Formulation and characterization of unfired clay bricks with plant aggregates

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Abstract

Construction is one of the most polluting sectors of industry, and this is why developing sustainable building materials is of world-wide interest. Earth is being increasingly studied as a building material because of its low environmental impact and its abilities to regulate indoor moisture and to improve the building occupants' comfort. Plant aggregates and fibers have been incorporated into the earth matrix for thousands of years to enhance its performance, but scientific studies began quite recently. Nowadays, the addition of renewable resources can be achieved with agricultural by-products, thus allowing carbon dioxide to be captured.

As part of the Bioterra project funded by the French National Research Agency (ANR), this thesis has the objective of contributing to the development of earth-based materials containing plant aggregates that can be used in bricks. After an extensive characterization of the different plant resources, namely barley straw, hemp shiv and corn cob, a comparative study of the use and durability properties of the composite materials is made.

A survey of the production and use of bio-resources in France showed good availability of the resources studied in the present research, although they are primarily used as human food and animal litter. In the experimental tests, a decrease of the mechanical strength was observed with the addition of plant aggregates. However, the ductility of the bio-composites increased. Thanks to its elongated shape, straw is the plant aggregate that gives the best results. Concerning hygrothermal properties, thermal conductivity is reduced and the vapor sorption capacity is slightly increased. Nevertheless, earth alone is very permeable. The addition of plant aggregates thus brings no benefit concerning the apparent water vapor permeability. Finally, plant aggregates improve some durability criteria, such as impact or erosion resistance, but limit abrasion resistance. With regard to fire, bio-composites are still not combustible, even if they contain a significant quantity of lignocellulosic matter. They are, however, transformed with firing, when the earth is fired and the plant material smolder. Lastly, the study on microbial growth contributed to the development of an experimental methodology. The incorporation of straw seems to facilitate mold growth in comparison with earth alone. However, proliferation appears only in the worst conditions: for material subjected to a temperature of 30°C and a relative humidity of 93%, after inoculation with *Aspergillus brasiliensis* strain.

To summarize, the addition of different plant aggregates in an earth matrix improves some properties but deteriorates others. The formulation of a composite material (particularly the nature and the content of the plant aggregate) will thus depend on its intended use in the building. Therefore, a compromise has to be found among the different properties.

Keywords

Compressed earth bricks, agro-resources, mechanical strength, hygrothermal properties, durability, mold

Résumé

Formulation et caractérisation de briques de terre crue avec granulats végétaux

La construction est l'un des secteurs de l'industrie les plus polluants. C'est la raison pour laquelle développer l'usage de matériaux de construction durables est un intérêt majeur. La terre crue est de plus en plus étudiée en tant que matériau de construction pour son faible impact environnemental, son abondance ou ses capacités à réguler l'humidité intérieure, améliorant ainsi le confort de l'occupant. Pour optimiser certaines de ses performances, des fibres ou granulats végétaux sont incorporés à la terre depuis des millénaires. Toutefois, les études scientifiques n'ont débuté qu'il y a une trentaine d'années, laissant une marge importante de compréhension du matériau. Actuellement, l'ajout de matière végétale peut s'effectuer par le biais de la valorisation d'agroressources, qui permet par ailleurs de piéger du dioxyde de carbone au sein des briques.

Cette thèse, qui s'inscrit dans le cadre du projet Bioterra financé par l'Agence Nationale de la Recherche (ANR), a pour objectif de contribuer au développement d'un matériau à base de terre crue et de granulats végétaux, pour une utilisation sous forme de briques. Après une caractérisation approfondie de différentes ressources végétales (paille d'orge, chènevotte et rafle de maïs), une approche comparative des propriétés d'usage et de la durabilité des matériaux composites est réalisée.

