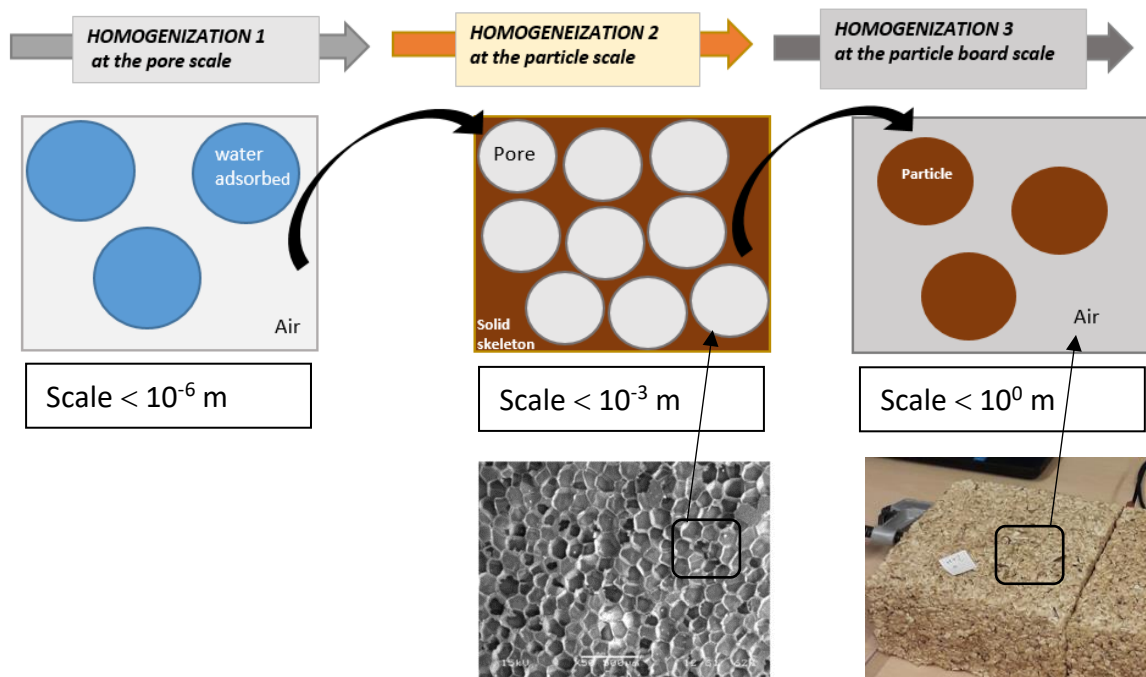


Figure 8. Steps of the triple homogenization proposed in this study and the corresponding real scale (SEM images from Magniont et al.[33] and image of our particleboards)

For easier understanding, Table 2 shows the input and output data used for each homogenization and how it provides the inputs and outputs of each homogenization step.

Table 2. Input and output data used for each homogenization

	Input data and constraint dependency	Reference	Output data
Homogenization 1	$\lambda_{air}(T, RH)$ $\lambda_{water}(T)$ $f_{water}(RH)$	[57] [58] Cf. section 2.1.5	$\lambda_{pore}(T, RH)$
Homogenization 2	$\lambda_{pore}(T, RH)$ $\lambda_{skeleton}(T)$ f_{pore}	According homogenization 1 [44] and section 3.1.3 Assimilated to intra-particulate porosity	$\lambda_{aggregate}(T, RH)$
Homogenization 3	$\lambda_{aggregate}(T, RH)$ $\lambda_{air}(T, RH)$ $f_{aggregate}$	According homogenization 2 [57] Depending on formulation	$\lambda_{particleboard}(T, RH)$

For each step, the analytical formula for λ^{hom} was applied as follows:

Step 1

$$\lambda_{pore}^{hom} = \lambda_{air} + \frac{3 f_{H_2O} \lambda_{air} (\lambda_{H_2O} - \lambda_{air})}{(1 - f_{H_2O}) \lambda_{H_2O} + (2 + f_{H_2O}) \lambda_{air}} \quad (10)$$

Step 2

$$\lambda_{particle}^{hom} = \lambda_{skeleton} + \frac{3 f_{pore} \lambda_{skeleton} (\lambda_{pore} - \lambda_{skeleton})}{(1 - f_{pore}) \lambda_{pore} + (2 + f_{pore}) \lambda_{skeleton}} \quad (11)$$

Step 3

$$\lambda_{bulk}^{hom} = \lambda_{air} + \frac{3 f_{particle} \lambda_{air} (\lambda_{particle} - \lambda_{air})}{(1 - f_{particle}) \lambda_{particle} + (2 + f_{particle}) \lambda_{air}} \quad (12)$$

The iterative use of known homogenization results gives access to macroscopic parameters by starting from microscopic parameters. The input data are characteristic of the material's microstructure, yet they are accessible through relatively simple and inexpensive characterization tests.

2.3.4 Effects of temperature and adsorbed water phase

The effect of temperature on the thermal conductivity of this type of material has been demonstrated in relatively recent work on agro-material composed of hemp shiv [59]. Consequently, one of the difficulties was also to consider this dual dependency. It should be noted that the thermal conductivity of air varies significantly with temperature and relative humidity [57] and the thermal conductivity of cellulose fibres is also temperature dependent [44]. It was therefore assumed that the solid skeleton of the plant aggregates, which, according to section 2.1.2, is mainly composed of cellulose, would also be temperature dependent. Finally, it should be emphasized that the study of the effects of temperature and

of the adsorbed water phase allows us to take account of the real loads to which bio-based materials are subjected under their conditions of use.

The high water sorption capacity of lignocellulosic materials and its effect on their thermal behaviour is well-known [60] [40]. For hemp composites, water sorption can increase by more than 0.002 W/mK for each 1% variation in relative humidity [19], [61]. Therefore, it appears necessary to consider the impact of adsorbed water on the thermal conductivity of plant-based materials.

In addition, cellulose fibres have been found to have a temperature-dependent thermal conductivity [44]. Since the solid skeleton of the plant particles is mainly composed of cellulose (section 2.1.2), sunflower pith particles must also be temperature dependent. Recent work on agro-material composed of hemp shiv demonstrated that thermal conductivity can increase by about 10 % between 10 °C and 40 °C in this type of materials [59].

Hence, to simulate the real loads to which bio-based materials are subjected under the conditions of use, our model has to consider this dual dependency on both temperature and adsorbed water. It is noteworthy that the thermal conductivity of air also varies significantly with temperature and relative humidity [57].

3. Results and discussion

3.1 Effective thermal conductivity of a single particle

i) Water adsorbed at the pore scale

In the first homogenization step, the matrix is air, and the inclusion is adsorbed water. Thus, this preliminary phase allows the water adsorbed in the pore space to be considered as a function of relative humidity (Figure 9).

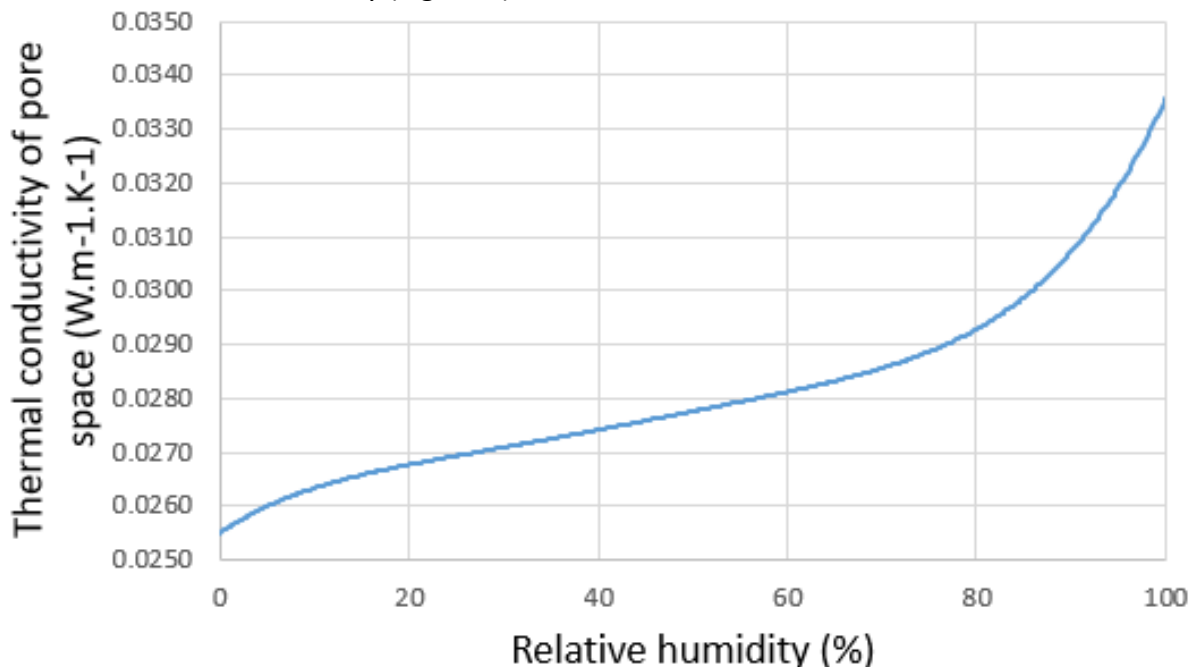


Figure 9. Thermal conductivity of the pore space depending on relative humidity from 1st homogenization step.

The curve indicates a significant increase in thermal conductivity after 80% RH. This is in line with what is observed in the water adsorption isotherms: above this value, the adsorption of

water is much greater than at lower humidity. At the pore scale, the model thus consistently reflects the impact of adsorbed water, which has a higher thermal conductivity than air.

ii) Impact of adsorbed water at the aggregate scale

Thermal conductivity results are presented for a single pith particle in Figure 10. Adsorbed water is taken into account at the pore scale as explained before. The maximum and minimum values correspond respectively to the minimum and maximum particle porosity (cf. section 2.1.3). It is important to note that the range on input data can have a significant impact on the output data at this particulate scale.

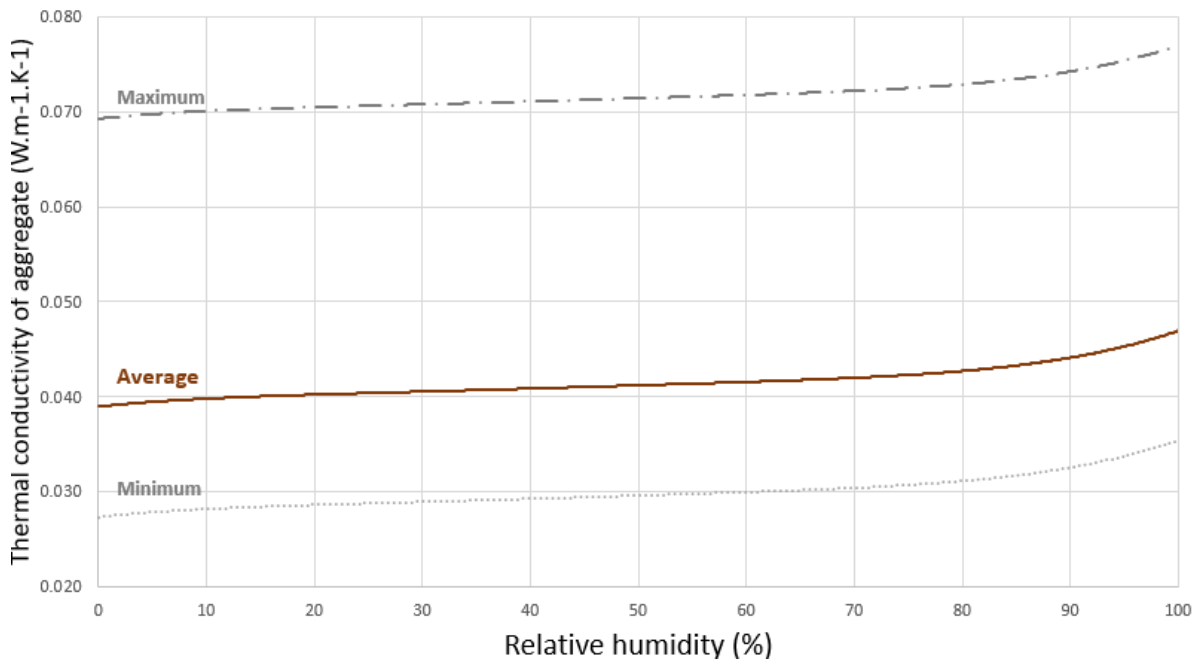


Figure 10. Thermal conductivity values of a sunflower pith particle (average, minimum and maximum) depending on ambient relative humidity, at 20 °C.

To obtain the dry values stated, since there was no adsorbed water, the first homogenization was not applied prior to the second one. At non-zero relative humidity, the first two steps of iterative homogenization were conducted. The value of 20°C was chosen as a reference since it is close to the average temperature in a building.

Finally, a comparison with the thermal conductivity of hemp shiv particles is interesting. It is about 0.1 W.m-1.K-1 according to the very few references in the literature [24], [25], [62]. Even under the most unfavourable conditions (high humidity), sunflower pith shows much better insulating capacities than hemp shiv.

iii) Impact of temperature

In order to visualize the range of particle thermal conductivities according to temperature, this value was calculated between the temperature extremes of 0°C and 50°C (Figure 11). If water is to be adsorbed, it must be in liquid form and therefore at a temperature of at least 0°C. The maximum temperature was chosen to reflect extreme conditions during a heat wave

with solar gain in a wall. The value of 50 % RH was chosen as a reference since it is close to the average relative humidity in a building.

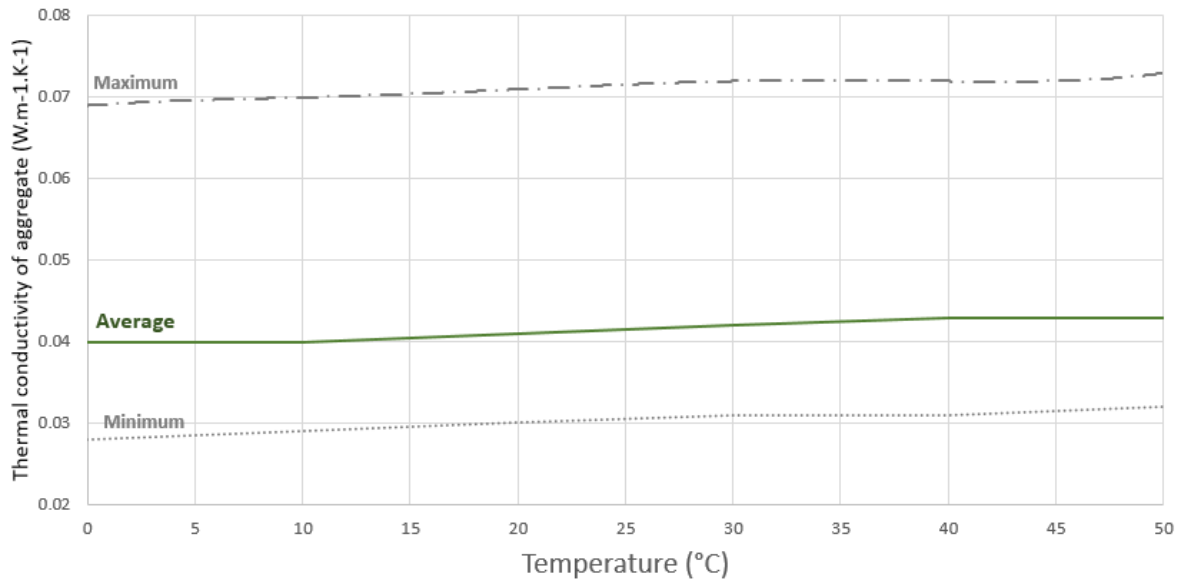


Figure 11. Thermal conductivity of a sunflower pith particle (average, minimum and maximum) depending on temperature, at 50 % RH.

According to these results, the particulate thermal conductivity varies, on average, by 10% with temperatures between 0 and 50 °C. This figure is close to the one found in a study conducted on hemp-based composites [59]. Although the aggregates are different, the temperature behaviour appears similar.

3.2 Measurement and isotropic hypothesis of particleboards

Table 3 compares the results of thermal conductivity measurements between the two configurations presented in section 2.1.2. The percentage of removed points (according to the protocol for measurement with the hot wire) is specified to enable the relevance of the measurements to be appreciated.

Table 3. Thermal conductivities of sunflower pith panels at 20±3°C: comparison of the two measuring directions.

Relative humidity	Configuration 1 ⊥ compaction direction		Configuration 2 // compaction direction		Relative gap between the configurations (%)
	Thermal conductivity (W.m ⁻¹ .K ⁻¹)	% points removed	Thermal conductivity (W.m ⁻¹ .K ⁻¹)	% points removed	
Dry state	0.031 ± 0.001	8	0.031 ± 0.001	5	0
30%	0.032 ± 0.001	7	0.032 ± 0.001	6	0
50%	0.035 ± 0.001	7	0.035 ± 0.001	6	0
60%	0.035 ± 0.001	8	0.034 ± 0.001	5	2
80%	0.041 ± 0.001	5	0.039 ± 0.001	5	4

Despite the compaction applied during panel manufacturing, the direction of the measurement does not seem to affect the result. This supports the isotropic hypothesis for this material. This may be surprising given the method of manufacturing, which leads to compaction and therefore probable deformation of the aggregates. However, we clearly observed an expansion of the material after compaction. The aggregates probably returned to their original shape once the compacting force was removed. Given the slight deviation of results between the two configurations, the average value is considered in the following. Lastly, the significant increase of thermal conductivity with relative humidity (almost 30% between the dry and wet states) is consistent with literature findings [51], [63], [64]. Thermal conductivity of water is much higher than the one of air and the sunflower pith has a high capacity to adsorb water from the ambient air (cf. section 2.1.4).

3.3 Particleboards: experimental and model values for different relative humidities

The thermal conductivity of particleboards made with sunflower pith was calculated according to the triple homogenization presented in section 2.3.3. In order to compare experimental (from the protocol given in section 2.1.2) and model values, measurements were carried out over the whole range of relative humidity. The evolution of the thermal conductivity of the panels as a function of relative humidity is presented in Figure 12. It is useful to recall that error bars for the theoretical values were calculated from the range of particulate porosity, which is assumed to be the factor having most impact on thermal conductivity values.

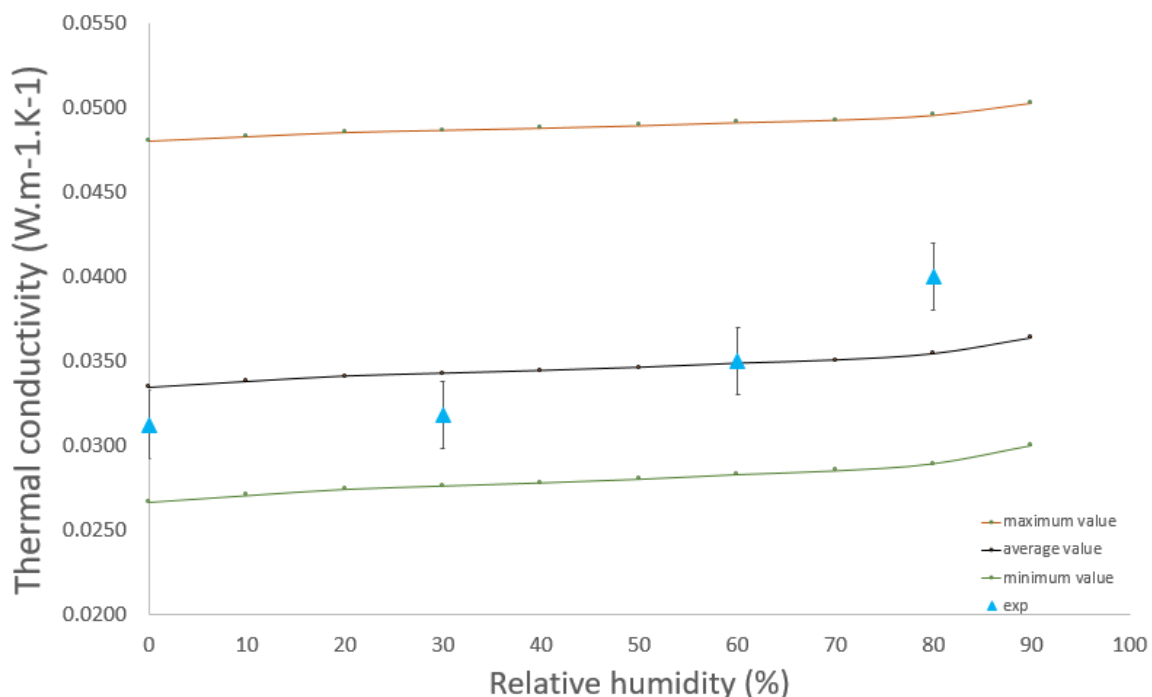


Figure 12. Thermal conductivity versus relative humidity: board of sunflower pith at 20 °C.

All experimental points are framed by the minimum and maximum modelled values. The extreme relevance of the model can be underlined here: it predicts the thermal conductivity of the pith panel over its whole range of use.

The maximum and minimum limits first defined (cf section 2.1.3) could be reduced given the quality of the prediction by the model: experimental results are within 15% of the average modeled value (from the average intra-particulate porosity). The developed model therefore allows to predict within 15% the thermal conductivity of particleboards made of sunflower pith over the entire range of relative humidity. It is worth noting that the only characteristic input data of the aggregate are intra-particle porosity and its sorption curve (cf section 2.3.3).

3.4 Particleboards: experimental and model values of the study for different temperatures

As in the previous section, the thermal conductivity of particleboards made with sunflower pith was calculated according to the triple homogenization presented in section 2.3.3. In order to compare experimental and model values, measurements were made over the whole range of temperature. This time, the relative humidity was set at 50% and the temperature dependence was investigated (Figure 13). This value was chosen since it is a common value for indoor air.

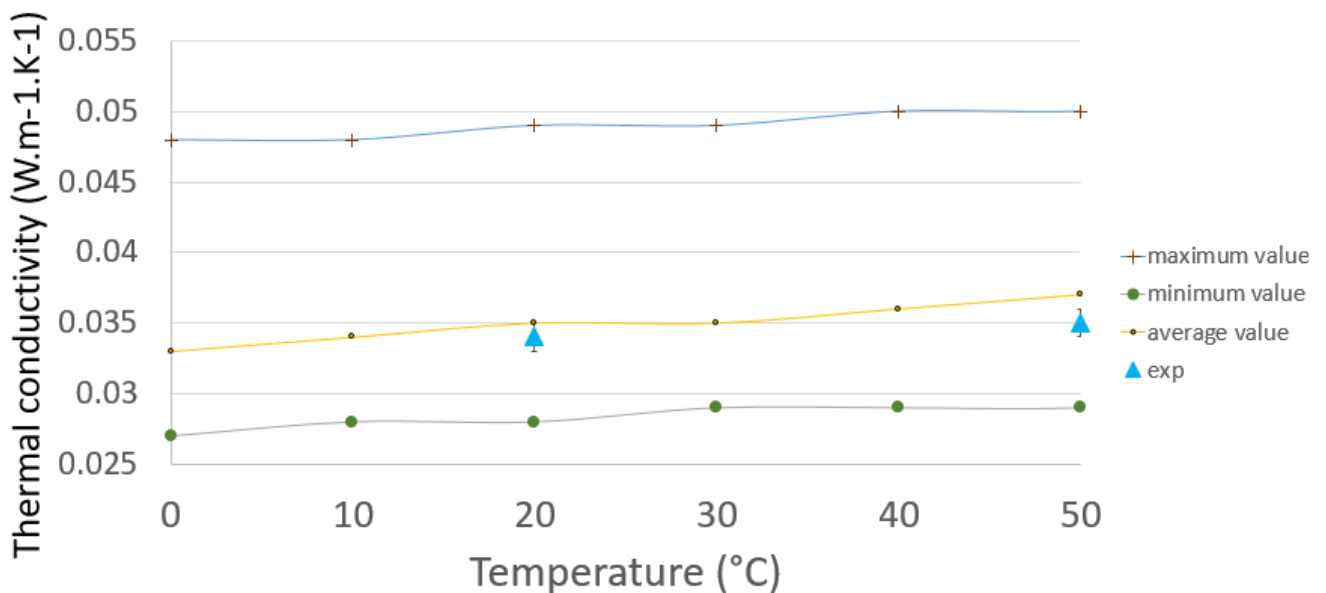


Figure 13. Thermal conductivity versus relative temperature: board of sunflower pith at 50 % HR.

The two experimental points are once again framed by the minimum and maximum modelled values. As in the previous section, the maximum and minimum limits first defined (cf section 2.1.3) could be reduced given the quality of the prediction by the model: experimental results are within 10% of the average modeled value (from the average intra-particulate porosity). The developed model therefore allows to predict within 10% the thermal conductivity of particleboards made of sunflower pith over the entire range of use temperature.

4. Conclusion

In this study, a new process has been proposed for manufacturing 100% bio-based insulating panelboards. It enables sunflower pith particles to be transformed into a self-supporting material, at room temperature and without the use of added binder. The insulating capacity of this bio-based panel was investigated in our study as this low-energy manufacturing process is very promising to reduce the building embodied energy.

The direct measurement of the thermal conductivity underlined the high potential of this sunflower-based product, which is close to conventional insulating products. In addition, a multi-scale model based on three iterative steps was developed to predict the thermal behaviour of the material under different conditions of use. The model takes the effects of temperature and relative humidity on thermal conductivity into account for the eco-material studied. Since the input values of the model had to be relatively accessible without neglecting the relevance of the model, a Mori-Tanaka scheme with isotropic consideration was chosen. Excellent agreement between experimental data and calculated data highlights the adequacy of the spherical geometry considered in the model to represent the microstructure of the sunflower pith. The key novelty of this study is the extremely relevance of the model to predict the thermal conductivity of the panel boards over its entire range of use from a few input data of the aggregates: intra-particulate porosity and sorption curve.

In order to extend the application of this model, the assumption of spherical inclusion - at the microscopic and macroscopic scale - might have to be revised depending on the type of aggregate (unless the spherical shape is sufficient, as suggested by a recent study [59]). Most other aggregates (hemp shiv, flax shiv, sunflower bark, rape straw, etc.) have both micro and macro structures closer to a cylinder than a sphere. It would be interesting to study whether cylindrical inclusions are more appropriate or not. Thanks to the single-particle scale developed here, our model could be transposed and applied to a wide range of plant aggregates. This would encourage the use of locally available plant aggregates, since a simple characterization is sufficient to feed our predictive model. Only sorption curves and particulate porosity are required. The latter is accessible thanks to the easy-to-implement methods recently developed by Ratsimbazafy [31]. The major interest of the model is to propose an efficient and sufficient multi-scale approach without the need for expensive equipment.

Thus, with the iterative approach proposed, it would be possible to take the variability of the raw material into account in terms of its density and/or its particulate porosity. This variability of particulate insulating capacity would impact the material scale through the multi-scale approach. Modelling will then open up new perspectives: the prediction of insulating capacities of materials even though they are made of different aggregates with intrinsic variability due to the plant species, geographical location, harvesting period, or method of production. Finally, future works are foreseen to investigate the properties during use (hygro-thermo-mechanical performances) of the pith-based insulating panels manufactured by the technique presented in this study. It will be necessary to explore more fully the potential of the binderless panels and to overcome any difficulties that may arise: sufficient strength for wall implementation, high sensitivity to humidity and long-term durability.

5. References

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Chapitre 6. Prise en compte de la variété de la ressource

Préambule

Les panneaux de moelle de tournesol, présentés dans le chapitre précédent, ne nécessitent pas de liant : ils peuvent donc être assimilés à du vrac. En faisant l'hypothèse que les granulats ne sont pas déformés par le processus de compactage (ou du moins qu'ils retrouvent leur forme originelle après compaction), ce chapitre propose d'étendre et de développer la méthode basée sur la triple homogénéisation à une plus large gamme de granulats végétaux. Ainsi, à partir des porosités intra particulières mesurées dans les travaux de thèse de H. Ratsimbazafy [22], il est possible de prédire la conductivité thermique en vrac (sous différentes conditions de température et d'humidité) pour une dizaine de co-produits agricoles différents.

Notons que la plupart des granulats végétaux ont une structure tubulaire, à l'image de la chènevotte, et non alvéolaire comme pour la moelle de tournesol. Ainsi, la morphologie particulière est un critère déterminant pour le type de modélisation proposé. L'objectif de cet article est de développer des modèles prédictifs « universels », à la fois pertinents vis-à-vis de la réelle microstructure du granulat considéré et relativement facile à exploiter. Deux modèles sont ainsi proposés, couvrant une large gamme de co-produits agricoles exploitables. Le but est de pouvoir valoriser le granulat disponible localement et selon la saisonnalité des récoltes, à partir d'un minimum de caractérisation.

Cette étude fournit ainsi la conductivité thermique particulière (sous différentes conditions de température et d'humidité relative) pour un grand nombre de granulats végétaux (chènevotte, anas de lin, moelle et écorce de tournesol, tige de miscanthus, sarment de vigne, paille de coriandre, paille et menue paille de blé, balle de maïs). Ceci est une base pour des travaux futurs de modélisation, basés sur de l'homogénéisation analytique ou numérique. Les résultats suggèrent, en outre, la possibilité d'une relation linéaire entre conductivité thermique particulière et masse volumique particulière. Une hypothèse qui pourrait aider à mesurer l'impact des pré-traitements ou encore du rouissage sur les performances thermiques des matériaux bio-sourcés. Enfin, dans un souci de pouvoir livrer des résultats directement exploitables en phase chantier, un tableau d'équivalence de performances thermiques de granulats en vrac est proposé. Il s'appuie sur les résultats de la modélisation. Une comparaison à des mesures expérimentales in situ sera nécessaire pour asseoir la validité du modèle proposé.

Article E. Bio-based building materials: A prediction of insulating properties for a wide range of agricultural by-products

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Highlights

- A particulate thermal conductivity is calculated for a wide range of bio-aggregates
- Multi-scale models are consistent with aggregate morphologies
- An equivalent insulation performance is possible despite the diversity of the resource

Abstract

Sustainable construction is a key solution in the light of today's climate and energy challenges. Indeed, the use of plant-based aggregates in the construction sector helps to improve both energy and environmental efficiency. However, the use of bio-based aggregates as loose-fill insulation remains limited despite the wide availability of raw materials (agricultural by-products or wild plants) and their easy implementation. One of the problems is the diversity of resources that complicates both characterization procedures as well as the understanding and prediction processes of bio-material behaviors. One way to overcome these obstacles is to offer a unified method which is applicable to a wide range of plant-based aggregates. This is the purpose of the following study with the prediction of the thermal conductivity of bulk aggregates. This paper initially focuses on the particle scale in order to identify commonalities across a wide range of aggregates. Based on a multi-scale study, then distinguishes two different types of particulate morphologies. Particulate values of thermal conductivity - unavailable in the literature - can thus be provided over the full range of temperature and relative humidity conditions. A linear relationship between thermal conductivity and density is suggested at the particle scale while this type of relationship was previously known at the material scale. This work will also demonstrate the possibility of anticipating and offering equivalent insulation performances depending on the local resource available for loose-fill insulation cases.

Keywords

Bio-aggregates, Thermal Conductivity, Multiscale Modelling, Intrinsic Variability

1. Introduction

Undoubtedly, one of the levers of the ecological transition is the building sector as it consumes 40% of the world's energy [1], [2]. Thus, Life Cycle Assessment (LCA) of construction materials is becoming an ever greater concern for this sector [3]. Locally available materials are to be developed and used to meet the environmental objectives [4]. Transport represents a non-negligible part of the environmental impact associated with construction projects [5], [6], [7]. In addition, manufacturing and installation techniques have to be as environmentally friendly as possible to optimize the use of bio-based resources. In this sense, using agricultural by-products as loose fill material is an efficient way of insulating buildings [8], [9], [10]. Bio-based materials also contribute to indoor thanks to their passive moisture capacities [11]. According to the FAO [12], global cereal production is approaching 3,000 million tons. This figure underlines the real potential of using agricultural by-products. With production set to increase further on [13], the use of agricultural by-products will become a major issue, so it makes sense to propose its use as an insulating material. Adding value to agricultural by-products is both a new source of income for farmers and a springboard for the local economy [14], [15], [16]. Indeed, many cereal crops generate waste that could be used in bio-sourced building materials. However, there is a lack of data on this raw material in literature. Its variability is an obstacle to its use [17], [18] while diversity and variety of the resource constitute scientific problems [19]. To date, the thermal conductivity of plant aggregates in bulk is neither predictable nor optimizable. Time and energy consumption have to be characterized. All sample need to be stabilized in terms of temperature and humidity. Then, to be representative, the measurement must be repeated several times, according to the Rilem TC236-BBM's recommendation [20]. Finally, the intrinsic variability of the resource, derived from the plant world, poses a significant challenge: how can the same performance be guaranteed from one site to another when the locally available resource varies ?

This article offers a method for predicting the thermal conductivity of bulk plant aggregates at different temperatures and relative humidities. The lock represented by resource variability was removed by analyzing the similarities at the particle scale for a wide range of plant aggregates. These elements are both based on the actual microstructure and on the macrostructure of plant aggregates. The developed methods will include several goals:

- To apply to all lignocellulosic aggregates
- To adopt easily accessible input data
- To consider the wide range of temperatures and relative humidity in use
- To provide particulate data for future modelling work.

The methods have been validated for a dozen plant aggregates. The aim is to promote and even optimize the use of agro-resources to propose bulk insulation processes with locally available aggregates. The point is to focus on a "universal" prediction method for plant aggregates. It is therefore an innovative step in the development of eco-materials, allowing the great variability of the resource to be overcome. This is made possible because the proposed method is based on a new scale, the one of aggregates.

In this study, a semi-experimental protocol is proposed to obtain an estimate of the thermal conductivity of particles for any type of lignocellulosic aggregate. It combines the results of a simple test performed on bulk particles [21] as well as known homogenization results. A

general approach will be presented herebelow, applicable and efficient for a wide variety of plant-aggregates.

First, this article presents the many aggregates selected for the study and focuses on their specificities. Then models are explained, and the input data are clearly defined. They are easily accessible thanks to recent works on plant aggregates [22], [23]. Model values are compared with those of the literature, for which all the input data are already known, and an extrapolation is made on certain aggregates. The relevance of this proposal is discussed. In addition, so as to extend use to other aggregates, a decision tree for choosing the adequate model is presented.

Finally, the ability of our models to find the known linear relationship between thermal conductivity and density of bio-based material [24], [25], [26] is highlighted. Thanks to modelling, the same type of linear relation is demonstrated at a new scale: the particulate one.

This study opens up new perspectives to predict and optimize insulating capacities of loose-fill insulation depending on the by-product locally available. It also provides particulate thermal conductivity values for numerous agricultural co-products which will become useful for numerous homogenization models. To date, only the thermal conductivity of hemp shiv has been either experimentally measured [27] or evaluated by inverse methods [28], [29].

2. Materials

2.1 Variety of aggregates: Criteria selection

By-products were first selected according to their availability in the authors' region as avoiding unnecessary transportation reduces the related environmental impact. Equally important was the diversity of aggregates, to appreciate the generalizability of results. Another fundamental criterion was access to both the data required for modelling and experimental values for the same samples. This made it possible to assess the model's relevance. To evaluate its possible extension, the model was also tested on aggregates for which not all the data were known. Data were taken from the literature concerning two studies. The first one [22] gave both the necessary input data and experimental values of thermal conductivity of hemp shiv, flax shiv, sunflower pith and sunflower bark in bulk (case 1). Partial input data and thermal conductivity of aggregates in bulk were also given for six other aggregates (case 2). All measurements are considered reliable as they were conducted according to the RILEM TC 236-BBM procedure [20] for thermal conductivity or by the Dynamic Vapour Sorption (DVS) device for sorption isotherms [22]. The second study [30] only provided part of the input data for other samples (case 3). Three groups of aggregates were therefore identified. For greater clarity, Table 1 summarizes the aggregates considered and the corresponding data used.

Table 1. Groups of aggregates considered depending on available data

Aggregate	Particularity
CASE 1	
Hemp shiv (HS-1)	All input data are known [21], [22]
Flax shiv (FS-1)	
Sunflower pith (SP-1)	
Sunflower bark (SB-1)	
CASE 2	
Miscanthus stem (MS-2)	Necessary input data are known [22] [21] except for the isotherm sorption curve. (Hypotheses are needed.)
Vine shoot (VS-2)	
Coriander straw (CS-1)	
Wheat straw (WS-2)	
Wheat chaff (WC-2)	
Maize husk (MH-2)	
CASE 3	
Hemp shiv (hs-3)	Intra particle porosity and skeletal density of particle are known [30]. Hypotheses are needed concerning isotherm sorption curves and particulate density
Sunflower pith (sp-3)	

This classification allowed discussion of the necessity of having access to certain input data for multiscale modelling. The following sections focus on the aggregates identified in cases 1 and 2, as experimental values are available to compare with modelled values. The aggregates of case 3 were considered only for further comparison of the modelled results (cf. section 4.1).

2.2 Chemical composition

Agricultural by-products are mainly cellulose-based ones since this biopolymer is the major component of plant cell walls. While the rate of crystallinity observed in cellulose is generally about 80% [31], the other main components of bio-aggregates, hemicellulose and lignin, are rather amorphous polymers [32]. In previous studies, chemical characterizations were carried-out concerning the selected bio-aggregates (of cases 1 and 2) [22]. Primary components (cellulose, hemicellulose, lignin and minerals) were tested but secondary components like pectin, wax, proteins or various extractives were not. Nevertheless, cellulose was assumed to be the only chemical component with crystalline properties. Thus, it was possible to deduce the likely crystalline cellulose content for each aggregate from the cellulose content. Then proportions of crystalline and amorphous polymers in the overall solid skeleton were compared (Table 2).

Table 2. Proportions of the crystalline cellulose and amorphous biopolymers in bio-aggregates (case 1) deduced from [22].

	% cellulose	% crystalline cellulose	% amorphous biopolymers
Hemp shiv (HS-1)	55.5	44.4	55.6
Flax shiv (FS-1)	52.1	41.7	58.3
Sunflower pith (SP-1)	45.5	36.3	63.7
Sunflower bark (SB-1)	53	42.4	57.6
Miscanthus stem (MS-2)	52.8	42.2	57.8
Vine shoot (VS-2)	40	32	68
Coriander straw (CS-2)	50.4	40.3	59.7
Wheat straw (WS-2)	43.1	34.5	65.5
Wheat chaff (WC-2)	35.9	28.7	71.3
Maize husk (MH-2)	38	30.4	69.6

These values were useful to discuss the assumptions made for our model.

2.3 Macroscopic shape

Shape factors can be determined in order to evaluate and compare the macroscopic shape of the plant aggregates [33]. Circularity (C) is a dimensionless number used to appreciate the degree of roundness of aggregates. It takes values between 0 (infinitely elongated particle) to 1 (circle) and is expressed as:

$$C = 4 \pi \frac{A}{P^2} \quad (1)$$

where A (mm²) and P (mm) are the sample's area and perimeter, respectively.

The aspect ratio (R) is defined as the major to minor axis ratio and is always greater than 1. The closer the value is to 1, the more circular or spherical the particle is. The measurements of these two parameters were obtained by image analysis according to the Rilem TC236-BBM's recommendation [34]. To assess the gap between the real macroscopic shape and a sphere for each plant aggregate, a new parameter D (deviation) was suggested as follows:

$$D = |(C - 1) + (R - 1)| \quad (2)$$

C being the circularity and R the aspect ratio.

For a perfect sphere, D is equal to 0. From the Ratsimbazafy's results [22] [21] and considering this new parameter, we can quantify the deviation of each sample from a perfect sphere (Table 3).

Table 3. Deviation of each plant aggregate calculated from the results of Ratsimbazafy [21], [22]

	Circularity C	Aspect Ratio R	Deviation D
Hemp shiv (HS-1)	0.34	3.30	1.64
Flax shiv (FS-1)	0.29	6.93	5.22
Sunflower pith (SP-1)	0.48	1.48	0.04
Sunflower bark (SB-1)	0.27	3.94	2.21
Miscanthus stem (MS-2)	0.28	4.63	2.91
Vine shoot (VS-2)	0.18	4.19	2.37
Coriander straw (CS-2)	0.17	6.67	4.84
Wheat straw (WS-2)	0.21	7.53	5.74
Wheat chaff (WC-2)	0.22	5.82	4.04
Maize husk (MH-2)	0.20	4.95	3.15

These results are consistent with the visual appearance of the different aggregates. Thus, the sunflower pith is objectively the most spherical granulate, while the others have rather a cylindrical or cuboid shape (Figure 1).

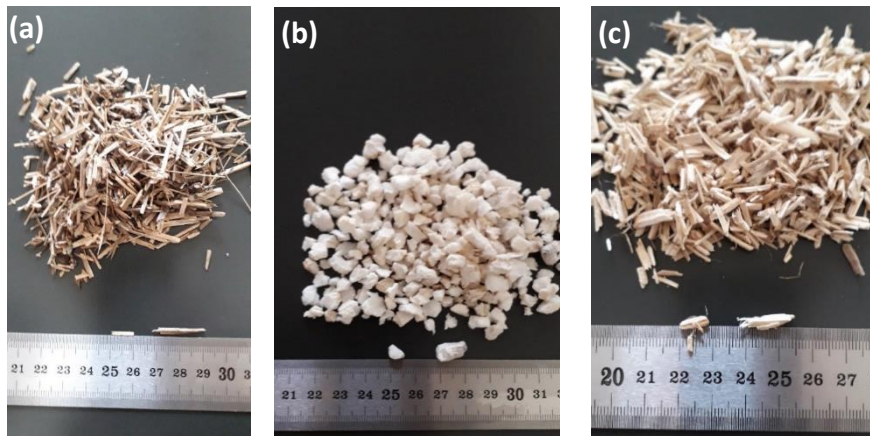


Figure 1. A selection of aggregates whose data whose data characterization was used for this study: Hemp shiv (a), sunflower pith (b) and bark (c)

The macroscopic shape of the aggregates will influence the modelling type to be considered in what follows. Two models will be studied in this article. Their relevance to the considered aggregate will use this new "Deviation from the sphere" criterion.

2.4 Microstructure

SEM observations revealed a porous microstructure for plant aggregates, with a solid skeleton and pores of differing shapes. Skeleton densities measured by Ratsimbazafy [22] and SEM images from the literature gave us a deeper understanding of the microstructure for each aggregate. The porous structure could also be observed and characterized by X-ray tomography images [35]. Table 4 lists the references in the literature that provide an overview of the microstructural properties of the selected aggregates.

Table 4. Microstructural characteristics of plant aggregates

	Skeleton density (kg.m⁻³) from [22]	Pore shape	References
Hemp shiv (HS-1)	1502	tubular	[36], [9], [37], [38],[39]
Flax shiv (FS-1)	1500	tubular	[36]
Sunflower pith (SP-1)	1536	honeycombed	[38], [40], [41]
Miscanthus stem (MS-1)	1509	tubular	[42]
Sunflower bark (SB-1)	1515	tubular	[40]
Vine shoot (VS-2)	1504	/	
Coriander straw (CS-2)	1500	/	
Wheat straw (WS-2)	1519	tubular	[36]
Wheat chaff (WC-2)	1515	tubular	[36]
Maize husk (MH-2)	1523	tubular	[35]

It should be noted that the average skeleton density for the aggregates (containing cellulose, hemicellulose, lignin, and mineral) was 1512 kg.m⁻³, while according to a recent literature review [22], the average density of cellulose in lignocellulosic materials is 1543 kg.m⁻³. The relative gap between these values is only 2%.

The porous structure contributes to the insulating capacity of bio-based composites [33]. Nevertheless, this porosity shows a non-negligible variation [43] for the same type of aggregates and is probably impacted by factors such as the variety of the plant, the place and time of harvesting, and possible pre-treatments. Particulate porosities considered in this study were extracted from [22] and are compared to a few data available in the literature (Table 5) to highlight the spread observed in the values.

Table 5. Particulate porosity of plant aggregates

	Particulate Porosity (%) from [22]	Particulate Porosity (%) from literature	Reference
Hemp shiv (HS-1)	83.5 ± 4.8	83.0-89.3	[43]
		89.3-91.3	[44]
		82.5-82.17	[45]
		61.5	[30]
Flax shiv (FS-1)	71.2 ± 4.4	45.3	[30]
		90.1	[46]
Sunflower pith (SP-1)	96.4 ± 7.8	95.8	[30]
Sunflower bark (SB-1)	71.9 ± 4.1	53.3	[30]
Miscanthus stem (MS-2)	74.4 ± 4.3	/	
Vine shoot (VS-2)	48.2 ± 2.8	/	
Coriander straw (CS-2)	81.7 ± 4.7	/	
Wheat straw (WS-2)	71.3 ± 5.1	/	
Wheat chaff (WC-2)	71.6 ± 5.2	/	
Maize/Corn husk (MH-2)	70.9 ± 5.5	/	

Particle porosity ϕ is calculated as follows:

$$\phi = 100 \cdot \left(1 - \frac{\rho_a}{\rho_s}\right) \quad (3)$$

ρ_a being is the particulate density of the given aggregate ($\text{kg}\cdot\text{m}^{-3}$) and ρ_s the density of its skeleton ($\text{kg}\cdot\text{m}^{-3}$).

It must be noted that, so far, the particulate density measurement has required elaborate equipment, such as mercury intrusion porosimetry (MIP) or X-ray tomography estimation. A new method was proposed in Ratsimbazafy's recent work [22]. It is efficient and easy to implement.

2.5 Adsorbed water

Because of their lignocellulosic nature, plant aggregates are able to absorb excess ambient water and desorb it when the humidity decreases. A hysteresis phenomenon is observable in this process [47]. Strictly speaking, the water content of the plant aggregate is different depending on whether the ambient humidity is increasing or decreasing. However, as only sorption curves are available for the aggregates in case 1 (cf. section 2.1), this phenomenon will not be considered here. Furthermore, for aggregates in case 2, an assumption had to be made since sorption curves were not available. The sorption curves utilized in the study show a highly similar behavior between the different aggregates, except for sunflower pith. In the following sections, if no reference has been found in the literature, it is assumed that the aggregates in case 2 and 3 adsorb water in the same way as hemp shiv. Taking account of adsorbed water in multi-scale modelling requires knowledge of the volume fraction of water as a function of relative humidity, so sorption isotherm data are exploited accordingly.

3. Methods

3.1 Models developed

A triple homogenization process was used from pore scale to loose particles in order to predict thermal conductivity of plant aggregates in bulk. This multiscale study is based on Eshelby's classical theory of [48], using a Mori-Tanaka homogenization scheme [49]. Bulk aggregates are complex heterogeneous materials since the distribution of the phases is not perfectly known. The effective thermal conductivity Λ^{hom} of the equivalent homogeneous material is described as follows:

$$\Lambda^{hom} = \sum_{i=0}^n f_i \Lambda_i \cdot \mathbf{A}_i = \Lambda_0 + \sum_{i=1}^n f_i (\Lambda_i - \Lambda_0) \cdot \mathbf{A}_i \quad (4)$$

Λ_0 being the thermal conductivity tensor of the matrix, f_i the volume fraction of the inclusion i ; \mathbf{A}_i being its second order concentration tensor and Λ_i its thermal conductivity tensor. The only values available in literature for comparison with modelled values are isotropic ones as thermal conductivity of particles in bulk is generally measured with a hot wire or hot plate (cf. section 4.2). Nevertheless, many recent studies highlight anisotropic thermal conductivity for bio-based composites [45], [50], [51]. Consequently, the anisotropy of plant particles is taken into account in one of the two models developed.

It should be pointed out that the chemical composition of the aggregates considered in this study is fairly alike. Most of them consist mainly of cellulose (cf. section 2.2). Their solid skeleton could be summed up since this chemical compound's thermal conductivity is available in the literature [52]. Such an assumption has proved its relevance in recent works on sunflower pith [53]. In addition, various analysis of the microstructure of agricultural co-products studied in the literature, whether by SEM or tomography imaging, reveal two distinct categories (cf Table 4 in section 2.4). Distinctions are made between aggregates with a honeycomb-type pore space and others with elongated and aligned pores. Macroscopically, the first ones are more spherically -shaped whereas the others are more elongated and can be compared to cylinders. Thanks to this analysis of both the common grounds and specific features of plant-based aggregates, two models are offered: the "R" model, where the plant aggregate is considered to be spherical (isotropic case for each step) and the "B" model where the plant particle is cylindrical (anisotropic case at the particle scale). Results from both models were compared to experimental isotropic values to assess their relevance. The overall process is summarized in Figure 2 for each model. For each homogenization step, the matrix and inclusions considered are respectively noted (m) and (i). The equivalent homogeneous medium considered in the k-th step becomes the inclusion of the heterogeneous medium considered in k+1-th homogenization process.

In practical terms, the first homogenization step is carried-out at the pore level to determine the effective thermal conductivity of a pore having adsorbed a certain quantity of ambient water. This pore is then included into the second homogenization step to calculate a particulate dry or wet thermal conductivity. Finally, this particle becomes the inclusion in the third homogenization to gain access to the effective thermal conductivity of bulk aggregates.

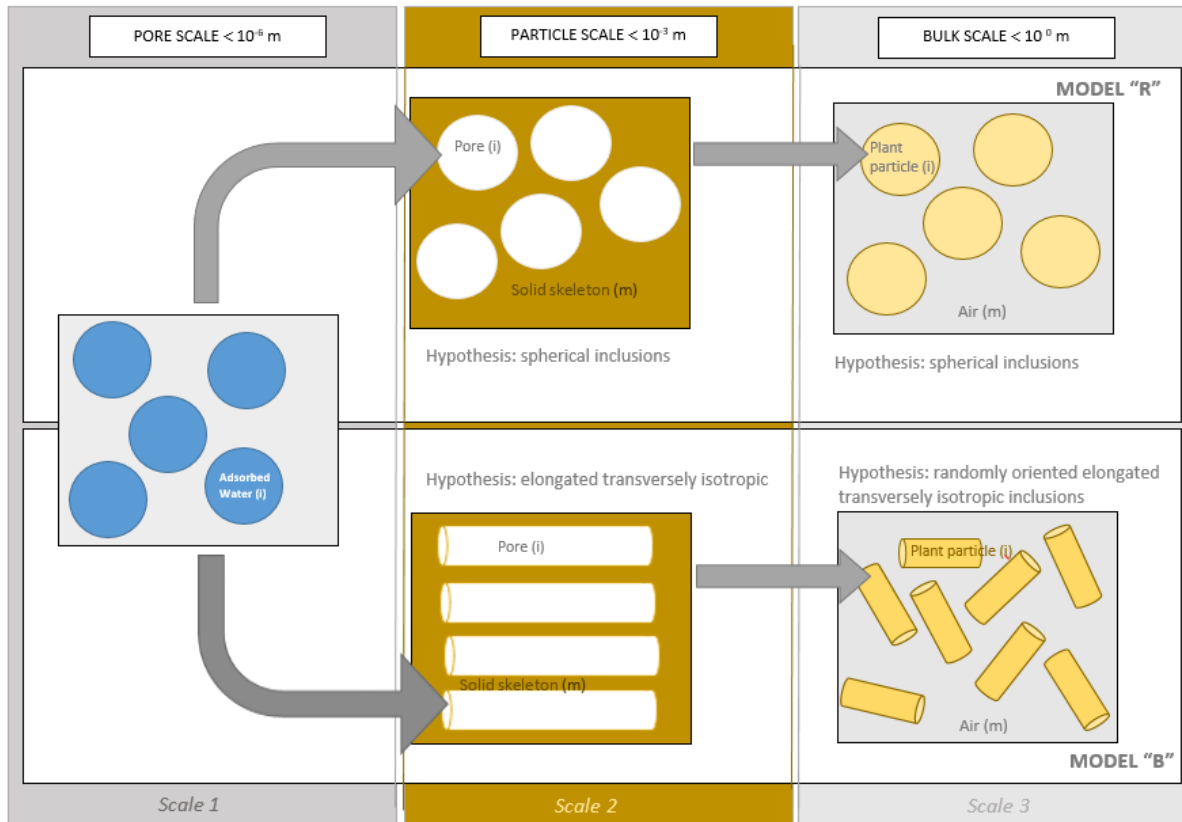


Figure 2. Description of the two models developed in this study

In the "R" model, thermal conductivity remains a scalar throughout the homogenization stage. In the "B" model, thermal conductivity is a tensor at the particle level. The formulae used for each iteration to access the effective thermal conductivity λ_{eff} are listed in Table 6. Input data from heterogeneous materials are needed for the thermal conductivity of the matrix λ_m and inclusions λ_i , and the volume fraction f_i of inclusions. The particle shape and orientation assumptions specified in Figure 2 are also considered. The iterative approach, used for the models developed here, has been validated in prior work to predict thermal conductivity of panel boards made of sunflower pith [53].

Table 6. Formulae used for each homogenization with m index for the matrix and i for the inclusion if not specified.

Iterative step	Formula	Commentary	Reference
“R” MODEL			
Homogenization 1, 2 and 3	$\lambda_{eff} = \lambda_m + \frac{3 f_i \lambda_m (\lambda_i - \lambda_m)}{(1 - f_i) \lambda_i + (2 + f_i) \lambda_i} \quad (5)$	Isotropic consideration: λ_{eff} is a scalar	Rosa Latapie et al. [53]
	where $\lambda_{eff} = \lambda_{pore}$ (step 1) $\lambda_{eff} = \lambda_{particle}$ (step 2) $\lambda_{eff} = \lambda_{pore}$ (step 3)		
“B” MODEL			
Homogenization 1	$\lambda_{pore}^{hom} = \lambda_{air} + \frac{3 f_{water} \lambda_{pore} (\lambda_{water} - \lambda_{air})}{(1 - f_{water}) \lambda_{water} + (2 + f_{water}) \lambda_{water}} \quad (6)$	Isotropic consideration: λ_{eff} is a scalar	Rosa Latapie et al.[53]
Homogenization 2	$\lambda_N = f_{pore} \lambda_{pore} + (1 - f_{pore}) \lambda_{skeleton} \quad (7)$	Anisotropic consideration: Λ is a tensor $\Lambda = \begin{bmatrix} \lambda_N & 0 & 0 \\ 0 & \lambda_T & 0 \\ 0 & 0 & \lambda_T \end{bmatrix}$	Tran-Le et al. [29] Nguyen et al [54]
	$\lambda_T = \left(\frac{f_{pore}}{\lambda_{pore} + \lambda_{skeleton}} + \frac{1 - f_{pore}}{2 \lambda_{skeleton}} \right)^{-1} \quad (8)$		
	The particulate thermal conductivity is calculated with an anisotropy factor of 1.5 according to Tran-Le et al. [42]. The equivalent value is based on normal and tangential values as follows:		
	$\lambda_{particle} = \frac{\lambda_N + 2 \lambda_T}{3} \quad (9)$		
	λ_N and λ_T being respectively the normal and tangential components of the thermal conductivity tensor.		
Homogenization 3	$\varphi \frac{\lambda_{air} - \lambda_{bulk}}{\lambda_{air} + 2 \lambda_{bulk}} + (1 - \varphi) \frac{\lambda_{particle} - \lambda_{bulk}}{\lambda_{particle} + 2 \lambda_{bulk}} = 0 \quad (10)$		Nguyen et al.[54]
	where φ (in %) is the porosity between particles, which is calculated from ρ_{bulk} the bulk density of particles and $\rho_{particle}$ the particulate density kg.m^{-3}) as follows:		
	$\varphi = 1 - \frac{\rho_{bulk}}{\rho_{particle}} \quad (11)$		

When plant aggregates are in loose bulk, it is assumed they are not subjected to sufficient stresses to justify the consideration of any crushing. Model B was therefore discarded for sunflower pith, since model R fulfilled the objective of taking the real microstructure into account. Consequently, the two approaches were systematically explored in order to determine the most relevant one for the aggregate considered, except for sunflower pith.

3.2 Input data

Thermal conductivity of air (λ_{air}) and of water (λ_{water}) were extracted from Tsilingiris' work [55] and Laurent and Guerre-Chaley [56], respectively. Their variation as a function of temperature is known. The other input data used for the models are detailed for each group of aggregates (cf. section 2.1) and for each step of the homogenization process.

i) Homogenization 1

In this first step, the pore of the aggregate - filled with air - is the matrix and the adsorbed water is the inclusion. The sorption isotherms were therefore used to translate the mass of adsorbed water per mass of aggregate into the volume fraction of water per aggregate. The references used are listed in Table 7.

Table 7. Input data used for the first homogenization stage

Aggregate	f_{water} reference	λ_{water} reference	λ_{air} reference
CASE 1			
Hemp shiv (HS-1)	From isotherm sorption curves of each aggregate [22]	Digitized curves to take temperature dependence into account [56]	Digitized curves to take temperature and humidity dependence into account [55]
Flax shiv (FS-1)			
Sunflower pith (SP-1)			
Sunflower bark (SB-1)			
CASE 2			
Miscanthus stem (MS-2)	Hypothesis: identical isotherm sorption curves as for hemp shiv (cf. section 2.5) [22]	Digitized curves to take temperature dependence into account [56]	Digitized curves to take temperature and humidity dependence into account [55]
Vine shoot (VS-2)			
Coriander straw (CS-2)			
Wheat straw (WS-2)			
Wheat chaff (WC-2)			
Maize husk (MH-2)			
CASE 3			
Hemp shiv (hs-3)	Hypothesis: identical isotherm sorption curves of hemp shiv as in [22] (cf. section 2.5)	Digitized curves to take temperature dependence into account [56]	Digitized curves to take temperature and humidity dependence into account [55]
Sunflower pith (sp-3)	Hypothesis: identical isotherm sorption curves as for hemp shiv in [22] (cf. section 2.5)		

ii) Homogenization 2

In this step, the result of the first homogenization process is exploited: the thermal conductivity of the pore is that of the inclusion. The thermal conductivity of the solid skeleton and the volume fraction of the inclusions, i.e. the pores, are also required. The volume fraction (f_i) is logically the intra-particle porosity. From this stage, it is possible to distinguish one aggregate from another although they come from the same type of plant. For the same type of plant aggregate, when the intra particle porosity is different, the particulate thermal conductivity differs. The same comments can apply to skeleton density. However, in order to avoid varying too many parameters at once, an assumption was made concerning the skeleton density of the aggregates considered in this study. The input values used in the second homogenization step are listed in Table 8.

Table 8. Input data used for the second homogenization stage

Aggregate	$\lambda_{skeleton}$	Reference	f_i (%)	Reference
CASE 1				
Hemp shiv (HS-1)	Calculated from cellulose density: 1543 kg.m-3 (cf. section 2.4)	[52].	83.5 ± 4.8	[22]
Flax shiv (FS-1)			71.2 ± 4.4	
Sunflower pith (SP-1)			96.4 ± 7.8	
Sunflower bark (SB-1)			71.9 ± 4.1	
CASE 2				
Miscanthus stem (MS-2)	Calculated from cellulose density: 1543 kg.m-3 (cf. section 2.4)	[52]	74.4 ± 4.3	[22]
Vine shoot (VS-2)			48.2 ± 2.8	
Coriander straw (CS-2)			81.7 ± 4.7	
Wheat straw (WS-2)			71.3 ± 5.1	
Wheat chaff (WC-2)			71.6 ± 5.2	
Maize husk (MH-2)			70.9 ± 5.5	
CASE 3				
Hemp shiv (hs-3)	Calculated from cellulose density: 1543 kg.m-3 (cf. section 2.4)	[52] [30]	61.5	[30]
Sunflower pith (sp-3)			95.8	

iii) Homogenization 3

In this last step, the result of the second homogenization process, the particulate thermal conductivity, dependent on temperature and relative humidity, is the essential input data. The volume fraction f_i of aggregates is also required. It is defined as follows:

$$f_i = \frac{V_{aggregates}}{V_{bulk}} \quad (12)$$

V_{bulk} being the total volume of the aggregates in bulk and $V_{aggregates}$ the one of aggregates.

The volume fraction f_i is not explicitly stated in the literature. The only data currently available is bulk density. In order to compare modelled values with the experimental ones for bulk thermal conductivity, the definition of density was used to modify (11) into:

$$f_g = \frac{\rho_{bulk}}{\rho_{particle}} \quad (13)$$

$\rho_{particule}$ and ρ_{bulk} being respectively the particulate density and the density of aggregate in bulk (kg.m^{-3}). The particulate densities used in the third homogenization step are listed in Table 9.

Table 9. Input data used for homogenization 3

Aggregate	$\rho_{particle}$ (kg.m^{-3})	Reference
CASE 1		
Hemp shiv (HS-1)	248	[22]
Flax shiv (FS-1)	433	
Sunflower pith (SP-1)	56	
Sunflower bark (SB-1)	425	
CASE 2		
Miscanthus stem (MS-2)	387	[22]
Vine shoot (VS-2)	779	
Coriander straw (CS-2)	275	
Wheat straw (WS-2)	436	
Wheat chaff (WC-2)	430	
Maize husk (MH-2)	444	
CASE 3		
Hemp shiv (hs-3)	394	[17]
Sunflower pith (sp-3)	144	

It is worth noting that the density of bulk aggregates ρ_{bulk} in the third homogenization step was chosen to fit the values of the literature in order to be able to compare modelled and experimental values.

4. Results and discussion

4.1 Effective thermal conductivity of a single particle

Particulate thermal conductivity is an intermediate result in the homogenization process. However, it is interesting to give these values because, to date, only particulate thermal conductivity of hemp shiv is available in the literature [24], [27], [28], [57]. Particulate thermal conductivities are listed at 20 °C (68°F), in the dry state and 50 % relative humidity. They correspond to current conditions for measurements (dry state) and of use (under ambient conditions). Results are listed in Table 10 so as to provide an exhaustive list of values that will enable other authors to use these data, which are missing from the literature. The number of data items is large, but this also enables the differences to be appreciated between individual aggregates in dry and wet states. In addition, it enables the effect of anisotropy on particulate thermal conductivity to be evaluated.

Table 10. Particle thermal conductivity for the plant aggregates considered in this study

Plant Aggregates	Model R		Model B						Relative gap between the models (%)	
	λ^R particle (W.m ⁻¹ .K ⁻¹)		λ_N^R particle (W.m ⁻¹ .K ⁻¹)		$\lambda_{T, particle}^R$ (W.m ⁻¹ .K ⁻¹)		λ^B particle (W.m ⁻¹ .K ⁻¹)			
	20°C Dry state	20°C 50 % RH	20°C Dry state	20°C 50 % RH	20°C Dry state	20°C 50% RH	20°C Dry state	20°C 50 % RH	20°C Dry state	20°C 50 % RH
Hemp shiv (HS-1)	0.0905	0.0908	0.1159	0.1162	0.0768	0.0771	0.0989	0.0901	0.7	0.8
Flax shiv (FS-1)	0.1439	0.1442	0.1834	0.1836	0.1211	0.1214	0.1418	0.1421	1.5	1.5
Sunflower pith (SP-1)	0.0391	0.0399	0.0452	0.0460	0.0360	0.0367	0.0391	0.0398	0	0.3
Sunflower bark (SB-1)	0.1408	0.1413	0.1795	0.1800	0.1184	0.1190	0.1388	0.1394	1.4	1.3
Miscanthus stem (MS-2)	0.1296	0.1299	0.1658	0.1660	0.1090	0.1093	0.1279	0.1282	1.3	1.3
Vine shoot (VS-2)	0.2572	0.2574	0.3094	0.3096	0.2225	0.2228	0.2515	0.2517	2.2	2.2
Coriander straw (CS-2)	0.0981	0.0984	0.1258	0.1260	0.0829	0.0832	0.0972	0.0975	0.9	0.9
Wheat straw (WS-2)	0.1435	0.1437	0.1828	0.1830	0.1207	0.1210	0.1414	0.1417	1.5	1.4
Wheat chaff (WC-2)	0.1421	0.1424	0.1812	0.1814	0.1196	0.1198	0.1401	0.1403	1.4	1.5
Maize husk (MH-2)	0.1453	0.1456	0.1850	0.1852	0.1222	0.1225	0.1432	0.1434	1.4	1.5
Hemp shiv (hs-3)	0.0941	0.0944	0.1122	0.1124	0.0832	0.0835	0.0929	0.0931	1.3	1.4
Sunflower bark (sb-3)	0.1005	0.1008	0.1173	0.1174	0.0900	0.9002	0.0991	0.0998	1.4	2.0
Sunflower pith (sp-3)	0.1042	0.1050	0.1414	0.1421	0.0852	0.0859	0.1040	0.1046	0.2	0.4
Flax shiv (fs-3)	0.1515	0.1517	0.1766	0.1767	0.1347	0.1350	0.1487	0.1489	1.8	1.8
Rape straw (rs-3)	0.1335	0.1333	0.1595	0.1596	0.1171	0.1174	0.1312	0.1314	1.7	1.4

First, the consistency of thermal particulate values is assessed by means of several findings:

- Particulate thermal values of hemp shiv are consistent with the literature [27], [28], [29], [57]
- Particulate thermal conductivity increases from dry to ambient conditions, taking account of the adsorption of water, which is much more conductive than air.

- The normal component is systematically higher than the transversal one due to the weaker connection of the solid skeleton in the transversal direction.

These values enable the insulating power of aggregates to be compared at the particle level. The general trend is that sunflower pith has a high insulating potential whatever the model. This is in line with comparative results in the literature [38], [58]. It is worth noting that the high density of the solid skeleton of the sunflower pith in Chabriac et al.'s study [30] (3400 kg.m^{-3} , which is almost twice that of cellulose) may explain the probably abnormally high thermal conductivity value for sunflower pith sp-3. This may be due to a particular sample or to measurement bias. Given the resource variability, it is difficult to settle this question. Straw-type aggregates have a thermal conductivity from 0.9 to $0.15 \text{ W.m}^{-1}\text{K}^{-1}$. Vine shoot is the least promising aggregate in terms of thermal insulation. This is consistent with a relatively low porosity combined with a relatively high solid skeleton density.

The impact of variability is significant for a given aggregate type. According to modelling, with an intra-granular porosity of over 30% and a solid skeleton density of over 60%, sunflower bark SB-1 has a particulate thermal which is 40% higher than sunflower bark sb-3.

The anisotropy of particulate thermal conductivity explains the differences in thermal conductivity, which have been reported and studied for hemp-based composites [38], [45], [51], [59], [60]. Depending on the processing method (compaction or spraying), the preferential orientation of plant aggregates logically induces anisotropy in thermal behavior at the material scale. From the values of the normal and tangential components given here for a wide range of aggregates, it is obvious that composites made from these aggregates will also exhibit anisotropic thermal behavior.

In the dry state, the values of models R and B differ by an average of 1.2% at the particle scale. Under 50% relative humidity, they differ by an average of 1.3%. At this point, one conclusion could be that the difference between the models is not significant. However, scale effects could occur at the bulk scale and the following homogenization step is different depending on the model, so neither model is favoured at this stage of the study.

4.2 Loose-fill insulation mode: comparison between model and experimental values

4.2.1 In dry state

The primary aim of this study was to be able to predict the thermal conductivity of plant aggregates used as loose-fill insulation from a minimum of data. The results of the triple homogenization stage (cf. section 3.1) were compared with those of the experimental study [21] in order to assess the relevance of the models to reach this objective.

Maximum and minimum values for intra-particle porosity were considered to give a range of values for the modelled thermal conductivities of aggregates in bulk. Aggregates were assumed to be in a dry state [22] at the time of thermal conductivity measurement and the density was equal to the "bulk density". No compaction was assumed at the time of measurement.

i) R Model

Thermal conductivities calculated using the R model are presented in Figure 3 for aggregates in bulk (case 1).

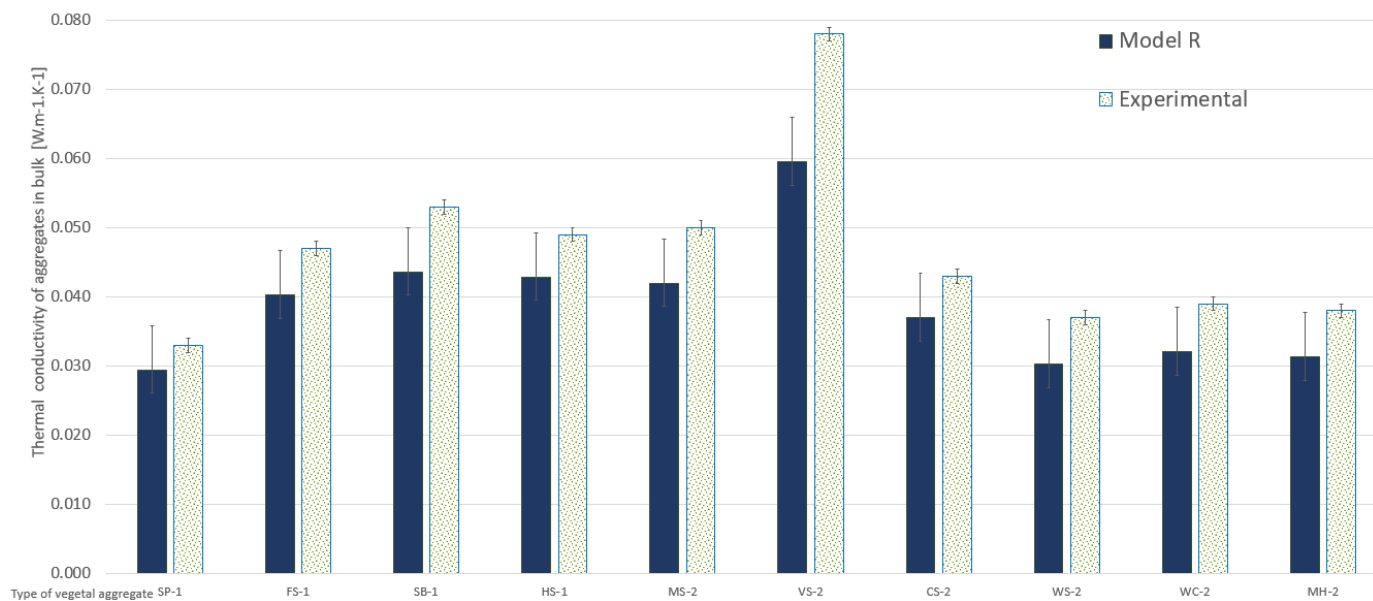


Figure 3. Experimental [21] and R-modelled values for 10 different aggregates (cases 1 and 2) at 20 °C and dry state.

The average difference between experimental and modelled values is 16%. It is worth noting that it is less than 10 % for sunflower pith. Model R, assuming spherical inclusions at each homogenization stage, therefore seems particularly well-suited to this aggregate. Nevertheless, the difference between modelling and experiment is over 20% for vine shoot. Thence, the model is less relevant for this particular plant aggregate. This can probably be explained by a combined effect of a microstructure very different from the one assumed by the above model, as well as a rather low cellulose content compared with other aggregates.

It is also noteworthy to highlight that the model value is systematically lower than the measured value. Several explanations can be put forward to understand and correct the gap:

- Measurements were carried-out on dry plant material at 20 °C, but with a relative humidity of $55 \pm 5\%$ according to the author of the study [22]. Depending on the time of measurement, the aggregates may have adsorbed water. In this condition, particle thermal conductivity and air thermal conductivity are actually higher than those considered due to this humidity.
- The plant aggregates were slightly compacted during the thermal conductivity measurement (in particular to limit the layer of air around the probe). Bulk densities could have been higher than those considered.
- For low densities, the effect of radiation can explain a different behavior from the one considered here (pure conduction) [61].

The R model is easy to use thanks to the accessibility of the input data. It is particularly relevant for sunflower pith since the spherical inclusion assumptions are in line with the real morphology of the aggregate at both microstructural and macroscopic scales. The R model demonstrates its extreme relevance and usability, first for sunflower pith but, more generally, for a wide range of lignocellulosic aggregates. This model is also relevant for a wide range of

lignocellulosic aggregates since it can predict bulk thermal conductivity with a margin of 16% in the dry state.

Finally, a comparison between the values of both the R and B models allow the effect of the anisotropic nature of the raw material to be quantified.

ii) B model

Thermal conductivities calculated in the B model are presented in Figure 4 for aggregates in bulk (case 1).

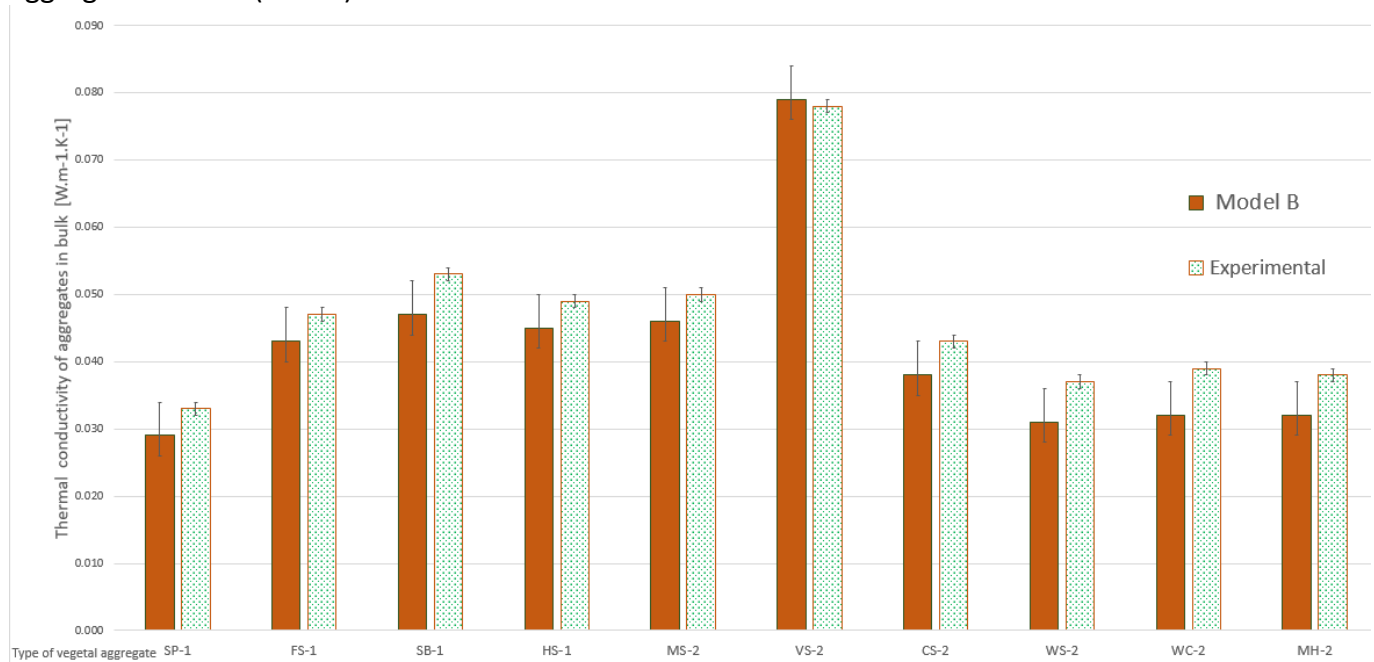


Figure 4. Experimental [21] and B-modelled values for 10 different aggregates (cases 1 and 2), at 20 °C and in dry state.

The average difference between experimental and modelled values is 11%. This model, which takes the anisotropy of thermal conductivity at the particle scale into account, is therefore relevant for all the aggregates considered here. Nevertheless, with a difference of over 12% (compared with less than 10% for model B) between the modelled value and the experimental one, it seems less relevant than model R for sunflower pith. The assumptions made here (cylindrical particle shape) deviate from the real morphology of this particular aggregate. Model B values are systematically lower than measured values (except for the shoot) as in Model R. The same explanations as above may account for this discrepancy. Model B is more complex to implement but the accessibility of the input data enables it to be used quite easily. This model is highly relevant for all lignocellulosic aggregates considered in the dry state, since it can predict bulk thermal conductivity with a margin of 10%.

iii) Comparison of models

In order to further quantify and appreciate the relevance of each model, a comparison was made between the deviation, D, from the sphere of each of the aggregates (cf. section 2.2.3) and the difference between the experimental value and the value given by each model (Figure 5).

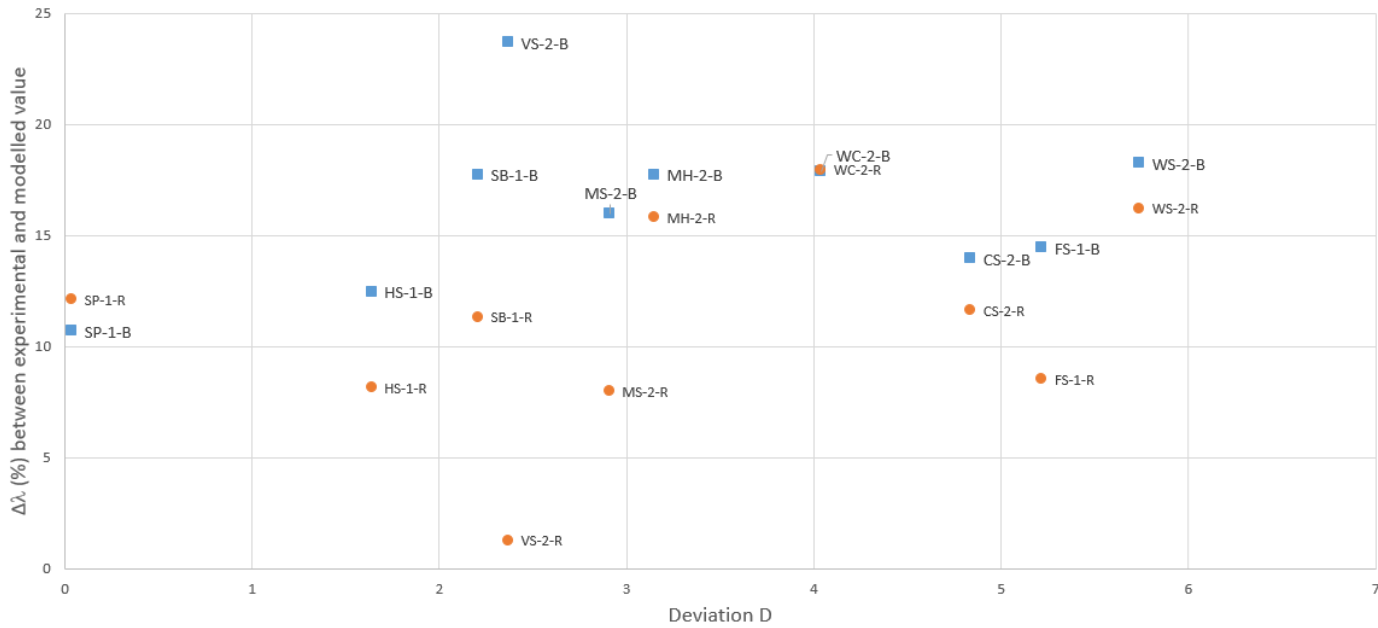


Figure 5. Experimental/model deviation as a function of Deviation D (from the sphere) for each plant aggregate and for each model (R and B).

Figure 5 shows that the difference measured between the experimental and modelled thermal conductivity values is, a priori, correlated with the deviation D defined in section 2.2.3, whereas model B assumes spherical inclusions at each homogenization stage. It can be concluded that the further the plant aggregate deviates geometrically from a sphere (on both microscopic and macroscopic scales), the less relevant the model becomes. Logically, model B remains more relevant for sunflower pith than model R. This is in line with its morphology. Model R is, therefore, to be recommended when considering this plant aggregate. However, the difference between the modelled and experimental values does not systematically increase with deviation D for Model B. This model is therefore simply more relevant for cylindrical aggregates (on both microscopic and macroscopic scales). Model B is to be recommended for aggregates of this kind according to the results of this study.

4.2.2 Extended comparison

For this section, we have selected bulk thermal conductivity measurements from the literature with available measurement conditions (temperature and relative humidity). These experimental values were compared with those given by our models. The input data were those used previously (cf. section 3.2) and those from the study by Chabriac and Al's study. [30] (case 3). Although the plant aggregates were of the same nature, they differed, because of the variability of the resource. Given the limited data available in the literature, only hemp shiv and sunflower pith are considered in this extended comparison of models.

i) Case of hemp shiv

The B model was applied to hemp shiv as it was considered more relevant for this aggregate (cf. Section 4.2.1). A wide range of experimental values of bulk thermal conductivities - under different temperature and relative humidity conditions - were compared with the model values obtained using the hemp-shiv HS-1 input data on the one hand and the hemp shiv hs-3 on the other (Table 11). In the case of HS-1, it is important to recall that the range of model values takes the maximum and minimum intra-particulate

porosity into account (cf. Section 4.2.1). It is worth reiterating that the input data for particle porosity and particle density are different between HS-1 and hs-3 (cf. Section 3.2).

Table 11. Experimental and model values for hemp shiv in bulk under different conditions of temperature and relative humidity.

HEMP SHIV						
λ_{bulk} (W.m ⁻¹ .K ⁻¹)						
Model Values		Experimental Values				
Input data from HS -1	Input data from hs-3	Value	Ref.	Experimental conditions	Measurement method	Details
0.047-0.059	0.057	0.048	[44]	23 °C, dry state	Hot plate	Hemp shiv 0–5 mm $\rho_{\text{bulk}} = 135 \text{ kg.m}^{-3}$
0.042-0.050	0.049	0.048	[44]	23 °C, dry state	Hot plate	Hemp shiv 0–20 mm $\rho_{\text{bulk}} = 110 \text{ kg.m}^{-3}$
0.050-0.064	0.058	0.055	[9]	25 °C, dry state	Hot plate	$\rho_{\text{bulk}} = 153 \text{ kg.m}^{-3}$
0.042-0.051	0.050	0.057 ± 0.006	[63]	20 °C, dry state	Hot wire	$\rho_{\text{bulk}} = 114 \text{ kg.m}^{-3}$
0.041-0.048	0.045	0.053	[36]	23 °C, dry state	Hot wire	$\rho_{\text{bulk}} = 96 \text{ kg.m}^{-3}$
0.043-0.051	0.050	0.072 ± 0.005	[63]	20 °C, 35 % RH	Hot wire	$\rho_{\text{bulk}} = 114 \text{ kg.m}^{-3}$
0.047-0.053	0.057	0.0545	[64]	25 °C, ambient RH (assumption: 60% RH)	Hot plate	$\rho_{\text{bulk}} = 134.8 \text{ kg.m}^{-3}$
0.046-0.057	0.069	0.0542	[64]	25 °C, ambient RH (assumption: 60% RH)	Hot plate	$\rho_{\text{bulk}} = 164.5 \text{ kg.m}^{-3}$

These data show a high degree of variability. The thermal conductivity of bulk hemp shiv varies experimentally from 0.048 to 0.072 $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ for a bulk density from 96 to 153 $\text{kg}\cdot\text{m}^{-3}$. This represents a 50% variation in insulating capacity. The model predictions are in line with this variability, as thermal conductivity is evaluated as increasing from 0.041 to 0.064 $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$, i.e. a variation of 56%, when input data from HS-1 is used. Using input data from hs-3 gives a variability of only 30% in bulk thermal conductivity. This is explained by the fact that the intrinsic variability of hemp shiv is not considered in this case; only the average intra-particulate porosity is considered. These results show the importance of evaluating and considering the range of particulate porosity rather than the average value, which masks the great variability of raw materials.

In addition, it is interesting to compare the average dry state thermal conductivity values given by the B model with the average of the experimental values in the following bar chart (Figure 6).

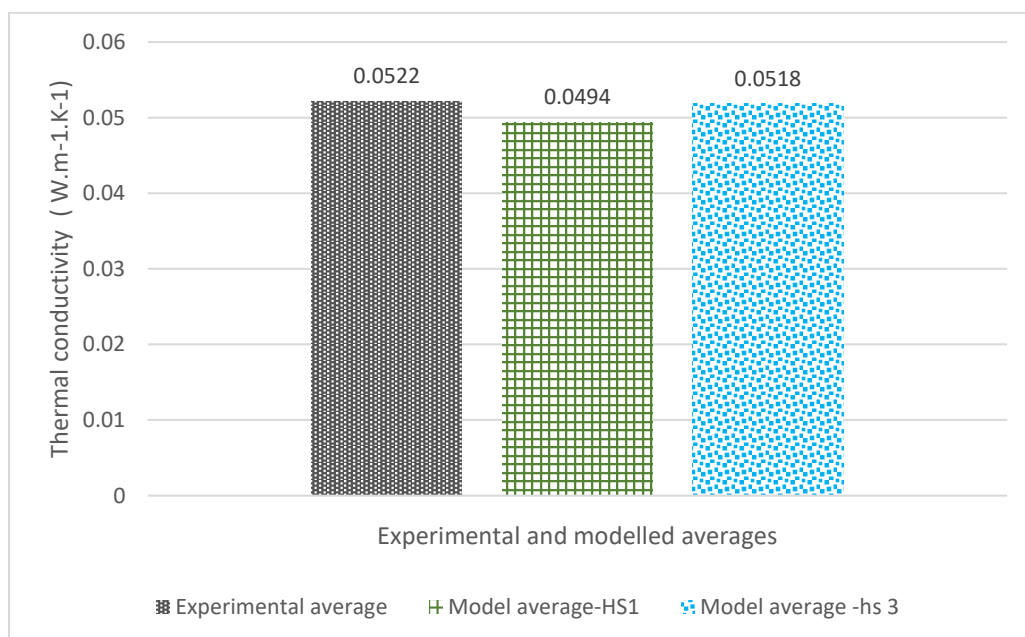


Figure 6. Experimental and modelled averages of thermal conductivity for hemp shiv in bulk in the dry state

When the average of predicted values over the range of bulk densities from 96 to 153 $\text{kg}\cdot\text{m}^{-3}$ is considered, the B model demonstrates its extreme relevance for the dry state. The maximum deviation between the average of modelled values and the average of experimental ones is 5%.

On the other hand, it is more difficult to draw conclusions concerning thermal conductivity prediction under certain relative humidity conditions, given the limited experimental data. Nevertheless, the model values differ from the experimental values by 25% with HS-1 input data and by 12% with hs-3 input data on average. This highlights the significant impact of particle density on the output data. These input data are therefore fundamental.

Our results were compared with those of several studies in the literature and highlighted the need to know the ranges of both the particulate density and the intra-particulate porosity for the aggregates considered when using model B.

Finally, to reflect the material's behavior in a wet atmosphere, the adsorbed water is considered from the pore scale in agreement with the sorption isotherm of the aggregate. This inevitably impacts the results of successive homogenizations. According to our models, bulk thermal conductivity increases on average by 8% between the dry and wet states (60% RH) for hemp shiv in bulk. These averaged figures should be taken with caution. Extreme rigor should imply a measurement on one sample both in dry, it would be necessary to have the measurement on the same sample in dry and wet state humidity owing to the variability of the resource.

ii) Case of sunflower pith

The R model was applied to sunflower pith as it was considered more relevant for this aggregate (cf. section 4.2.1). A range of experimental values for bulk thermal conductivities - under different temperature and relative humidity conditions - were compared with the model values found with the sunflower pith SP-1 input data on the one hand and the sunflower pith sp-3 on the other one (Table 12).

SUNFLOWER PITH						
λ_{bulk} (W.m ⁻¹ .K ⁻¹)						
Model Values		Experimental Values				
Input data from SP -1	Input data from SP-3	Value	Ref.	Experimental conditions	Measurement method	Details
0.028-0.046	0.034	0.051	[64]	23 °C, ambient RH (hypothesis: 60% RH)	Hot plate	$\rho_{\text{bulk}} = 20 \text{ kg.m}^{-3}$ ($\rho_{\text{aggregate}} = 35 \text{ kg.m}^{-3}$)
0.027-0.041	0.033	0.036 ± 0.001	[65]	23 °C, dry state	Hot wire	$\rho_{\text{bulk}} = 15.9 \text{ kg.m}^{-3}$
0.027-0.039	0.032	0.050	[58]	25°C, 60% RH	Hot wire	$\rho_{\text{bulk}} = 14 \text{ kg.m}^{-3}$

Table 12. Experimental and model values for sunflower pith in bulk under different conditions of temperature and relative humidity

As in the case of hemp shiv, these data show a high degree of variability. The thermal conductivity of bulk sunflower pith varies experimentally from 0.036 to 0.051 W.m⁻¹.K⁻¹ for a bulk density from 14 to 20 kg.m⁻³. This represents a variation in insulating capacity of about 40%. The model predictions overestimate this variability, as thermal conductivity is evaluated from 0.027 to 0.046 W.m⁻¹.K⁻¹, i.e. a variation of more than 60 % from when input data from SP-1 is used. Using input data from sp-1 gives a variability of less than 10 % in bulk thermal conductivity. The high porosity of this aggregate (over 95% in the case of SP-1 and sp-3) implies

that variations in bulk density have less impact on bulk thermal conductivity than in the case of hemp shiv (the particle porosity of which is closer to 80%). The proportion of air in this extremely porous material is so high that the impact of conduction in the solid skeleton is very low.

Model values in Table 12 are systematically lower than experimental ones when modelling with sp-3 input data. The difference is almost 25%. With SP-1 input data, the difference is about 22%. Nevertheless, it should be noted that the model values are within the range of the experimental value in one of the cases reported [65]. If the particulate density is corrected by applying that of the sunflower pith studied by Magniont [64] as input data, the model's predictions are improved. As an illustration, using a particle density of 35 kg.m^{-3} and assuming an intra-particle porosity of $90 \pm 5 \%$, the prediction of thermal conductivity is $0.036\text{-}0.051 \text{ W.m}^{-1}.\text{K}^{-1}$ under 60% RH and 23°C , and $0.036\text{-}0.052 \text{ W.m}^{-1}.\text{K}^{-1}$ under 60% RH and at 25°C . This is consistent with the experimental values of 0.051 and $0.050 \text{ W.m}^{-1}.\text{K}^{-1}$ [58], [64]. To make good predictions, the model needs reliable input data on the sunflower pith used for measuring.

From this extensive comparison of our modelled values with experimental values, it can be concluded that the fundamental input data required to ensure the model's relevance are:

- the range of intra particulate porosity.
- the particulate density.

4.4 Thermal conductivity versus density

4.3.1 At the bulk scale

The literature reports a linear relationship between measured thermal conductivity $\lambda_{bulk \text{ exp}}$ and bulk aggregate density ρ_{bulk} [21] as follows:

$$\lambda_{bulk \text{ exp } 1} = 0.0001 \rho_{bulk} + 0.0311 \quad (14)$$

Under the same conditions (dry state, 23°C), Viel [36] found a similar relation according to experimental values from a wide range of bio-aggregates:

$$\lambda_{bulk \text{ exp } 2} = 0.0001 \rho_{bulk} + 0.0398 \quad (15)$$

These correlations are based on experimental results. In order to assess the predictive potential of our models, the thermal conductivity of ten dry bulk aggregates (cases 1 and 2) at 23°C was plotted as a function of bulk density. Acknowledging the findings of the previous sections, Model B was used for sunflower pith and Model R for the other plant aggregates to ensure a coherent approach. The results are presented in Figure 7.

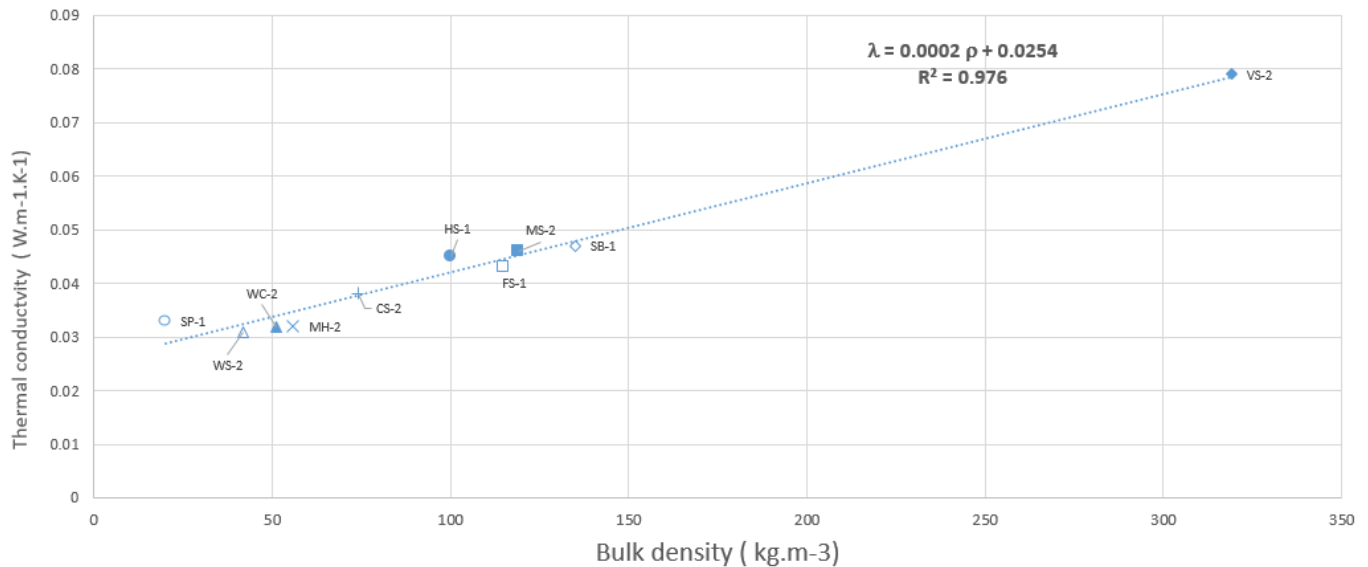


Figure 7. Plant aggregates' thermal conductivity as a function of their bulk density: modelling values at a dry stage, 23 °C

The linear relationship obtained with modelling is in line with that reported in the literature:

$$\lambda_{bulk\ model} = 0.0002 \rho_{bulk} + 0.0254 \quad (16)$$

$\lambda_{bulk\ model}$ (W.m⁻¹.K⁻¹) being the thermal conductivity of bulk aggregates and ρ_{bulk} (kg.m⁻³) the bulk density.

According to the model equations, the y-intercept represents the case where the plant aggregate volume fraction is zero. This is the borderline case where there is only air. From this perspective, the value we obtained by modelling is entirely consistent, since the thermal conductivity of dry air at 23°C (73.4 °F) is 0.026 W.m⁻¹.K⁻¹ [55]. The values of 0.0311 and 0.0398 W.m⁻¹.K⁻¹ - obtained from experimental data – differ by 20% and 50 %, respectively, from this value.

Our model is therefore efficient to predict the thermal conductivity of bulk insulation from bulk density for a wide range of cellulosic aggregates. It can thus anticipate and optimize the thermal conductivity of several aggregates from a loose or compacted implementation.

To progress further, a comparison is made with the relations commonly given for bio-based concretes (bio-aggregates and binder) [24]:

$$\lambda_{bio-based\ composite} = 0.0002 \rho_{composite} + 0.0194 \quad (17)$$

$\lambda_{bio-based\ composite}$ (W.m⁻¹.K⁻¹) being the thermal conductivity of bio-based composites (vegetal concretes) and $\rho_{composite}$ (kg.m⁻³) their density.

The same remark applies to the intercept as described above. In this case, it differs by 30% from the thermal conductivity of air. In addition, discussion is needed on a possible impact of the binder. Comparing relations (16) and (17) may demonstrate that a difference of binder only leads to a translation of the line (same slope).