Une étude sur la disponibilité des bio-ressources en France a montré que les coproduits de l'agriculture utilisés dans ce travail de recherche sont disponibles en quantités importantes, bien que leur utilisation pour l'alimentation humaine ou animale soit prioritaire. Les résultats des essais expérimentaux ont montré que les résistances mécaniques sont diminuées avec l'ajout de végétaux, mais que la ductilité est améliorée. La paille, grâce à sa forme allongée, donne toutefois de meilleurs résultats que les autres agroressources. En ce qui concerne les propriétés hygrothermiques, la conductivité thermique est améliorée et la capacité de sorption de vapeur est légèrement augmentée. Toutefois, la terre seule étant très perméable à l'eau, l'ajout de particules végétales n'a pas d'effet bénéfique sur la perméabilité apparente des composites à la vapeur d'eau. Finalement, les granulats végétaux améliorent certains critères de durabilité comme la résistance à l'impact ou l'érosion à l'eau, mais limitent la résistance à l'abrasion. Vis-à-vis de la résistance au feu, les bio-composites, bien que contenant une quantité importante de matière ligno-cellulosique, sont toujours incombustibles. Ils sont toutefois transformés avec la cuisson de la terre et la consumation des végétaux. Enfin, l'étude de la prolifération de micro-organismes a abouti à la mise en place d'une méthodologie expérimentale. L'incorporation de paille semble faciliter l'apparition de moisissures par rapport à la terre seule. Néanmoins, la prolifération apparaît uniquement dans des conditions optimales de 30°C et 93% d'humidité relative après inoculation de souche d'*Aspergillus brasiliensis*.

L'addition de différentes ressources végétales dans une matrice de terre améliorera donc certaines propriétés d'usage mais en dégradera d'autres. La formulation du matériau composite (nature et dosage en granulats végétaux notamment) sera donc conditionnée par sa destination dans le bâtiment. Un compromis devra être trouvé entre les différentes propriétés.

Mots-clés

Brique de terre crue, agro-ressources, résistance mécanique, propriétés hygrothermiques, durabilité, moisissures

Table of contents

Table of contents

List of figures

List of tables

List of tables

General introduction

1. Context of the study

Given the context of global warming, the climate action network of the European Commission has set targets for reducing greenhouse gas (GHG) emissions by 2020 and 2050. One of the key objectives is for the member countries to reduce GHG emissions by 20% in 2020 and 80% in 2050, with respect to their 1990 levels.

The building sector is responsible for a significant share of the global environmental footprint. For example, in France in 2013, buildings were responsible for 45% of the energy consumption, mainly with heating and air-conditioning, and around 20% of GHG emissions (Service de l'observation et des statistiques, 2014). There are thus major environmental challenges, as well as health and comfort challenges, to be taken up by the construction sector and, more specifically, the makers and users of building materials. A recent collective enlightenment is leading to search for alternative materials. The environmental impact of these eco-materials has to be considered from the production of the raw materials to the end of life. Moreover, with the exhaustion of natural resources, recyclability or resource valorization is of major interest and is being increasingly studied.

One of the oldest materials, raw earth, would meet some of these expectations. Earth has been used worldwide as a building material for thousands of years. Many examples could be quoted: some parts of the Great Wall of China, erected over 4000 years ago, or the city of Shibam in Yemen, built in the XVIth century. Around 30% of the world's population still lives in earth dwellings (Minke, 2006). Building with earth was also common throughout France, before being abandoned after World War II in favor of concrete or steel. Nevertheless, a renewed interest in earth constructions has been observed in recent decades.

Unfired earth presents many advantages that allow to meet the current targets mentioned above. Although it is not an innovative material, earth is, by its nature, a very sustainable construction material thanks to its:

- Contribution to reducing energy consumption thanks to its passive regulation: moisture buffering and thermal inertia;
- Low carbon emission: by using material available locally (reduction of transport) and not firing the earth, a reduction of 86% of the embodied energy can be reached in comparison to fired clay (Morton, 2006);
- Abundance and recyclability (when not stabilized);
- Natural and health aspects.