Finally, it should be pointed out that experimental linear relationships cannot be exploited to predict thermal conductivity over the wide range of conditions of use (temperatures and relative humidity). Given the resource variability and the time necessary for characterizations, modelling is a fundamental and necessary tool for predicting thermal conductivity of bio-based building materials.

4.3.2 At the particle scale

The linear relationship between thermal conductivity and density has been highlighted in the literature for both aggregates in bulk [21] and bio-based composites [66], [67]. In the process of this multi-scale study, identification of a possible linear relationship at the particle scale was attempted as models give access to particulate thermal conductivities (cf. section 4.1). The particulate thermal conductivities as a function of the particulate densities have been plotted in Figure 8.

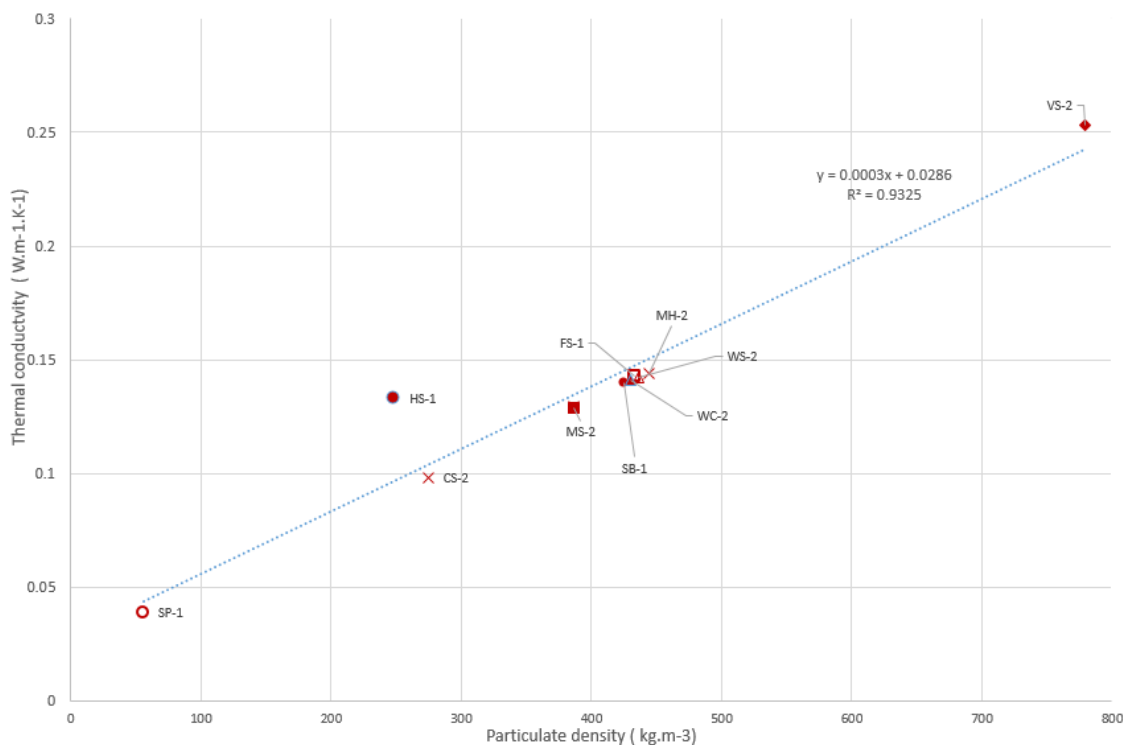


Figure 8. Particulate thermal conductivity as a function of particulate density: modelling values in dry state, 23 °C.

The results demonstrate that the linear relationship between thermal conductivity and density can be written from the particle scale. As in the previous section, the y-intercept corresponds to the value of the thermal conductivity of dry air at 23 °C:

$$\lambda_{particle} = 0.0003 \rho_{particle} + 0.0286 \quad (18)$$

$\lambda_{particle}$ (W.m⁻¹.K⁻¹) being the particulate thermal conductivity and $\rho_{particle}$ (kg.m⁻³) its density.

The denser the particle is, the higher is its thermal conductivity. This is logical given the mode of propagation of heat by conduction. It is clear that this link is linear at the particle scale. Consequently, any variation in the particulate density has a strong impact on the particulate thermal conductivity and therefore on the insulating capacity of the material made of this plant aggregate.

Thanks to these results, it is now possible to quantify the effect of any treatment (chemical or retting) or ageing of the particulate solid skeleton [68] (change in particulate density) on the thermal conductivity of plant aggregates through the effect on particle density. It would also be interesting to study the impact of the plant variety, harvesting period or retting on the physicochemical composition of the agricultural co-product that impacts particulate density. Thus, both prediction and optimization of its insulating capacity could be considered.

4.5 Equivalent walls

It is interesting to compare insulating properties on a particulate scale (cf. section 4.1) or on a bulk scale [69]. However, in order to offer equivalent insulation applications depending on the local availability of raw material, it is helpful to consider thermal resistance. Thanks to the models developed in this study, it is possible to propose thermally equivalent walls depending on the local availability of aggregates. The thermal resistance of $7 \text{ m}^2 \cdot \text{K} / \text{W}$ was chosen as it corresponds to the value recommended for lost attic space in France, where new thermal regulations have just come into force (RE 2020) [70]. Considering this target value for thermal resistance, the thickness of bulk aggregate required was determined (at $23 \text{ }^\circ\text{C}$ and in the dry state). The results are summarized in Table 13.

Plant aggregate	Bulk density considered [$\text{kg} \cdot \text{m}^{-3}$]	Thickness required for loose-fill insulation to obtain $7 \text{ m}^2 \cdot \text{K} / \text{W}$ according to modelling [cm]
HS-1	100	31.5
FS-1	115	30.1
SP-1	20	20.3
SB-1	135	32.9
MS-2	119	32.2
VS-2	319	55.3
CS-2	74	26.6
WS-2	42	21.7
WC-2	56	22.4
MH-2	51	22.4

Table 13. Thickness to be implemented for each type of aggregate to obtain a thermal resistance of $7 \text{ m}^2 \cdot \text{K} / \text{W}$ according to modelling results.

These values are consistent with a lost attic space implementation. Only the relatively dense vine shoot seems unsuitable for this type of insulation, given the thickness (and therefore the corresponding load) required to obtain good insulating capacity.

It should be recalled that these values are derived exclusively from the following input data: aggregate particle porosity and particle density. For simplification, the bulk material has been considered unpacked (bulk density), but the model also allows particle compaction to be considered. Once the basic characterization has been carried-out, several thermally

equivalent solutions can be supplied using different aggregates depending on their availability.

Finally, to take the discrepancy highlighted between the experimental and modelled values for model R (used for sunflower pith) and model B (used for the other aggregates) into account, increasing the thicknesses in Table 13 by 10 % may be recommended.

For clarification and to summarize the procedure that led to the proposal of equivalent constructive solutions from different plant aggregates, a diagram is provided in Figure 9.

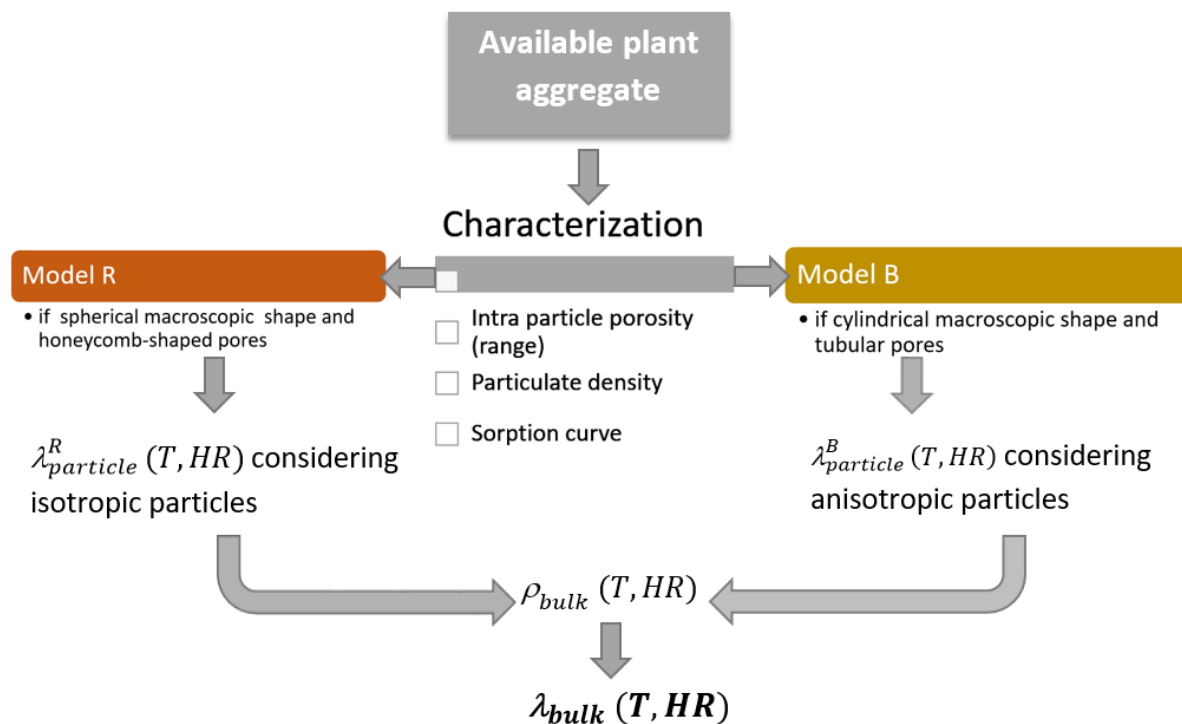


Figure 9. Summary diagram of the proposed approach for predicting the thermal conductivity of bulk aggregates based on a basic characterization of the aggregate under consideration.

5. Conclusion

In this work, a multiscale study has been developed that enables issues from the pore scale to the bulk material scale to be addressed for plant aggregates. The consideration of the original scale of a single particle of plant aggregate extends the scope of modelling of the insulating capacity of bio-based building materials. A wide range of plant granules has been considered (hemp shiv, flax shiv, sunflower pith and bark, miscanthus stem, coriander straw, etc.). Intra particulate porosity, particulate density and sorption curve of one cellulosic aggregate were exploited to estimate the thermal particulate conductivities of a single particle. Previously, only the value for hemp shiv was available and the ambient conditions were not systematically specified. Given the lack of data available in the literature, this study provides valuable data that can be widely used in other models of thermal conductivity of bio-composites for the entire range of temperatures and relative humidity encountered in use.

Based on particle thermal conductivity and bulk density (loose or compacted), a final homogenization step predicts bulk thermal conductivity. These bulk particles can be used to insulate lost attic spaces or intermediate floors with loose filling. Two models have been developed and discussed to predict the thermal conductivity of loose-fill insulation with lignocellulosic aggregates. They take both the microstructure and macrostructure of plant aggregates into account to best fit the available raw material. Their relevance is clear since the difference between the modelled and experimental values is only 10% in the dry state. Given the lack of available data in the literature, comparison under several relative humidities is rather difficult for the moment. Other experimental works, concerning several aggregates and different relative humidities, may complete this study and support the proposed approach.

Nevertheless, the assumptions and methods used in this study apply to a large range of plant aggregates. The models developed are applicable provided that basic characterizations have been made. It is worth noting that particulate porosity is accessible thanks to the easy-to-implement, inexpensive methods recently developed by Ratsimbazafy [22]. The major interest of our models is to propose an efficient and sufficient multi-scale approach without the need for expensive equipment. Thus, the work aims to encourage the use of locally available plant aggregates since a simple characterization is sufficient to feed the predictive model.

In addition, the modelling results presented here have led to a new theoretical justification of the well-known linear relation between the thermal conductivity and the density of a given material. The role of the binder in bio-based composites was raised and discussed from a new perspective. Lastly, it is suggested that the linear relation is also true from the particle scale. As a result, we can now quantify the impact of changes in the density of the solid particulate skeleton (treatment, ageing, etc.) on the insulating capacity of the plant particle.

This iterative approach provides the opportunity to integrate the effect of variability of the raw material on its insulating capacity. Such a variability consideration of particulate insulating capacity (depending on ambient conditions) is studied both at microscopic and macroscopic scale through the multi-scale modelling approach. It opens up new perspectives for the prediction of insulating capacities of materials even though they are made of different aggregates with intrinsic variability (due to plant species, geographical location, harvesting period, type of crops, harvesting techniques or production methods). To summarize, this study provides an answer to the problem of taking the variability of the primary resource into account. A simple characterization of aggregates provides the input data necessary for the successive homogenization presented here. The analytical relationships make the model easier to apply and should allow for wider applications. It is now possible to predict the same performance from one site to another even when the locally available raw material is different.

Finally, the particle thermal conductivity data provided in this article could serve as a basis for numerous other works. It is now possible to quantify the impact of a modification of the microstructure (intra-particle porosity) on the insulating capacity of bio-materials. As soon as the thermal conductivity of chemical components other than cellulose is made available, it is also possible to estimate how a modification of the particulate solid skeleton (density and/or chemical composition) impacts the thermal properties. Another area of work could be to predict the thermal conductivity at the material scale, such as vegetal concrete or lightened

earth by mean-field homogenization. This method has demonstrated its relevance in this study. Moreover, it offers a financial advantage, both in terms of material and time consuming costs.

This work therefore opens up numerous perspectives concerning modelling thermal behavior of bio-based building materials. It makes it possible to consider other aggregates than hemp shiv, which is widely studied but hardly available in many territories.

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Chapitre 7. Vers une optimisation des formulations

7.1 Prédire le comportement thermique face au climat futur

L'utilisation de l'isolation en vrac, traitée dans le chapitre précédent, est une solution low-tech car elle ne nécessite pas de process industriel lourd. De plus, de nombreux co-produits agricoles sont valorisables par cette technique, ce qui peut faire émerger des filières locales et amener les acteurs territoriaux à se saisir de cette opportunité. Sur le plan économique et environnemental, c'est une bonne alternative aux isolants conventionnels. Toutefois, face au changement climatique annoncé et d'ores et déjà ressenti dans beaucoup de territoires, il est légitime de se poser la question de la pertinence de ce mode d'isolation face aux vagues de chaleur. Ces dernières seront vraisemblablement de plus en plus fréquentes et intenses dans le futur. Ainsi, ce premier article se positionne sous les conditions de la canicule subie en France métropolitaine en 2003 : des températures dépassant ponctuellement 45 °C et une humidité relative relativement faible (de moins de 30 %). Les phénomènes de sorption d'eau peuvent ainsi être négligés. Seule la variation de la conductivité thermique en fonction de la température est prise en compte au niveau particulaire (air situé dans l'espace poral intra particulaire) puis à l'échelle des particules en vrac (air situé dans l'espace inter particulaire). L'impact de la température sur le squelette solide de la particule végétale est également considéré.

L'étude se concentre sur la chènevotte en vrac. C'est le seul granulat végétal pour lequel des données de conductivité thermique en vrac, sous conditions précisées de température et d'humidité relative, sont disponibles dans la littérature. Ces dernières servent à apprécier la cohérence des résultats de l'homogénéisation proposée à l'échelle particulaire puis à l'échelle du matériau en vrac. Grâce à l'étude multi-échelle menée, l'évolution de la conductivité thermique du vrac en fonction de la température serait de l'ordre de 10% entre 0°C et 50 °C. Cela peut questionner la pertinence de la caractérisation de la conductivité thermique des matériaux bio-sourcés, généralement conduite à 23°C.

Enfin, il s'agit de discuter des critères de confort d'été vis-à-vis d'une isolation en vrac. La diffusivité et l'effusivité sont les grandeurs physiques qui permettent d'apprécier l'inertie du matériau de construction. Ainsi, leur évolution en fonction de la température est évaluée sur la base de celle de la conductivité thermique. Il s'avère que l'isolation en vrac, par de la chènevotte, est probablement plus pertinente pour une isolation par l'intérieur, améliorant le confort face à une vague de chaleur. En ce qui concerne l'isolation par l'extérieur, le confort d'été peut être amélioré par un matériau à faible diffusivité. Le recours à une matrice liante, en terre crue ou minérale, peut être une solution pour augmenter la masse volumique du matériau et diminuer la diffusivité qui reste relativement élevée pour de l'isolation en vrac.

Article F. Etude multi-échelle des transferts thermiques d'une paroi biosourcée : effet d'un épisode de fortes chaleurs sur la conductivité thermique.

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Résumé. Le secteur du bâtiment doit aujourd'hui opérer une transition efficace et pérenne sur le plan environnemental tout en assurant un confort d'été du bâti. Les matériaux biosourcés peuvent répondre à ces enjeux mais leur caractérisation s'avère relativement chronophage. Dans ce contexte, nous proposons une nouvelle méthode de modélisation afin d'appréhender la variabilité de la conductivité thermique de ces matériaux vis-à-vis de la température. Celle-ci prend en compte la nature du squelette ainsi que la porosité de la particule végétale. Il s'agit donc de se positionner à une nouvelle échelle : celle du granulat. Une approche d'homogénéisation basée sur l'estimation de Mori-Tanaka, différente du schéma auto-cohérent couramment utilisé dans la littérature pour ces matériaux, nous permet de quantifier l'impact de la température sur la conductivité thermique particulaire. Une nouvelle homogénéisation est ensuite réalisée pour appréhender les effets de la température à l'échelle du matériau. Enfin cette étude a permis d'appréhender la pertinence des granulats végétaux, mis en œuvre en tant qu'isolant en vrac, vis-à-vis du confort d'été. Nos résultats mettent en avant l'intérêt de prendre en compte la température sur les performances thermiques des parois biosourcées. Ils pourront être exploités pour prédire le comportement d'une large gamme de bétons végétaux.

Mots-clefs Multi-échelle, biosourcé, thermique, modélisation, confort d'été

1. Introduction

Les co-produits agricoles utilisés en tant que granulats dans les matériaux biosourcés ont la particularité de présenter une structure poreuse importante. En effet, de nombreux capillaires permettent d'assurer la circulation de la sève dans la tige de la plante, pendant sa phase de croissance. Après récolte et séchage, le granulat issu de la tige (moelle et/ou écorce selon les cas) présente alors un réseau capillaire rempli d'air. Cette porosité particulière s'étend de près de 50% à plus de 95% selon le type de granulat considéré (Ratsimbazafy, 2022). Cette porosité ouverte, que l'on retrouve à l'échelle matériau, est le siège de transferts de masse d'eau lorsque l'humidité relative ambiante varie. Ce phénomène est à l'origine d'une augmentation de la conductivité thermique des bio-composites avec l'humidité relative, puisque l'eau est un meilleur conducteur thermique que l'air (Samri, 2008) (Gourlay and Arnaud, 2010). Or, l'air présente également une variation significative de sa conductivité thermique en fonction de la température et de l'humidité relative (Tsilingiris, 2008). De plus, le squelette solide particulière de ces agrégats végétaux est composé principalement de cellulose (Ratsimbazafy, 2022) dont la conductivité thermique dépend également de la température (Antlauf et al., 2021). Cette étude propose donc dans un premier temps de modéliser les effets de la température sur la conductivité thermique d'une particule végétale seule, puis dans un second temps à l'échelle macroscopique d'un agglomérat de particules (vrac) en considérant ces deux aspects.

Néanmoins, une première difficulté résidait dans le choix du modèle permettant d'appréhender l'impact de la température à l'échelle de la conductivité thermique particulière. En effet, il est important de souligner que les méthodes de mesure actuelles ne permettent pas d'avoir accès à la valeur de conductivité thermique particulière. Les valeurs que l'on trouve dans la littérature proviennent de méthodes inverses ou numériques (Cérézo, 2005) (Tran-Le et al., 2019). Enfin, la caractérisation thermique des granulats végétaux est en général réalisée sous conditions normées (23°C, 50%HR) ce qui limite la disponibilité des données sous d'autres conditions. Afin de lever ces différents verrous scientifiques, cette étude propose une nouvelle méthode de détermination de la conductivité thermique en se basant à une échelle microstructurale, celle de la particule végétale. Par ailleurs, le caractère hygroscopique des granulats végétaux nécessite en toute rigueur de considérer une phase d'eau adsorbée dès lors que l'on se place à une humidité relative non nulle. Nous avons pourtant choisi de considérer des particules sèches dans cette étude. Ceci présuppose deux cas de figure : soit les particules végétales sont placées dans un air ambiant à 0% d'HR (cas idéal non représentatif des conditions d'usage), soit la cinétique de sorption (Reuge et al., 2021) implique que l'état d'équilibre n'est pas atteint et que les molécules d'eau présentes dans l'air ambiant n'ont pas encore été fixées par le squelette solide. Cette simplification a du sens dans la mesure où l'étude s'intéresse ici à la conductivité thermique d'une paroi biosourcée sous des sollicitations caractéristiques d'une forte période de chaleur. L'hypothèse faite est également soutenue par un dernier élément. En effet, les relevés météorologiques de la canicule de 2003 en France métropolitaine ont montré des taux d'humidité relative de moins de 30% (Bessemoulin et al., 2004) (Cantat, 2005) (Wahl et al., 2005) . Or, les phénomènes de sorption au niveau des granulats végétaux ont lieu à des humidités relatives moyennes (Lenormand et al., 2014) et les quantités d'eau adsorbée diminuent lorsque la

température croît (Maalouf et al., 2015). Seule la variation d'humidité relative au niveau de l'air a donc été considérée ici.

Dans un premier temps, cet article expose les raisons qui ont conduit à considérer le granulat de chanvre d'une part et des modèles d'homogénéisation issus de la théorie d'Eshelby d'autre part. Ainsi une nouvelle méthode de détermination de la conductivité thermique d'une paroi biosourcée est présentée : il s'agit d'un modèle numérique qui s'appuie sur des données intrinsèques de la particule végétale. Ensuite, nous présentons les résultats récents de la littérature qui ont permis de proposer, via notre modèle, une projection de la variabilité de la conductivité thermique d'une paroi biosourcée en fonction de la température. Enfin, les résultats de cette étude multi-échelle sont discutés afin d'étudier la pertinence de prendre en compte l'impact de la température sur la conductivité thermique de ce type de paroi, notamment lors d'un épisode de fortes chaleurs. A ce titre, les conditions particulières de forte chaleur et d'humidité relative faible sont considérées ici, correspondant à celles de la canicule de 2003 en Europe.

2. Matériaux et méthodes

2.1 Choix du granulat étudié

Le granulat végétal choisi dans cette étude est la chènevotte. En effet, c'est le seul granulat pour lequel des valeurs de conductivité thermique particulière sont disponibles dans la littérature (Céréo, 2005) (Pham et al., 2012) (Dartois et al., 2017) et permettent de vérifier la cohérence de nos valeurs obtenues par homogénéisation. De plus, le modèle proposé nécessite des données d'entrées qui sont disponibles pour la chènevotte dans les travaux récents de (Ratsimbazafy, 2022). Il s'agit de la porosité particulière et de la teneur en cellulose de son squelette solide.

2.2 Choix du VER

Le schéma d'homogénéisation utilisé suppose de définir au préalable un Volume Élémentaire Représentatif pertinent. Ainsi, pour respecter le principe fondamental de « séparation d'échelle » et se positionner à une échelle suffisamment grande devant les hétérogénéités microstructurales, nous nous sommes placés à l'échelle d'une particule de chènevotte (dimensions de l'ordre du centimètre) puis à l'échelle du vrac (dimensions de l'ordre du décimètre). L'étude a été menée en considérant un VER bi-phasique, sans considérer l'eau adsorbée par le granulat végétal. La matrice (phase m) est constituée par le squelette solide de la particule végétale, les inclusions (pores, phase i) sont supposées sphériques (Jeancolas, 2018). Dans le cas du vrac, la matrice du milieu hétérogène est représentée par l'air inter-particulaire (phase m), les granulats végétaux constituent les inclusions (phase i) également supposées sphériques. L'anisotropie due à une orientation préférentielle des particules dans le vrac n'est pas étudiée ici.

2.3 Choix du schéma d'homogénéisation

Le matériau hétérogène considéré, dans les deux cas de figure, est donc un composite de type « matrice + inclusions ». Or, la porosité des particules de chènevotte étudiée est de plus de 80 % (Ratsimbazafy, 2022) et le volume occupé par les granulats de chènevotte en vrac

représente plus de 40% du volume total. Ainsi, la fraction volumique des inclusions, pour les deux VER considérés dans l'étude, est trop importante pour utiliser un schéma dilué (Jeancolas, 2018). Nous avons donc opté pour le schéma de (Mori and Tanaka, 1973) pour tenir compte de l'effet des inclusions entre elles. Ces dernières sont considérées sphériques et de même taille. Ainsi, selon la théorie classique d'Eshelby, le tenseur de conductivité thermique homogénéisé Λ_{hom} peut être ramené sous forme d'un scalaire comme suit :

$$\Lambda_{\text{hom}} = \lambda_{\text{hom}} \mathbf{I} \quad (1)$$

Où \mathbf{I} est le tenseur unitaire d'ordre 2 et λ_{hom} la conductivité thermique homogénéisée en $\text{W.m}^{-1}.\text{K}^{-1}$.

Avec :

$$\lambda_{\text{hom}} = \lambda_m + \frac{3f_i(\lambda_i - \lambda_m)\lambda_m}{(1 - f_i)\lambda_i + \lambda_m(2 + f_i)} \quad (2)$$

Où f_i est la fraction volumique des inclusions.

Les conductivités thermiques de la matrice et des inclusions sont respectivement notées λ_m et λ_i et s'expriment en $\text{W.m}^{-1}.\text{K}^{-1}$. A l'échelle de la particule, le squelette solide et les pores représentent respectivement la matrice et les inclusions, f_i est égale à la porosité particulaire. A l'échelle des granulats en vrac, l'air et les granulats végétaux représentent respectivement la matrice et les inclusions, f_i est le rapport entre le volume de chènevotte et le volume total du vrac.

Enfin, la valeur de conductivité thermique déterminée pour une particule de chènevotte à sec et sous 20°C a été prise comme valeur de référence et une extrapolation a été faite pour sa valeur sous d'autres conditions de température et d'humidité relative.

2.4 Nouvelle méthode de détermination de la conductivité thermique

La conductivité thermique λ_m de la matrice solide particulaire est difficile à appréhender. Seuls des travaux récents sur la cellulose (Antlauf et al., 2021) permettent d'évaluer la conductivité thermique de cette macromolécule et il est intéressant de noter qu'elle dépend de la température. En revanche, aucune donnée de la littérature n'est disponible sur la conductivité thermique de l'hémicellulose et la lignine, molécules également constitutives de la chènevotte. Sachant que la cellulose est le constituant majoritaire de la chènevotte (Mazhoud, 2017) (Ratsimbazafy, 2022), mais également le seul qui présente une structure cristalline, il est légitime de considérer qu'il participe majoritairement au phénomène de conduction thermique (Schick, 2011). Ainsi, nous avons fait l'hypothèse d'un squelette solide constitué exclusivement de cellulose pour déterminer la conductivité thermique λ_m de la matrice particulaire (squelette solide). Les valeurs obtenues sont de l'ordre de grandeur de celle estimée par méthode inverse par (Nguyen et al., 2016) ce qui nous permet de valider la cohérence de nos résultats pour la valeur de conductivité thermique du squelette solide (matrice). Ensuite, une validation a été faite à l'échelle de la particule végétale puis du vrac. Enfin, pour bien expliquer la méthode utilisée, le principe de la double homogénéisation réalisée est illustré dans la figure 1.

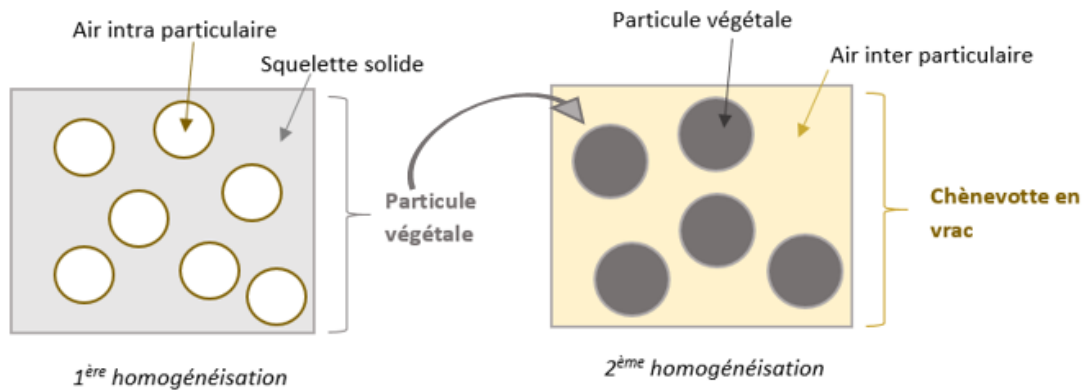


Figure 1. Principe de la méthode utilisée : de l'échelle particulaire à celle de particules en vrac

2.5 Impact de la température sur la conductivité thermique du squelette solide

En considérant la variation de la conductivité thermique de la cellulose en fonction de la température, nous obtenons la figure 2. Ainsi, la conductivité thermique du squelette solide augmente d'environ 5% de 0°C à 50°C selon une relation linéaire. Cette dernière a été intégrée dans la relation (2) pour étudier l'impact de l'élévation de la température ambiante sur la conductivité thermique particulaire.

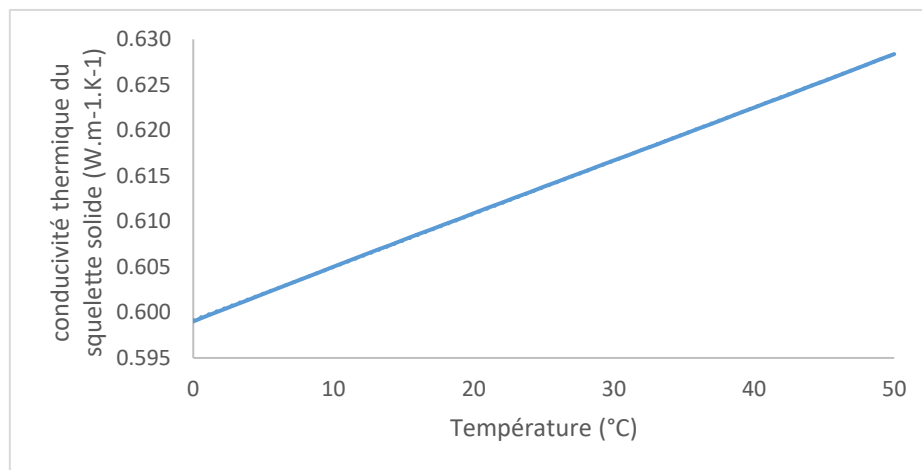


Figure 2. Evolution de la conductivité thermique de la matrice particulaire en fonction de la température d'après la relation polynomiale donnée par (Antlauf et al., 2021)

2.6 Impact de la température sur la conductivité thermique de l'air

Dans la littérature, la valeur de 0,026 W.m⁻¹.K⁻¹ à 20 °C pour la conductivité thermique de l'air est couramment utilisée (Cérézo, 2005) (Ducoulombier, 2014). Cependant, les travaux de (Tsilingiris, 2008) montrent que cette valeur varie de manière non négligeable en fonction des conditions de température et d'humidité relative de l'air. Pour s'approcher au maximum des conditions réelles, nous avons tenu compte des relevés effectués pendant la canicule de 2003. Ainsi, nous avons considéré des valeurs comprises entre 0 et 30 % d'humidité relative pour l'air ambiant. La dépendance de la conductivité thermique de l'air en fonction de la température et de l'humidité relative (Fig 3) a été obtenue à partir de la numérisation des résultats de (Tsilingiris, 2008) puis par régression polynomiale pour pouvoir être intégrés à la relation (2). Ainsi la conductivité thermique de l'air augmente de près de 15% entre 0°C et 50°C sur la gamme d'humidité relative considérée dans l'étude.

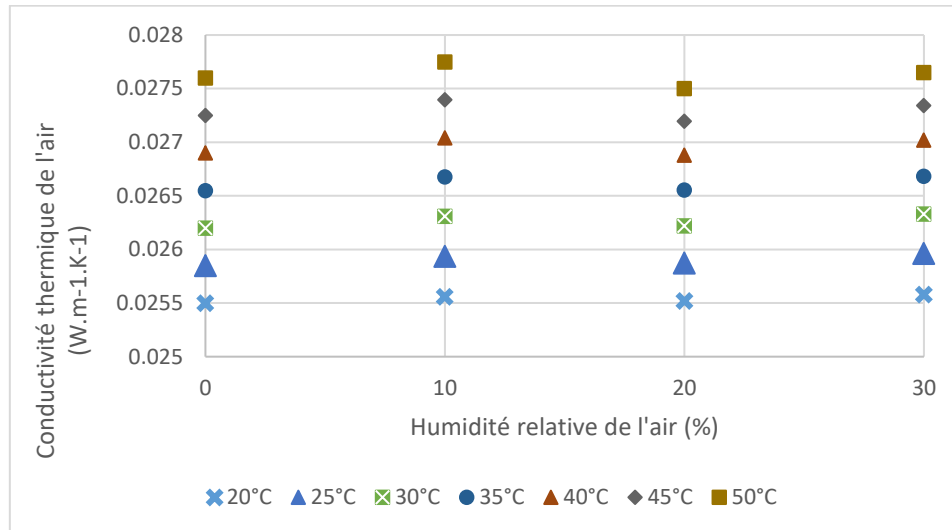


Figure 3. Evolution de la conductivité thermique de l'air en fonction de l'humidité relative et pour différentes températures d'après le traitement des données de (Tsilingiris, 2008)

Dans la mesure où la conductivité thermique dépend de la température, les grandeurs physiques qui sont calculées à partir de cette valeur sont logiquement impactées. Ainsi, pour étudier le confort d'été, mis en avant notamment dans la RE 2020, entrée en vigueur au 1^{er} Janvier 2022 pour les maisons individuelles et logements collectifs (Pestre, 2021), il est important de considérer la capacité du bâti à maintenir une température intérieure agréable malgré une température extérieure élevée. Pour cela, l'inertie thermique des parois doit permettre de déphaser et d'amortir les sollicitations extérieures. On distingue l'inertie de transmission -diffusivité a - qui caractérise la vitesse à laquelle la chaleur se propage par conduction dans le matériau (équation 4). D'autre part, l'inertie d'adsorption -effusivité b - traduit la capacité du matériau à stocker de la chaleur sans se réchauffer de manière notable en surface (équation 5). Cette dernière caractérise plutôt les parois intérieures, traduisant ainsi une sensation de paroi froide ou de paroi chaude. Plus l'effusivité est grande, plus la chaleur de l'air intérieur sera absorbée rapidement par la paroi limitant ainsi l'élévation de température et donc l'inconfort lors de périodes de canicule. Pour contribuer au confort d'été, il convient donc que l'enveloppe extérieure du bâti soit la moins diffusive possible et l'enveloppe intérieure la plus effusive possible (Munaretto, 2014).

$$a = \frac{\lambda}{\rho C} \quad (4) \qquad b = \sqrt{\lambda \rho C} \quad (5)$$

Avec a diffusivité (en $\text{m}^2.\text{s}^{-1}$), b effusivité (en $\text{J}.\text{m}^{-2}.\text{K}^{-1}.\text{s}^{-1/2}$), λ conductivité thermique du matériau (en $\text{W}.\text{m}^{-1}.\text{K}^{-1}$), ρ sa masse volumique ($\text{kg}.\text{m}^{-3}$) et C sa chaleur spécifique (en $\text{J}.\text{K}^{-1}.\text{kg}^{-1}$).

3. Résultats et discussion

3.1 Impact de la température sur la conductivité thermique particulière

La conductivité thermique particulière de la chènevotte, selon notre modèle, est en moyenne de $0,095 \text{ W}.\text{m}^{-1}.\text{K}^{-1}$ ce qui est cohérent avec les valeurs de la littérature (Cérézo, 2005) (Dartois et al., 2017). Selon nos résultats, sa valeur varie de manière relativement

significative en fonction de la température. Le calcul a été mené pour les deux valeurs extrêmes de 0 % et 30 % d'HR (Fig 4).

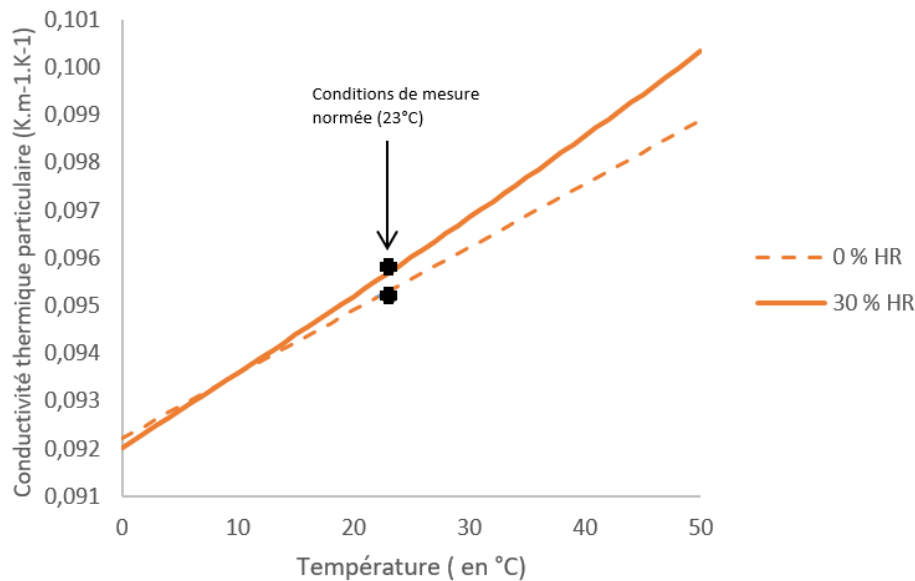


Figure 4. Evolution de la conductivité thermique particulaire en fonction de la température pour de faibles humidités relatives, selon le modèle proposé

La conductivité thermique particulaire augmente avec la température, ce qui est cohérent avec les relations données aux sections II.E et II.F et utilisées dans le modèle. Notons, par ailleurs, que le taux de variation de la conductivité thermique particulaire augmente avec l'humidité relative. Selon notre modèle, entre 0 $^{\circ}\text{C}$ (conditions hivernales) et 50 $^{\circ}\text{C}$ (conditions extrêmes sous un épisode caniculaire avec apport solaire), la valeur de conductivité thermique particulaire augmente de plus de 6% sous une humidité relative de 10% et de plus de 8% sous une humidité relative de 30 %. Aussi, jusqu'à environ 20 $^{\circ}\text{C}$, température à laquelle la caractérisation thermique des matériaux se fait en général, l'impact de l'humidité relative sur la conductivité thermique est relativement faible. Pour des températures plus élevées, il est plus marqué. Cela s'explique par les effets conjugués de l'humidité relative et de la température sur le squelette solide (cellulose) et l'air poral. On peut également remarquer que la mesure faite sous conditions normées (23 $^{\circ}\text{C}$ et 50% d'HR) ne permet pas de caractériser fidèlement la conductivité thermique particulaire sur toute la gamme de température à laquelle le matériau est soumis en condition d'usage et de surcroît lors de périodes de forte chaleur.

3.2 Impact de la température sur la chènevotte en vrac

A partir de la conductivité thermique particulaire, nous avons déterminé la conductivité thermique d'un arrangement en vrac, pour une gamme de fractions volumiques de chènevotte de 40 à 80%, ce qui correspond respectivement à du vrac non compacté et fortement compacté. La figure 5 montre clairement que la fraction volumique de 80% entraîne la conductivité thermique la plus défavorable vis-à-vis d'une utilisation en isolation. Finalement, de ce point de vue, c'est la fraction volumique de 40 % qui présente les meilleures capacités d'isolation. Ces résultats s'expliquent par une conductivité thermique particulaire qui est en moyenne de $0,095 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ (cf section III.A) face à une conductivité thermique de

l'air de l'ordre de $0,027 \text{ W.m}^{-1}\text{.K}^{-1}$ (cf section II.F). Logiquement, un vrac de particules non tassé contient un volume d'air plus important qu'un vrac tassé, il est donc plus isolant.

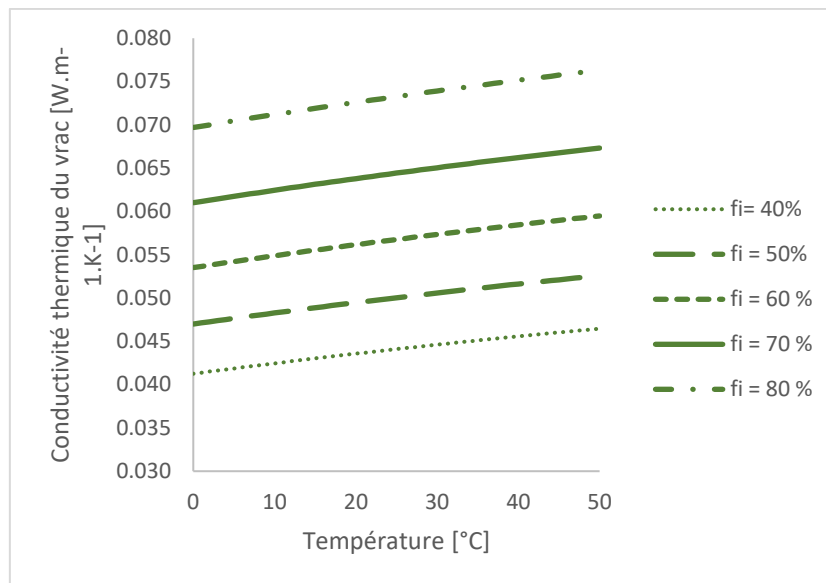


Figure 5. Evolution de la conductivité thermique de chènevotte en vrac en fonction de la température pour différentes fractions volumiques de granulat, à 30 % d'HR, selon le modèle proposé.

De plus, la conductivité thermique du vrac varie linéairement avec la température et augmente de 10 % entre 0°C et 50°C , quelle que soit la fraction volumique de granulat considérée. Ces tendances données par notre modèle sont en accord avec l'étude menée par (Bourdote et al., 2017) sur des composites à base de chènevotte et d'amidon. Enfin, la valeur moyenne donnée par notre modèle pour la conductivité thermique en vrac est de $0,050 \text{ W.m}^{-1}\text{.K}^{-1}$ sur la gamme de température $20\text{-}25^\circ\text{C}$ ce qui est en accord avec les valeurs données par la littérature (Tableau 1).

Tableau 1. Valeur moyenne de la conductivité thermique de la chènevotte en vrac donnée par notre modèle et comparée aux résultats de la littérature.

$\lambda_{\text{vrac}} \text{ (W.m}^{-1}\text{.K}^{-1}) \text{ de chènevotte}$			
Modèle proposé	Résultats expérimentaux issus de la littérature		
Moyenne 0%-30%HR sur la gamme 20°C - 25°C	Valeur	Référence	Température de mesure
0,050	0,048	(Bourdote et al., 2017)	23°C
	0,049	(Ratsimbazafy, 2022)	20°C
	0,055	(Magniont et al., 2012)	25°C
	0,058	(Dinh, 2014)	25°C

3.3 Discussion sur le confort d'été

En considérant que le vrac à 40 % de fraction volumique est le plus isolant donc le plus opportun vis-à-vis de l'usage, nous avons étudié l'impact de la température sur les coefficients de diffusivité a et d'effusivité b pour ce cas de figure. Les résultats sont présentés dans la figure 6. Ainsi, lorsque la diffusivité augmente d'un peu plus de 10% entre 0°C et 50 °C, l'effusivité du même mélange en vrac augmente de seulement 5%. Cet écart entre les deux grandeurs s'explique par une pondération différente de la conductivité thermique dans les formules de diffusivité et d'effusivité. Aussi, selon nos résultats, une paroi dans laquelle de la chènevotte a été insufflée en vrac voit ses capacités de transmission augmenter deux fois plus que ses capacités d'adsorption lors d'une élévation de la température ambiante. Il est donc probablement plus opportun d'utiliser ce matériau en paroi intérieure plutôt que pour de l'isolation extérieure. En effet, avec une effusivité croissante avec la température, la paroi absorbera plus efficacement les apports internes pour les transmettre au cœur ce celle-ci, favorisant ainsi le confort d'été.

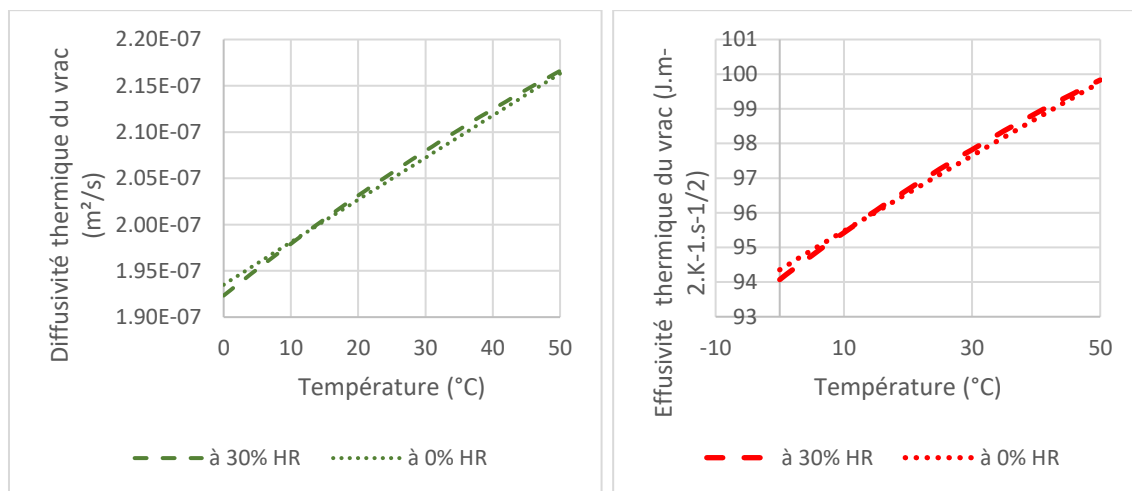


Figure 6. Evolution la diffusivité (à gauche) et de l'effusivité (à droite) de la chènevotte en vrac en fonction de la température à 0% et 30 % d'HR, selon le modèle proposé

4. Conclusion

Cet article propose une nouvelle démarche de détermination de la conductivité thermique de parois biosourcées. En effet, la modélisation détaillée ici repose sur une connaissance préalable de la porosité particulière du granulat végétal, de la conductivité thermique de l'air et de celle du squelette solide particulière. A partir de ces données d'entrée nous obtenons la conductivité thermique particulière puis, par une nouvelle homogénéisation, celle de la chènevotte en vrac. Ainsi nous sommes capables de proposer une estimation, à différentes échelles, de la variation de la conductivité thermique en fonction de la température, ce pour de faibles humidités relatives. Une étape chronophage de caractérisation thermique pourrait ainsi être évitée, ouvrant la perspective pour une application à d'autres types de granulats végétaux.

Les résultats de cette étude montrent d'une part que la caractérisation thermique des matériaux biosourcés sous 23°C conduit à une valeur de conductivité thermique qui ne peut, en toute rigueur, rendre compte du comportement réel du matériau soumis à des conditions

extrêmes. En effet, une variation moyenne de 10% des valeurs de conductivité thermique du matériau en vrac est observée entre 0°C et 50°C, ce qui correspond aux conditions d'usage dans le bâti, soumis à des conditions hivernales ou caniculaires. D'autre part, l'impact de la température sur les paramètres d'inertie du matériau est également mis en relief par nos résultats. Ainsi, ils permettent de s'interroger sur la pertinence d'une utilisation de ce type de matériau en isolation par l'extérieur ou l'intérieur. Plus la température est élevée, plus leur diffusivité est grande augmentant ainsi l'échange thermique entre le climat extérieur et le climat intérieur. Tandis que l'augmentation de la conductivité thermique avec la température a donc un effet néfaste sur l'inertie par transmission, elle permet d'améliorer l'inertie par adsorption. Ainsi, des parois internes constituées de chènevottes en vrac permettront de s'opposer plus facilement aux variations internes de température, en période de forte chaleur, participant ainsi au confort d'été.

Par ailleurs, selon des relevés météorologiques existants (Martin, 2022) et certaines projections climatiques, des périodes de chaleurs peuvent s'accompagner d'humidités relatives bien au-delà des 30 % considérés ici. Cette étude devra être complétée par une prise en compte de la phase d'eau adsorbée au sein de la particule végétale pour étudier la variabilité de la conductivité thermique avec la température dans toute la gamme des humidités relatives. Cela permettra de discuter plus largement de l'impact sur le confort d'été mais également du comportement de ce type de paroi dans des conditions hivernales.

Enfin, la méthode appliquée ici sur des granulats végétaux pourra être étendue à l'échelle de composites bio-sourcés comme les bétons végétaux. Pour cela, certains verrous scientifiques, tels que la fraction volumique de granulats dans le matériau ou encore la porosité au niveau de l'interface granulat/liant devront être levés pour proposer une étude pertinente.

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7.2 Valider les modèles de prédiction dans le cas de matériaux à base de terre crue incorporant des granulats végétaux

Les terres allégées, composées de granulats végétaux enrobés par une matrice terre crue, permettent d'assurer des fonctions d'isolation tout en offrant un matériau plus dense que l'isolation en vrac. Les performances du bâti, en termes de confort d'été, sont ainsi améliorées et peuvent plus facilement répondre aux objectifs nouvellement fixés par la RE 2020 en France. Les terres allégées sont donc des matériaux de construction particulièrement prometteurs. L'incorporation de granulats végétaux dans des blocs de terre crue, dans une plus faible proportion que pour les terres allégées, peut se justifier également pour gagner en capacité d'isolation tout en visant des propriétés mécaniques proches du bloc de terre crue dépourvu d'addition végétale. De plus, aucune réaction délétère entre matrice terre crue et granulats végétaux n'est rapportée à ce jour dans la littérature, contrairement aux matrices hydrauliques. Les matériaux à base de terre crue incorporant des granulats végétaux sont donc d'un intérêt tout particulier, d'où le choix de les considérer en priorité pour proposer et valider des modèles de prédiction du comportement thermique.

Cet article se focalise sur la prédiction de la conductivité thermique de différentes terres allégées et blocs de terre crue incorporant des granulats végétaux. Des travaux expérimentaux de la littérature ont été rigoureusement sélectionnés pour servir de base à l'étude. Deux types de volumes élémentaires représentatifs (V.E.R.) sont ainsi testés pour évaluer la conductivité thermique effective de chacun des matériaux, par homogénéisation. Le but est d'identifier la description la plus pertinente, en adéquation avec la réelle morphologie du matériau (forme et orientation des granulats végétaux, fissuration après séchage). Lorsque des inclusions d'air sont considérées dans les V.E.R., elles représentent à la fois les fissurations au sein de la matrice et la porosité à l'interface entre la matrice et les granulats végétaux. Une attention particulière est portée sur l'accessibilité et la cohérence des données d'entrée des modèles, bien que la proportion de la fissuration des composites demeure inconnue dans les travaux sélectionnés. Ce manque de données impose de formuler des hypothèses de travail.

Les résultats présentés ici montrent que l'utilisation du modèle de Mori-Tanaka est pertinent à l'échelle matériau, comme à l'échelle particulaire (chap. 4) ou celle du vrac (chap. 7). Par ailleurs, même pour des fractions volumiques d'inclusions relativement importantes, les résultats fournis par schéma de Mori-Tanaka demeure cohérent. Les VER proposés, en adéquation avec la réelle microstructure des matériaux, ainsi que le travail réalisé précédemment sur la conductivité thermique particulaire (chap. 4 et 5) permettent de prédire de manière cohérente la conductivité thermique des matériaux en terre crue incorporant des granulats végétaux. Cette étude souligne l'intérêt de poursuivre les travaux pour évaluer l'impact de modifications dans la microstructure sur les performances thermiques des matériaux. L'objectif est de pouvoir guider les essais, en amont de la fabrication, et contribuer ainsi à l'optimisation de la formulation des matériaux bio et géo-sourcés.

Article G. Matériaux terre crue-granulats végétaux : prédiction de la conductivité thermique par modélisation multi-échelle

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Résumé. La terre crue est un matériau de construction exemplaire sur bien des aspects : disponibilité, recyclabilité, faible impact environnemental, performances mécaniques et hydriques reconnues. L'incorporation de granulats végétaux permet d'améliorer les performances thermiques. Cependant la diversité des co-produits agricoles ajoutée à celle des sols utilisés en tant que matrice liante conduit à des performances très variables selon les formulations et la nature des constituants. L'homogénéisation par champs moyens (schéma de Mori Tanaka) est ici proposée et évaluée en tant qu'outil de prédiction de la conductivité thermique de ces matériaux. S'appuyant sur une détermination adéquate des données d'entrée et un modèle inspiré de la structure du matériau, cette étude valide des modèles de prédiction en s'appuyant sur plusieurs campagnes expérimentales de la littérature. Ces travaux ouvrent la voie vers la possibilité d'une optimisation de la conductivité thermique d'un matériau terre-crue incorporant des granulats végétaux avant même l'étape de fabrication.

Mots-clefs Modélisation multi-échelle, terre crue, granulats végétaux

1. Introduction

Le réchauffement climatique et ses conséquences sont désormais une réalité visible à l'échelle mondiale (Adélaïde et al., 2022). Les réponses apportées peuvent diverger en fonction de la philosophie adoptée, qu'il s'agisse d'une approche axée sur le « culte de la nature sauvage », de l'« éco-efficacité » ou encore de l'« écologisme populaire » (Martinez-Alier, 2023). L'utilisation ancestrale de la terre crue dans la construction, ses capacités reconnues de tampon hydrique, les possibilités d'exploitation locale à moindre coût et son faible impact environnemental constituent des avantages majeurs qui devraient favoriser un consensus quant à son utilisation en tant que matériau de construction. Par ailleurs, l'incorporation de granulats végétaux dans une matrice terre crue permet d'atteindre de bonnes performances thermiques (Laborel-Préneron et al., 2018) et ainsi de contribuer à la performance énergétique du bâti. Devant le besoin d'optimiser les formulations et d'utiliser des co-produits locaux - pour minimiser l'impact environnemental et favoriser l'économie locale-, des travaux de modélisation des propriétés effectives de matériaux à base de terre crue incorporant des granulats végétaux sont disponibles dans la littérature (Belayachi et al., 2022). Cependant, les auteurs concluent à la pertinence de l'homogénéisation numérique tout en soulignant les limites des modèles conceptuels. Il convient de noter que ces modèles ont pourtant démontré leur pertinence dans le domaine des matériaux composites (Shanmuga Priyan and Kanmani Subbu, 2023). Le réel défi réside dans la pertinence des données d'entrée des constituants. La conductivité thermique effective, calculée par une analyse multi-échelle, nécessite notamment d'avoir accès à la conductivité thermique des inclusions considérées, c'est-à-dire celle des granulats végétaux. Ces valeurs font défaut dans la littérature mais des travaux récents ont permis de les estimer efficacement (Rosa Latapie et al., 2023a). Dans cette étude, une homogénéisation semi-analytique par champs moyens (Mori and Tanaka, 1973) est menée en utilisant les valeurs de conductivité thermique particulière. Les valeurs de sortie des modèles - les conductivités thermiques de composites terre crue-granulats végétal- sont comparées aux valeurs expérimentales. Cela permet de démontrer et d'asseoir la pertinence du recours à la modélisation multi-échelle pour les matériaux de construction géo-sourcés dès lors que les données d'entrée sont correctement déterminées.

2. Matériaux et méthodes

2.1 Matériaux à base de terre crue avec incorporation de granulats végétaux.

Plusieurs matériaux ont été sélectionnés dans la littérature pour cette étude. La contrainte était d'avoir toutes les informations pertinentes pour pouvoir en déduire les données d'entrée nécessaires à la modélisation. Les matériaux retenus, leurs caractéristiques extraites des campagnes expérimentales et les références associées sont rassemblées dans le tableau 1.

Tableau 1. Composites sélectionnés pour l'étude : caractéristiques et références

Matériau composite (Notation)	Références	Masse volumique du composite (kg.m ⁻³)	Conductivité thermique du composite (W.m ⁻¹ .K ⁻¹)	Masse volumique de la matrice (kg.m ⁻³)	Conductivité thermique de la matrice (W.m ⁻¹ .K ⁻¹)	Conductivité thermique du granulat en vrac (W.m ⁻¹ .K ⁻¹) / Masse volumique en vrac (kg.m ⁻³)	Méthode de mesure
Argile/Moelle de tournesol (AM)	(Belayachi et al., 2022)	235	0.055	900	0.27	0.036 / 15.9	Fil chaud (régime transitoire)
Argile/Ecorce de tournesol (AE)	(Brouard et al., 2018)	714	0.158			0.053 / 129	
Fines de lavage/Chènevotte (FC3)	(Laborel-Préneron et al., 2018)	1519	0.20	1891	0.57	0.051 / 153	Plaque chaude gardée (régime permanent)
Fines de lavage/Chènevotte (FC6)		1271	0.30				

Notons que les mesures expérimentales font l'hypothèse d'un transfert thermique unidirectionnel, i.e l'anisotropie du matériau n'est pas prise en compte. Les masses volumiques des composites terre crue-granulat végétal sont très variables ainsi que leurs conductivités thermiques. Aussi, dans l'optique d'une modélisation fidèle à la réalité structurelle des matériaux, une classification est proposée. En effet, selon le ratio granulat/liant et la méthode de fabrication notamment, la structure de ces éco-matériaux est clairement différente . Elle peut être décrite et schématisée (Figure 1) comme suit :

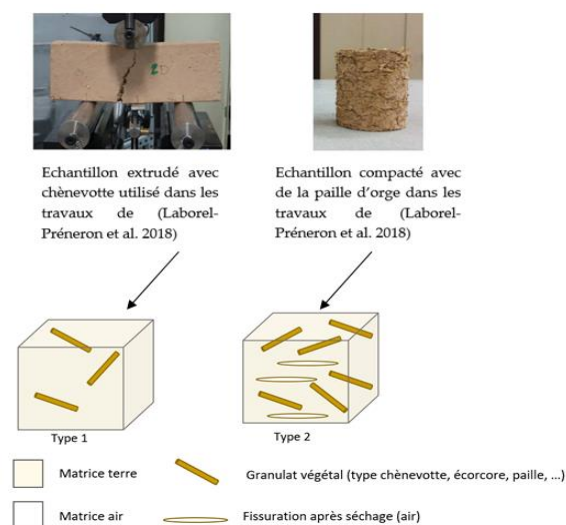


Figure 1. Schématisation des différentes structures de composites terre crue-granulats végétaux (cas des granulats de forme cylindrique) et exemples illustratifs correspondants extraits de la littérature (Laborel-Préneron et al., 2018).

Lorsque les granulats sont complètement noyés dans la matrice liante, ils constituent des « inclusions » au sens strict du terme. C'est particulièrement le cas avec un procédé de fabrication par extrusion (Laborel-Préneron et al., 2017) et une faible proportion de granulats (type 1).

Dans le cadre d'une mise en oeuvre par compression, une porosité additionnelle notable se crée dans le plan perpendiculaire à la compression (Laborel-Préneron et al., 2017) après la mise en oeuvre. C'est probablement l'effet combiné d'une fissuration de la matrice liante au séchage (Anglade, 2022) et des granulats végétaux qui reprennent leur forme initiale après déformation (type 2). D'autre part, une orientation préférentielle des granulats se fait dans un plan perpendiculaire à la compression. Dans le cas éventuel d'une projection, on retrouve le même phénomène d'orientation préférentielle dans le plan perpendiculaire à la direction de projection.

2.2 Modélisation par champs moyens

Le schéma de Mori Tanaka (Mori and Tanaka, 1973) est utilisé pour les calculs d'homogénéisation sur tous les types modèles de composites. Cela permet de prendre en compte notamment l'effet des inclusions entre elles. Il s'agit de considérer des éléments de volumes représentatifs (VER) constitués de plusieurs phases (décrites dans la figure 1) dont il faut connaître la fraction volumique et la conductivité thermique. Pour les inclusions (granulat végétal ou fissuration), il est nécessaire également de définir une forme (sphère, cylindre, ellipsoïde), un facteur de forme (fixe ou défini par une loi de distribution) et une orientation (aléatoire 3D, aléatoire 2D ou définie par les composantes d'un tenseur). Une relation analytique permet ensuite de calculer la conductivité thermique d'un milieu homogène équivalent (celui du composite) à partir de celles des particules végétales et de la matrice terre (Rosa Latapie et al., 2024, 2023a) . Ce schéma d'homogénéisation peut toutefois montrer des limites dans le cas d'une grande fraction volumique (Jeancolas, 2018) surtout si le contraste est particulièrement marqué entre l'inclusion et la matrice (Ghossein and Lévesque, 2011).

2.3 Conductivité thermique particulière

L'analyse macro et microstructurale des inclusions permet d'accéder à leur conductivité thermique particulière selon que le granulat est plutôt isotrope (type moelle de tournesol) ou transversalement isotrope (type paille, chènevotte) grâce à deux méthodes développées dans des travaux précédents (Rosa Latapie et al., 2023a, 2023b). Dans les références choisies pour cette étude, la conductivité thermique des granulats en vrac a été mesurée avant la mise en oeuvre des matériaux ce qui a permis de remonter, par méthode inverse, à la conductivité thermique particulière des granulats végétaux utilisés. Cependant, ces calculs nécessitent également la connaissance des masses volumiques particulières. Étant donné que ces données n'étaient pas disponibles dans les campagnes expérimentales sélectionnées, les valeurs ont été extraites d'autres travaux de la littérature (Ratsimbazafy et al., 2023).

En outre, pour les granulats de forme cylindrique, une hypothèse sur la valeur du paramètre d'anisotropie γ était nécessaire : le choix a été de considérer une valeur de 1.5 qui semble faire consensus pour la chènevotte (Nguyen et al., 2016; Rosa Latapie et al., 2023a) bien qu'elle n'ait pas été déterminée pour d'autres granulats. Ce choix est justifié par la similitude apparente de l'aspect des pailles avec celui de la chènevotte, tant à l'échelle macroscopique

que microscopique. Ainsi, deux composantes de la conductivité thermique particulaire, la composante tangentielle λ_T et la composante normale λ_N , sont déterminées (figure 2).

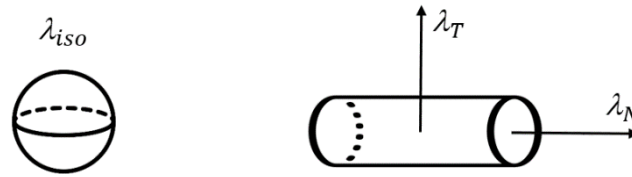


Figure 2. Conductivité thermique particulaire selon le type de granulat végétal envisagé : cas isotrope (moelle de tournesol) ou anisotrope (chènevotte, paille de blé/colza/orge, ...)

Enfin, dans la mesure où les conductivités thermiques ont été mesurées sur des échantillons préalablement séchés en étuve, la conductivité thermique particulaire a été calculée à l'état sec en amont de la modélisation. Les valeurs particulières utilisées pour l'homogénéisation sont récapitulées dans le tableau 2.

Tableau 2. Détermination des conductivités thermiques particulières en amont de la modélisation

Type de granulat (Notation)	Masse volumique particulaire (kg.m^{-3})	Référence	Conductivité thermique particulaire à l'état sec utilisée dans l'étude ($\text{W.m}^{-1}\text{K}^{-1}$)			Valeur comparative de la littérature -cas isotrope- ($\text{W.m}^{-1}\text{K}^{-1}$) (Référence)
			λ_{iso}	λ_T	λ_N	
Moelle de tournesol (M)	56	(Ratsimbazafy et al., 2023)	0.08 (0.04)	/	/	0.036 (Belayachi et al., 2022)
Ecorce de tournesol (E)	425		/	0.045	0.068	0.053 (Belayachi et al., 2022)
Chènevotte (C)	248		/	0.044	0.067	0.110 (Dartois et al., 2017)

La valeur de $0.08 \text{ W.m}^{-1}\text{K}^{-1}$ pour la conductivité thermique particulaire de la moelle de tournesol - déduite de la mesure faite sur le vrac selon l'étude sélectionnée - est relativement élevée alors même que ce granulat est connu pour être l'un des plus poreux (Abbas et al., 2020). Il devrait donc logiquement avoir une conductivité thermique plus faible que l'écorce de tournesol ou la chènevotte. Une précédente étude sur la conductivité thermique particulaire de ce granulat met en évidence une valeur deux fois moins importante, de l'ordre de $0.04 \text{ W.m}^{-1}\text{K}^{-1}$, validée à l'échelle de panneau isolants (Rosa Latapie et al., 2023b). Les deux valeurs ont été considérées ici pour être discutées ensuite au niveau des résultats et sont référencées sur les noms des composites par les suffixes - λ_8 ou - λ_4 .

2.3. Hypothèses et démarche de l'étude

Les hypothèses retenues pour cette étude sont les suivantes :

- L'eau éventuellement adsorbée par le bio-composite au niveau de la terre crue et du granulat végétal n'est pas prise en compte.

- Les granulats végétaux sont considérés comme des inclusions isotropes (sphères) ou transversalement isotropes (cylindres) avec une orientation aléatoire selon les trois directions de l'espace (figure 3) ou dans le plan perpendiculaire à la compression

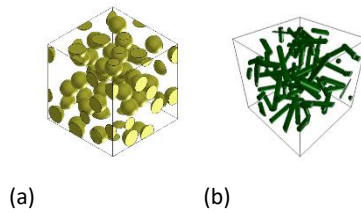


Figure 3. VER considérés selon le type de granulat végétal : moelle de tournesol (a) ou type paille /chènevotte (b) avec l'hypothèse de deux phases : inclusions (orientation aléatoire) et matrice liante.

Par ailleurs, pour apprécier la pertinence des descriptions proposées à la section II.A, l'étude a été menée en considérant les deux types de structure définies dans la figure 1 :

- la porosité matrice/inclusion (soit liant/granulat végétal) est négligée, seules sont prises en compte les porosités intra liant et intra granulaire via les conductivités thermiques respectives du liant et du granulat végétal (type 1).
- la porosité matrice/inclusion et la porosité additionnelle qui se crée après le processus de fabrication sont considérées à travers une porosité spécifique (orientation perpendiculaire à la compression et facteur de forme correspondant à une ellipse aplatie), fixée arbitrairement à 10 % du volume total du VER (type 2), qui est une valeur vraisemblable car ces composites sont très cohésifs (Menibus et al., 2018).

Une discussion est menée sur la base de ces simulations, par comparaison aux résultats expérimentaux. Il est à noter cependant que ces derniers sont des scalaires (notés $\lambda_{\text{composite}}$), issus des appareils de mesure de conductivité thermique (fil chaud, plaque chaude gardée) alors que la modélisation donne accès à un tenseur de conductivité thermique $\underline{\underline{\lambda}}_{\text{composite}}$ qui prend en compte les différentes composantes définies par la loi de Fourier pour un matériau anisotrope :

$$\underline{j} = - \underline{\underline{\lambda}}_{\text{composite}} \underline{\text{grad T}} \quad (1)$$

Avec \underline{j} , le vecteur de densité de flux thermique macroscopique (en W.m^{-2}), $\underline{\underline{\lambda}}_{\text{composite}}$ le tenseur de conductivité thermique (en $\text{W.m}^{-1}.\text{K}^{-1}$) et $\underline{\text{grad T}}$ le gradient de température (en K.m^{-1}). Le tenseur de conductivité thermique est composé de trois composantes notées comme suit :

$$\underline{\underline{\lambda}}_{\text{composite}} = \begin{bmatrix} \lambda_1 & 0 & 0 \\ 0 & \lambda_2 & 0 \\ 0 & 0 & \lambda_3 \end{bmatrix} \quad (2)$$

Dans le but de pouvoir comparer les valeurs expérimentales (une seule valeur scalaire) aux valeurs résultant de l'homogénéisation (composantes λ_{ij} du tenseur), la moyenne des composantes données par la modélisation a été calculée. Dans le cas où le matériau est

isotrope, les composantes λ_{ij} sont égales à $\lambda_{\text{composite}}$. Ces composantes sont calculées par l'homogénéisation par champs moyens décrite dans la partie B.

2.4 Données d'entrée de la modélisation

Des données d'entrée spécifiques à la matrice et aux inclusions sont nécessaires (masse volumique et conductivité thermique) ainsi que la fraction volumique des phases dans le composite. Cette donnée constitue un point délicat car elle est rarement explicitée ou mesurée dans la littérature. Elle peut se calculer -pour la structure de type 1- à partir des masses volumiques de chaque phase et du volume de l'échantillon. Les rapports de forme (moyenne des analyses granulométriques) sont extraits, si possible, des références sélectionnées. A défaut, ils sont issus de la moyenne des valeurs données dans une étude bibliographique récente sur les granulats végétaux (Ratsimbazafy et al., 2021). Le tableau 3 précise ainsi toutes les données d'entrées complémentaires utilisées pour la modélisation.

Tableau 3. Données d'entrée complémentaires aux conductivités thermiques particulières

Composite Matrice/Inclusion (Notation)	Rapport de forme de l'inclusion (Référence)	Fraction volumique de l'inclusion
Argile/Moelle de tournesol (AM)	1.5 (Ratsimbazafy et al., 2021)	0.79
Argile/Ecorce de tournesol (AE)	3.87 (Ratsimbazafy et al., 2021)	0.39
Fines de lavage/Chènevotte (FC1)	3.3 (Laborel-Préneron et al., 2018)	0.22
Fines de lavage/Chènevotte (FC2)		0.37

L'étude multi-échelle donne ainsi accès à la conductivité thermique effective du matériau. Il est démontré empiriquement que cette valeur décroît avec la masse volumique du matériau (Laborel-Préneron et al., 2018).

3. Résultats et discussion

Pour bien distinguer les différents essais menés et dans le but de comparer de manière exhaustive les résultats expérimentaux aux résultats obtenus par homogénéisation, la liste des modèles testés est récapitulée dans le tableau 4. Cela permet d'analyser les différentes variantes considérées pour un même matériau et de visualiser l'impact des hypothèses faites sur les résultats.

Tableau 4 Liste des modèles testés avec le schéma de Mori Tanaka et résultats associés

Type 1			
Composite modélisé	Orientation des inclusions	Spécificités du modèle	Conductivité thermique du composite à l'état sec - λ_{iso} ou composantes $\lambda_1, \lambda_2, \lambda_3$ - (W.m⁻¹.K⁻¹)
AM-1- λ_8	/	$\lambda_{iso} = 0.08 \text{ W.m}^{-1}\text{K}^{-1}$	0.112
AM-1- λ_4	/	$\lambda_{iso} = 0.04 \text{ W.m}^{-1}\text{K}^{-1}$	0.077
AE-1-3D	Aléatoire 3D	/	0.166
AE- 1-2D	Aléatoire 2D dans plan perpendiculaire à la direction de compaction	/	0.172/0.172/0.156
FC1-1-3D	Aléatoire 3D	/	0.421
FC1-1-2D	Aléatoire 2D dans plan perpendiculaire à la direction de compaction	/	0.430/0.430/0.413
FC2-1-3D	Aléatoire 3D	/	0.362
FC2-1-2D	Aléatoire 2D dans plan perpendiculaire à la direction de compaction	/	0.341/0.341/0.323

Type 2			
AM-2- λ_8	/	Inclusions (air) de facteur de forme 20 (allongé de type fissure) aléatoirement disposées dans un plan perpendiculaire au plan de compaction	0.091/0.091/0.090
AM-2- λ_4	/		0.057 /0.057/0.056
AE-2-3D	Aléatoire 3D		0.142/0.142/0.139
AE- 2-2D	Aléatoire 2D dans plan perpendiculaire à la direction de compaction		0.147/0.147/0.129
FC1-2-3D	Aléatoire 3D		0.360/0.360/0.340
FC1-2-2D	Aléatoire 2D dans plan perpendiculaire à la direction de compaction		0.360/0.360/0.330
FC2-2-3D	Aléatoire 3D		0.375/0.275/0.266
FC2-2-2D	Aléatoire 2D dans plan perpendiculaire à la direction de compaction		0.284/0.284/0.248

Pour chaque composite, les résultats obtenus grâce à ces différentes modélisations ont été comparés aux valeurs expérimentales (figure 4).

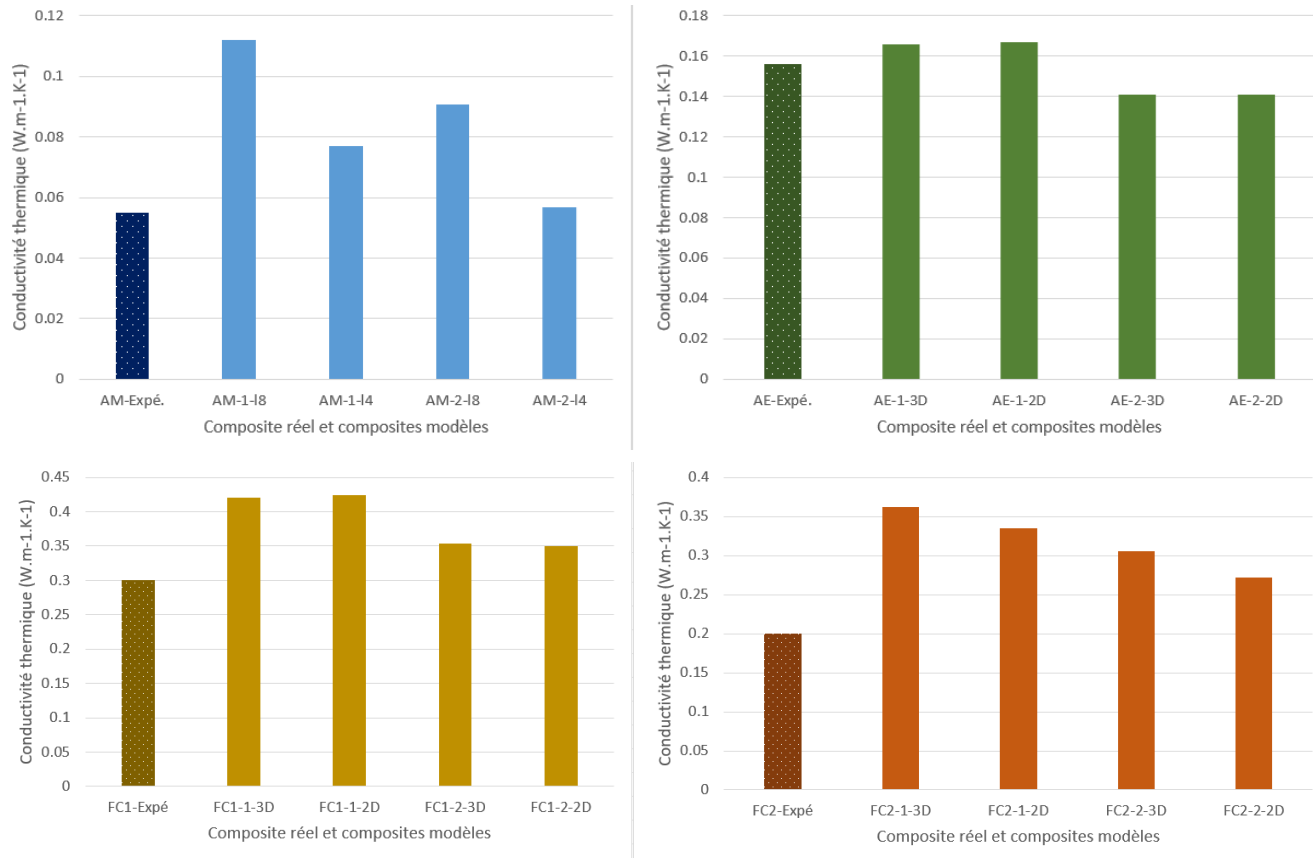


Figure 4. Comparaison des valeurs de conductivité thermique mesurées sur chaque composite aux valeurs calculées par homogénéisation selon les différents matériaux considérés : AM, AE, FC1 et FC 2.

Pour le composite AM (argile / moelle de tournesol), on constate que toutes les modélisations considérant une conductivité thermique particulière de $0,08 \text{ W.m}^{-1}.\text{K}^{-1}$ surévaluent largement celle mesurée sur le composite réel. En revanche, en considérant la valeur particulière de $0,04 \text{ W.m}^{-1}.\text{K}^{-1}$, les résultats sont nettement améliorés (écart relatif de moins de 5% entre la valeur modélisée AM-2- λ_4 et la valeur expérimentale). Cette valeur est donc vraisemblablement plus pertinente. Par ailleurs, il est intéressant de noter que les modélisations type 2 donnent des meilleurs résultats : la prise en compte de la fissuration après séchage semble se justifier indépendamment de la valeur de la conductivité thermique du granulat.

Pour le composite AE (argile/écorce de tournesol), les modélisations qui ne tiennent pas compte de la porosité additionnelle due à la fissuration au séchage surévaluent légèrement la conductivité thermique du composite, ce qui est cohérent. Avec une porosité additionnelle de 10% du volume total, les valeurs du modèle sont en-deçà de la valeur réelle (environ 10 % en dessous). La porosité additionnelle réelle est donc vraisemblablement inférieure à cette valeur fixée arbitrairement (faute d'avoir accès à cette donnée dans la littérature).

Pour le composite FC1 (fines de lavage/chènevotte), la modélisation la plus pertinente est celle de type 2. La surestimation de ce modèle (de l'ordre de 15%) montre que la porosité additionnelle est probablement plus importante que la valeur de 10 % fixée dans l'étude. Comme pour le matériau AE, la prise en compte de l'orientation préférentielle des particules

de chènevotte dans une orientation perpendiculaire au plan de compactage ne semble pas nécessaire. En effet, les résultats qui considèrent une orientation aléatoire dans les trois directions de l'espace donnent des résultats tout aussi pertinents.

Pour le modèle FC 2 (fines de lavage/chènevotte), la modélisation la plus pertinente reste celle de type 2. La prise en compte de l'orientation préférentielle des particules due à la mise en œuvre du matériau améliore le résultat prédictif. Cet effet notoire sur la conductivité thermique du composite s'explique par une proportion volumique de granulats importante : la proportion volumique de chènevotte dans le composite FC 2 est de 37% contre 22% pour le composite FC 1. Structurellement, la fraction volumique de matrice est donc moins importante mais il est difficile d'en évaluer la part de fissuration d'où un écart plus marqué dans ce cas entre valeur théorique et valeur expérimentale. Cependant, on pourrait envisager une fissuration moins importante pour FC2 que pour FC 1 par un possible pontage réalisé par les particules de chènevottes. Plus nombreuses dans le composite FC 2 que dans FC 1, elles pourraient être à l'origine d'une limitation de la fissuration au séchage. En effet, ce type de phénomène a été mis en évidence sur des travaux concernant des mortiers (Saad et al., 2022).

4. Conclusion

Cette étude démontre qu'une bonne définition des données d'entrée, couplée à une définition structurelle fidèle du composite réel, permet de prédire la conductivité thermique à sec de composites terre crue-granulats végétaux de manière très pertinente grâce à l'homogénéisation par champ moyens utilisant le modèle de Mori Tanaka. Malgré des fractions volumiques relativement élevés, les résultats sont pertinents. Cela s'explique probablement par un contraste peu marqué entre les conductivités thermiques particulières et celle de la matrice. Pour la grande majorité des composites sélectionnés dans l'étude, la valeur modélisée de la conductivité thermique n'excède pas 15 % d'écart avec la valeur réelle. Pour améliorer le modèle prédictif, il conviendra d'apporter une attention particulière sur la caractérisation préalable des constituants du matériau que l'on souhaite mettre en œuvre. Il s'agit d'avoir accès à la masse volumique et la conductivité thermique de la matrice seule. Concernant les granulats végétaux, la mesure de la conductivité thermique en vrac, sur un mélange dont on connaît la fraction volumique de granulats, est souhaitable. A défaut, la masse volumique en vrac est exploitable si la masse volumique particulière est connue pour en déduire la fraction volumique. En conséquence, le procédé de fabrication doit permettre de contrôler la proportion volumique des granulats incorporés mais également l'orientation préférentielle de ces derniers. En effet, cette dernière peut avoir un impact non négligeable sur la conductivité thermique du composite.

Il est à noter que les composites modèles étudiés dans ces travaux ouvrent la possibilité de pouvoir déterminer la conductivité thermique par homogénéisation en amont même de la fabrication. Il est donc possible d'envisager une optimisation de la formulation en modifiant l'orientation des granulats et/ou leur facteur de forme dans une phase préalable à la mise en œuvre. L'impact de modifications microstructurales pourrait ainsi être évalué sur les performances thermiques des géo-composites. Toutefois, le manque de campagnes expérimentales dans la littérature donnant accès aux valeurs nécessaires à la modélisation est

notable. Une étude élargie pourrait permettre -par comparaison entre les différents modèles et la pertinence de la valeur de conductivité thermique prédite- de définir des « cas type » de modélisation suivant la technique de fabrication et la formulation (type et proportion de granulats).

Enfin, le fait de mener une étude multi-échelle dès celle du granulat permet d'envisager également, dans la modélisation, un changement de granulat dans le composite en prenant en compte la disponibilité locale des co-produits agricoles (filière existante ou non, saisonnalité des récoltes). Ce dernier élément est fondamental pour optimiser l'impact environnemental des matériaux terre crue-granulats végétaux et répondre ainsi aux enjeux énergétiques, climatiques et sociétaux actuels. Cette perspective pourra faire l'objet de travaux futurs.

5. Remerciements

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7.3 La modélisation multi-échelle pour prédire et optimiser

Ce dernier chapitre explicite la méthodologie adoptée pour prédire la conductivité thermique de bétons végétaux et de terres allégées. Il s'appuie sur les démarches réalisées dans les chapitres précédents et une nouvelle fois sur des travaux expérimentaux rigoureusement sélectionnés dans la littérature.

Dans un premier temps, il s'agit d'identifier le lien entre les données des formulations et les volumes élémentaires représentatifs (VER) à considérer dans les modèles prédictifs. En plus du schéma de Mori-Tanaka utilisé jusqu'ici dans les travaux de thèse, le schéma de double inclusion est également considéré dans la modélisation. Cette proposition est motivée par la recherche d'une modélisation toujours plus cohérente, en adéquation avec la morphologie du matériau étudié. Ainsi, pour chaque type de matériau, trois différents VER sont testés et évalués. Le modèle le plus pertinent est identifié et, selon la disponibilité des données à l'échelle microstructurale, une calibration est envisagée afin de poursuivre l'étude d'optimisation.

Dans un deuxième temps, la modélisation permet de réaliser des modifications au niveau de la microstructure des matériaux, qui seraient relativement difficiles à explorer sur le plan expérimental (faisabilité et/ou caractère chronophage). Ces modifications permettent d'étudier l'impact d'un changement de granulats (type ou forme), de leur orientation (induite par le procédé de mise en œuvre) ou encore d'un changement de liant sur la conductivité thermique à l'échelle macroscopique du matériau. Un premier pas est ainsi franchi dans l'objectif d'optimisation de la formulation vis-à-vis des performances thermiques visées. Enfin, pour élargir la réflexion et considérer les bétons végétaux, dans une dimension plus globale en termes de performance, une analyse multi-critère est proposée. Elle ne saurait être exhaustive et souffre objectivement d'un manque de données quantitatives, faute de références suffisantes dans la littérature. Elle doit être considérée comme les prémices de travaux à mener à long terme pour contribuer au développement des matériaux de construction bio et géo-sourcés.

7.3.1 Cas des terres allégées

Les travaux de prédiction et d'optimisation se concentrent d'abord sur les performances thermiques de terres allégées, en approfondissant ceux proposés dans la section 7.2. En effet, le modèle de Mori-Tanaka montre ses limites dès lors que la proportion de granulats végétaux incorporés dans la matrice terre crue devient significativement importante. Le modèle de double inclusion se révèle alors plus pertinent. Il est important de souligner le manque d'images microstructurales dans le cas des terres allégées. L'appui d'images par tomographie à rayons X, en correspondance à une formulation donnée, permettrait de consolider les hypothèses faites en termes de fractions volumiques sur les différentes phases des volumes élémentaires représentatifs choisis dans cette étude. Il serait également important de pouvoir avoir des informations fiables sur l'orientation des particules végétales dans la matrice terre crue pour affiner les modèles prédictifs.

Article H. Thermal performance of lightweight earth: from prediction to optimization through multiscale modeling

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Highlights:

- Predictive models: Experimental data are used to develop and calibrate thermal conductivity prediction models.
- Real morphology: Models incorporate the morphology of lightweight earth materials.
- Optimization: Thermal performance is optimized according to various criteria, enhancing the material's efficiency and applicability.

Abstract: This study investigates the prediction of the thermal conductivity of lightweight earth and raw earth blocks incorporating plant aggregates. Given the high variability of raw materials, it is not currently possible to predict the thermal performance of this type of material before sample production. This is a major obstacle to using these eco-materials, although their use is widely encouraged to improve building performance under evolving regulatory frameworks such as The French RE2020 standard. The incorporation of plant aggregates into earth-based materials offers improved insulation properties without compromising their mechanical integrity, positioning them as promising sustainable alternatives. Mean-field homogenization techniques, including the Mori-Tanaka as well as double inclusion models, are used to develop predictive tools for thermal behavior, using rigorously selected experimental data. The selected methods are particularly relevant. The Mori-Tanaka model appears to be better suited when the proportion of aggregates is limited, whereas the double inclusion scheme proves its worth when a higher proportion of aggregates is incorporated. This study emphasizes the influence of aggregate types and processing methods on thermal conductivity, highlighting the need for precise formulation and processing techniques to optimize performance. This paper demonstrates the relevance of the applied homogenization techniques applied. It enables the real morphology of the materials studied, such as aggregate shape and intrinsic cracking, to be taken into account. It contributes to the advancement of eco-material modeling toward predictive digital twins, with the goal of simulating and optimizing complex material behavior under various environmental conditions.

Keywords: multiscale modeling; geo-based building materials; thermal conductivity; optimization

1. Introduction

Composed of plant aggregates coated in a matrix of raw earth, lightweight earth provides insulation while offering a denser material than loose insulation. Such a property not only improves the performance of buildings in terms of summer comfort, but it makes it easier to meet the new targets set by the RE 2020 French standard as well. Lightweight earth is therefore a particularly promising building material. The incorporation of plant aggregates in raw earth blocks, in a lower proportion than in lightweight earth, can also be justified to improve insulation capacity. Interestingly, this incorporation can lead to mechanical properties close to those of raw earth blocks without plant additives. So far, the literature does not contain any report of any deleterious reaction between the raw earth matrix and the plant aggregate, unlike what occurs with hydraulic matrices used in plant-based concretes [1]. Raw earth-based materials incorporating plant aggregates are therefore of particular interest, hence the decision to consider them as a priority when proposing and validating models for predicting thermal behavior.

This article focuses on thermal conductivity prediction for various lightweight earths and raw earth blocks incorporating plant aggregates. Recent work carried out under the RILEM TC 274-TCE program highlights the difficulty of characterizing this type of composite material, especially from a thermal viewpoint [2]. Temperature and relative humidity conditions influence measurements, although there is no consensus on a well-defined measurement protocol to date [3]. The wide variety of formulations (nature of the plant aggregate, volume fraction of aggregates, etc.) equally complicates an identification of the most significant criteria and increases the growing number of campaigns as confirmed by the literature. Such a variability component probably explains the lack of modeling references in the field of raw earth building materials to date [4]. Authors are led to consider model materials in an attempt to understand the specific nature of these materials [5]. Meanwhile, few modeling studies have explored the possibility of predicting their insulating capacity from formulation data specifically. This study uses mean-field homogenization techniques (the Mori-Tanaka [6] and double inclusion schemes [7]) to offer a tool for predicting the thermal conductivity of these eco-materials.

In this sense, experimental work from the literature is rigorously selected to serve as a basis for the study. Two types of representative volume elements (RVE) are tested to assess the effective thermal conductivity of each of the materials, through a homogenization process. The aim is to identify the most relevant description in line with the actual morphology of the material (shape and orientation of the plant aggregates, cracking stage after the drying phase). Additionally, the lack of comprehensive porosity data requires further calibration to account for "global cracking". This concept includes both drying cracking in a plane perpendicular to compaction and binder/aggregate porosity. When air inclusions are considered in the RVE, they represent both the cracks within the matrix and the porosity at the matrix/aggregate interface. Particular attention is given to the accessibility and consistency of model input data, although the proportion of composite cracking remains unknown in the selected works. Consequently, this lack of data makes it necessary to formulate working hypotheses.

The proposed RVEs, which are consistent with the actual microstructure of the materials, make it possible to predict the thermal conductivity of rammed earth materials incorporating plant aggregates in a consistent manner. Thence, this study highlights the need for further work to assess the impact of changes in microstructure on the thermal performance of

materials. One of the identified goals is to be able to guide pre-production testing and thus contribute to optimizing the formulation of geo-sourced materials. Modeling also enables an exploration of the impact of other parameters such as aggregate shape or orientation. Thence, another goal is to provide modeling results to serve as a basis for discussion on the establishment of thermal conductivity measurement procedures for earth-based building materials that will result in a consensus in the scientific community.

2. Material Description

2.1. Lightweight Earths

Lightweight earths made from plant aggregates and raw earth binders offer interesting hygrothermal properties and are particularly environmentally friendly, given the local availability, recyclability, and ecological nature of their constituents [8,9]. They provide a very low-impact construction solution. Lightweight earths are manufactured by incorporating plant aggregates (e.g., agricultural by-products) into pre-wetted earth. This earth binder can be obtained from quarry fines from aggregate washing processes coming from the chemical or concrete industries [10]. Excavated earth can also be used directly on the construction site to produce earth-based building materials [11]. These materials are particularly heterogeneous due to the binding phase, which coats the plant aggregates to a greater or lesser degree. Additionally, the aggregates vary in shape and size (see Figure 1).

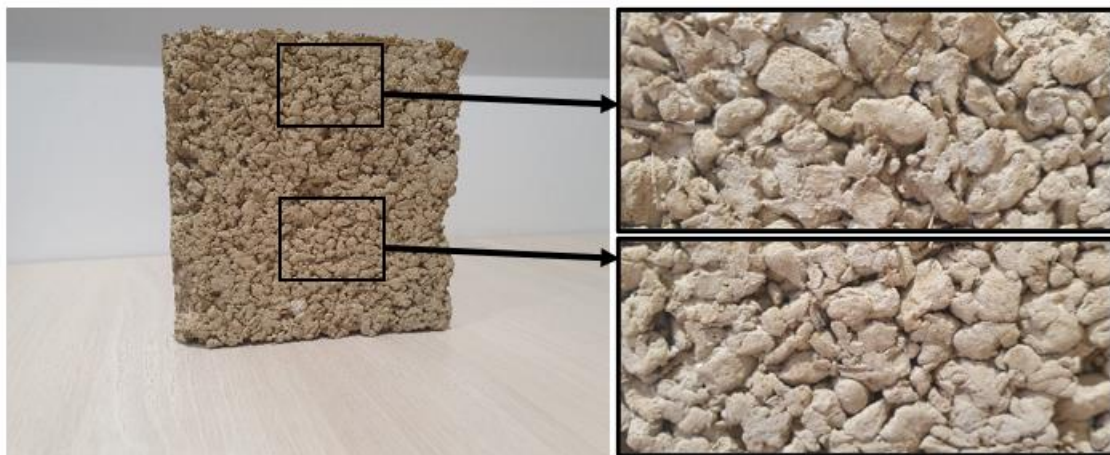


Figure 1. Picture (left) and several close-ups (right) of lightweight earth (sunflower pith in earth-based binder matrix).

Macroscopically, these materials can be described in two distinct ways. Depending on the formulation, the earth-based binder may be predominant with a few visible aggregates [10]. Alternatively, the aggregates are coated with earth and bonded together (see Figure 1).

2.2. Earth-Based Binder

A change in raw-earth binders could be used to predict the thermal behavior of composites so as to help optimize formulations. The aim is to explore how the intrinsic variability of earth can be taken into account on the scale of the resource (earth alone) to explore later on the composite scale. A study is therefore carried out on a wide range of earths and on their thermal conductivities, in an attempt to identify trends that could then be used in modeling.

2.3. Plant-Aggregates

The plant aggregates selected for this study in earth-based composites are hemp shiv, sunflower bark, and sunflower pith. Hemp shiv and sunflower bark exhibit macroscopic cylindrical shapes and tubular pores. The aspect ratios were determined through particle size analysis in the studies that provided the thermal conductivities of the composites. Otherwise, they were extracted by the same authors in preliminary works [10], ensuring consistent input data and mitigating the impact of resource variability [12].

The aspect ratio of hemp shiv is 3.3 as reported by Laborel-Préneron et al. (2018) [13], and the aspect ratio of sunflower bark is 3.4 according to Lagouin et al. (2019) [1]. Sunflower pith, characterized by honeycomb pores, is predominantly spherical (see as described by Lagouin et al. (2020) [14], Magniont et al. (2012) [15] and Arufe et al. (2021) [16]) at the macroscopic scale [17] with an aspect ratio very close to 1. The assumption of spherical inclusions for sunflower pith in mean-field homogenization was validated in previous research [18].

2.4. Earth-Based Materials Incorporating Plant Aggregates

Experimental data for lightweight earth are selected according to various criteria. Finding studies that provide all the necessary information for the homogenization approach proved challenging. The selected composites and the associated data are listed in Table 1.

Table 1. Earth composites selected for the study: experimental characteristics.

Earth Composite	Reference	Binder/Aggregate	Density (kg.m ⁻³)	Volume Fraction of the Aggregate	Porosity of the Composite (%)	Thermal Conductivity (W.m ⁻¹ .K ⁻¹)	Measurement Conditions
FH3-Laborel	Laborel Préneron et al. 2018 [8]	FWAS/Hemp shiv	1519 ± 38	0.22	/	0.30 ± 0.01	25 °C, dry state
FH6-Laborel		FWAS/Hemp shiv	1271 ± 16	0.37	/	0.20 ± 0.01	
CSP-Belayachi	Brouard et al. 2018 [19]	Clay/Sunflower Pith	235	/	90	0.055	Dry state
CSB-Belayachi	Belayachi et al. 2022 [20]	Clay/Sunflower Bark	714	/	64	0.158	

These materials are used as a basis for comparing the different modeling tools mentioned in Section 3. It is worth noting that the aggregate volume fraction in the study by Laborel-Préneron et al. [8] is determined under the assumption of a binder matrix containing aggregates free from additional porosity. Indeed, these values can be found using the density of the washing fines (binder) and the density and the mass proportion of plant aggregates of each composite. However, the nature of the porosity reported in the work of Belayachi et al. [20] is not specified. For the sunflower pith and clay composite, given its very high value, total porosity (including intra-granular and inter-granular) was assumed.

Furthermore, in order to link the thermal conductivity of the stabilized material to its formulation, i.e., the material in its fresh state, it is valuable to specify the volume fraction of each component. The literature provides information on formulations, summarized in Table 2.

Table 2. Formulation of selected earth-based composites from Labore-Préneron et al. [10] and Brouard et al. [19].

Lightweight Earth	Binder (%)	Aggregate (%)	Water (%)
FH3-Laborel	/	3	17
FH6-Laborel	/	6	20
	Binder/aggregate weight ratio	Water/binder weight ratio	Aggregate/clay volume ratio
CSP-Belayachi	2.4	1	26
CSB-Belayachi	2.4	1	4.7

It is worth noting that authors use varying terminology when discussing formulation, complicating the extraction of input data for modeling purposes. Initially, aggregate volume fractions, calculated from density and mass proportions, did not take additional porosity into account. Subsequently, a 10% porosity was considered to account for the cracking post-drying phase with an adequate preferential orientation. This adjustment is based on literature indicating a high cohesion of these materials [21].

Finally, when applying the double inclusion model, the volume fraction of the coating part becomes a critical input. Though no specific data are available in the literature, a credible value is set at 20%. The percentage of coating is adjusted in the modeling process.

3. Methodology

3.1. Mean-Field Homogenization (MFH) Methods: Double-Inclusion and Mori-Tanaka Models

To determine the effective thermal conductivity tensor, mean-field theories utilize concentration tensors that relate the averaged fields at the microstructure level (i.e., inclusions and matrix) with the corresponding macroscopic fields. The double-inclusion (D-I) model, based on the theory by Hori and Nemat-Nasser [7], is employed. This model assumes that each inclusion in each phase is surrounded by a hollow inclusion embedded in an outer matrix. Additionally, it is assumed that both inclusions share altogether the same aspect ratio, symmetry axis, center, and volume ratio as the inclusions and matrix in the composite.

By setting the coating phase with the same properties as the matrix, the Mori-Tanaka (M-T) model can be derived from the double-inclusion model. The analytical approach presented here—via the Mori-Tanaka and double inclusion models—is a well-known technique of mean-field homogenization. It is usually applied in disciplines other than earth-based building materials [16–18]. Although such a technique is generally used for elasticity tensor, it can be efficiently transposed to the effective conductivity tensor (refer to its relevance in numerous works [18–22].)

The M-T model is based on an approximate use of Eshelby’s solution. Inclusions are embedded in a matrix. Each inclusion is surrounded by a region of the matrix with the interaction between the inclusions and the matrix being averaged. This relationship can be expressed through the following equations [22] :

$$\underline{\underline{\Lambda}}_{hom} = \sum_{r=1}^N f_r \underline{\underline{\Lambda}}_r \cdot \underline{\underline{\Lambda}}_r^{MT} \text{ with } \underline{\underline{\Lambda}}_r^{MT} = \underline{\underline{\Lambda}}_r^O \cdot \left(\sum_{r=0}^n f_i \cdot \underline{\underline{\Lambda}}_r^O \right)^{-1} \quad (1)$$

where $\underline{\underline{\lambda}}_{hom}$ is the effective thermal conductivity tensor of the representative volume element (RVE), $\underline{\underline{\lambda}}_r$ is the thermal conductivity tensors of the r-th phase (matrix or inclusion), f_r is the volume fraction of the r-th phase, and $\underline{\underline{A}}_r$ is the concentration tensor of the r-th phase. The concentration tensor is noted $\underline{\underline{A}}_0^0$ for the matrix, and it is equal to the $\underline{\underline{\delta}}$ Kronecker symbol. The inclusions are supposed to have identical properties: their concentration tensor $\underline{\underline{A}}_r^0$ is determined as follows:

$$\underline{\underline{A}}_r^0 = \left[\underline{\underline{\delta}} + \underline{\underline{P}}_r \cdot \left(\underline{\underline{\lambda}}_i - \underline{\underline{\lambda}}_0 \right) \right]^{-1} \quad (2)$$

where $\underline{\underline{P}}_r$ is the second-order interaction tensor and $\underline{\underline{\lambda}}_i$ and $\underline{\underline{\lambda}}_0$ are, respectively, the thermal conductivity tensor of the i inclusion and of the 0 matrix.

The inclusion shape is taken into account thanks to the $\underline{\underline{S}}_r$ depolarization tensor which is linked to $\underline{\underline{P}}_r$ through the relationship :

$$\underline{\underline{P}}_r = \underline{\underline{S}}_r \cdot \left(\underline{\underline{\lambda}}_0 \right)^{-1} \quad (3)$$

3.2. Challenges in Homogenization

One of the challenges of homogenization deals with heterogeneous materials with random microstructures. The arrangement of phases is not perfectly known; the microstructure of geo-based building materials is particularly complex, featuring multiscale porosities [23]. Information on phase arrangement at the microscopic level can only be provided through the volume fractions f_r of the constituents as follows [24]:

$$f_r = \frac{V_r}{V_{tot}} \text{ with } V_{tot} = \sum_r^N V_r + V_m \quad (4)$$

where V_r and V_m are, respectively, the volumes of inclusions and of the matrix in the total volume V_{tot} of the RVE.

The application of homogenization models onto the materials considered here is no trivial matter. However, in the literature, material formulations typically only specify the mass relationships between the constituents. This poses a significant challenge in determining the input data for homogenization and, consequently, in calculating the effective thermal conductivity tensor. To address this point, a correspondence between mass ratios and volume ratios can be explored, using X-ray tomography images [25]. In the absence of information for earth-building materials, assumptions about volume fractions at the material scale are necessary, followed by calibration. At the particle scale, previously validated methods are used to account for relevant volume fractions [26], such as intra-particle porosity [27,28].

3.3. Particulate Orientation

The concentration tensor in Equation (1) depends on a Hill tensor, which takes the variability of the microstructure in terms of the orientation and the shape of the inclusions into account [28].

In the analytical approach proposed here, the orientation of aggregates in composites can be characterized by two angles, with Θ the angle between the primary axis of the inclusion and the Z-axis in global coordinates and ϕ the angle between the projection of the inclusion onto the XY plane and the Y-axis. Image analysis based on microstructure data could provide

information on the actual orientation of particles in the composites studied. While some work on plant-based concretes has produced initial results [14], such data are not available for lightweight earth. Due to this lack of precise data on the orientation of aggregates within the studied materials, this study adopts two key assumptions.

First, it assumes a fully random orientation of aggregates in all spatial directions, named the “random 3D” assumption as if the implementation technique was free on any no impact on the orientation of the inclusions. Secondly, it assumes that the compaction process during placement results in a random orientation of aggregates confined to the *XY* plane, termed the “random 2D” assumption. The high compaction rate of the techniques used may support this second hypothesis [10].

3.4. Particulate Thermal Conductivity tensor

The effective thermal conductivity, influenced by the anisotropy of each RVE, is described by a tensor with components as follows:

$$\underline{\underline{\lambda}}_{EB} = \begin{pmatrix} \lambda_i^{EB} & 0 & 0 \\ 0 & \lambda_j^{EB} & 0 \\ 0 & 0 & \lambda_k^{EB} \end{pmatrix} \quad (5)$$

These components may vary depending on the orientation of particles, which is significant in the case of earth-based composites. The manufacturing process can influence the particles’ preferred orientation and therefore the properties of the final material [10]. Thus, in order to evaluate the impact of particle shape and orientation on the effective thermal conductivity of the composite, plant aggregates are considered as anisotropic inclusions in this study.

3.5. Representative Volume Element (RVE)

Each constitutive model is based on an RVE to conduct the homogenization process. Consequently, each RVE is carefully defined to take the specific characteristics of lightweight earths into account, in particular the development of cracks perpendicular to compaction after drying [10]. Homogenization enables the determination of effective thermal conductivity tensors, depending on the calculation assumptions. It should be pointed out that various RVE configurations can be considered by drawing inspiration from both the macro- and microstructure of materials.

First, under the assumption of a low volume fraction of aggregates, the earth matrix contains aggregates as inclusions, with no additional porosity apart from particulate and binder intrinsic porosities. This type of RVE is designated as “EB-1”.

In the manufacturing process of lightweight earth, the literature reports the occurrence of cracks perpendicular to compaction after compaction and drying. Cracking in a stabilized state is taken into account by spheroidal air inclusions with an aspect ratio of 20 for consistency (5 to 15 times longer than aggregates), randomly oriented in the plane perpendicular to compaction [10]. This type of RVE is referred to as “EB-2”.

Finally, plant particles may appear to be simply embedded within the earth matrix, in a relatively random arrangement. In this context, an RVE describing coated particles bathed in an air matrix looks relevant as well (see Figure 1) This latter case is noted as “HB-3”. Figure 2 provides further illustration of the various models defined in this study.

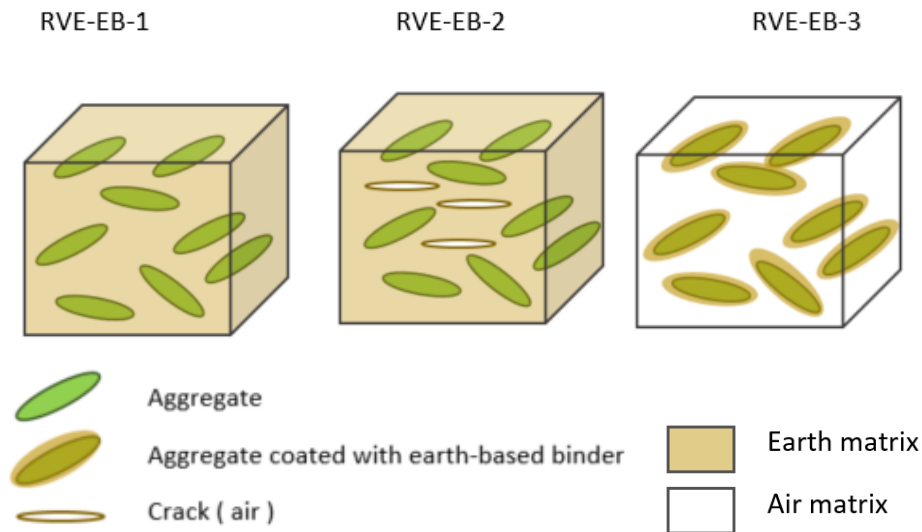


Figure 2. Different types of RVE considered for the analytical homogenization of earth-based materials.

The Mori-Tanaka scheme is applied in the RVE-EB-1 and the RVE-EB-2 patterns. The double inclusion scheme is used for the RVE-EB-3 structure. The two analytical approaches are based on different homogenization assumptions (cf. Section 3.1). In addition, the representative volume elements are significantly different. In the Mori-Tanaka scheme, the binding matrix is represented by earth. In the case of the double inclusion scheme the matrix enclosing the embedded inclusions is made up of air.

3.6. Overall Study Strategy

In order to clarify the overall approach carried out in this study, a flowchart has been designed (Figure 3). It highlights three main steps: the input data determination for the homogenization process, the model validation, and the optimization phase according to different criteria.

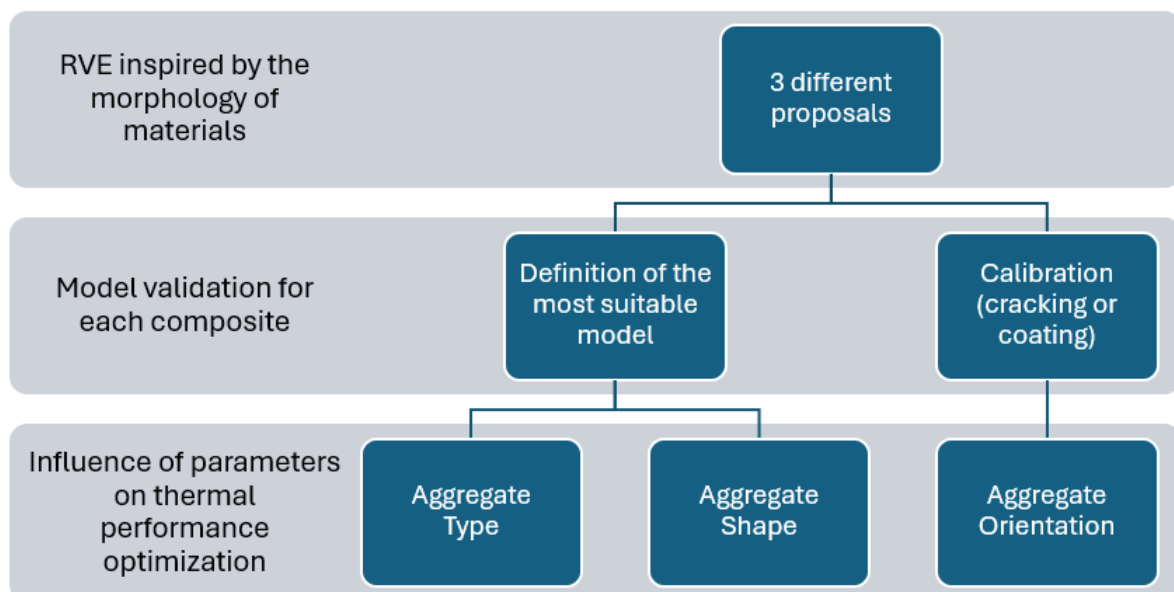


Figure 3. Flowchart of the methodology used in this study.

4. Application, Validation and Optimization

4.1. Modeling Input Data

4.1.1. Particulate Thermal Conductivity

Thanks to previous work [26,27], particulate thermal conductivity can be determined by both the density of the bulk particles and the thermal conductivity value measured on the same bulk particles. Since the required data are available in the selected references, each component of the particle thermal conductivity tensors is calculated. Hence, these dry reference values at the temperature of 20 °C are selected as reference values. If needed, corrections are made according to the experimental conditions of the thermal conductivity measurements on composites since thermal conductivity depends on temperature and relative humidity. The particulate values calculated and used as input data in the homogenization process are listed in Table 3.

Table 3. Particulate thermal conductivities values calculated under 20 °C and in a dry state.

Reference	Type of Aggregate	Calculation Method in Reference to Rosa Latapie et al., 2023 [27–31]	Modeled Particulate Thermal Conductivity (W.m ⁻¹ .K ⁻¹)	
			λ_T	λ_N
Laborel-Préneron et al., 2018 [10]	Hemp shiv	From measurement on bulk particles	0.044	0.066
Brouard et al., 2022 [19]	Sunflower pith		$\lambda_{iso} = 0.08$	
	Sunflower bark	0.045	0.068	

4.1.2. Binder Thermal Conductivity

A correlation is commonly highlighted in the literature between the density of a bio-sourced material and its thermal conductivity [14]. The purpose of this section is to explore whether this trend is confirmed on the specific scale of earth-based binders. A compilation of literature data [8,32–38] clearly demonstrates that no trend emerges between thermal conductivity and material density (Figure 4). It is therefore necessary to systematically measure the thermal conductivity of the earth material alone used as binder, in order to have this input data for modeling.

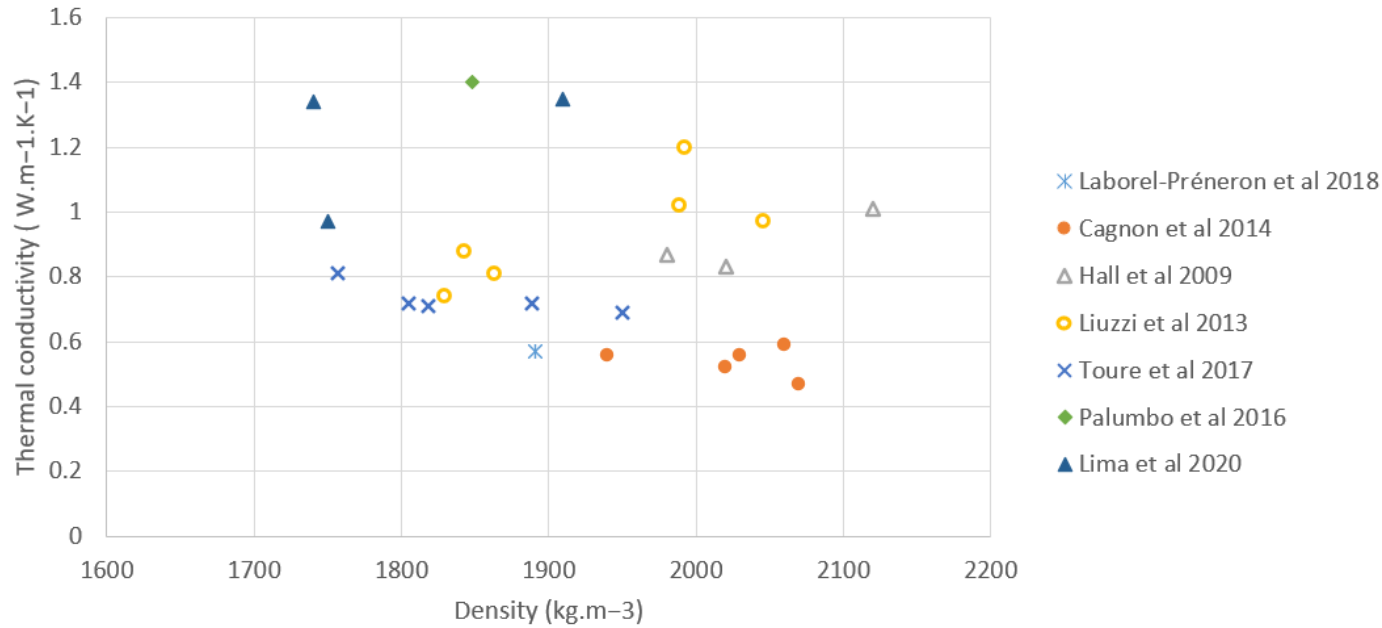


Figure 4. Thermal conductivity of an earth-based binder as a function of its density [29], [30], [31], [32], [33], [34]

These observations are consistent with various studies that have highlighted the significant variability of earth materials (constituents, clay content, etc.) and the difficulty of predicting their thermal conductivity [39,40].

Thereafter, it is assumed that any change in an earth-based binder requires a thermal conductivity measurement before prediction and optimization. Consequently, changes to raw earth binders are therefore not considered in this study. The original earth binders are maintained for the optimization phase.

4.1.3. Volume Fractions

The particulate thermal conductivities determined in the dry state are not corrected (cf. Table 3) since the experimental thermal conductivity measurements are taken on pre-dried composites. The thermal conductivity of the binder is considered constant, although the literature reports sorption—particularly in raw earth materials—and consequent changes in thermal conductivity [34]. Finally, the volume fractions of the aggregates are either extracted from the experimental study [8] or calculated from the mass ratios and densities of the constituents, initially assuming no additional porosity within the final composite [20].

In the case of lightweight earth, the input data—required for the various homogenization calculations carried out in this study—are listed in Table 4.

Table 4. Input data common to modeling for earth-based composites at a dry state and at a temperature of below 25 °C.

Composite	Earth-Based Binder			Aggregate		Volume Fraction	Aspect Ratio
	Density (kg.m ⁻³)	Thermal Conductivity (W.m ⁻¹ .K ⁻¹)	Particulate Density (kg.m ⁻³)	Particulate Thermal Conductivity (W.m ⁻¹ .K ⁻¹)			
				λ_T	λ_N		
FH3-Laborel	1891 (FWAS)	0.57 ± 0.03	248	0.044	0.066	0.22	3.3
FH6-Laborel						0.37	
CSP-Belayachi	900 (clay)	0.27	56	$\lambda_N = \lambda_T = 0.08$ or 0.04		0.79	1
CSB-Belayachi			425	0.045	0.068	0.39	3.4

4.2. Validation

4.2.1. Fine-Based Composites

- FH3-Laborel composite

For the first earth-based building material reference modeled in this study, referred to as HF3-Laborel, the results of the analytical homogenizations are gathered in Figure 5.

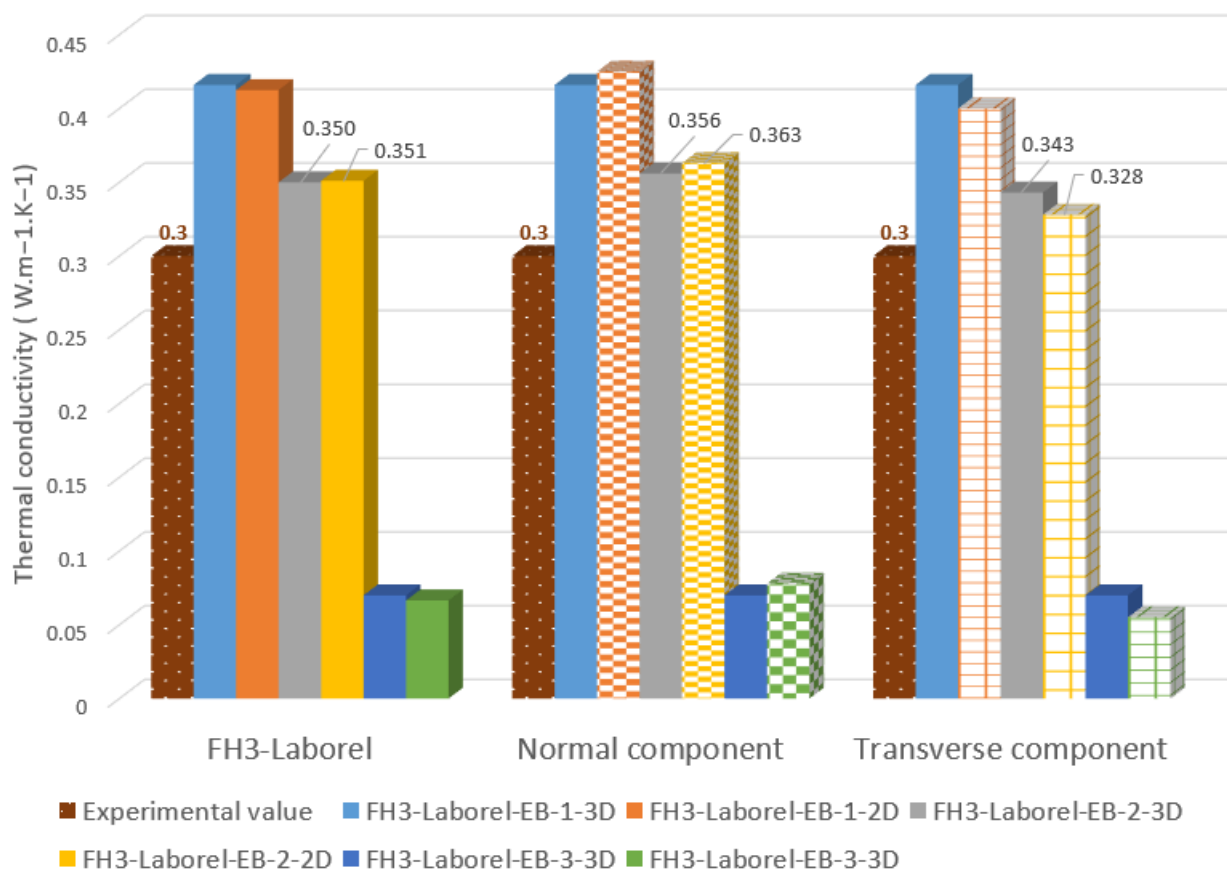


Figure 5. Results of predicted values concerning FH3-Laborel composite compared to the experimental value.

Several aspects can be highlighted in these results: Models using the double inclusion scheme give inconsistent results, diverging from the experimental value by almost 80%. Avoiding the

cracking after drying stages results in an overestimation of the experimental value by around 40%.

Moreover, the most appropriate model is undoubtedly the one that considers cracks in a plane perpendicular to compaction, with a deviation of around 15% from the target value. Consequently, the FH3-Laborel-3 model is retained for the rest of the study.

It is noted that the difference between 3D and 2D models is insignificant. The percentage of cracking in this case was initially set arbitrarily at 10%.

The following step is to refine this percentage to calibrate the model from experimental data. It was carried out by considering the average of the components given by the homogenization process to simplify the comparison with the experimental single scalar value (Figure 6).

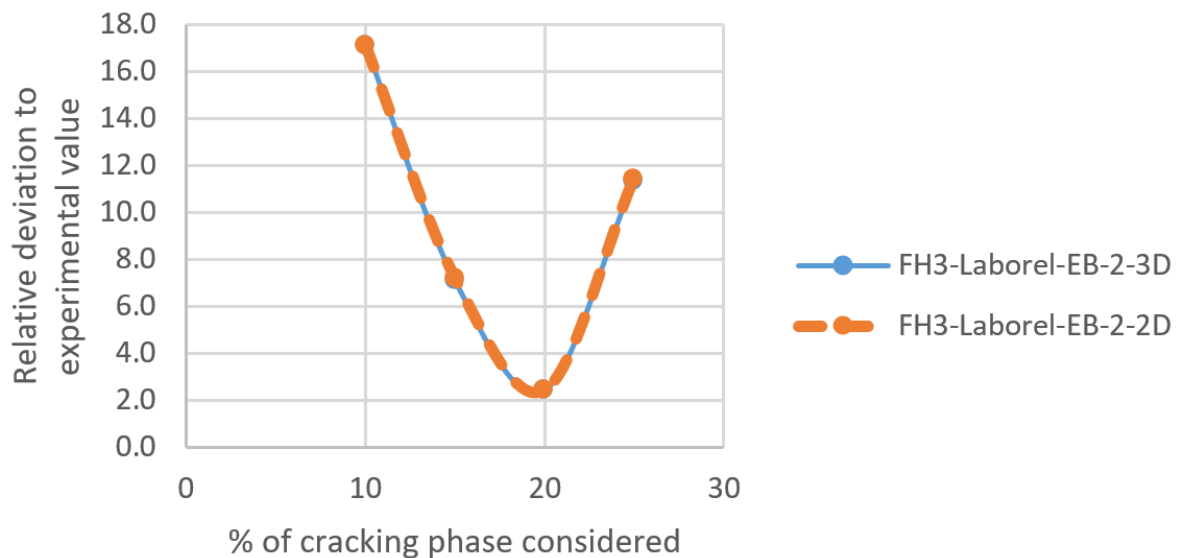


Figure 6. Calibration of cracking percentage in FH3-Laborel-EB-2 type models.

The graph in Figure 6 reveals that with an unassuming cracking percentage of 18%, the model predicts a value within 2% of the experimental one. Thence, this calibrated percentage is adopted for subsequent analysis.

Interestingly enough, the orientation of particles, whether considered or not, has a minimal impact on the model's predicted value, likely due to the cracking's predominant influence perpendicular to compaction. The influence of plant aggregate orientation would therefore be negligible when considering the volume fractions of the actual composite. The FH3-Laborel-EB-2 model is thus retained for this composite to further study the subsequent impact of particle orientation and aspect ratio.

- FH6-Laborel composite

For the FH6-Laborel composite, in which the mass proportion of hemp shiv is twice that of the FH3 composite, the homogenization results also demonstrate the beneficial impact of including cracking on the Mori-Tanaka model (Figure 7).

The key findings include:

- Models only considering the raw earth matrix and aggregates overestimate the effective thermal conductivity of lightweight earth by almost 50%.

- Models based on the double inclusion scheme underestimate the experimental value by an average of 60%.

The most relevant model is clearly the EB-2 model, with a relative deviation from the experimental value of around 20%.

As in the previous section, the adjustment of the volume fraction of air in the composite (i.e., cracking) is necessary before using this model further in the study (Figure 8).

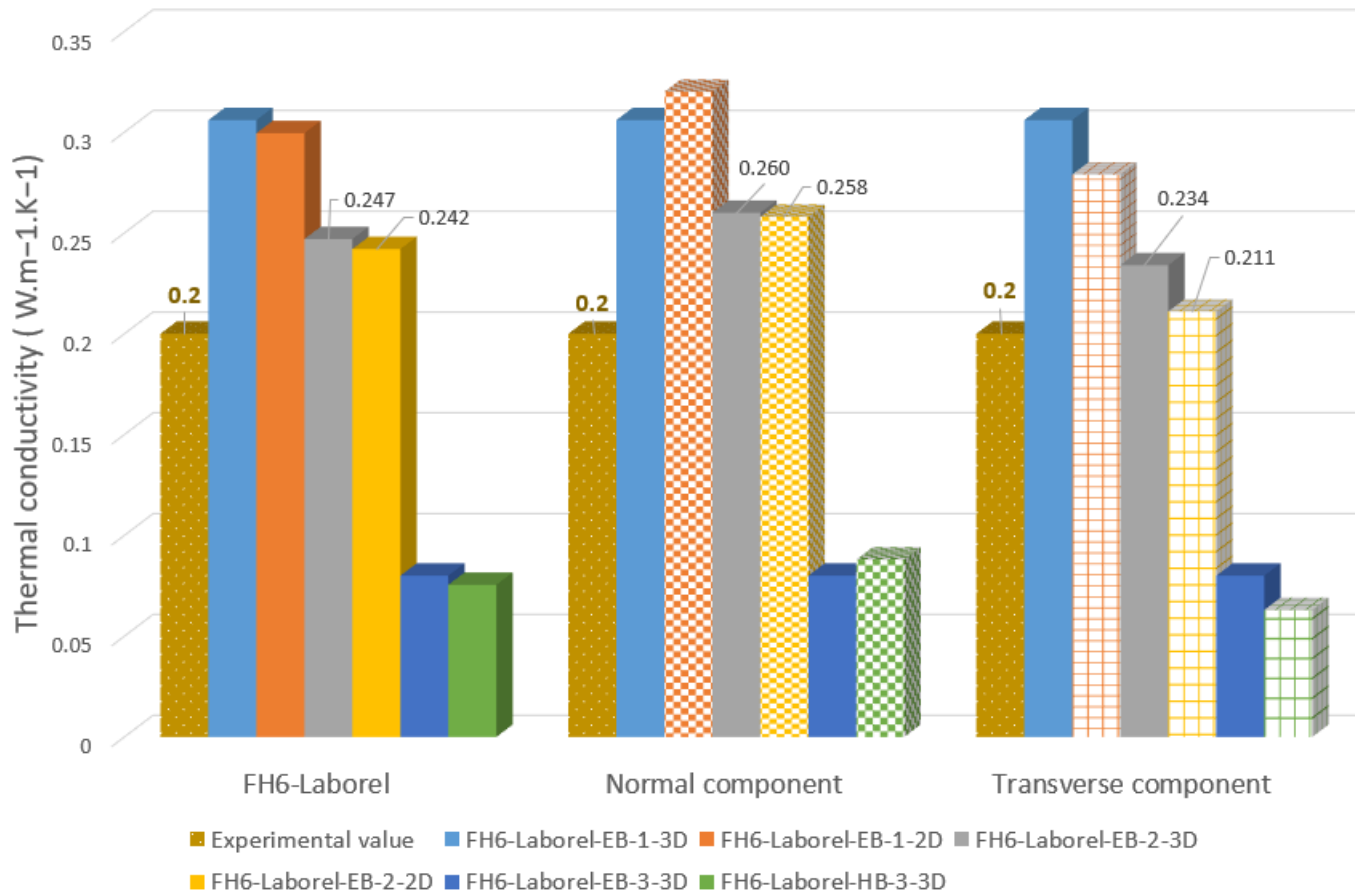


Figure 7. Results of predicted values concerning the FH6-Laborel composite compared to the experimental value.

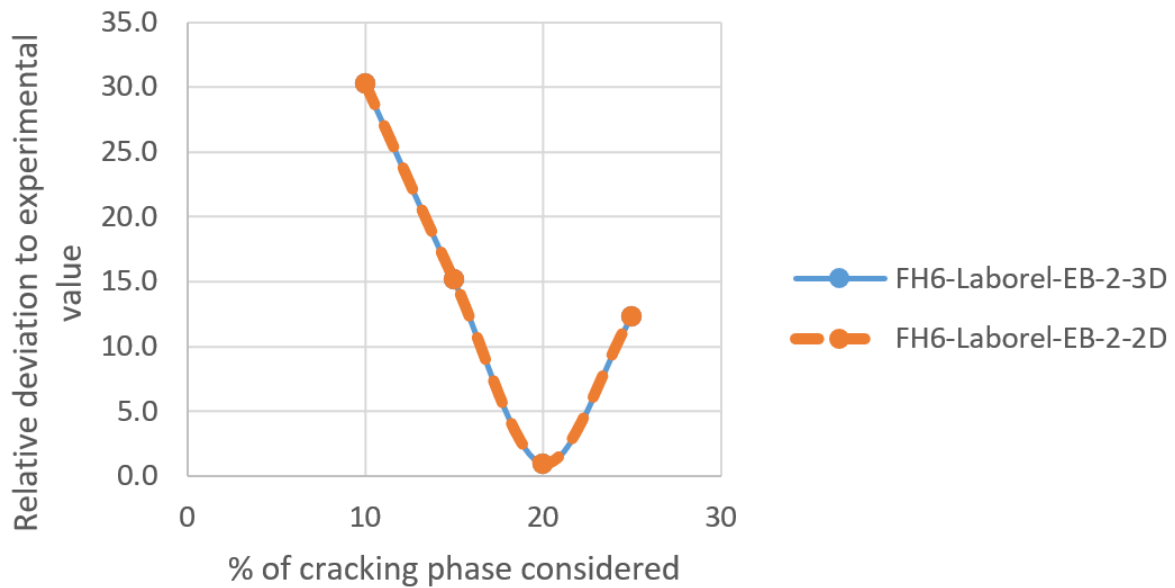


Figure 8. Calibration of cracking percentage in FH6-Laborel-EB-2 type models.

Assuming a percentage of 24% cracking, Figure 8 reveals that the model predicts a value within 1% of the experimental value. Consequently, this calibrated value is adopted, and the FH6-Laborel-EB-2 model is selected to represent the FH6-Laborel composite.

With the calibration system, it is worth noting that with calibration a slightly higher cracking value is obtained in the case of the FH 6 composite due to its higher aggregate content. That element may potentially affect air space due to binder matrix bridging observed in some instances [37].

4.2.2. Clay-Based Composites

- CSP-Belayachi

In the case of sunflower pith composites, the components of the thermal conductivity tensor are strictly equal for the EB-1 and EB-3 models due to particle isotropy. The consideration of cracking in the case of EB-2 induces a slight anisotropy which is neglected. The values of the normal and tangential components differ by less than 5%. Furthermore, as discussed in the section, the two particulate values of 0.04 and $0.08 \text{ W.m}^{-1}\text{.K}^{-1}$ have been tested in the modeling. The predicted values of the different models are compared with the experimental one in Figure 9.

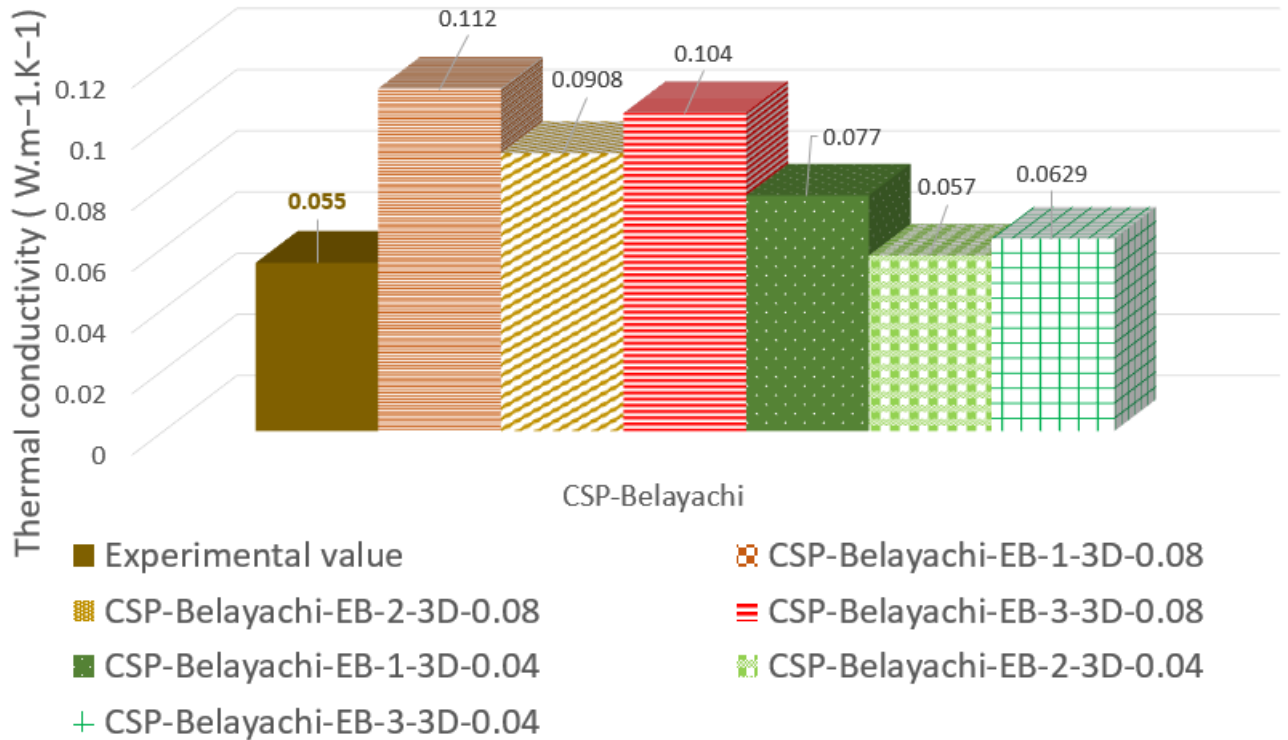


Figure 9. Results of predicted values concerning the CSP-Belayachi composite compared to the experimental value.

Regardless of the model considered, taking into account the particle value of $0.08 \text{ W.m}^{-1}.\text{K}^{-1}$ systematically overestimates the experimental value by more than 50%. The value of $0.04 \text{ W.m}^{-1}.\text{K}^{-1}$ is adopted for the rest of the study as it is considered more relevant in light of these results along with previous work [18]. The EB-1 type model overestimates the experimental value by 40% and is therefore considered unsuitable. Meanwhile, The EB-2 and EB-3 models—based, respectively, on the Mori-Tanaka Model (with the cracking phase) and the double inclusion model—approach the experimental value to less than 15%. Both types of models appear relevant. It may be difficult at this stage to consider any value more valid than the other. Given the lack of data available in the literature, both include arbitrary but plausible values. As a reminder, type EB-2 uses a cracking value of 10%, whereas type EB-3 uses a coating proportion of 20%. Insofar as the aim of the developed models is to best reflect the morphology of the material studied, the double inclusion model is adopted, and the proportion of coating adjusted (Figure 10). Considering the volume fraction of plant aggregates as being close to 80%, the CSP-Belayachi composite appears to be closer to a description in which the coated aggregates are immersed in an air matrix.

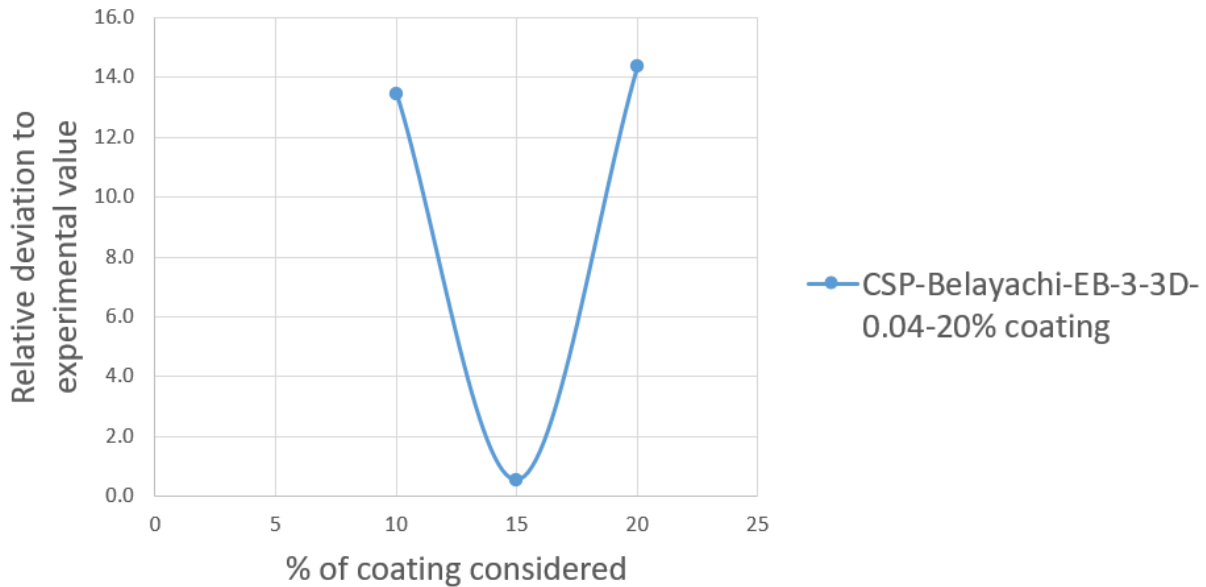


Figure 10. Calibration of coating percentage in CSP-Belayachi-EB-3-3D model.

In the following sections, particle coating is estimated at 20% for the CSP-Belayachi composite as it provides a deviation between model and experimental values of less than 1%.

- CSB-Belayachi

The results of thermal conductivity prediction for the CSB-Belayachi composite are presented in Figure 11.

For this composite, the EB-3 type models underestimate the experimental value by over 60%. They are consequently rejected. The EB-1 model, which takes no cracking into account, overestimates the experimental value by 6%. Furthermore, the measurement uncertainty is not specified in the selected references [13,14], but this difference could probably be included in it. A cracking adjustment on the EB-2 model shows that an error of $\pm 1\%$ is reached between the modeled value and the experimental value when considering a cracking of 3% instead of 10% (initial value arbitrarily set). This point is illustrated in Figure 12.

These results demonstrate the consistency of the two types of modeling and the possibility for the given composite to neglect the cracking phenomenon that appears, to a certain extent, after drying. Hereafter, the authors have agreed to adopt the model offering a predicted value closest to that measured one. For this reason, the EB-2-2D model with 3% cracking has been retained for the CSB-Belayachi composite.

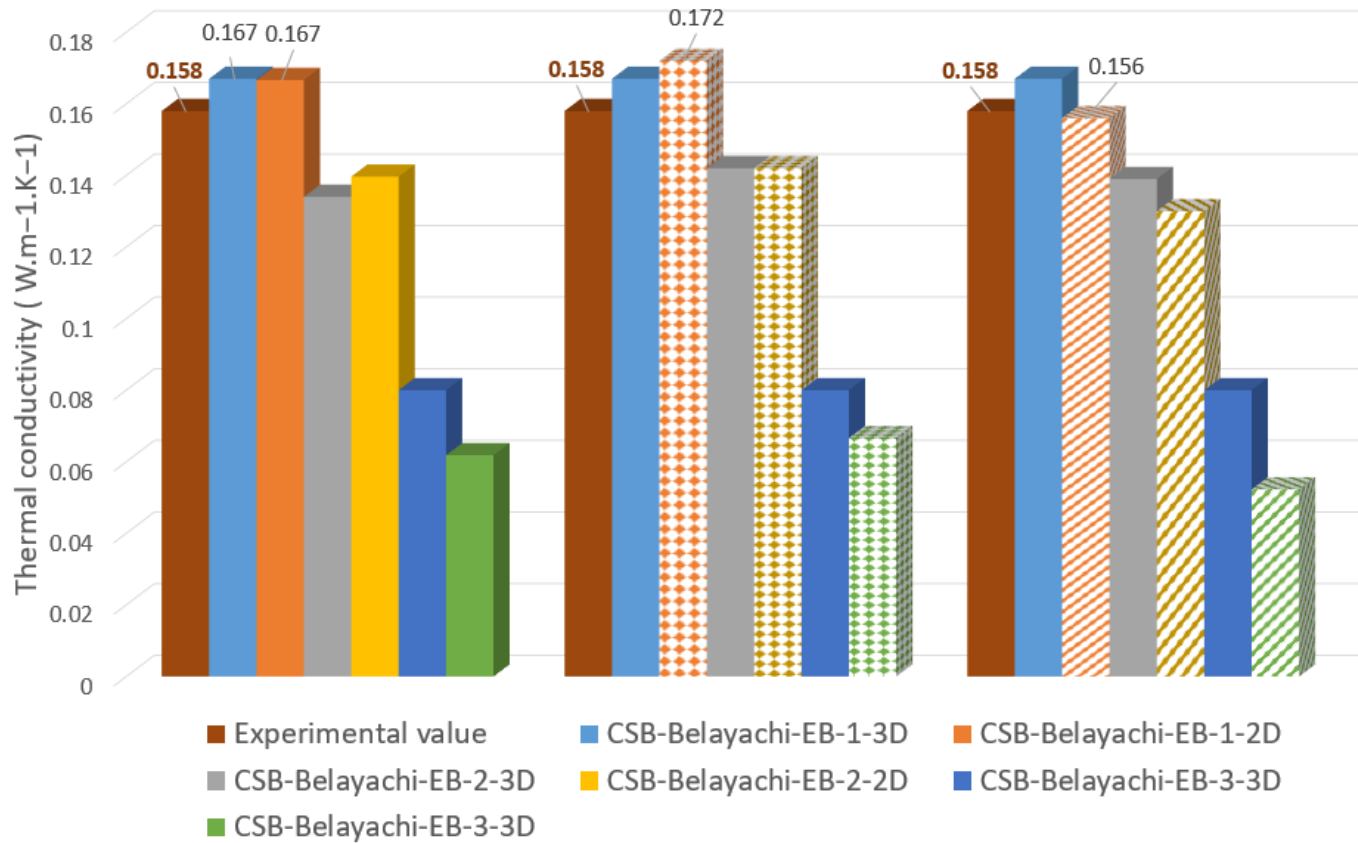


Figure 11. Results of predicted values concerning the CSB-Belayachi composite compared to the experimental value.

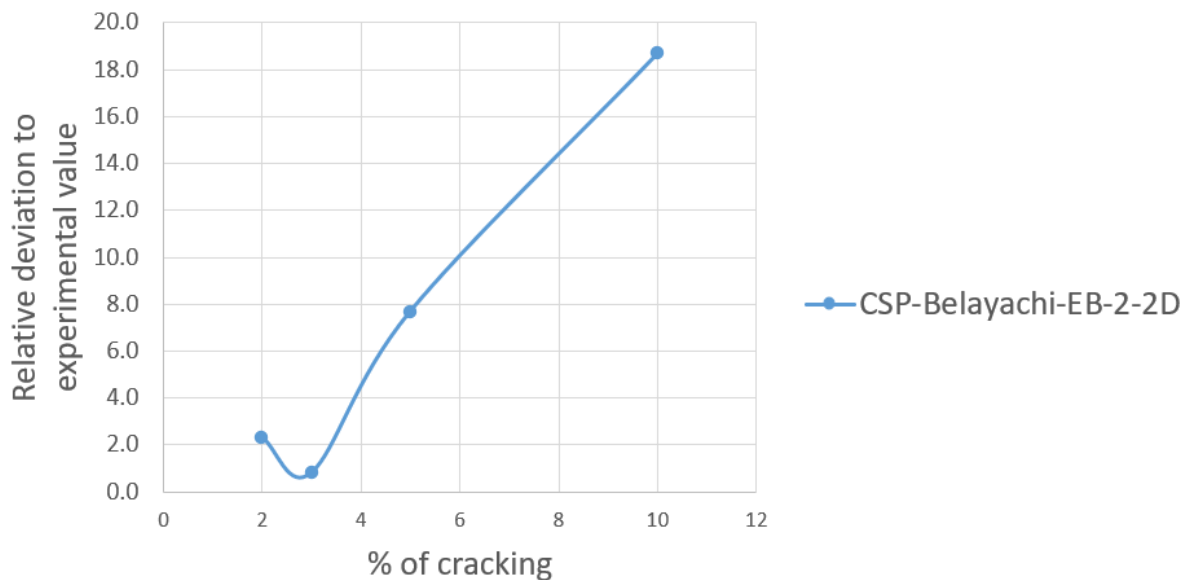


Figure 12. Calibration of cracking percentage in CSB-Belayachi-EB-2- models.

4.3. Optimization Criteria

To optimize the effective thermal conductivity, this section will be investigating three specific parameters that can influence this property.

4.3.1. Influence of Aggregate Type on Thermal Properties

The use of an analytical homogenization approach to investigate how different aggregates influence composite materials is favored. By incorporating sunflower pith, sunflower bark, or hemp shiv, while keeping other parameters constant—such as formulation and processing conditions—the study aims to optimize the material formulation. Undeniably, the impact of these aggregates on effective thermal properties is crucial for enhancing the performance and efficiency of the composite material.

4.3.2. Influence of Aspect Ratio Variation on Thermal Conductivity

In the modeling process, the particle aspect ratio is fixed in the first instance. The input values are those specified in Section 2.2. Due to their natural origin and extraction methods, aggregates actually come in a wide range of sizes; this is extensively documented by Granulometric analyses in the literature [40–44]. However, for optimization purposes, it is crucial to examine the impact of the various aspect ratios of the aggregates.

Selecting plant aggregates with uniform shapes for experimental studies is particularly challenging. Indeed, modeling enables the investigation of how plant particle aspect ratios affect thermal properties. Within this scope, the range of aspect ratios selected for this study aligns with data reported in the literature. For instance, hemp shiv has an aspect ratio ranging from 2.28 to 8.75, sunflower bark ranges from 2.99 to 4.74, and sunflower pith varies between 1.00 and 1.50 (Ratsimbazafy et al., 2021 [17]).

4.3.3. Influence of Preferential Orientation on Thermal Behavior

Due to the anisotropic thermal behavior of plant particles, the shape of the aggregate necessarily influences the effective thermal conductivity of the resulting geo-sourced composite. In addition, the preferential orientation of these particles within the material—whether due to compaction or spray application [38,39]—adds to the phenomenon and probably amplifies or compensates for the inherent anisotropy of the particles.

Regarding particle orientation, various cases are tested, ranging from random orientations to the extreme case of the perfect alignment of all particles in the same direction. It is important to note that while perfect alignment does not correspond to any practical implementation seen to date, exploring this extreme case allows for a more comprehensive discussion of the material's thermal behavior.

5. Results

5.1. Initial Modeling

In each study within this section, only the calibrated model appropriate for each composite is utilized. When particle orientation is not specifically examined, a random distribution is adopted if it results in a smaller relative deviation from the experimental value compared to orientation in a plane. To investigate the effect of changing aggregates (either in nature or shape factor), it is assumed that all other modeling parameters remain constant. During the optimization process, the reference thermal conductivity, denoted as $\lambda_{model}^{composite}$, is determined by the modeling results. The thermal conductivities associated with selected models and their calibration values are detailed in Table 5.

Table 5. Recapitulation of the models chosen for each composite and the reference thermal conductivity values before optimization.

Composite	FH3-Laborel	FH6-Laborel	CSP-Belayachi	CSB-Belayachi
Model type	EB-2-2D	EB-2-2D	EB-3-3D	EB-1-2D
Calibrated value	18% cracking	24% cracking	15% coating	/
$\lambda_{\text{composite}}$ [W.m ⁻¹ .K ⁻¹]	0.3	0.2	0.055	0.158
$\lambda_{\text{model}}^{\text{composite}}$ [W.m ⁻¹ .K ⁻¹]	0.304	0.201	0.055	0.159
Relative deviation	3%	1%	1%	1%

Calibration allows for a consistent prediction of modeled thermal conductivity to below 5% of the experimentally measured value. Based on this value, simulations are conducted by varying several criteria: the type of plant aggregate, the aggregate shape factor, and the aggregate orientation.

5.2. Influence of Aggregate Type on Thermal Conductivity

Simulation results allow for assessing the impact of each composite's substituted aggregate on thermal conductivity (Figure 13). For instance, in a composite, hemp shiv is replaced by sunflower pith or sunflower bark, as described in Section 4.3.1. The composites are abbreviated with letters here below: H for hemp shiv, SP for sunflower pith, and SB for sunflower bark.

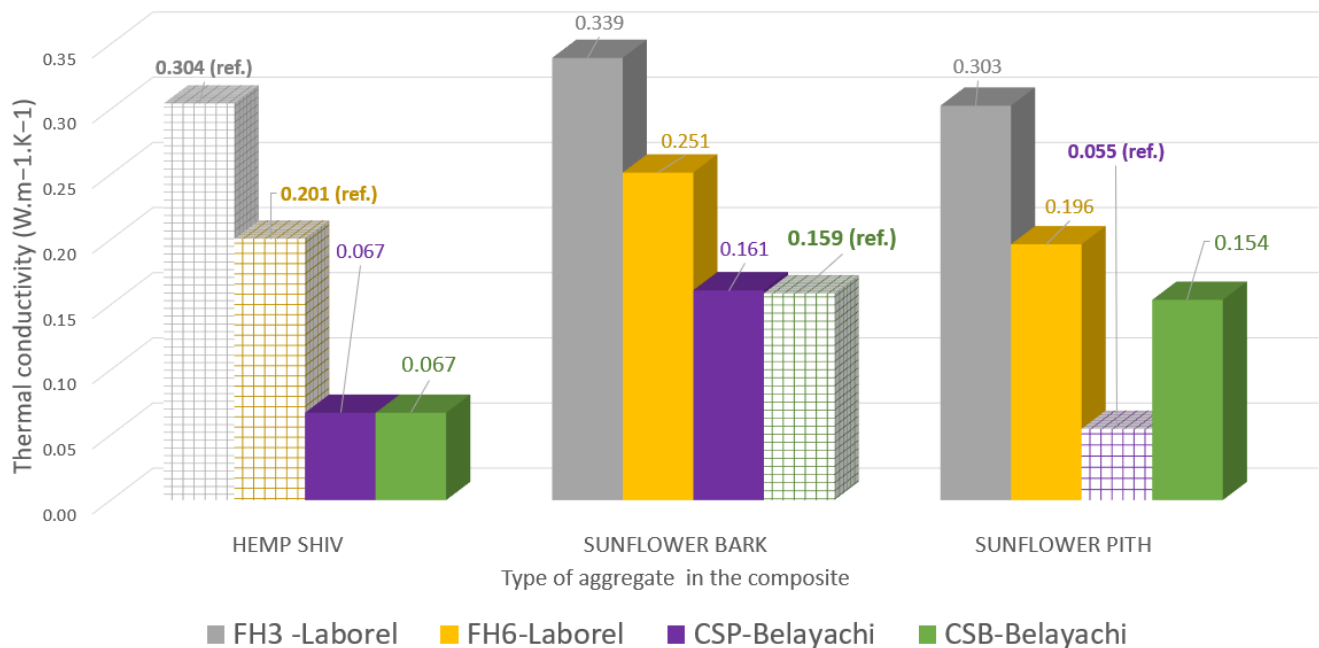


Figure 13. Impact of a plant aggregate change in lightweight earth.

The sunflower pith stands out as the optimal plant-based aggregate for thermal performance across all composite types due to its consistently low particulate thermal conductivity. Conversely, composites incorporating sunflower bark exhibit the lowest thermal efficiency, which is attributed to its significantly higher particulate thermal conductivity compared to hemp shiv and sunflower pith.

In FH-Laborel composites, substituting hemp shiv with sunflower pith does not notably alter thermal conductivity. This is likely due to the similar particulate thermal conductivities of the aggregates and the relatively low aggregate volume fractions in FH3 and FH6 composites (less than 50%). However, if sunflower bark is used, thermal conductivity increases by 10% in FH3 and 20% in FH6, consistent with the higher mass fraction in FH6.

5.3. Effect of Aggregate Shape on Thermal Conductivity

Due to the variability in resources, the literature reports different aspect ratio values for a given type of plant aggregate [17]. Therefore, assuming a uniform particulate thermal conductivity, the aspect ratios of aggregates in each composite are adjusted across the range documented in the literature (see Section 2.2). These aspect ratios are input data for the homogenization process. The aim is to examine the impact of this variation on each composite's effective thermal conductivity. The outcomes of these homogenization calculations are depicted in Figure 14.

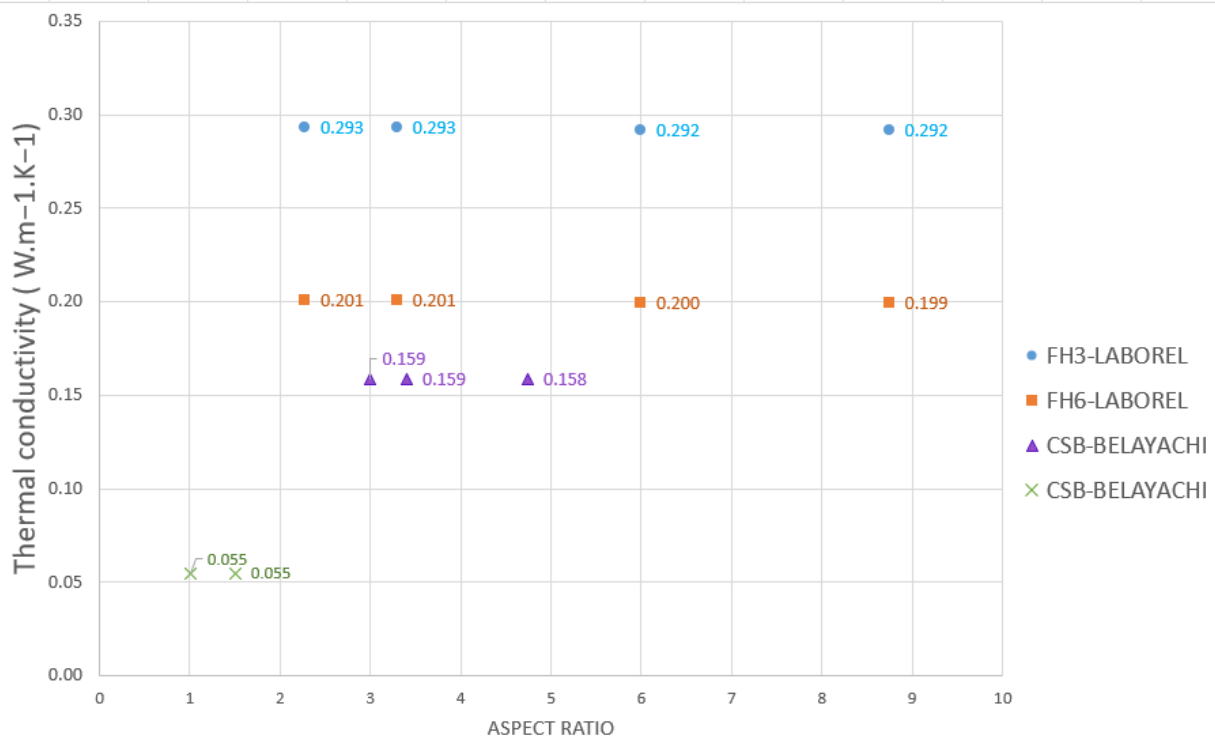


Figure 14. Impact of an aggregate aspect ratio change in modeled composites.

Based on our homogenization calculations across the selected composites, the aspect ratio can influence thermal conductivity notably when a wide range of values prevail due to variability in raw materials. However, in comparison to measurement uncertainties, this influence is generally negligible regarding the thermal conductivity of the composites under study. It is substantial to highlight that our modeling assumed fixed aspect ratios, whereas in reality plant aggregates exhibit a specific size distribution, which can be quantified through particle size analysis [40]. This study could therefore be extended by incorporating the results of a granulometric analysis of aggregates used in lightweight earth materials.

5.4. Impact of Aggregate Orientation on Thermal Conductivity

Current processing techniques tend to favor a preferential orientation of plant particles in the plane perpendicular to either compaction or projection. In order to consider the development of new techniques that would enable plant aggregates to be oriented differently, the full range of possible orientations has been considered in this study. Different values of the angles described in Section 3.3 are considered and the first results are presented in Figure 15.

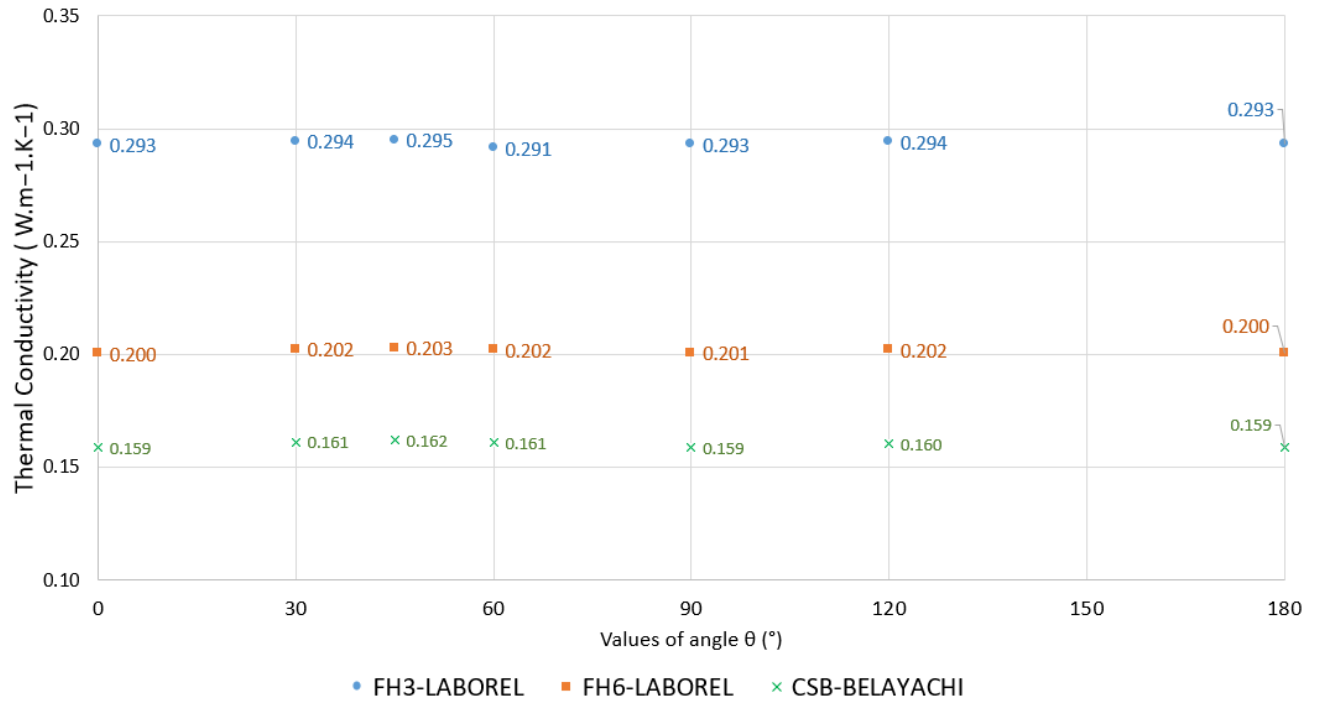


Figure 15. Impact of a granulate orientation change in modeled composites: case of average thermal conductivity values.

When focusing only on average thermal conductivity values, particle orientation seems to exert minimal influence on thermal conductivity in the studied composites. However, exploring the variability in component values of the thermal conductivity tensor with respect to particle orientation is insightful. Figure 16 illustrates these findings for the considered materials.

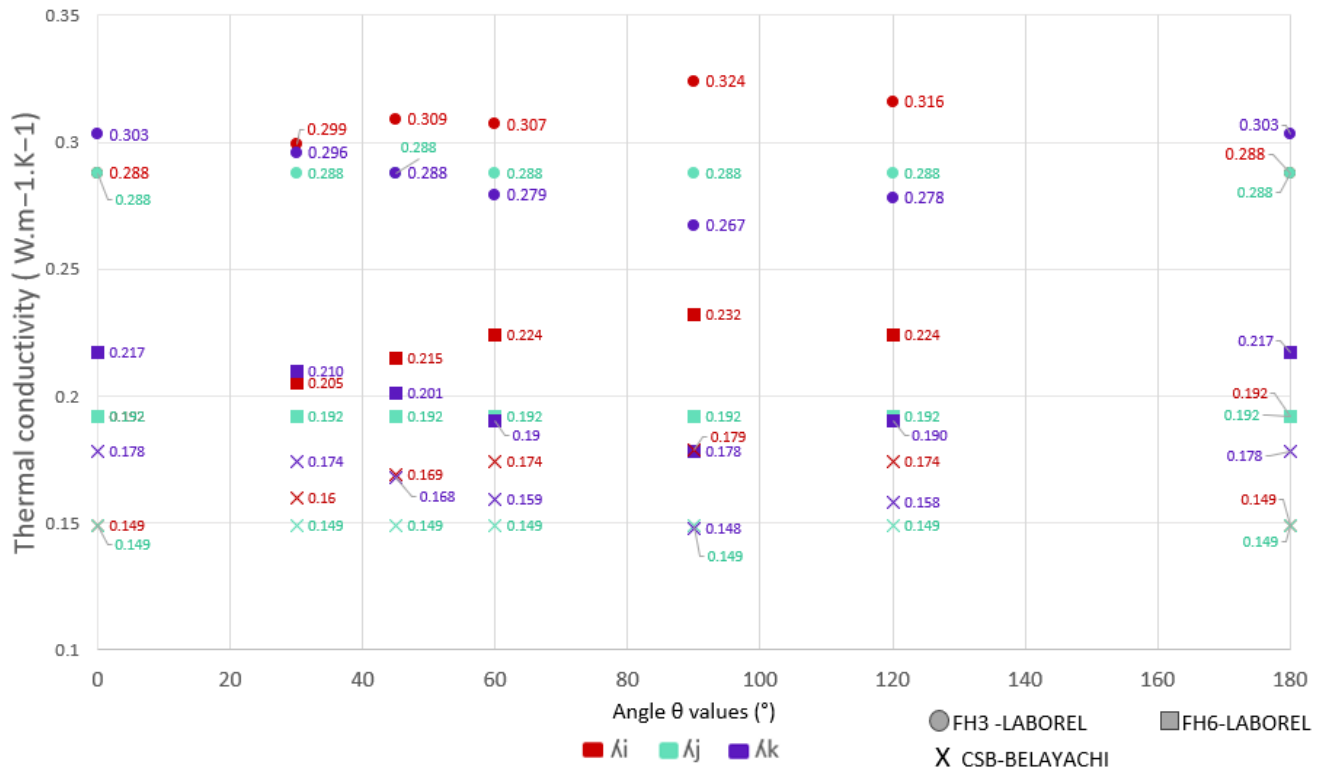
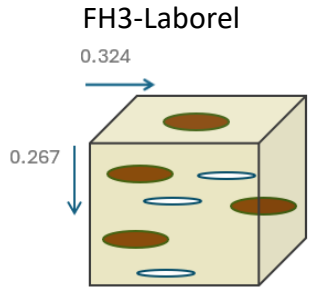
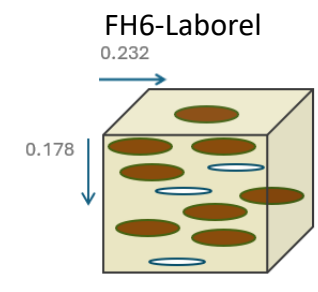
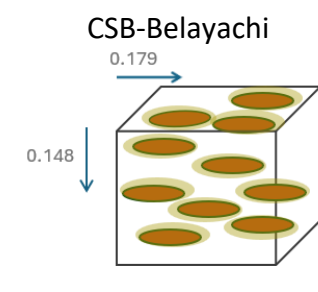


Figure 16. Impact of a granulate orientation change in modeled lightweight earth: case of thermal conductivity tensor components.

Depending on the orientation of the plant particles, the composite’s thermal conductivity in a specific direction can vary about 20% in lightweight earths. In Laborel’s FH3 and FH 6 lightweight earths, the low relative volume fraction of aggregates—by volume and, respectively, 22% and 37%—and of cracking in the plane perpendicular to compaction attenuate the influence of particle orientation on thermal conductivity. Finally, in the case of the CSB-Belayachi composite, both the relatively moderate volume fraction of sunflower bark (below 40%) and the effect of the low-conductivity matrix (air) explain the low degree of anisotropy.

In order to be more explicit about the most favorable or unfavorable cases for each of the composites, the following table summarizes the extremum results (Table 6).

Table 6. Extreme thermal conductivities for each modeled composite.

Composite	FH3-Laborel	FH6-Laborel	CSB-Belayachi
			
$\lambda_{\max}/\lambda_{\min}$	1.2	1.3	1.2

No value can be found in the literature concerning sunflower bark-based composites to assess the relevance of these results. However, in the case of hemp-based composites, the ratio

between normal and tangential thermal conductivity reported by experimental work is about 1.5 for non-optimized composites [27] i.e., where particle alignment is not necessarily identical. According to this work, the ratio is from 1.2 to 1.3 when the hemp particles are aligned in the same direction. The influence of the binder is not considered, as it has an isotropic thermal behavior according to our assumptions. Eventually, modeling results are consistent with the orders of magnitude given in the literature; the results highlight that processing techniques controlling particle orientation could be used to optimize the thermal properties of a geo-sourced material with a given formulation.

6. Conclusions

6.1. Relevance of Methods Used

In this study, the goal is to develop a reliable tool to predict the thermal conductivity of lightweight earth and raw earth blocks incorporating plant aggregates. Four composites selected from the literature are used to support the modeling work. To ensure consistency, the proposed models take both the macrostructure and the microstructure of the selected materials into account. In cases where data are missing—such as the proportion of cracking after drying or the extent of aggregate coating by the binder matrix—model calibration is proposed.

The approach presented here has proven effective for lightweight earth, maintaining a discrepancy between experimental and modeled thermal conductivity values within 5%, which falls within measurement uncertainties. However, the Mori-Tanaka model may become inconsistent with larger volume fractions of aggregate, making the double inclusion model more appropriate. Overall, the results indicate that mean-field homogenization is a valuable tool for modeling the thermal behavior of earth materials, paving the way for predictive tools to guide formulation choices.

6.2. Optimization of Formulations

By validating models for each studied composite, the impact of various criteria on thermal conductivity is evaluated, identifying the type of aggregate as a key factor. This is a significant result with a view to optimizing the formulation of geo-based materials for insulating purposes. While particle orientation seems to exert minimal influence on thermal conductivity in the studied composites, aspect ratio can notably affect thermal conductivity when there is significant variability in raw materials. However, this influence is generally negligible compared to measurement uncertainties. Additionally, the difference between normal and tangential components (along or perpendicular to the compaction axis) can reach 40%, underscoring the importance of appropriate processing techniques in optimizing the thermal performance of geo-sourced materials. These findings highlight the need for a careful consideration of aggregate type and processing methods to achieve optimal thermal efficiency in these composites. In this way, the conclusions of this study could inspire new manufacturing techniques for lightweight earth.

6.3. Future Outlooks

This work is a follow-up to the modeling work carried out at the scale of the plant aggregate particle [26], a key element in the homogenization process presented here. The addition of the binder phase constituted by the earth adds difficulty in determining the volume

proportion of the binder phase as well as the one due to cracking after drying. Microstructure images such as X-ray tomography images could provide valuable information to avoid the calibration step currently required. To enhance robustness, this approach should be applied to a broader range of geo-sourced materials, as the current study's conclusions are limited to the selected materials.

Additionally, this research suggests the potential for mixing different plant aggregates to accommodate local and seasonal availability. Recent studies indicate that plant aggregates can swell or shrink in response to relative humidity [41]. Quantifying this behavior could enhance the accuracy of models predicting thermal insulation performance. Additionally, models could be further refined by incorporating the variability in aspect ratio values for the same type of aggregate, addressing the intrinsic variability that has not been considered in this study.

Finally, the developed virtual models represent real geo-based materials and could be further enhanced with new experimental data, such as fire resistance and sustainability indicators. This work serves as a precursor to digital twins, enabling the prediction of the complex behavior of eco-materials under various conditions. For this purpose, the modeling process needs to incorporate extensive experimental information [42]. Given the vast diversity of possible formulations for geo-sourced materials, advanced data exploitation techniques—such as Bayesian networks—may help address the inevitable data gaps [43], [44].

7. Nomenclature

Latin symbols

$\underline{\underline{\lambda}}$	Thermal conductivity tensor
$\underline{\underline{\mathbb{A}}}_r$	Concentration tensor
$\underline{\underline{P}}_R$	Interaction tensor
$\underline{\underline{\lambda}}_i$	Thermal conductivity tensor of the inclusion i and the matrix 0 .
$\underline{\underline{\lambda}}_0$	Thermal conductivity tensor of the matrix 0
$\underline{\underline{S}}_R$	Depolarization tensor $\underline{\underline{S}}_R$
f_r	Volume fraction of r phase
V_r	Volume of r phase

Greek symbols

λ	Thermal conductivity (W/(mK))
$\underline{\underline{\delta}}$	Kronecker symbol

Superscripts

MT	Mori-Tanaka scheme
EB	Earth-based

Subscripts

R	r -th phase
m	Earth-based binder
i,j,k	Reference to the Cartesian coordinate space vector base
tot	Total
hom	Homogeneous materials
T	Parallel to the compaction (for the earth composites) or fiber axis (for aggregates)
N	Perpendicular to the compaction (for earth composites) or fiber axis (for aggregates)

Abbreviations

RVE	Representative Volume Element
FWAS	Quarry fines from aggregate washing processing
FH3	Composite with FWAS and hemp shiv
FH6	Composite with FWAS and hemp shiv
CSP	Composite with clay and sunflower pith
CSB	Composite with clay and sunflower bark

Author Contributions: Conceptualization, S.R.L.; methodology, S.R.L.; software, S.R.L.; validation, S.R.L., V.S. and A.A.-C.; formal analysis, S.R.L.; investigation, S.R.L.; resources, S.R.L.; data curation, S.R.L.; writing—original draft preparation, S.R.L. and A.A.-C.; writing—review and editing, S.R.L. and A.A.-C.; visualization, S.R.L.; supervision, A.A.-C.; project administration, S.R.L.; funding acquisition, S.R.L. All authors have read and agreed to the published version of the manuscript

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7.3.2 Cas des bétons végétaux

La démarche adoptée précédemment (proposition de modèles de prédiction des performances thermiques, validation des modèles et optimisation) a été transposée au cas des bétons végétaux. Deux points particuliers distinguent cependant ces résultats de ceux présentés à la section 7.3.1. D'abord, grâce aux informations fournies dans les travaux expérimentaux sélectionnés dans la littérature (notamment la porosité ouverte du composite et des images microstructurales par tomographie à rayons X), les hypothèses faites dans la modélisation concernant les fractions volumiques des différentes phases sont confortées. De plus, en s'appuyant sur des nombreuses références de la littérature, ces travaux proposent une évaluation de la conductivité thermique de la matrice liante hydraulique des bétons végétaux à partir de la connaissance de leur masse volumique. Cette donnée d'entrée permet ainsi, grâce aux modèles prédictifs proposés, d'envisager un changement de liant en amont de l'étape de fabrication. C'est une différence fondamentale par rapport aux travaux sur les terres allégées pour lesquelles la variabilité de la ressource a montré qu'il était à ce stade difficile de corréler conductivité thermique de la matrice et masse volumique.

Article I. *Vegetal Concretes: Towards Formulation Optimization For Hygro-Thermal Applications thanks to Multiscale Modeling*

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Highlights

- Thermal conductivity prediction models are developed according to experimental data.
- Models are based on the real morphology of bio-based materials.
- Thermal performance can be optimized according to various criteria which are difficult to experimentally investigate.

Abstract

The construction sector is one of the most environmentally impactful. Over the last few years, new materials are emerging to contribute to this sector's ecological transition. These include bio-based materials, in particular plant-based concretes. They hold great promise as insulating materials due to their local availability and combined attributes of low embodied energy, good thermal performance and natural hygroscopicity. A growing number of campaigns to measure their thermal conductivity are reported in the literature, but the wide variety of formulations (nature of the plant aggregate, nature of the binder, proportion of aggregate) complicates the identification of the most significant criteria. Meanwhile, few modeling studies explore the possibility of predicting their insulating capacity notably from formulation data. This study uses mean-field homogenization techniques (Mori Tanaka and double inclusion schemes) to propose a new tool for predicting the thermal conductivity of these eco-materials. Experimental data on plant-based concretes have been rigorously selected to carry out and validate this modeling work since it needs specific microstructure data. The developed models consider the formulation, the orientation of the aggregates induced by the laying technique, as well as the actual morphology of the materials at both microscopic and macroscopic scales. The models are also used to analyze the factors optimizing their insulating properties which is currently impossible to do on an experimental stage: aggregate type, aspect ratio and orientation. Finally, a multi-criteria analysis enables broader factors to be considered, such as changes in binder or aggregate proportion, hygric and mechanical aspects and even raw material availability. This work extends the possibility of optimizing the thermal conductivity of materials incorporating plant aggregates even before the manufacturing stage. This modeling work should serve to encourage the

development of new manufacturing techniques to optimize the thermal performance of bio-based building materials, which clearly offer answers to the current climate challenge.

Keywords

Multiscale modeling, Vegetal concrete, Thermal conductivity, Optimization

1. Introduction

The construction sector's carbon footprint requires a rapid and sustained transition to limit the environmental and social consequences of global warming [1], [2]. Bio-based building materials are increasingly recognized for their low environmental impact, providing viable alternatives to conventional high-impact materials [3], [4]. Despite their potential, large-scale adoption of these materials remains limited to date due to various technical, political and societal barriers [5].

Plant-based aggregates in building materials demonstrate great potential for enhancing hygrothermal comfort [6],[7]. Their unique thermo-hydric coupling enables them to serve as both insulators and passive regulators of ambient humidity, significantly improving building energy efficiency [8]. This explains the recent interest in concretes incorporating plant aggregates- known as vegetal concretes -and the large number of experimental studies evaluating their thermal conductivity [9], [10], [11], [12], [13], [14]. Indeed, the incorporation of plant aggregates in cementitious matrices reduces thermal conductivity, achieving good thermal performance [15]. Despite their interesting thermal properties but the development of vegetal concretes is hindered by marked anisotropy and multi-scale porosity, complicating understanding of their behavior [16], [17], [18]. The effect of aggregate size has been studied in mechanical terms [19], but not in thermal ones. Measurement methods do not easily permit the study of this aspect and do not systematically provide access to the different components of thermal conductivity, which is in fact, a tensor in the case of anisotropic material [20].

On the one hand, thermal conductivity varies according to formulation (type of aggregate, nature of binder and binder/plant aggregate ratio) [21]. Some authors conclude from experimental data that the binder has little influence [15] while others indicate that it has a significant impact on the material's thermal behavior [22]. Similarly, questions arise about the influence of the type of plant aggregate used [23]. The consensus seems to be that thermal conductivity is linearly dependent on material density [15], [24], [25] [26], although other empirical exponential formulas can be put forward [27]. Thermal conductivity may decrease with density and with the concentration of plant aggregates [20], without the influence of either the binder or the plant aggregate being clearly identified. However, recent studies underline that density alone is not a good predictor of thermal performance of bio-based materials [28]. Consequently, emerging research aims to efficiently predict the thermal conductivity of vegetal concretes [29]. To date, there is no established correlation between the formulation (material implemented in the wet state) and the thermal conductivity of the stabilized material (i.e. in the wall before significant aging and evolution of its physical properties).

On the other hand, it is important to highlight that the experimental linear relationship established between dry density do not consider the anisotropy of vegetal concretes [30]. The self-coherent model (n-sphere model) has been widely used in the literature [24], [25], [31], but its isotropic assumption contradicts the anisotropy observed both at the particle scale [16]

and at the material scale [32]. Modeling must be yet a powerful tool to study the impact of the preferential orientation of plant particles generated by processing techniques [23] [13]. In addition, the few models found in literature have been developed for hemp concretes [32], [33], [34] while numerous studies demonstrate the relevance of using locally available aggregates, such as pith or sunflower bark, which are more available than hemp shiv [35]. No literature references propose a predictive model for the thermal conductivity of plant-based concrete using other agricultural by-products than hemp shiv.

In addition, thermal characterization of plant-based concretes can be time-consuming, and measurement methods are questionable [36]. Moreover, it is experimentally difficult to measure the effect of plant particle form factor or orientation, given the variability of plant aggregates and current manufacturing techniques [37]. Lastly, optimizing formulations is complex because reducing material density improves insulation but degrades mechanical properties [38], [39].

The aim of this article is to gain a better understanding of the various parameters influencing the thermal properties of plant-based concretes, and to supplement the information provided by the numerous experimental studies in the literature [40]. The purpose of this modeling work is to refine thermal conductivity prediction in order to contribute to the development of plant-based concretes. Specific optimization points are studied and proposed to optimize their thermal properties where experimental work is difficult to achieve. Finally, this study stands out notably from the work carried out in the literature to date since it considers vegetal concretes incorporating sunflower barks.

Among the challenges that need to be overcome to encourage the emergence and use of vegetal concretes, the following are worth mentioning to understand the approach taken of this article.

- The wide variety of plant aggregates, their availability, seasonal harvests, and the range of possible binders (hydraulic or raw-earth binders) become more complex formulation parameters and their characterization [41], [21].
- The stabilization times in various environments combined with varying thermal conductivities depending on formulation and external conditions (temperature and ambient relative humidity), make characterization challenging [42] [43].
- Anisotropic behavior at both the material and aggregate scales, influenced by implementation technique and the microstructure of aggregates complicates thermal performance assessment [20], [32], [33], [34], [44].
- There is to date lack of predicting tools concerning thermal conductivity of vegetal concretes from formulation's data.

This article aims to tackle these challenges through multi-scale modeling by considering:

- the variability of both the aggregate and the binder to easily study and compare different formulations.
- a relevant and fast-calculating modeling method to complete and guide time-consuming characterization processes.
- the anisotropic thermal behavior both at the particle and the vegetal concrete scales.

- different models to link formulation and morphology data to the thermal conductivity of a stabilized vegetal concrete

This study proposes two predictive models of thermal conductivity of plant-based concretes. Given the selected experimental data, they are applied to sunflower bark-based vegetal concretes made of lime- or metakaolin-based binders. But there is a fundamental need to precise that they are both applicable to different hydraulic binders and aggregates thanks to the proposed methodology. The modeling process enables comparisons of different formulations regarding the targeted thermal properties, even before the manufacturing stage.

Firstly, analytical homogenization techniques (such as Mori Tanaka [45] and double inclusion models [46]) are used as a continuation of work already carried out at the particle and material scales [20], [47]. Particular attention is paid to the accessibility of modeling input data and the previous techniques are applied to bio-based building materials made of a binding matrix. Experimental data from the literature have been rigorously selected, allowing discussions of modeling results in relation to the experimental measurements for plant-based concretes [21] under given measurement conditions. The variability of thermal conductivity according to external loads (effects of temperature and relative humidity) and different criteria is taken into consideration. Models are previously evaluated for adequacy, and the most appropriate models are selected for further study.

The selected models are used as optimization tools to study the effect of particle orientation, nature and shape. The aim of the approach is to illustrate that modeling enables to control both the size and orientation of aggregates to study their effects, optimizing not only the formulation but also the implementation.

But formulation optimization cannot be confined to the thermal aspects of building materials. Other factors are taken into account such as mechanical or hygroscopic aspects or resource availability) to achieve a more global approach to optimizing formulations in line with the composite's expected performance in use. Consequently, a multi-criteria approach is suggested, combining several factors studied in the literature such as mechanical properties, hygric properties and the availability of plant aggregates. The intent is to be able to assess the performance of plant-based concretes on a broader scale, beyond their thermal properties.

2. Materials and methods

2.1 Homogenization methods

2.1.1 Choice of the homogenization method

Several modeling tools are available, such as numerical or analytical homogenization to get access to the thermal conductivity of a heterogeneous material such as vegetal concrete. The choice of method used generally depends on the expected computation time, the accuracy of the prediction, and the availability of input data.

2.1.2 Heat conservation equation

According to the first law of thermodynamics, energy is conserved over time for a closed system. The three-dimensional steady-state heat equation, with no internal heat supply, can be expressed as follows [48]:

$$\rho C \frac{dT(x)}{dt} = - \vec{\nabla} \cdot \vec{Q} \quad (1)$$

where ρ is the density of the material ($\text{kg}\cdot\text{m}^{-3}$), C its specific heat ($\text{J}\cdot\text{kg}^{-1}\cdot\text{K}^{-1}$), T the temperature (K), t the time (s), $\vec{\nabla}$ the gradient operator and \vec{Q} the thermal flux ($\text{W}\cdot\text{m}^{-2}$).

If only pure conduction is considered for heat transfer, Fourier's law applies:

$$\vec{Q} = -\underline{\underline{\Lambda}} \cdot \vec{\nabla} T \quad (2)$$

Where $\underline{\underline{\Lambda}}$ is the thermal conductivity tensor of the material.

In the case of an isotropic material, the thermal conductivity tensor can be reduced to its norm, i.e., the scalar value of each tensor component. If the material's thermal conductivity is the same in any direction, the thermal conductivity is expressed as follows:

- Isotropic

$$\underline{\underline{\Lambda}} = \Lambda \begin{pmatrix} 1 & 0 & 0 \\ 0 & 1 & 0 \\ 0 & 0 & 1 \end{pmatrix} \quad (3)$$

Where Λ is in $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$.

If the material behavior is anisotropic regarding thermal conduction, two cases can be distinguished where the value of thermal conductivity depends on the direction considered:

- Transversely isotropic

$$\underline{\underline{\Lambda}} = \begin{pmatrix} \lambda_N & 0 & 0 \\ 0 & \lambda_T & 0 \\ 0 & 0 & \lambda_T \end{pmatrix} \quad (4)$$

Where λ_N and λ_T are respectively the normal and tangential components ($\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$).

- Orthotropic

$$\underline{\underline{\Lambda}} = \begin{pmatrix} \lambda_i & 0 & 0 \\ 0 & \lambda_j & 0 \\ 0 & 0 & \lambda_k \end{pmatrix} \quad (5)$$

Where λ_i , λ_j and λ_k are respectively the components in the three directions of an orthonormal reference frame ($\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$).

2.1.3 A homogenization-based effective thermal conductivity

The aim of homogenization methods is to determine the effective thermal behavior of the material. An external macroscopic load (temperature field) is applied to determine its thermal conductivity tensor, which requires information on the material's microstructure. A Representative Volume Element (RVE) of the considered material has to be defined. This corresponds to the sample of vegetal concrete on which the experimental thermal conductivity measurements were carried out. The dimensions must be sufficiently large in relation to microstructural heterogeneities for the R.V.E. to be statistically representative of the real material. A macroscopic uniform thermal gradient field is applied to the boundary of the R.V. E. In this case, the thermal conduction problem can be written using the microscopic (6) and macroscopic (7) forms of Fourier's law as follows:

$$\vec{q} = -\underline{\underline{\lambda}} \cdot \vec{\nabla} T \quad (6)$$

$$\vec{Q} = -\underline{\underline{\Lambda}}_{hom} \cdot \vec{E} \quad (7)$$

Where \vec{q} and \vec{Q} are respectively the microscopic and the macroscopic heat fluxes, $\underline{\underline{\lambda}}$ is the microscopic the effective thermal conductivity tensor, $\underline{\underline{\Lambda}}_{hom}$ is the effective thermal

conductivity tensor, $\vec{\nabla} T$ and \vec{E} are the microscopic and macroscopic heat gradient fields, respectively.

Based on this, different methods can be used to determine the effective thermal conductivity tensor $\underline{\underline{\Lambda}}_{hom}$.

One of the difficulties of homogenization is that heterogeneous materials with random microstructure are to be considered. The arrangement of phases is not perfectly known : the microstructure of bio- based building materials is notably complex with multiscale porosities [49]. Information on phase arrangement at the microscopic level can only be given through volume fractions f_i of the constituents:

$$f_i = \frac{V_i}{V_{tot}} \text{ with } V_{tot} = \sum_i^r V_i + V_m \quad (8)$$

Where V_i and V_m are the volumes of inclusions and the matrix, respectively, in the total volume V_{tot} of the RVE.

The entire RVE microstructure is thus defined by the combination of volume fractions:

$$\sum_i^r f_i + f_m = 1 \quad (9)$$

Where f_i and f_m are the volume fractions of the inclusions and the matrix, respectively.

However, in the literature, only the mass relationships between the constituents are typically specified in the material formulation. This presents a major obstacle to determining the input data for homogenization, and thus calculating the effective thermal conductivity tensor. A correspondence between mass ratios and volume ratios is therefore sought as soon as possible, often using X-ray tomography images from the literature [50]. In the absence of this information, assumptions about volume fractions are required at the material scale, and calibration is then applied. For the particle scale, previously validated methods enable the consideration of relevant volume fractions (such as intra-particle porosity) [20], [47].

- Mori Tanaka scheme

The Mori-Tanaka model simplifies the localization problem by representing inclusions of the same shape, orientation and behavior as a single ellipsoidal inclusion embedded in an infinite medium (Figure 1).

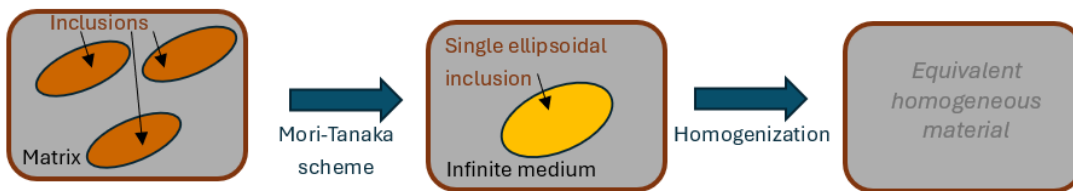


Figure 1. Schematic of homogenization using Mori-Tanaka model

Interactions between inclusions are included in Mori Tanaka's scheme. The infinite medium has the properties of the matrix and is subjected to the average deformation of the matrix within the composite. It derives the following expression:

$$\underline{\underline{\Lambda}}_{hom} = \sum_{r=1}^N f_r \underline{\underline{\Lambda}}_r \cdot \underline{\underline{A}}_r^{MT} \text{ with } \underline{\underline{A}}_r^{MT} = \underline{\underline{A}}_r^O \cdot \left(\sum_{r=0}^n f_i \cdot \underline{\underline{A}}_r^O \right)^{-1} \quad (11)$$

This scheme has demonstrated its effectiveness in determining the effective thermal conductivity of building materials [32], [52], [53]. This study thus explores its applicability to composite materials where the inclusions are plant aggregates incorporated into hydraulic binders. In particular, the relevance of the Mori-Tanaka scheme with a high volume fraction of inclusions is examined, as extensively discussed in the literature [54], [55].

- Double inclusion scheme

Hori and Nemat-Nasser [46] generalized Eshelby's formula and Mori–Tanaka's average theory to propose a different model known as the double-inclusion model. In this model, inclusions are embedded in a fictitious reference matrix and coated with another material matrix to predict effective properties of composites considering interphase effects (Figure 2).

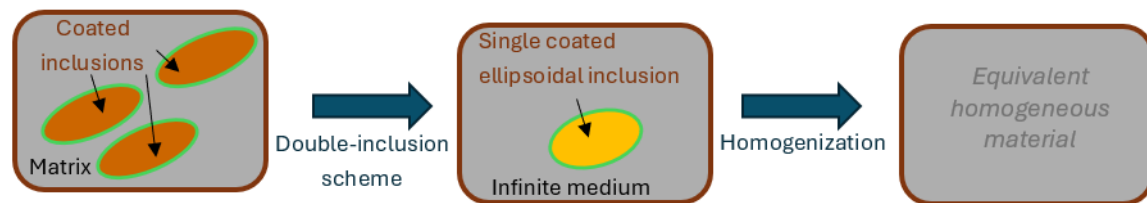


Figure 2. Schematic of homogenization using double inclusion model

The double inclusion model considers the shape and orientation of the double inclusion and the properties of the reference medium, which must be specified based on morphological and microstructural considerations. The external geometry of the double inclusion should reflect the arrangement of inclusions within the material. The coating phase (binder) has exactly the same shape and orientation as the original inclusion (plant aggregate). This new coated inclusion is then processed using the Mori-Tanaka model [56]. The results differ from the previous model since the matrix enveloping the coated inclusions is air and not hydraulic binder. In the case of two-phase composites, the volume fraction of the double inclusion equals the volume fraction of the inclusion [57]. The double inclusion model is particularly suited for multiphase materials [46], [57], [58], [59], [60]. However, applying it to bio-based building materials poses challenges in assessing the thickness of the coating, which is not well-documented in the literature. Nevertheless, the macroscopic granular appearance of plant-based concretes is evident when the volume fraction of aggregates is significant; plant aggregates are coated by the binder and bonded together (Figure 3).

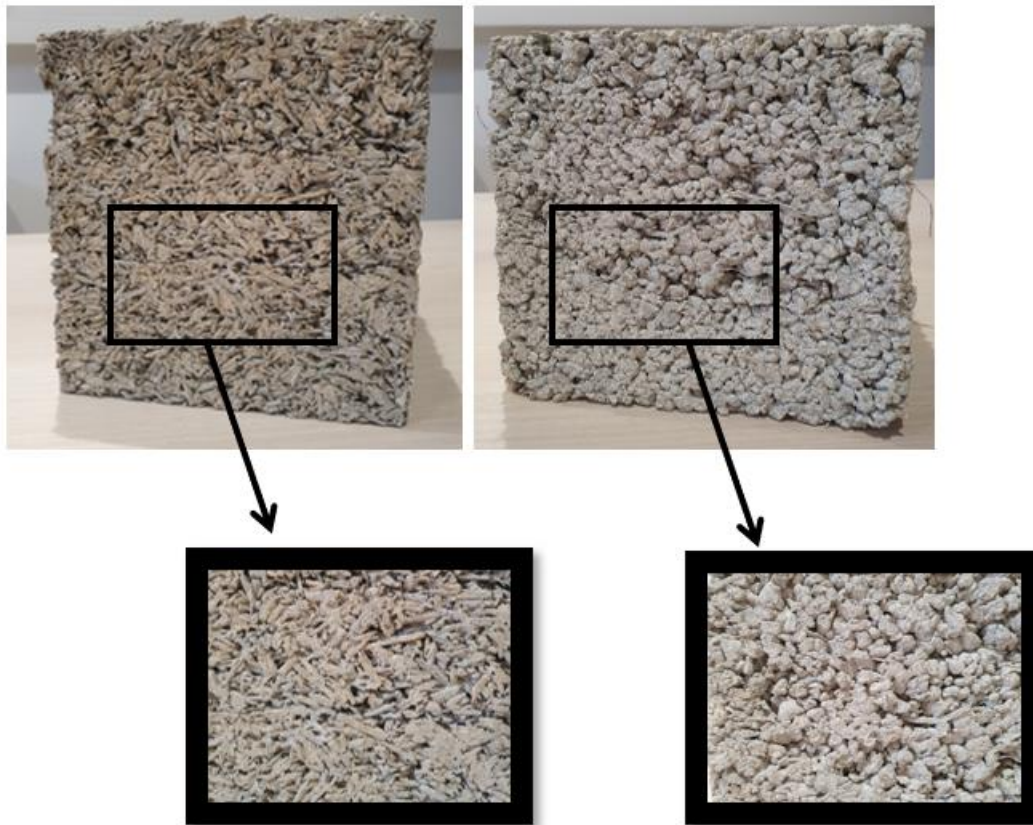


Figure 3. Close-up photos of vegetal concretes

The apparent relevance of the double inclusion model's description can be emphasized. This study focuses on comparing the results of this model and the Mori Tanaka model in predicting the thermal conductivity of the materials considered.




2.2 Aggregate scale

2.2.1 Particulate shape

The plant aggregates selected for this study in the bio-based composites are hemp shiv, sunflower bark, and pith. It is worth noting that the vegetal concretes selected in the literature are made of hemp shiv and sunflower bark. Sunflower pith is considered in the optimization phase of this study because of its exceptional insulating properties at the particle level [47]. Hemp shiv and sunflower bark exhibit macroscopic cylindrical shapes and tubular pores. The aspect ratios of hemp shiv and sunflower bark were determined through particle size analysis in the same study that provided thermal conductivities of the composites or by the same authors in preliminary works [21], [61], ensuring consistent input data and mitigating the impact of resource variability [62], [63]. Sunflower pith, characterized by honeycomb pores, is predominantly spherical at the macroscopic scale [64]. The assumption of spherical inclusions for sunflower pith in mean-field homogenization was validated in previous research [47].

Table 1 summarizes the aspect ratios and shapes considered for the plant aggregates in medium field homogenization.

Table 1. Particulate values determined using data from literature references (at dry state, under 20 °C).

Aggregate	Aspect ratio	Assumption	Reference	Shape in analytical models
Hemp shiv	3.3	Transversely isotropic	Laborel-Preneron et al 2018 [61]	
Sunflower Bark	3.4	Transversely isotropic	Lagouin et al 2019 [21]	
Sunflower pith	1	Isotropic	Magniont et al 2012 [65] Arufe et al 2021 [42] Rosa Latapie et al 2023 [47]	

The ellipsoidal shape is assumed for hemp shiv and sunflower bark as per the mean-field homogenization hypotheses (cf. section 2.1.3). Sunflower pith, on the other hand, is modeled as a spherical, i.e. isotropic inclusion.

2.2.2 Particulate orientation

The orientation of aggregates in composites can be defined by two angles. Θ is the angle between the main axis of the inclusion and the Z-axis in global coordinates, while ϕ denotes the angle between the projection of the inclusion in the XY plane and the Y axis. Due to the lack of precise knowledge regarding the actual orientation of aggregates in the materials under consideration, this study makes two assumptions. Firstly, it assumes that aggregates are randomly oriented in all directions ("random 3D" assumption). Secondly, it assumes that compaction during placement induces random orientation only in the XY plane ("random 2D" assumption), illustrated in Figure 4.

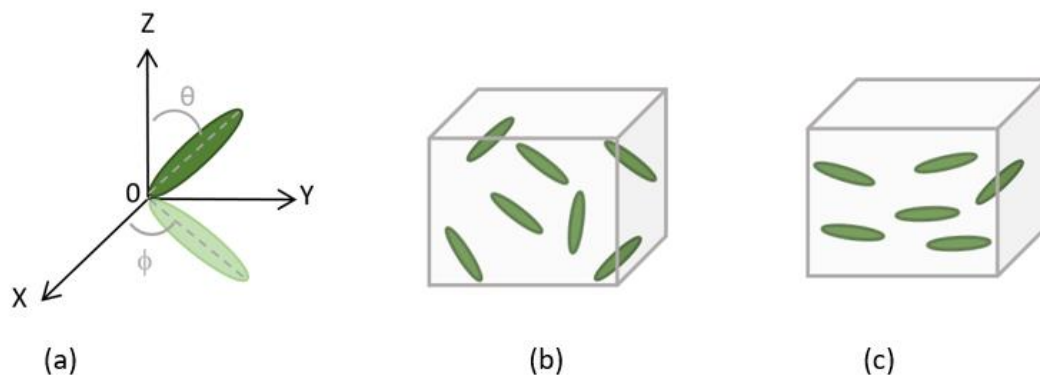


Figure 4 . Orientation of plant aggregates in composites: definition of inclusion direction (a), "Random 3D" composite, "Random 2D" composite (i.e $\theta = 90^\circ$)

Considering these two hypotheses allows to discuss the relevance of accounting for anisotropy at the composite scale, quantifying it, and to comparing it with experimental values reported for hemp shiv-based composites in the literature [32], [66], [67], [68], [69], [70].

2.1.4 Homogenization schemes

In order to gain access to the effective thermal conductivity tensor, mean-field theories use concentration tensors that relate the averaged fields at the microstructure level (i.e. inclusions and matrix) with the corresponding macroscopic fields. Considering an n-phase heterogeneous material, the effective thermal conductivity tensor $\underline{\underline{\Lambda}}_{hom}$ of the RVE is defined, according to the classical theory of Eshelby, as follows [51] :

$$\underline{\underline{\Lambda}}_{hom} = \sum_{r=1}^N f_r \underline{\underline{\Lambda}}_r \cdot \mathbb{A}_r \quad (10)$$

Where $\underline{\underline{\Lambda}}_r$ is the thermal conductivity tensors of the r-th phase (matrix or inclusion), f_r is the volume fractions of the r-th phase and \mathbb{A}_r the concentration tensor of the r-th phase.

The concentration tensor itself depends on a Hill tensor and a depolarization tensor, which take into account the variability of the microstructure in terms of the orientation and shape of the inclusions [34]. This is particularly relevant for bio-based building materials, where the implementation technique induces a preferential orientation of the plant aggregates (inclusions) [52]. An advantage of mean-field homogenization methods is that they provide analytical results requiring little computing time and memory. Knowledge of the concentration tensors \mathbb{A}_i allows for the resolution of the homogenization problem, provided the volume fractions of each phase and their thermal conductivity tensors are well defined. Several estimation schemes can then be used to perform the resolution.

2.2.3 Particulate thermal conductivity

Particulate thermal conductivity is an essential input to the homogenization process as it translates local thermal behavior at the inclusion level. In the literature, only the thermal conductivity of the hemp shiv particles has been measured experimentally [44]. Given the difficulty of the measurement protocol at the particle scale and the variability of the resource, it is possible to determine the particle thermal conductivity from particle porosity or, by inverse method, from the bulk thermal conductivity [20].

The macroscopic shape of the aggregates studied necessitates considering normal and tangential components for hemp shiv and sunflower bark: their thermal conductivity is a tensor with the assumption of a transversely isotropic inclusion (cf. section 2.1.1). Sunflower pith, considered as an isotropic inclusion, is characterized by a single particulate thermal conductivity value, thus reducing its thermal conductivity to a scalar value (cf. section 2.1.1). In the following, details of each component of particle thermal conductivity will be given with reference to the tensors in Figure 5.



$$\underline{\underline{\Lambda}}_{sunflower\ bark} = \begin{pmatrix} \lambda_N & 0 & 0 \\ 0 & \lambda_T & 0 \\ 0 & 0 & \lambda_T \end{pmatrix} \quad \underline{\underline{\Lambda}}_{hemp\ shiv} = \begin{pmatrix} \lambda_N & 0 & 0 \\ 0 & \lambda_T & 0 \\ 0 & 0 & \lambda_T \end{pmatrix} \quad \underline{\underline{\Lambda}}_{sunflower\ pith} = \lambda_{iso} \begin{pmatrix} 1 & 0 & 0 \\ 0 & 1 & 0 \\ 0 & 0 & 1 \end{pmatrix}$$

Figure 5. Plant aggregates considered in the study: photo and components of the particulate thermal conductivity tensors in the transverse isotropic case (hemp shiv and sunflower bark) and isotropic case (sunflower pith)

Plant aggregates are hygroscopic materials and naturally fix water when ambient humidity rises [49], [71], [72]. Part of the air trapped inside the particle, which contributes to its insulating capacity, is then replaced by water, whose thermal conductivity is almost twenty times greater than that of air. This significantly impacts particle thermal conductivity [25], [73]. To consider this phenomenon, it is necessary to quantify the water adsorbed in the plant aggregate using sorption isotherms. The sorption curves for hemp shiv and sunflower pith are selected from Ratsimbazafy's work [74] to deduce the volume fraction of water in the plant particle at a given ambient humidity. Based on this water volume fraction determined in the pore space of the plant particle, an analytical homogenization on this scale is used to correct the particulate thermal conductivity from the dry state.

Since both the thermal conductivity of air and the particle's solid skeleton increase with temperature, a correction is also made as necessary. Particle thermal conductivities are first determined in dry conditions and at 20°C (bulk thermal conductivity measurement conditions). They are then corrected according to the temperature and relative humidity conditions specified in the selected reference works for thermal conductivity measurements on the composite.

The method applied here has already demonstrated its suitability for predicting the evolution of the thermal conductivity in sunflower pith panels over the entire temperature and relative humidity range of use [47]. It is assumed that water binding takes place only at the level of the plant particles, so any sorption at the level of the binder is neglected in this study. Similarly, any variation in the thermal conductivity of the binder as a function of temperature is not considered.

2.3 Hydraulic binder

Plant-based concretes are made from a mixture of water, plant particles and lime or metakaolin-based binders. The choice of binder is crucial as it determines the mechanical, economic and environmental properties of the composite [69], [75]. Its impact on thermal conductivity is still under discussion in the literature [15], [22]. Modeling can be a useful addition to the discussion.

It is beneficial to gather extensive data from the literature and examine how to consider a change of binder in the developed models. In order to model and predict the thermal conductivity of similar composites (in terms of proportions, type and nature of plant aggregate) where only binder type differs, an analysis of the consequences of such a change is carried out on the effective thermal properties calculated.

2.4 Plant-based concretes

2.4.1 Experimental data

The studies selected for comparison with the models are chosen based on several criteria. Firstly, specific data for each scale's material is required as it forms the basis of the input data for modeling. This includes the thermal conductivity of the binder alone and that of the bulk aggregates (or their particular porosity if thermal conductivity is not available). Additionally, the temperature and relative humidity conditions for measuring the composite's thermal conductivity have to be specified which is not systematically done in the literature. An

assessment, even if incomplete, of the volume proportions of each phase of the composite (inclusion, solid phase or total porosity) has to be provided in those references. Moreover, the material implementation must be clearly explained to assess the possibility of preferential orientation of plant particles. Given these constraints, few works in the literature can be retained. The selected experimental work is one of the few to provide the key elements needed for modeling.

The chosen composites and corresponding characteristics for vegetal concretes are listed in **Table 2**. It is interesting to note that the materials chosen have similar densities and thermal conductivities, but different binders. This enables to discuss the influence or otherwise of the binder on the thermal conductivity of a composite - a point on which there is no consensus in the literature - and to verify that the modeling gives comparable results in the two cases.

Table 2. Vegetal concretes selected for the study: experimental characteristics.

Vegetal concrete	Reference	Binder/Aggregate	Density (kg·m ⁻³)	Open porosity (%)	Thermal conductivity (W·m ⁻¹ ·K ⁻¹)	Measurement conditions
LSB-Lagouin	Lagouin et al. 2019 [21]	Lime/Sunflower Bark	539.64 ± 51.56	68.6 ± 1.7	0.127 ± 0.008	25°C, 50 % RH
MSB-Lagouin		Metakaolin/Sunflower Bark	511.07 ± 61.86	71.5 ± 5.3	0.128 ± 0.009	

These materials are used as a basis for comparing the different modeling tools mentioned in section 2.1. Open porosity is associated with the air volume fraction in the composite. Since porosity, which depends significantly on formulation [21], has a major impact on composite's insulating capacity, it is important to specify the formulation of the selected plant-based concretes (Table 3).

Table 3. Formulation of selected vegetal concretes from Lagouin et al. [21] to obtain one cubic meter of material.

Vegetal concrete	Binder (kg)	Aggregate (kg)	Water (kg)	Water/binder	Aggregate/Binder
LSB-Lagouin	374.3	161.9	390.4	1.11	0.43
MSB-Lagouin	374.3	161.9	371.7	0.99	0.43

It should be noted that the authors provide the mass proportion of each component to obtain one cubic meter of vegetal concrete. In addition to open porosity, these indications are valuable for modeling to determine the phase volume ratio of the materials under study. Furthermore, X-ray tomography scans and analysis of the orientation of sunflower bark particles reveal that the particles in the MSB-Lagouin composite do not have a preferential direction in a plane direction, but rather an inclination of less than 40° to the horizontal [21].

In addition, for a given binder/aggregate mass ratio, it is important to assess the volume fraction of each phase in the final composite. Using tomographic scans, Bennai et al. [50] applied a wall-type formulation for vegetal concrete, similar to the work of Lagouin et al [21]. The correspondence between mass ratio and volume ratio is clearly dependent on the type of aggregate used in the formulation. However, since the bulk densities of the particles are close for hemp shiv ($250\text{-}320\text{ kg}\cdot\text{m}^{-3}$) and sunflower bark ($296\text{ kg}\cdot\text{m}^{-3}$) [62], these figures provide an initial estimate of the volume fractions applicable to the MSB-Lagouin composite.

The volume fractions of each phase are defined using the abovementioned references [50] [21] as the values are in line and based both on a wall-type formulation and on the material's real microstructure. Finally, the volume fraction of the coating is a necessary input when applying the double inclusion model. Unfortunately, no literature references provide SEM analyses of cross-sections of coated plant aggregates that could be used to assess the thickness of the binder coating the particle [76]. Given this lack of data, a first arbitrary - but credible- value of 20% was set in this study. This value is then adjusted in the models by comparing with experimental values.

2.4.2 Representative Volume Element

Determining the RVE a critical point in homogenization. Given the complexity of plant-based concretes, several possibilities are conceivable. The choices made for analytical homogenization are detailed below. Depending on the isotropic or anisotropic nature of the inclusions and their orientation, homogenization provides access to the effective thermal conductivity in scalar or tensor form.

Firstly, open porosity could be derived mainly from the intrinsic porosity of the binder. It can be as high as over 50%, whether measured by Water Immersion Porosimetry (WIP) or Mercury Intrusion Porosimetry (MIP) [21]. In this case, this porosity is taken into account in the thermal conductivity of the binder. A RVE consisting of a binder matrix with only aggregate inclusions is a first-line solution. It is particularly relevant when the volume fraction of plant aggregates is low as it can be assumed that they are largely enclosed by the binder matrix in the composite. This type of RVE is referred to as "HB-1".

When the aggregate volume fraction is more important, the additional porosity between the aggregate and the binder is more difficult to neglect. Indeed, the literature commonly refers to tri-modal porosity in the case of hemp concrete (intra-binder porosity, intra-particle porosity and porosity at the interface between the binder and the plant particle) [21], [49]. In this case, additional porosity is defined compared to the previous case. Without any particular indication found in the literature, the additional porosity (air) was considered to be anisotropic (as a spherical inclusion) in the case of plant-based concretes. This type of RVE is referred as "HB-2".

Finally, the plant particles may appear to be simply embedded in the binder and arranged in a relatively random way. From this point of view, an RVE describing coated particles bathed in an air matrix also seems relevant (cf. figure 3 in section 2.1.3). The thickness of the binder layer around the particle has already been discussed in the section 2.4.1., representing in the first instance 20% of the total volume of the coated inclusion. This latter case is noted "HB-3". To provide a better understanding of the different RVE cases considered in this study for vegetal concretes, an illustration is provided (Figure 6).

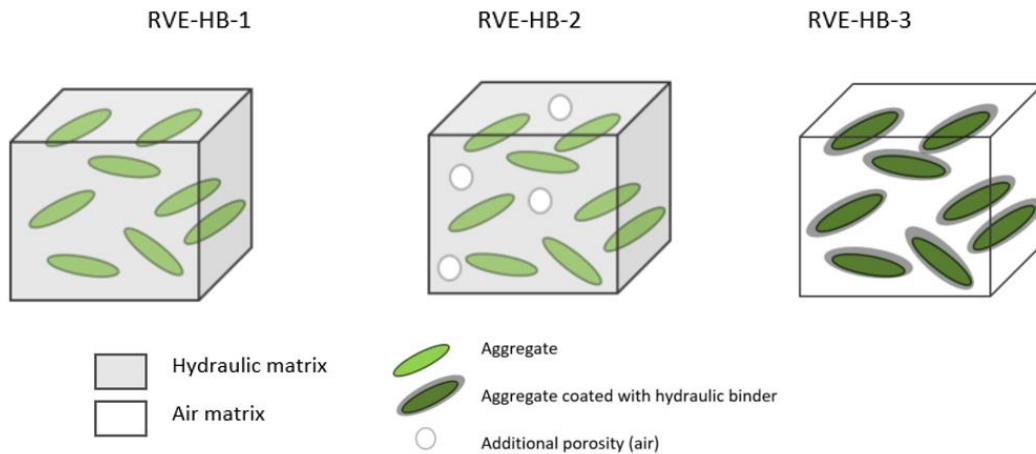


Figure 6. Different types of RVE considered for the homogenization process of plant-based concretes.

The effective thermal conductivity, due to the anisotropy of each RVE, is a tensor whose components are described as follows:

$$\underline{\underline{\Lambda}}_{HB} = \begin{pmatrix} \lambda_i^{HB} & 0 & 0 \\ 0 & \lambda_j^{HB} & 0 \\ 0 & 0 & \lambda_k^{HB} \end{pmatrix} \quad (12)$$

Depending on the orientation of the particles, these components can differ or not. In the following, the tangential components (λ_T) are those in the (O, \vec{i}, \vec{j}) plane, while the normal component (λ_N) is that along the (O, \vec{k}) axis.

2.6 Optimization factors of effective thermal conductivity

2.6.1 Type of plant aggregate

Modeling is a powerful tool for simulating various RVE configurations and evaluating their impact on effective material properties. The analytical homogenization approach chosen in this study enables to study the impact of a change in aggregate on a composite with relative ease and efficiency. Specifically, the incorporation of sunflower pith, sunflower bark or hemp shiv is tested while keeping the other parameters constant in the modeling, i.e assuming both a same formulation and processing.

2.6.2 Aspect ratio

Selecting plant aggregates with identical shapes for experimental study presents a significant challenge. Due to their natural origin and extraction methods, aggregates typically exhibit a certain size distribution, widely highlighted by granulometric analyses in the literature [17], [35], [77]. Modeling allows to investigate the effects of plant particle aspect ratio on thermal properties. The range of aspect ratios chosen in this study aligns with data reported in the literature (Table 4).

Table 4. *Variability of the resource regarding the aspect ratio from Ratsimbazafy et al 2021 [62]*

Aggregate	Range of Aspect ratio
Hemp shiv	2.28 – 8.75
Sunflower Bark	2.99 – 4.74
Sunflower pith	1-1.50

2.6.3 Preferential orientation

Due to the anisotropic thermal behavior of plant particles (cf. section 2.2.3), the shape of the aggregate necessarily influences the effective thermal conductivity of the resulting bio-composite. In addition, the preferential orientation of these particles within the material – whether due to compaction or spray application [40], [78]- adds to the phenomenon and probably amplifies or compensates for the inherent anisotropy of the particles.

Regarding particle orientation, various cases are tested, ranging from random orientations to the extreme case of perfect alignment of all particles in the same direction. It's important to note that while perfect alignment does not correspond to any practical implementation seen to date, exploring this extreme case allows for a more comprehensive discussion of the material's thermal behavior.

2.7 Multi-criteria optimization parameters

While materials that incorporate plant aggregates hydraulic matrix are mainly used for distributed insulation [79], [80], it is crucial to consider other criteria when optimizing their formulation. This section suggests considering different aspects linked to the material's extended performance, including mechanical and hygric properties, as well as environmental considerations related to the local availability of aggregates.

2.7.1 Mechanical aspect

The binder content seems to be fundamental from a mechanical point of view for vegetal concretes : for a given type of hydraulic binder, the higher the concentrations of binder the lower are deformation levels [81]. Experimental studies have also demonstrated that smaller average aggregate sizes lead to improve mechanical performance [81], [82]. In addition, the use of metakaolin-based binders improves the mechanical performance of plant-based concrete [83] [84].

Due to the incomplete availability of mechanical data for all materials under consideration, this study relies on trends reported in the literature. Nevertheless, to provide a visual multi-criteria analysis of the influence of varying binder or aggregate volume fractions, arbitrary values have been assigned to the mechanical (where a higher index indicates a greater value of the criterion). These parameter values are included in radar diagrams for comparative purposes between different formulations.

2.7.2 Hygroscopic aspect

Plant-based concretes are capable of capturing, storing, and transporting moisture from the environment, a property related to their morphology, pore connectivity, and pore size [85]. As they naturally moderate indoor relative humidity fluctuations through water adsorption, these bio-composites contribute to the hygric comfort of buildings. The Moisture Buffer Value (MBV) reflects this moisture regulation capacity.

Experiment findings suggest that the effect of formulations on sorption curves is fairly limited, as long as the composites are similar in terms of porosity [21], [86]. The extent to which these materials can regulate moisture depends on their sorption curve. It can be argued that, for the same binder, the use of a more hygroscopic aggregate (i.e. one with a steeper sorption curve) increases the MBV, as it is proportional to hygric effusivity - a factor related to the slope of the material's sorption curve- [27]. On the other hand, it has been established that using a binder based on metakaolin- rather than a hydraulic binder improves the hygric regulating capacity of the composite [21], [87].

Furthermore, to compare the effect of plant aggregate type on storage dynamics, Abbas et al.[23] introduced an interesting indicator, the ideal storage capacity of aggregates $\zeta_{aggregate}^{ideal}$. : This value was compared with a reference value for the dynamic storage capacity of the composite, noted $\zeta_{aggregate}^{ref}$ assuming no modification of the aggregate microstructure. Their study suggested that sunflower pith suffered microstructural alteration due to interactions with the hydraulic binder, whereas hemp shiv did not show such alterations. However, it is difficult to generalize this trend. Consequently, for simplicity, it is assumed here that aggregates with higher sorption/desorption capacities are more hygroscopic, based on the sorption curves of the aggregates considered in this study: hemp shiv, sunflower bark, and pith [88], [89].

Given the incomplete availability of hygroscopic property data for the materials in this study, only trends reported from the literature have been considered. As for mechanical properties, arbitrary values have been assigned to hygroscopic criteria, based on the works of Ratsimbafazy et al. [62].The aim is to provide comparison across several formulations once thermal properties have been optimized.

2.7.3 Resource availability criteria

The local availability of raw materials is an important factor in the life-cycle analysis of building materials [90]. Therefore, the local supply of plant aggregates used in vegetal concretes have to be examined. This study considers availability on a European scale, based on values reported in the literature [91], [92]. Ideally , it would be necessary to have data on the quantities of waste available, both geographically (i.e. as close as possible to the construction site) and according to the seasonality of harvests (i.e. at the time of the construction phase) [93]. Nevertheless, the aim here is to compare different formulations, including the choice of aggregate used.

Since specific data on the proportion of waste for a given crop is not available, a rate of return was calculated based on the number of hectares cultivated and the quantity of plant produced [103], [92]. A higher ratio indicates more waste available per cultivated hectare. It is also important to assess the proportion of each crop compared to European cereal production, which was 543 061 000 tonnes in 2019 [94]. These indicators allow for a comparison of the potential of each agro-resource (Table 5).

Table 5. Indicators calculated for each agro-resource considered in the study based on literature data.

Raw material	Cultivated area in Europe (ha)	Annual european production (t)	Reference	Rate of return (t/ha)	Proportion compared to European total cereal production (%)
Hemp	15 700	85 000	Carus et al. 2016 [92]	5,4	0.01
Sunflower	18 073 170	68 655 795	Debaeke et al 2017 [91]	3.8	12.6

In practice, separating pith from bark requires a mechanical process that must be repeated to improve pith purity (i.e. effective removal of traces of bark) [35], [74], [95]. The proportion of pith and bark obtained from a certain quantity of harvested stem waste is not well-documented. However, it can be stressed that this process necessarily impacts the availability of pure pith. In this study, hemp by-products are considered less available globally compared to those from sunflower, in line with literature [96]. In the case of sunflower, the bark is more readily available, it can be extracted more easily than the pith.

2.8 Modeling input data

2.8.1 Particulate thermal conductivity

From the data available in the selected reference, each component of the particle thermal conductivity tensors of sunflower bark is calculated in this work [20], [64]. These dry reference values at 20°C were chosen as the reference values. Corrections are made according to the experimental conditions of the thermal conductivity measurements on composites. In addition, to be able to consider a change of aggregates in the modeling, the particulate thermal conductivity values of the hemp shiv are also calculated from experimental results [97]. For sunflower pith, we have retained values from previous work [47]. It should be recalled that the morphology of this aggregate justifies considering it as spherical and not an anisotropic one as for hemp shive and sunflower bark. The method used and the dry reference values are listed in Table 6.

Table 6. Particulate values calculated under 20°C and in a dry state.

Reference	Type of aggregate	Calculation method	Modeled particulate thermal conductivity (W·m ⁻¹ ·K ⁻¹)	
			λ_T	λ_N
Lagouin et al. 2019 [98]	Sunflower Bark	From intra-particle porosity	0.139	0.208
Laborel et al. 2018 [97]	Hemp shiv	From measurement on bulk particles	0.044	0.066
Rosa Latapie e al. 2023 [47]	Sunflower pith	From intra-particle porosity	$\lambda_{iso} = 0.04$	

The results for hemp shiv are relatively consistent with those found in the literature [20], [32], [66]. However, it is slightly lower, which can be explained by a slightly higher porosity of the aggregate used compared to the average of hemp aggregates. To our knowledge, no experimental reference exists for the particulate conductivity of sunflower bark. However, since sunflower bark is less porous than hemp shiv, the result is consistent.

The very low thermal conductivity and therefore the high potential of sunflower pith should be emphasized, which is consistent with an intra-particle porosity of the order of 95% intra-particle porosity [6], [74], [99].

2.8.2 Binder thermal conductivity

In order to investigate the possible correlation between density and thermal conductivity of hydraulic binders (based on lime, metakaolin and/or pouzolanne), several references were selected from the literature [21], [69], [75], [100], [101], [102], [103], [104]. The results are shown in Figure 7.

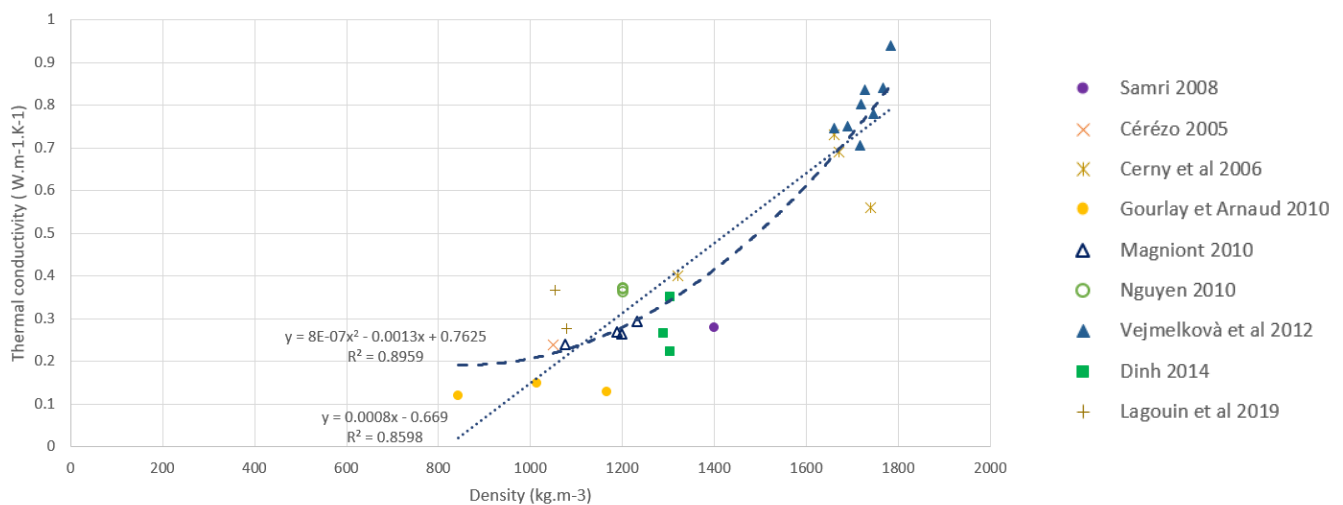


Figure 7. Thermal conductivity of a hydraulic binder as a function of its density [21], [75], [81], [104], [105], [106], [107], [108]

Experimental references[21], [69], [75], [81], [102], [103], [104], [105], [106] indicate that hydraulic binders usually range in density from 800 to 1800 kg.m⁻³. While a linear correlation appears to be a good approximation, a polynomial equation, more accurately relates thermal conductivity to the density of a hydraulic binder:

$$\lambda_{hydraulic\ binder} = 8.10^{-7} \rho^2 - 13.10^{-4} \rho + 0.7625 \quad (12)$$

In the modeling, this relationship is used to study the impact of a binder change on the thermal conductivity of this phase in each RVE. It should be noted that this thermal conductivity necessarily includes the intra-binder porosity. To remain consistent with experimental data, three values of hydraulic binder density are tested: 800, 1300 and 1800 kg.m⁻³. It leads to discuss the influence of the binder on the effective thermal conductivity. The corresponding thermal conductivity is calculated using the relationship (12).

2.8.3 Initial input data

The dry particulate thermal conductivities are corrected to suit the temperature and relative humidity conditions used to measure the composite's thermal conductivity. The thermal conductivity of the binder is considered constant (i.e. independent of ambient humidity and temperature). Finally, the volume proportions of the aggregates are calculated from the mass ratios and densities of the constituents, initially assuming no additional porosity in the final composite.

The input data required for the various homogenization calculations carried out in this study are listed in Table 7.

Table 7. Initial input data common for vegetal concretes under 25 °C and 50 % HR

Composite	Hydraulic binder		Aggregate				
	Density (kg.m ⁻³)	Thermal conductivity (W.m ⁻¹ .K ⁻¹)	Particulate density (kg.m ⁻³)	Particulate thermal conductivity (W.m ⁻¹ .K ⁻¹)		Volume fraction	Aspect ratio
				λ_T	λ_N		
LSB-Lagouin	1052	0.366	425	0.139	0.209	0.82	3.4
MSB-Lagouin	1079	0.277				0.87	

3. Results and discussion

3.1 Effective thermal conductivity of Vegetal Concretes

One of the difficulties in analyzing results is that experimental measurements reported in the literature typically provide a single value for thermal conductivity, whereas the modeling can provide up to three components (in each direction of space) when considering an anisotropic distribution of plant particles (cf. section 2.2.2). In the presentation of the results, the weighted average value of these components obtained through homogenization is compared

with the measured value. Additionally, each component of the effective thermal conductivity tensor is compared with the experimental value to assess the relevance of accounting for anisotropy in the models. For randomly oriented composites (cf. section 2.4.2), the thermal conductivity components are provided as a single common value for comparative purposes.

3.1.1 Lime-based composite

For the first plant-based reference concrete modelled in this study, denoted LSB-Lagouin, the results of the analytical homogenizations are gathered in Figure 8. The aim of this preliminary work is to identify the model that gives the most consistent result when compared with the experimental value. This model is then used to optimize the thermal properties of the actual composite (section 3.2).

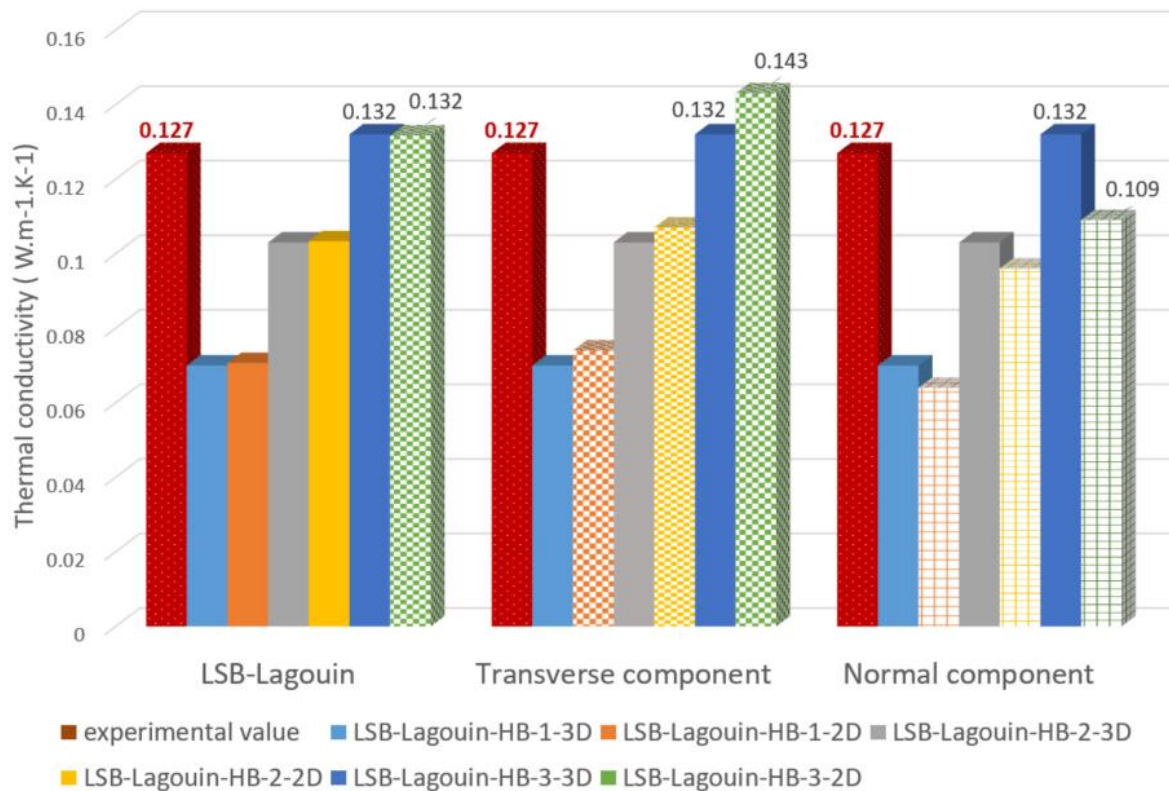


Figure 8. Results of predicted values for LSB-Lagouin models compared to the experimental value.

The main conclusions that can be drawn are:

- RVE HB-1 models: models without additional porosity show a significant relative deviation of over 40 % from the experimental target value, indicating their limited relevance.
- RVE HB-2 Models: these models exhibit better results with a deviation of around 20% from the experiment value but systematically below it. This could indicate a bias in the model
- Double Inclusion Scheme (HB-3): the most relevant results are obtained with homogenization considering the double inclusion scheme, with a deviation of less than 15% across all components. Given the measurement uncertainty, the thermal conductivity of this composite is estimated between 0.119 W·m⁻¹·K⁻¹ and 0.135 W·m⁻¹·K⁻¹. The predicted values of 0.132 W·m⁻¹·K⁻¹ by both the HB-3-3D and HB-3-2D models (averaged) fit within this range.

The excellent results of the double inclusion model are probably due to a very good correspondence between the model's assumptions and the real morphology of the material. Therefore, the LSB-Lagouin-HB-3 model is chosen for the rest of the study.

In addition, the anisotropy ratios between the normal and tangential components are 1.2, 1.1 and 1.3 respectively for the HB-1-2D, HB-2-2D et HB-3-2D models. The literature reports an anisotropy effect on hemp concretes consistent with these values [68], [109], [110]. However, the composites considered under consideration use sunflower bark instead of hemp shiv. It is interesting to note that despite a minor 3% difference in aspect ratio between these two aggregates, the ratio of tangential to normal thermal conductivity components remains the same ($\lambda_N/\lambda_T = 1.5$ for both aggregates according to preliminary particle scale calculations, see section 2.8.1).

Although, anisotropic effects vary depending on the formulation and the degree of compaction during processing [111]. Therefore, focusing on particle-scale anisotropy is crucial for predicting composite behavior. The impact of both form factor and particle-scale anisotropy has thus been considered in predictive models to optimize the insulating power of plant-based concrete. This approach could potentially guide the choice of aggregates used for manufacturing, enhancing the material's overall performance.

3.1.2 Metakaolin-based composite

The results of the analytical homogenizations for the MSB-Lagouin composite are presented in Figure 9 to select the model to be retained for the optimization phase :

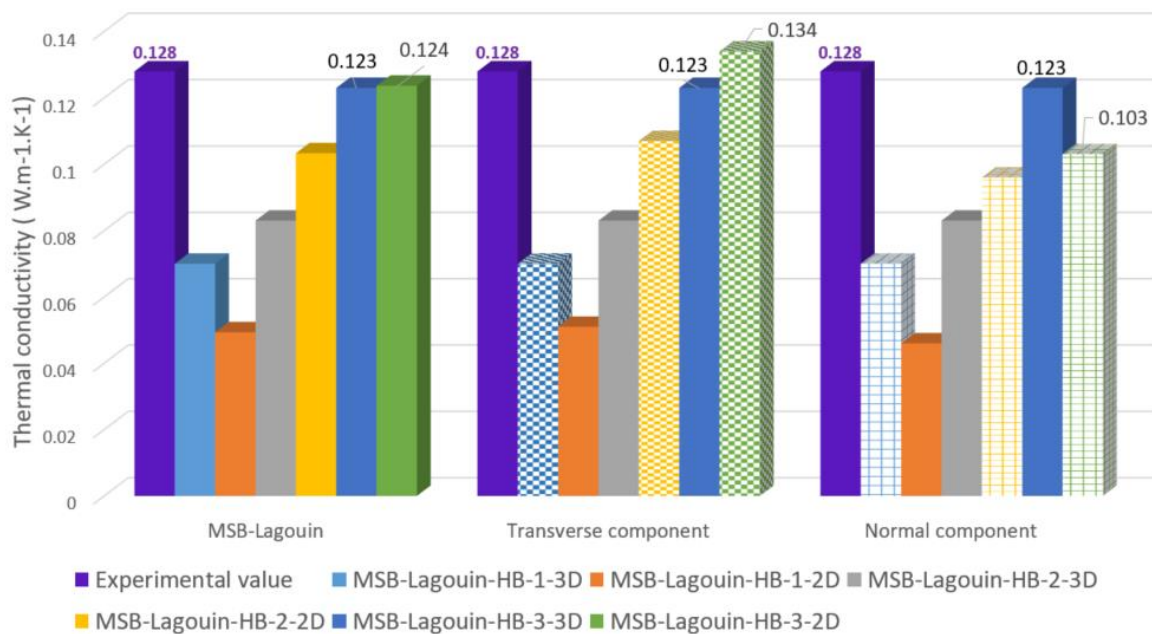


Figure 9. Results of predicted values concerning MSB-Lagouin models compared to the experimental value.

- The conclusions are similar to previous ones (for lime-based composite) :RVE HB-1 models are again clearly irrelevant with a relative deviation of over 60% from the experimental target value, indicating their lack of relevance.
- Composites modeled by RVE HB-2 give also unsatisfactory results with a deviation of around 40%., systematically below the experimental value.
- Better results are obtained with homogenization using the double inclusion scheme, where both the HB-3-3D and HB-3-2D models show a deviation of less than 5%

(considering the average value). Given the measurement uncertainty, this composite has a thermal conductivity value between $0.119 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ and $0.137 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$. The values of 0.123 and $0.124 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ predicted by the MSB-Lagouin-HB-3 models are therefore entirely appropriate. The LMB-Lagouin-HB-3 is consequently retained for the optimization process. Similar conclusions can be drawn to those of the LSB composite concerning the alignment between the chosen model and the material structure, explaining the excellent fit between model and real composite behavior.

Finally, the anisotropy ratio between the normal and tangential components is 1.1 for the HB-1-2D and the HB-2-2D models and 1.3 for the HB-3-2D models, based on the experimental values from the literature [68], [109], [110]. It supports the idea that analytical homogenization is promising for predicting anisotropic effects in plant-based concretes. The double-inclusion model is the most relevant for each of the studied plant-based concretes, showing its potential suitability for this type of composite. Logically, this model is retained and calibrated (concerning the coating proportion applied on the plant aggregate) before the optimization phases.

3.2 Optimization of thermal conductivity

3.2.1 Reference values and models

For each study in this section, only the appropriate calibrated model for each composite is considered. When particle orientation is not investigated, a random distribution is retained if it offers a smaller relative deviation from the experimental value than in the case of orientation in a plane. To study the effect of an aggregate change (nature or shape factor), it was assumed that the other modeling parameters remained unchanged. For the optimizing process, the reference thermal conductivity- noted $\lambda_{model}^{composite}$ for each model- is the one given by modeling.

The results given by the selected models and their calibration values (concerning the percentage of coating) are listed and compared to the experimental data in Table 8.

Table 8. Recapitulation of the models chosen for each composite and the reference thermal conductivity values before optimization.

Plant-based concretes		
Composite	LSB-Lagouin	MSB-Lagouin
Model type	HB-3-2D	HB-3-2D
Calibrated value	20% coating	20% coating
$\lambda_{composite} [\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}]$	0.127	0.128
$\lambda_{model}^{composite} [\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}]$	0.132	0.124
Relative deviation	4 %	4 %

From this point, simulations were carried out by varying various criteria: nature of the plant aggregate, aggregate shape factor and aggregate orientation. Assuming that the other parameters remain unaltered and the selected models reliable, thermal conductivity values modelled in the various optimization cases can be expected to be within 7 % of the real value, i.e. the value experimentally measured. This is entirely consistent with the measurement

uncertainties given by the reference chosen for plant-based concretes [21] , which is 6-7% depending on the composite considered.

3.2.2 Depending on nature of aggregate

The simulations enable to envisage a change of aggregate of each composite in order to compare the effect on the material's thermal conductivity. The initial composites, LSB-Lagouin and MSB-Lagouin, are made from sunflower bark. Thanks to knowledge about the thermal conductivity value of plant aggregates (cf. section 2.8.1) such as hemp shiv or sunflower pith, it is possible to simulate a change of aggregate in the composite. The names of the modeled composites indicate the letters H for hemp shiv, SP for sunflower pith instead of the initial SB in reference of sunflower bark. The simulation results are summarized in Figure 10.

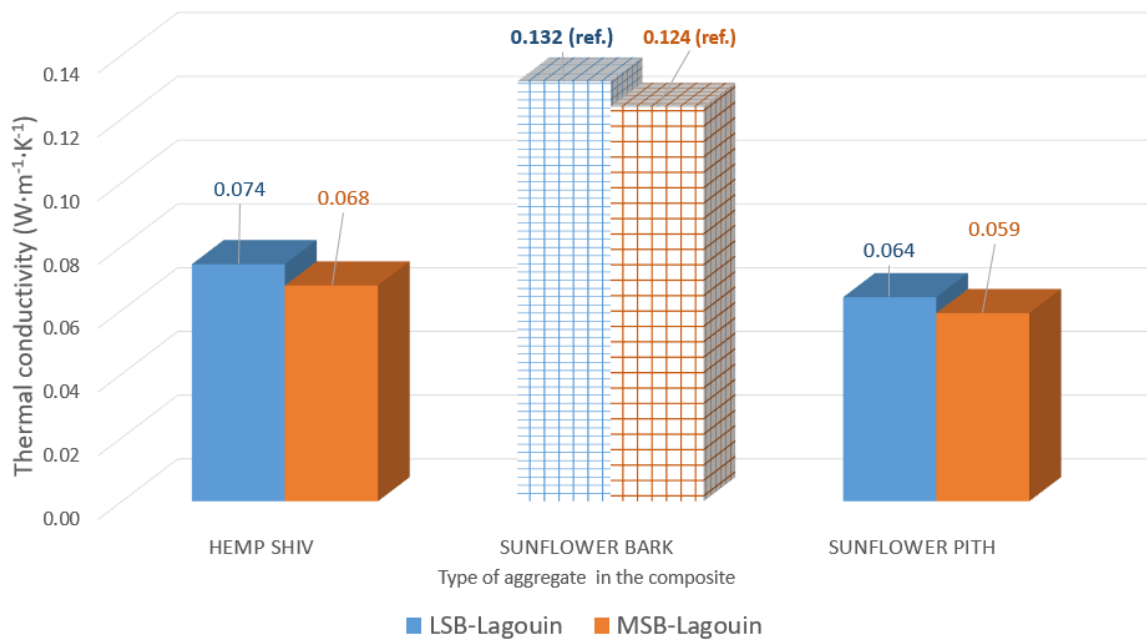


Figure 10. Impact of a plant aggregate change in modeled composites.

According to the modeled values, replacing the bark in the LSB- Lagouin composite with sunflower pith divides by two the initial material's thermal conductivity. The use of hemp shiv instead of sunflower bark reduces the initial value by respectively 44% and 45 % for LSB and MSB-Lagouin composites. The situation is very similar for both composites which is consistent with their initial comparable thermal conductivities (cf. section 2.4). The use of sunflower pith allows to divide the initial value by two.

The use of sunflower pith as a plant-based aggregate offers the best thermal performance whatever the composite considered. It is consistent since this aggregate has the lowest particulate thermal conductivity. In contrast, composites with sunflower bark are the least thermally efficient due to the significantly higher particulate thermal conductivity of this aggregate compared to hemp shiv and sunflower pith.

For further consideration, it is interesting to compare the results with hemp shiv, as it is the granulate reference in today's literature. While sunflower pith aggregate is 30% more thermally efficient than hemp shiv, when it replaces it in plant-based concrete, the thermal gain is only 15% on average for the two formulations studied here (metakaolin-based and

lime-based). This is due to the binder phase, which counterbalances the effect of the change in aggregate.

Furthermore, while sunflower bark aggregate is three times less thermally efficient than hemp shiv, when it replaces the latter in plant-based concrete, thermal performance is halved. Once again, the effect of the binder attenuates the disparities associated with aggregates. However, it is important to note that the choice of aggregate is a key element in the expected performance, and therefore a key factor in the choice of formulation.

3.2.3 Depending on aggregate shape

Because of the variability of the resource, the literature reports different aspect ratio values for a given type of plant aggregate. Thus, assuming the same particulate thermal conductivity, the shape factors of the aggregates in each composite were varied over the range reported in the literature (cf. section 2.6). The results of the homogenization calculations are presented in Figure 11.

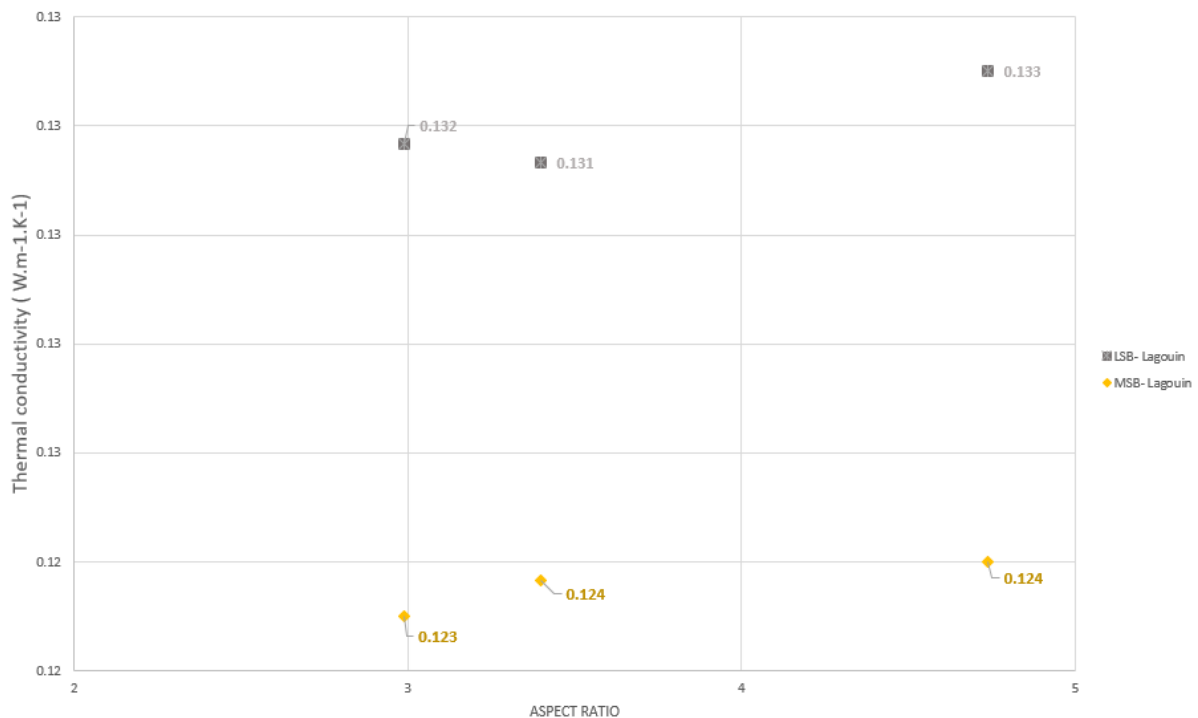


Figure 11. Impact of an aggregate aspect ratio change in modeled composites.

According to our homogenization calculations on the selected composites, the aspect ratio seems to have very limited influence on their thermal conductivity. However, when compared with measurement uncertainties (cf. section 3.2), this impact is largely negligible concerning the thermal conductivity of the studied composites. It is worth noting that the modeling was carried out with fixed aspect ratios, whereas in reality a certain size distribution exists in plant aggregates, which can be assessed by particle size analysis[77]. This study could therefore be extended by considering the results of granulometric analysis of aggregates used in plant-based concretes.

3.2.4 Depending on aggregate orientation

Current processing techniques tend to favor a preferential orientation of plant particles in the plane perpendicular to compaction or projection. In order to consider the development of new techniques that would enable plant aggregates to be oriented differently, the full range of possible orientations has been considered in this study. For obvious reasons of symmetry, only the range from 0 to 90° is presented in this section. The results for average thermal conductivities values are first presented in Figure 12.

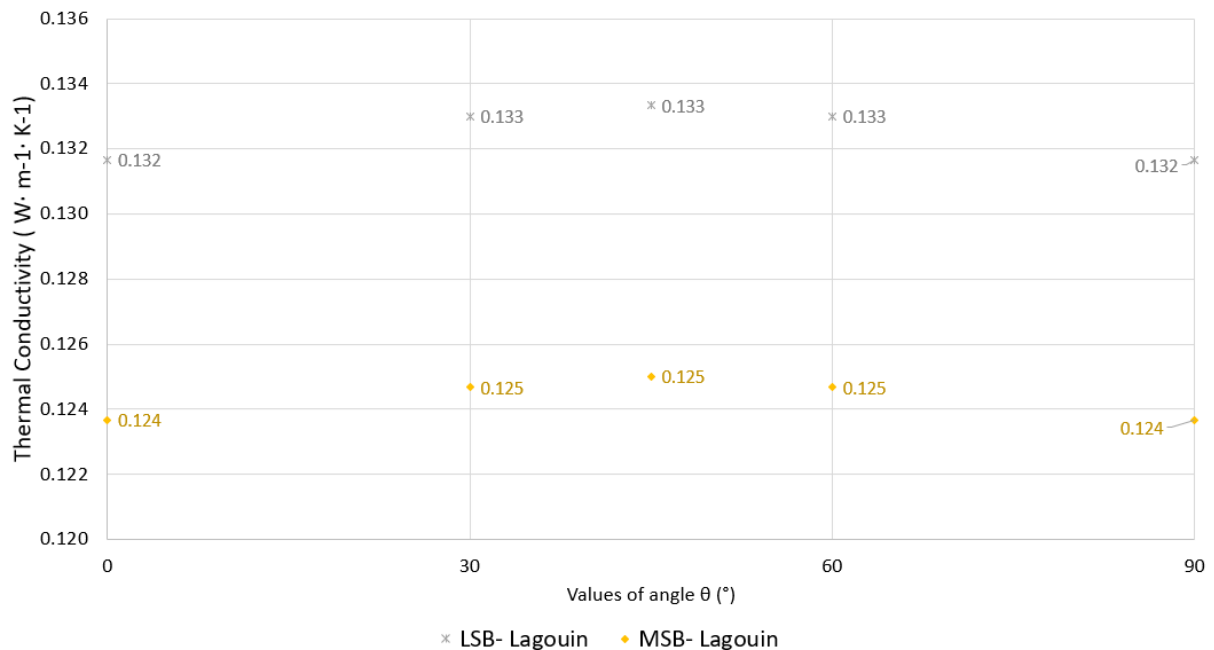


Figure 12 . Impact of a granulate orientation change in modeled composites: case of average thermal conductivity values.

Considering only average thermal conductivity values, particle orientation appears to have little influence on thermal conductivity in the case of the composites studied (less than 1% difference between the different values).

Nevertheless, it is interesting to examine the variation in the component values of the thermal conductivity tensor as a function of particle orientation. For each space component (defined in section 2.42.), the results are presented in the Figure 13.

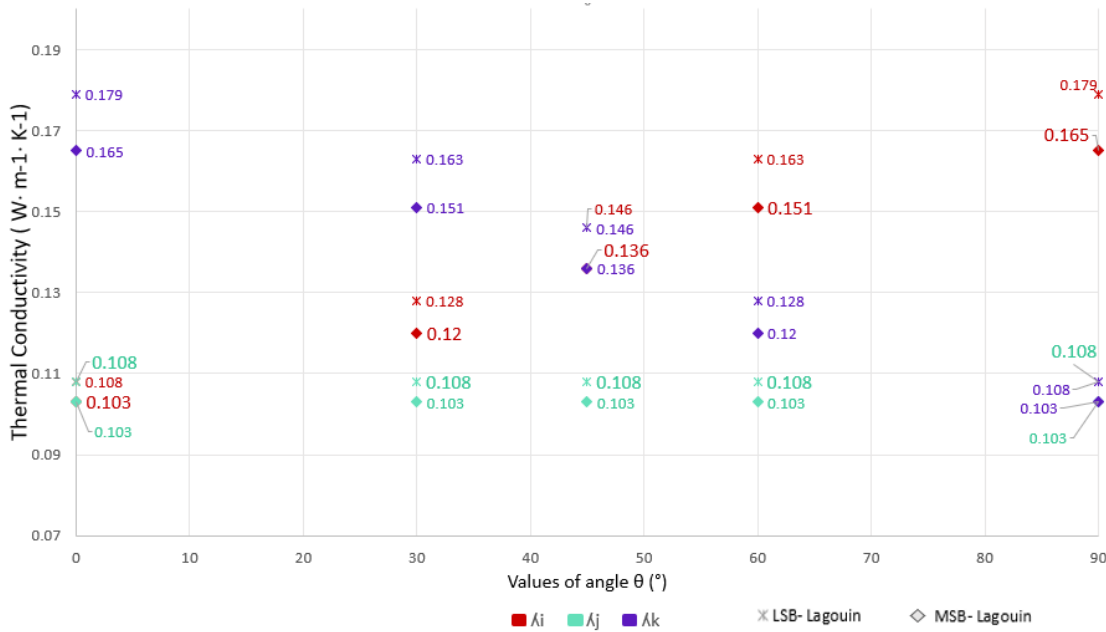


Figure 13. Impact of a granulate orientation change in modeled vegetal concretes: case of thermal conductivity tensor components.

Depending on the orientation of the plant particles, the composite's thermal conductivity in a specific direction can vary about 60% in both plant-based concretes. The marked anisotropy of thermal behavior may help to optimize the orientation of a prefabricated block of plant concrete in a wall, for example, to ensure efficient insulation performance. In order to be more explicit about the most favorable or unfavorable cases for each of the composites, the following table summarizes the extremum results (Table 9).

Table 9. Extreme thermal conductivities for each modeled composite

Plant-based concretes		
Composite (λ in $W \cdot m^{-1} \cdot K^{-1}$)	LSB-Lagouin	MSB-Lagouin
$\lambda_{max}/\lambda_{min}$	1.7	1.6

No values can be found in the literature concerning sunflower bark-based composites to assess the relevance of these results. However in the case of hemp-based composites based,

the ratio between normal and tangential thermal conductivity reported by experimental work is about 1.5 for non-optimized composites [29], [112], [113], i.e. where particle alignment is not necessarily the same. The results presented here are consequently likely to be consistent. The influence of the binder is not considered as it has an isotropic thermal behavior according to our assumptions

Finally, the results of this section highlight that processing techniques controlling particle orientation could be used to optimize the thermal properties of a bio-based material with a given formulation. These modeling results demonstrate how the exploration of a controlled preferential orientation could help optimize the thermal performance of plant-based concretes.

3.2.5 Depending on hydraulic binder

The change of hydraulic binder was tested on the LSB-Lagouin composite. The modeling results for three hydraulic binders differing in density and hence thermal conductivity are presented in Figure 14.

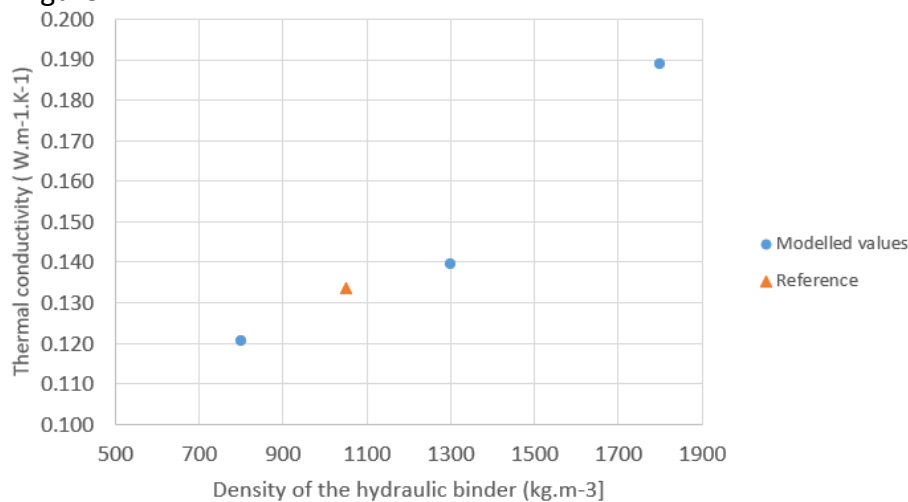


Figure 14. Thermal conductivity of LSB-Lagouin composite according to hydraulic binder density based on modeling results.

The results suggest that the binder can significantly influence the thermal conductivity of plant-based concrete in wall formulations. When the hydraulic binder is increased from 800 to 1800 kg.m⁻³, the thermal conductivity of the plant-based concrete increases by almost 60%. However, over a range from 800 to 1300 kg.m⁻³, this variation is only 16%. It can therefore be hypothesized that experimental observations and conclusions on the influence of binder on the thermal conductivity of hydraulic matrix plant-based concretes depend on the range of binder densities involved. This point could explain the different views expressed in the literature on the influence of binder type on the thermal conductivity of plant-based concretes [15], [22].

3.3 Multi-criteria optimization of the formulation

In order to compare different possible formulations based on locally available raw materials, optimizing thermal properties does not simply mean achieving the lowest thermal conductivity. It involves weighing up a whole range of more or less dependent criteria. A multi-criteria analysis is therefore proposed to suggest criteria for optimizing formulations prior to

the manufacturing stage. It is based on the thermal behavior results presented in this article, as well as the mechanical, hygroscopic and availability aspects discussed in section 2.7. The optimization process is based on exploring the possibility of changing the plant aggregate in the initial composite. Since the only difference between LSB and MSB Lagouin composites is the binder type, the results are combined in a single graph to study the effect of both a change in aggregate and a change in binder (Figure 15).

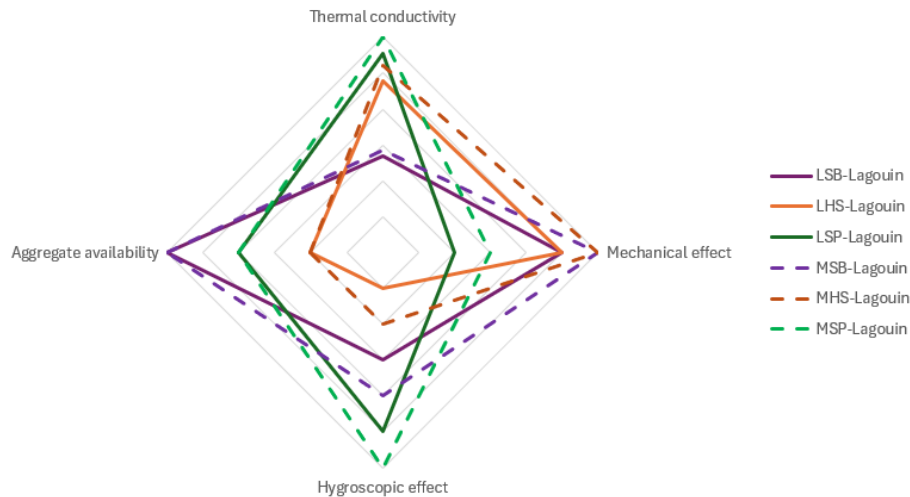


Figure 15. Multi-criteria comparison of vegetal concretes (change of aggregate and binder).

This study provides a basis for guiding choices upstream of a manufacturing stage depending on the objectives being pursued. With a view to using widely available raw materials to minimize environmental impact, sunflower-based materials are of particular interest. In these circumstances, if mechanical performance is not a priority and a material with high hygro-thermal performance is required, the MSP-Lagouin composite- based on metakaolin and sunflower pith- is the best candidate. To obtain a material with good mechanical, hydric and thermal performances, MSB-type composites, i.e. based on metakaolin and sunflower bark, are the most appropriate. Finally, it is interesting to note that the result discussed in section 3.4.5 on the influence of the binder is similar in this case. With a change of binder but retaining the same aggregate (lines of the same color in solid or dotted lines), the influence on the thermal conductivity of the composite is relatively slight for binders with a density of around $1000 \text{ kg}\cdot\text{m}^{-3}$.

4. Conclusion

In this study, the aim is to propose a relevant tool for predicting and optimizing the thermal conductivity of plant-based concretes. This work demonstrates that modeling opens the way to a efficient tool to guide the formulation choice. Indeed, modeling is used to address a number of strategic points that are currently difficult to study under experimental conditions Two composites rigorously selected from the literature are used to support the modeling work: one limed-based and another metakaolin- based, both using sunflower bark. This is particularly original in view of the few modeling literature studies, which only consider composites based on hemp shiv.

To ensure consistency, the proposed models consider the macrostructure as well as the microstructure of the chosen materials. In addition, to contribute to the tool's wide diffusion

and use, modeling input data are readily available thanks to recent work at the particle scale [64]. In the case of missing data, such as the proportion of aggregate coating by the binder matrix, model calibration is proposed. The presented approach is relevant as it ensures a difference between experimental and modelled thermal conductivity values of less than 5%. This is within the range of measurement uncertainties. The Mori Tanaka model reveals its limitations in the studied composites with a high proportion of inclusions. In this case, the double inclusion model is much more appropriate. The results reveal that mean-field homogenization is an effective tool for modeling the thermal behavior of bio-sourced materials. It could also be interesting to evaluate the Mori-Tanaka model with a matrix made up of air and an aggregate whose effective thermal conductivity (considering aggregate and hydraulic matrix) is assessed by initial homogenization. Furthermore, to gain in robustness, this approach will need to be applied to a wider range of bio-based materials. However, this remains difficult today due to a notable lack of data, particularly concerning the material's microstructure. At this stage, the study's conclusions are consequently limited to the materials selected. Experimental and modeling work carried out in parallel would enable to broaden the conclusions of this study.

However, with a view to optimizing the formulation for thermal performance, this study sheds light on the following interesting points:

- i) The nature of the aggregate is a fundamental criterion. Replacing sunflower bark with sunflower pith can halve the thermal conductivity of a bio-based composite, especially when the aggregate proportion in the formulation is particularly significant as in vegetal concretes.
- ii) The aggregate shape has a minor impact on the composite thermal conductivity on a macroscopic scale for the same type of aggregate.
- iii) The orientation of plant particles has a significant impact on thermal conductivity of the vegetal concrete. The difference between normal and tangential components (along or perpendicular to the compaction axis) can be as high as 60%. These results underline that appropriate processing techniques can make a major contribution to optimizing the thermal performance of bio-based materials.
- iv) The impact of a binder change is limited as long as it remains within the same density range, since it has been revealed - on the basis of a collection of experimental values - that a polynomial relationship exists between the thermal conductivity of the binder and its density.

It is especially important to note that, as long as the input data is available, those important conclusions are obtained without any time-consuming calculation step thanks to the developed tool.

Finally, a simplified multi-criteria study provides clearer picture of the impact (environmental impact, thermal, hygric and mechanical performance) of the choices concerning formulation that can be made upstream of the manufacturing phase.

As a continuation of this work, exploring the possibility of mixing plant aggregates to meet local and seasonal availability of aggregates is an interesting perspective. In addition, the models can be refined by considering the dispersion of aspect ratio values for the same type of aggregate. Moreover, the nature of aggregates imposes an intrinsic variability that has not

been included here (aspect ratios are considered constant). Recent studies have also demonstrated that plant aggregates swell or shrink under the effect of relative humidity [114] : this phenomenon has to be integrated with quantitative terms in order to help models to measure the impact of water sorption on thermal insulation.

In addition and to go beyond the material scale considered here, the wall scale would be interesting to consider. Indeed, as thermal comfort in buildings is directly linked to the thermo-hygric coupling within bio-based building materials [115], the projection of the modeling tool to a larger scale is probably very promising.

Finally, the developed virtual models represent real bio-based materials: they could be complexified and fed with new experimental data, such as fire resistance and sustainability indicators. This work is the precursor of digital twins that could enable us to anticipate the complex behavior of eco-materials under various types of use. To achieve this, the modeling process will have to be able to incorporate a lot of information from experimentation [116]. However, given the extreme diversity of possible formulations concerning bio-based materials, new techniques of data exploitation - such as Bayesian networks - will probably help to overcome the inevitable lack of data [117], [118] .

5. Acknowledgements

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Chapitre 8. Conclusion, synthèse et perspectives

8.1 Bilan scientifique et synthèse des résultats

Ces travaux de thèse se concentrent sur les matériaux de construction bio-sourcés valorisant des co-produits agricoles. Une attention particulière est portée sur le cas du tournesol, disponible localement dans le Sud-Ouest de la France mais également largement cultivé à travers le monde.

L'objectif premier était de pouvoir prédire le comportement thermique – i.e. la conductivité thermique effective – d'un matériau de construction bio-sourcé (panneau isolant, terre allégée ou béton végétal) en amont des étapes de fabrication. Il s'agissait donc de lever certains verrous scientifiques liés à la variabilité de la ressource [1] tout en proposant des méthodes pertinentes et facilement applicables pour un transfert efficace du laboratoire au chantier. Cet aspect, particulièrement important pour pouvoir promouvoir ces éco-matériaux, est un des principes fondateurs du travail de modélisation proposé. L'accessibilité des données d'entrée permet ainsi une applicabilité rapide de cette contribution, au plus près des besoins et des attentes des acteurs des économies locales de la construction [2]. Face à l'urgence climatique et environnementale à laquelle nos sociétés sont confrontées, ces travaux s'inscrivent pleinement dans une démarche de valorisation et de développement des matériaux bio-sourcés dans le secteur de la construction.

La démarche multi-échelle adoptée dans ces travaux permet d'explorer différentes spécificités, de l'échelle moléculaire à l'échelle du matériau (vrac, panneau de particules, béton végétal, terre allégée...), en passant par l'échelle stratégique du granulats végétal. Cette échelle particulière permet notamment de traiter de différents points sensibles comme la variabilité de la ressource ou encore la variation de la conductivité thermique en fonction des sollicitations extérieures, i.e. de la température et de l'humidité relative. Ainsi, chaque échelle étudiée permet de contourner certaines difficultés rapportées jusqu'ici dans la littérature, comme il est spécifié dans la figure 1.

Echelle moléculaire

- Prise en compte de la nature biologique des granulats végétaux pour mieux comprendre leur caractère hygroscopique.
- Mise en évidence d'un couplage hydro-mécanique à l'échelle moléculaire avec des conséquences macroscopiques à l'échelle matériau.

Echelle particulaire

- Détermination semi-expérimentale de la conductivité thermique particulaire d'une large gamme de co-produits agricoles.
- Etude de l'impact des conditions d'usage (température et humidité relative) sur la conductivité thermique particulaire.
- Prise en compte du comportement anisotrope en considérant la réelle microstructure des granulats végétaux.

Echelle matériau

- Mise en oeuvre d'un panneau isolant 100% biosourcé (sans liant ni process énergivore) à base de granulats localement disponibles.
- Détermination de performances thermiques équivalentes pour l'isolation en vrac utilisant différents co-produits agricoles.
- Prédiction de la conductivité thermique de matériaux à base de granulats végétaux incorporés dans une matrice liante (béton végétal, terre allégée...) à partir de leur morphologie et de leur formulation.
- Etude de critères d'optimisation de la conductivité thermique de matériaux bio et géo-sourcés.

Figure. 1. Synthèse des travaux menés et des contributions associées aux différentes échelles.

L'articulation de l'échelle microscopique à l'échelle macroscopique permet en outre de dissocier certains phénomènes physico-chimiques et de se concentrer sur ceux qui prédominent selon le niveau considéré [3]. C'est une méthode qui s'avère efficace pour traiter de la grande complexité - tant sur le plan du comportement que de la morphologie [4],[5] - des matériaux étudiés. Un des premiers constats est que l'étude de la microstructure des matériaux de construction bio-sourcés est un axe de recherche prometteur pour progresser dans la connaissance de ces derniers [6]. La prise en compte de cette échelle, plus fine que celle généralement traitée dans la littérature, nécessite de croiser des compétences dans plusieurs domaines scientifiques pour comprendre et explorer au mieux les différents phénomènes mis en jeu.

Ce travail interdisciplinaire, basé sur une étude multi-échelle, fournit des résultats significatifs, rassemblés dans la figure 2. Ils permettent notamment de mettre en lumière la portée de ces travaux de thèse, que ce soit pour de futurs travaux expérimentaux ou de modélisation.

Enfin, il est important de noter l'applicabilité directe de cette contribution ce qui devrait pouvoir encourager de manière significative à l'utilisation des matériaux bio et géo-sourcés dans les projets de construction d'aujourd'hui et de demain.

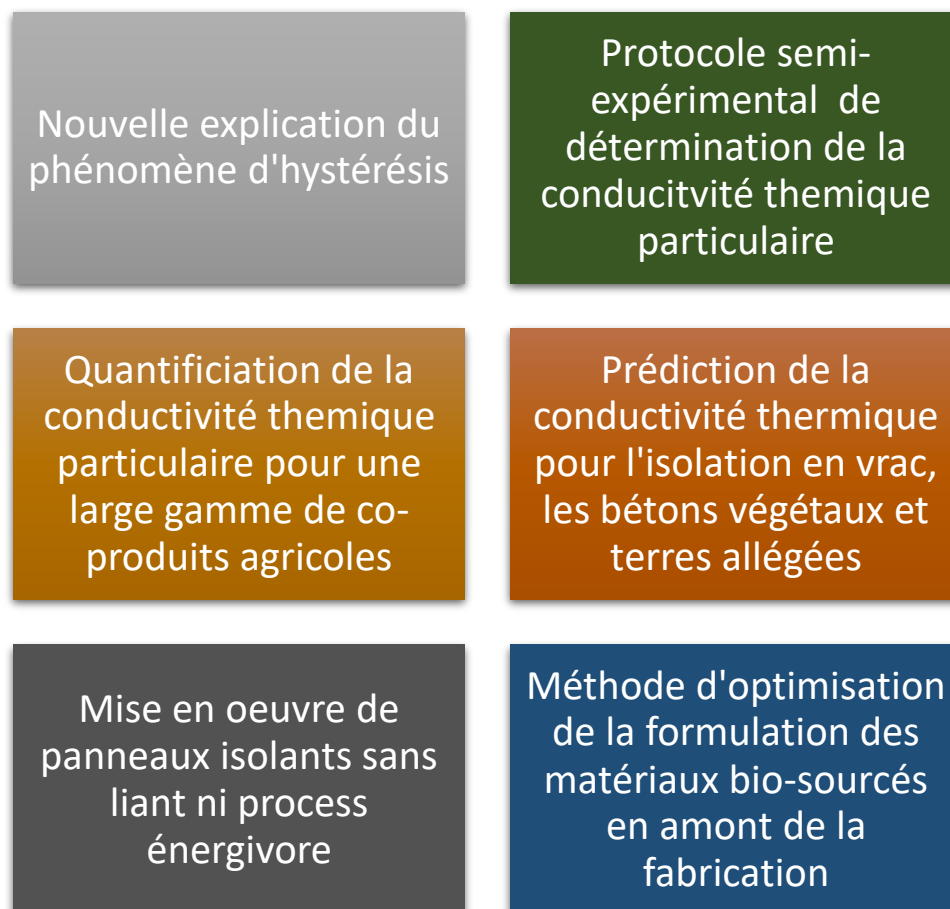


Figure 2. Principaux résultats de ces travaux de thèse

8.2 Axes de développement futur

8.2.1 Prolonger les axes de travail de la thèse

La considération de l'échelle moléculaire (chap. 3) montre combien les interactions à cette échelle peuvent influencer le comportement macroscopique des matériaux bio-sourcés. Il serait donc intéressant de poursuivre l'exploration des phénomènes qui se jouent à ce niveau, que ce soit sous l'effet de l'humidité ou encore de la température. Les techniques d'imagerie couplées à la simulation moléculaire pourraient livrer de précieux enseignements sur le comportement des éco-matériaux étudiés dans ces travaux de thèse. D'ailleurs, ces derniers se fondent sur un principe récemment démontré par simulation moléculaire [7] pour proposer un nouvel éclairage sur un phénomène largement observé dans les matériaux bio-sourcés, l'hystérèse (chap. 3). Cependant, de nombreuses interrogations subsistent sur ce mécanisme. Dans de nombreux domaines, comme l'optique ou l'électromagnétisme, l'aire du cycle d'hystérésis fournit des informations sur l'état du système étudié [8], [9], [10]. Une étude approfondie sur ce point pourrait donc permettre de mieux comprendre les conséquences d'une hystérèse, plus ou moins marquée, au niveau du matériau étudié.

Une hypothèse forte a été faite à l'échelle particulaire (chap. 4, 5 et 6) concernant la conductivité thermique du squelette solide des granulats végétaux. Seule la conductivité thermique de la cellulose - et sa dépendance en température - est disponible dans la littérature [11]. Bien que ce composé chimique soit majoritaire dans les granulats végétaux issus de co-produits agricoles [12], il serait intéressant d'avoir accès à la conductivité thermique des autres composants chimiques - comme l'hémicellulose ou encore la lignine - pour affiner les résultats présentés dans ce manuscrit. A défaut, il est possible de remonter à la conductivité thermique particulaire par méthode inverse à condition que la fraction volumique des granulats végétaux soit connue au moment de la mesure. Sur la base des méthodes semi-expérimentales proposées dans ces travaux (chap. 4), l'application d'une méthode de caractérisation des granulats disponibles localement pourrait permettre d'accéder à leur conductivité thermique particulaire. L'objectif est de favoriser l'utilisation des ressources locales pour une valorisation des déchets agricoles au plus près du chantier de construction.

Concernant le panneau de particules à base de moelle de tournesol, développé et étudié dans le chapitre 5, les particules végétales ont été considérées isotropes (assimilées à une sphère). Ceci est particulièrement pertinent quand la particule de moelle est considérée seule et soumise à aucune contrainte [13]. Cela est également justifiable pour un mélange en vrac, dès lors que le tassement dû au poids des particules reste négligeable. En toute rigueur, un certain écrasement est inévitable dans le cas des panneaux de moelle à cause de la technique de mise en œuvre (compactage sous presse hydraulique). Bien que macroscopiquement, le panneau regagne en volume après compactage, les particules pourraient ne pas retrouver leur configuration initiale à cause d'une déformation non élastique de leur squelette solide. Cela pourrait induire, notamment, une anisotropie non négligeable dans le comportement thermique du granulat. Il s'agirait alors de prendre en compte cette déformation dans la modélisation proposée pour affiner les résultats prédictifs. Une étude à ce sujet permettrait de vérifier ou d'infirmer l'hypothèse faite dans ces travaux qui consiste à négliger une éventuelle déformation résiduelle après compactage. Le but étant de pouvoir estimer au

mieux la conductivité thermique particulière d'une part, puis celle du panneau dans un second temps, afin de pouvoir garantir une gamme de conductivité thermique selon les conditions d'usage (humidité et température) et prendre en compte les cas les plus défavorables. A terme, il s'agit d'anticiper les performances thermiques d'un tel matériau et donc les consommations énergétiques associées à l'échelle du bâti. Par ailleurs, les résultats donnés par le modèle prédictif concernant le panneau de moelle de tournesol ont été comparés à des valeurs mesurées par la technique du fil chaud. Cette technique expérimentale pourrait cependant montrer des limites [14] et il serait pertinent de comparer les résultats obtenus à des valeurs mesurées à la plaque chaude gardée pour compléter l'étude proposée. La mesure expérimentale de la conductivité thermique des matériaux bio-sourcés est aujourd'hui un point délicat [15],[16]. Les travaux menés dans cette thèse soulignent que les conditions de mesure devraient être systématiquement explicitées (conditions de température, d'humidité relative et fraction volumique de l'échantillon non précisée) pour fournir les jeux de données indispensables à la modélisation et à la prédiction du comportement thermique. Modélisateurs et expérimentateurs doivent collaborer davantage pour développer des méthodes de caractérisation fiables et pertinentes afin d'alimenter des bases de données exploitables, notamment pour les techniques d'homogénéisation. Enfin, mis en œuvre sans aucun liant, les panneaux à base de moelle de tournesol doivent faire l'objet de caractérisations supplémentaires pour comprendre, dans un premier temps, la cohésion naturelle qui se forme entre les granulats de moelle après humidification. Aussi, la quantité d'eau nécessaire doit être évaluée et optimisée en vue d'une possible étape d'industrialisation d'un tel process. En complément, l'étude de la durabilité de ces panneaux low-tech, et notamment la question de la cohésion à long terme des particules, reste un sujet à explorer.

En ce qui concerne la variabilité de la ressource (chap. 6), les travaux peuvent être poursuivis par une campagne de caractérisation expérimentale menée sur une large gamme de co-produits agricoles. A ce jour, aucune étude expérimentale ne fournit des données fiables et complètes à visée comparative pour des granulats végétaux issus de co-produits agricoles. Il faut pourtant pouvoir comparer les caractéristiques particulières (porosité intra particulaire, facteur de forme, composition chimique du squelette solide, ...) au sein d'un même type de plante (même plante, même variété, même zone de récolte) et faire varier différents paramètres comme l'année, la période ou encore la technique de récolte. L'impact du process de valorisation des co-produits agricoles (traitement, transformation, adjuvantation) est également un point important à traiter. La difficulté réside dans le grand nombre d'échantillons à traiter pour pouvoir avoir des conclusions suffisamment robustes sur le plan statistique. Par ailleurs, une mesure conjointe de la conductivité thermique particulière par thermographie infrarouge [17] permettrait de consolider ou non les modèles proposés pour les différents types de granulats (modèles R et B du chapitre 6). Une fois de plus, modélisation et expérimentation sont complémentaires : une collaboration étroite est nécessaire pour développer des modèles prédictifs du comportement thermique des matériaux bio et géo-sourcés malgré la variabilité inhérente aux matières premières.

Le chapitre 7 propose des méthodes de prédiction puis d'optimisation des performances thermiques des matériaux bio et géo-sourcés en s'appuyant sur des travaux expérimentaux de la littérature. Bien que ces derniers aient été choisis avec la plus grande rigueur, pour limiter au maximum les biais dû à la variabilité des matières premières, une étude ciblée et

menée conjointement sur le plan expérimental et numérique permettrait de consolider les conclusions proposées. Il s'agirait de fabriquer différents échantillons de terres allégées et de bétons végétaux avec une caractérisation thermique préalable de chacune des phases constitutives (liant, granulat). L'information sur les fractions volumiques de chacune des phases ainsi que l'orientation des particules végétales serait confirmée par analyse d'image (via la tomographie à rayons X par exemple [18]). A l'image de la caractérisation thermique, une caractérisation mécanique des phases seules (par micro indentation par exemple [19], [20]) puis des composites permettrait de développer des modèles multi-échelles pour déterminer les propriétés mécaniques effectives des matériaux. Pour compléter l'analyse multicritère amorcée dans le chapitre 7, des informations supplémentaires concernant les propriétés hygriques des matériaux seraient nécessaires. Plusieurs verrous restent à lever pour utiliser la modélisation multi-échelle à des fins de prédiction de la perméabilité effective des matériaux bio et géo-sourcés, en raison notamment de la complexité induite par la phase fluide contenant de l'eau et de l'air (changement d'état de l'eau présente dans le matériau en fonction de l'humidité ambiante et de la température). En outre, l'ajout de critères liés au comportement au feu [21] et aux performances acoustiques [22] des matériaux considérés permettrait d'avoir une vision encore plus globale. Cela aiderait ainsi à la prise de décision vis à vis des formulations à privilégier selon l'usage visé. Enfin, un recensement large de la disponibilité (y compris de la saisonnalité) des co-produits agricoles à l'échelle du territoire (français, européen et mondial), favorisera l'utilisation des matériaux à base de ressources locales, en adéquation avec l'approvisionnement en matières premières. Des économies locales de la construction pourraient émerger en se saisissant de l'opportunité de construire avec des matériaux locaux. Cela serait à la fois une solution à des problématiques de gestion des terres d'excavation [23] ou des fines de lavage des granulats [24] et un complément de revenus potentiel pour les agriculteurs par la valorisation des déchets agricoles [25]. Avec une vision globale et objective des possibilités de valorisation des ressources locales (co-produits agricoles, terre crue), à la fois en termes de performances techniques et environnementales, l'optimisation des techniques de mise en œuvre sera un atout supplémentaire pour une diffusion large des matériaux bio et géo-sourcés dans l'industrie de la construction.

8.2.2 Pallier le manque de données dans la littérature

Outre l'aspect multi-échelle, il est à noter que la méthodologie adoptée dans ces travaux de modélisation a nécessité de poser un certain nombre d'hypothèses. Bien que discutées dans les différents chapitres, il est important s'insister sur celles qui relèvent d'un manque de données dans la littérature. Ainsi, deux éléments paraissent fondamentaux pour affiner les résultats présentés dans ces travaux.

D'abord, la conductivité thermique de la cellulose, au sens squelette solide (ou plus précisément microfibrilles), et sa dépendance en température sont disponibles uniquement pour un échantillon extrait de la pâte de bouleau [11]. La disposition de ces microfibrilles dépend de la croissance de la plante. Chaque configuration impacte inévitablement les propriétés mécaniques mais cela pourrait, en toute vraisemblance, avoir également des conséquences sur les propriétés thermiques, bien que la dépendance en fonction de la densité semble limitée [26]. Par ailleurs, il serait pertinent de pouvoir avoir accès à la conductivité thermique (et ses éventuelles dépendances en température) des autres

composés chimiques que l'on trouve dans les parois végétales des co-produits agricoles, comme l'hémicellulose ou la lignine [27]. Enfin, l'impact du vieillissement sur le squelette solide particulaire est un élément clé pour pouvoir appréhender l'évolution des propriétés thermiques des matériaux bio-sourcés à long terme, sous contraintes d'usage [28].

En outre, les modèles multi-échelles, obtenus par des techniques d'homogénéisation, nécessitent l'accès à la fraction volumique de chaque phase du matériau considéré [29]. Que l'approche soit analytique ou numérique, cette donnée d'entrée est fondamentale [30], [31], [32]. La littérature fournit des rapports massiques sur lesquels il est difficile de s'appuyer pour déterminer les rapports volumiques, en raison des porosités additionnelles de l'échelle matériau (porosité inter particulaire, porosité à l'interface liant- granulat végétal). Accroître le nombre d'images de la microstructure pourrait servir à constituer une base dans le but d'établir une correspondance pertinente entre formulation (rapports massiques) et proportion volumique des phases (porosité, liant, granulat) en présence.

Enfin, devant le manque de données expérimentales et face à l'extrême variabilité des matières premières, une approche bayésienne pourrait aider à lever certains verrous. En effet, dans des disciplines très diverses, elle permet de compenser le manque de données par une approche probabiliste [33], [33], [34].

8.2.3 Mener une analyse de sensibilité

Les modèles développés dans ces travaux permettent d'estimer la conductivité thermique à l'échelle particulaire puis à l'échelle matériaux. Pourtant, à chacune de ces échelles, la variabilité de la ressource impacte nécessairement la valeur des données d'entrée. Cette variabilité a été prise en compte en considérant la plage de porosité particulaire déterminée dans les travaux d'Haga Ratsimbazafy [35]. Pour une approche à la fois plus globale et plus objective, une étude de sensibilité permettra de mieux appréhender l'impact de la variabilité des données d'entrée sur les données de sortie [36]. En outre, il s'agirait d'évaluer la qualité des modèles proposés dans ces travaux vis-à-vis de l'influence de chacun des paramètres d'entrée, par comparaison aux observables des travaux d'expérimentation. La détermination des paramètres particulièrement influents est une information précieuse pour affiner les phases de caractérisation des matières premières. Ainsi, l'identification des paramètres d'entrée fondamentaux (vis-à-vis de la sensibilité des modèles) encouragera la mise en place de protocoles dédiés, spécifiques aux matériaux étudiés et à leur variabilité intrinsèque. Enfin, dans la perspective de viser un passage à l'étape industrielle, l'étude de sensibilité permettra de mieux appréhender comment relier les performances attendues sur le matériau fini et les propriétés des matières premières, malgré leur variabilité et la complexité de leur comportement [37].

8.2.4 Explorer d'autres méthodes d'homogénéisation

Dans ces travaux, les outils d'homogénéisation ont été choisis pour être pertinents tout en limitant le temps de calcul. En outre, ils livrent des relations analytiques permettant de passer alternativement de l'échelle microscopique à l'échelle macroscopique afin d'appréhender la complexité morphologique des matériaux étudiés et contourner le manque

de données concernant certaines phases (porosité particulière, interface granulat/liant notamment). Cependant, il existe d'autres méthodes d'homogénéisation qui pourraient se révéler intéressantes, comme celles utilisées pour l'évaluation de l'endommagement [38]. Ces dernières pourraient être particulièrement efficaces et transposables à la modélisation de la fissuration des éco-matériaux, que ce soit au niveau des terres allégées [39] ou des enduits [40]. Il est à noter que l'évaluation et la prédiction des propriétés effectives des matériaux multiphasiques est une problématique spécifique [41]. Les différents outils développés par cette discipline peuvent fournir des perspectives pertinentes pour progresser dans la connaissance et la compréhension des matériaux bio et géo-sourcés. Ainsi, l'approche analytique proposée dans ces travaux pourra être étendue via d'autres schémas d'homogénéisation, comme celui de Ponte Castenada et Willis [42], et complétée par une approche numérique [43]. Il conviendra de rechercher le meilleur compromis en termes de temps de calcul, de pertinence mais également d'applicabilité des modèles prédictifs. Le transfert de compétences du laboratoire aux acteurs de terrain (bureaux d'études, artisans...) est un maillon essentiel pour contribuer de manière significative au développement des matériaux bio et géo-sourcés.

8.2.4 Etendre l'étude multi-échelle

Ces travaux démontrent l'intérêt d'explorer différentes échelles. L'échelle moléculaire, et notamment les interactions physico-chimiques qui se produisent à ce niveau, peuvent aider à la compréhension et donc à la caractérisation des interfaces liant/granulat notamment. La modification de la composition chimique du squelette particulière, par les effets de pré-traitements, de rouissage ou encore par les techniques de récolte, sont également à considérer à cette échelle.

En complément de la mesure de la conductivité thermique, les techniques de micro et nano-indentation pourront fournir de précieuses informations sur le module d'Young local [19], [44], [45], toujours dans le but de fournir des informations sur les différents phases des matériaux. Informations nécessaires aux techniques d'homogénéisation.

L'échelle de la particule végétale constitue une strate stratégique, notamment pour prendre en compte les effets des impuretés de la matière première. En effet, concernant la moelle de tournesol, les techniques de séparation peuvent s'avérer énergivores [46] pour obtenir de la moelle pure, plus poreuse que l'écorce donc plus performante sur le plan thermique. Il sera donc important de pouvoir prendre en compte l'impact, sur le comportement thermique, des impuretés contenues dans le co-produit agricole récolté localement (traces d'écorce dans la moelle dans l'exemple précis du tournesol). Le processus de fabrication du matériau bio-sourcé, utilisant l'agro-ressource concernée, pourra être ainsi optimisé vis-à-vis des performances attendues tout en minimisant l'impact carbone. Ceci dans le but d'améliorer les indicateurs de l'analyse de cycle de vie (ACV) du bâti [47], de plus en plus important à considérer avec l'entrée en vigueur récente de la RE 2020 sur le territoire français. C'est aussi à l'échelle de la particule que la pertinence de l'utilisation d'un granulat végétal recyclé pourrait être étudiée. A cause des traces résiduelles de matrice minérale, la conductivité thermique particulière est fatalement impactée. La quantification de ces résidus minéraux sur le comportement thermique est un élément clé dans l'étude du réemploi des bétons végétaux.

La principale difficulté de l'échelle matériau est vraisemblablement l'information manquante sur chacune des phases. Des campagnes expérimentales ciblées, faisant le lien entre formulation et caractérisation des phases (fractions volumiques, orientation des particules, interface, fissuration...), permettront d'améliorer la fiabilité des modèles prédictifs à cette échelle. Des études récentes démontrent d'ailleurs la pertinence de cette démarche sur la laine de chanvre [48].

La compréhension des phénomènes mis en jeu ainsi que la validation des modèles prédictifs aux échelles précédentes, permettront de poursuivre efficacement les travaux à l'échelle de la paroi puis du bâti. Un des enjeux majeur est de pouvoir alimenter des bases de données pour mener efficacement un calcul d'ACV dynamique en intégrant les spécificités des matériaux bio et géo-sourcés [49].

8.2.5 Favoriser l'interdisciplinarité

A l'image de la réflexion menée sur le phénomène d'hystérésis, l'interdisciplinarité est probablement un des leviers les plus prometteurs pour progresser dans la connaissance des matériaux bio et géo-sourcés. C'est également une des conclusions principales de l'analyse bibliométrique présentée en début de ce manuscrit.

Le champ exploratoire est évidemment très vaste mais dans la continuité de ces travaux de thèse, il paraît intéressant d'explorer, à chaque échelle et pour chaque type de matériau, la globalité des phénomènes de transfert [50]. Par ailleurs, l'apport de la physique des transferts, couplée aux méthodes de Monte Carlo [51], [52], [53], [54], pourrait permettre de gagner en efficacité au niveau des changements d'échelle.

Les matériaux bio-sourcés sont connus pour être hygroscopiques et participer ainsi à la qualité de l'air intérieur. Il serait intéressant de poursuivre les investigations, pour explorer une possible adsorption des polluants par ces matériaux, comme les Composés Organiques Volatils (COV) [55].

De par leur nature organique, les matériaux bio et géo-sourcés nécessitent une attention particulière quant à l'évaluation de leur durabilité [56]. Aussi, une collaboration active avec des équipes de bio-chimie et de microbiologie notamment, devrait aider à mieux comprendre les mécanismes de dégradation de ces éco-matériaux. L'apport des techniques exploratoires utilisées dans les sciences du vivant [57] et des phénomènes de biodégradation spécifiques aux matières organiques [58], constitueront une base solide pour établir les préconisations de mise en œuvre et d'entretien du bâti.

Devant la grande complexité des matériaux bio et géo-sourcés, quelle que soit l'échelle considérée, des outils puissants issus de l'Intelligence Artificielle pourront probablement repousser les limites des connaissances dans ces domaines. Réseaux de neurones [59], [60], [61] et jumeaux numériques [62], [63], [64] commencent à montrer leur pertinence dans le domaine du génie civil : ce sont donc des pistes prometteuses pour des travaux futurs.

Enfin, les enjeux sociétaux visés par les matériaux de construction bio et géo-sourcés ne peuvent être décorrélées des sciences humaines et sociales [65], [66], [67]. De la difficulté à structurer des filières locales [68], en passant par les obstacles à l'écoconstruction [69], l'apport des sciences humaines est un levier indispensable à la démocratisation des matériaux bio et géo-sourcés.

8.3 Références

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Titre : Modélisation et optimisation des performances thermiques des matériaux bio et géosourcés par approche multi-échelle : apport à la valorisation d'une large gamme de co-produits agricoles.

Mots clés : Matériaux de construction bio-sourcés, Modélisation multi-échelle, Propriétés thermiques, Granulat végétal, Matériaux de construction géo-sourcés, Microstructure

Résumé : Face aux défis du 21^e siècle, les questions énergétiques et environnementales sont au cœur des préoccupations de nos sociétés. Le secteur du bâtiment, parmi les plus impactants, doit s'emparer de cette réalité pour opérer une transition à la fois rapide, pertinente et durable. L'utilisation de matériaux de construction bio et géo-sourcés permet d'améliorer le confort intérieur et l'efficacité énergétique du bâti tout diminuant l'impact environnemental de la construction. Dans ce cadre, le béton de chanvre est une alternative prometteuse qui se développe depuis plusieurs années. Cependant, de nombreux co-produits agricoles -autres que la chènevotte- peuvent être valorisés dans les matériaux de construction. Ces derniers sont, de surcroît, largement disponibles grâce aux différentes cultures implantées localement (tournesol, colza, lin, ...). Toutefois, de nombreux freins expliquent l'assurabilité délicate de ces éco-matériaux, ce qui limite aujourd'hui leur utilisation à grande échelle à des fins d'isolation répartie. Leur comportement complexe, face aux variations de température et d'humidité relative, est probablement un des principaux verrous à lever. Ces travaux de thèse visent donc à mieux comprendre les phénomènes physiques qui s'opèrent dans ces matériaux, à les modéliser et à proposer des modèles de prédiction de leur comportement thermique. Ils s'appuient principalement sur des techniques d'homogénéisation analytique (Mori Tanaka et Double Inclusion) permettant de considérer la variabilité de la conductivité thermique sous les contraintes d'usage. La considération d'une échelle stratégique, celle de la particule végétale, permet une application étendue à une large gamme de co-produits agricoles. Ainsi, l'analyse multi-échelle proposée permet de prédire et d'optimiser le comportement thermo-hygrique de ces éco-matériaux avant même l'étape de fabrication et en appui des travaux expérimentaux. Ces travaux devraient favoriser l'émergence d'économies locales autour de matériaux de construction sains, efficaces et écologique. Ils constituent des leviers stratégiques à la réduction des émissions de gaz à effet de serre visée par le Pacte Vert pour l'Europe, d'ici 2030.

Title: Thermal performance modeling and optimization of bio- and geo-based materials using a multi-scale approach: contribution to the valorization of a wide range of agricultural co-products.

Key words: Bio-based building materials, Multiscale modeling, Thermal properties, Bio-aggregate, Geo-based building materials, Microstructure

Abstract: The challenges of the 21st century require energy and environmental issues to be central concerns for society. The building sector, one of the most environmentally-impacting, must seize this opportunity to ensure a rapid, relevant and sustainable transition. The use of bio- and geo-based building materials allows improvements in indoor comfort and energy efficiency to be achieved, while reducing the building environmental impact. Hemp concrete is a promising alternative which has been developing for several years. However, many agricultural by-products - other than hemp shives - can be used in construction materials. Moreover, they are widely available thanks to the various local crops (sunflower, rapeseed, flax, etc.). Nevertheless, numerous obstacles explain the delicate insurability of these eco-materials, which currently limits their large-scale use for distributed insulation. Their complex behavior, when subjected to temperature and relative humidity variations, is probably one of the main obstacles to be overcome. The aim of this thesis work is therefore to gain a better understanding of the physical phenomena involved in these materials, to model them and to propose models for predicting their thermal behavior. It is mainly based on analytical homogenization techniques (Mori Tanaka and Double Inclusion) allowing the variability of thermal conductivity to be taken into account under use conditions. By considering a strategic scale, the plant particle one, it is possible to extend the approach to a wide range of agricultural co-products. The proposed multi-scale analysis enables the thermo-hygric behavior of these eco-materials to be predicted and optimized even before the manufacturing stage, and as a support for experimental work. This research is expected to encourage the emergence of local economies based on healthy, efficient and environmentally-friendly construction materials. They represent strategic levers in the reduction of greenhouse gas emissions targeted by the Green Pact for Europe between now and 2030.