Still, earth faces many obstacles, such as its poor image, low durability to water and the difficulty of making it meet modern productivity or quality standards, with some tests designed for conventional industrial materials.

Furthermore, plant by-products have also always been used in construction. They could be valorized in building materials, to improve the thermal insulation of an earth brick for instance. Their main advantages are low environmental impact and renewability.

This thesis, which aims to contribute to the development of an earth-based material containing plant aggregates, is part of the Bioterra project. Bioterra is a collaborative project, funded by the French National Research Agency, and carried out by five laboratories, three industrial partners and one technical center. These partners and their roles are:

- LMDC (Toulouse), coordinator of the project, is the host laboratory of this thesis. Its aim is to develop and characterize earth-based bricks with plant aggregates.
- CEREMA (Lyon) focuses on bio-based earth plaster properties under thermo-hygro-mechanical stress.
- LGCB (Lyon) investigates the use properties of earth plasters in terms of mechanical and hygrothermal performance.
- LGC (Toulouse) deals with microbial sampling in situ and on prepared earth specimens, and quantifying the presence of microorganisms by usual culture techniques.
- LRSV (Toulouse) aims to use genome sequencing techniques to quantify the flora found in building materials (specimens and in situ).
- CTMNC (Limoges) has the goal of assessing the extrudability of earth containing various amounts of plant aggregates.
- Agencement Structure (Bordeaux) aims to build an earth wall prototype inside their building in order to observe its hygrothermal buffering capacity by monitoring the wall and the room.
- Agronutrition SAS (Toulouse) aims to participate in the culture of a positive microflora to fight against microbial proliferation, in collaboration with LGC and LRSV.
- Carrières du Boulonnais (Ferques) is the supplier of the earth used in the project, which is an industrial by-product. Its aim is hence to participate in the valorization of this product and to propose diverse plaster formulations for study.

Thus, the main objectives of this research project are to identify, characterize and provide solutions to microbial growth on earthen bio-based bricks or plasters, used in the construction and renovation of healthy, sustainable buildings.

This experimental thesis deals with earth bricks containing plant by-products, compressed or extruded, as a load-bearing material. As they can be used for external or inside walls, the tests to characterize the material will depend on the application. Different plant aggregates are studied in this work as additions in an earth matrix. Their influence on the material properties will thus be investigated. Experimental procedures will also be developed and adapted to this kind of material.

2. Research objectives

The main objective of this work is to help to improve the understanding of the influence of plant aggregate incorporation in an earth matrix, with regard to the mechanical, hygrothermal and durability properties. The main points are summarized below:

- Formulation of earth bricks with plant aggregates that are more or less common in this type of material (barley straw, hemp shiv, corn cob, cork granules, rice husk);
- Manufacturing the composite materials according to two methods: compression, which is a current technique notably used for Compressed Earth Blocks, or extrusion, less common for unfired clay, but a production method that could be readily industrialized as it is already used for fired bricks;
- Assessment of the availability of by-products from agriculture to be valorized in building materials;
- Characterization of plant aggregates by adapting and developing testing procedures for these lignocellulosic resources;
- Investigation of the type of plant aggregate with respect to every property;
- Study of the influence of the plant aggregate content (0, 3 and 6% by weight) on every property;
- Comparison and discussion of two procedures for testing the compressive strength of ductile materials: the first, commonly used, technique involves direct contact of the specimen with the press, while the second implements a Teflon capping on the specimen to reduce friction at the interface with the press;
- Investigation of the effect of a treatment (surfactant adjuvant) on the plant aggregates to improve their adhesion to the matrix;
- Comparison and discussion of two techniques for assessing sorption-desorption isotherms: the Saturated Salt Solution (SSS) method and the Dynamic Vapor Sorption (DVS) method;
- Discussion on the relevancy of various durability tests to this type of material;
- Investigation of the influence of a plant matter addition regarding the behavior of the material with respect to fire;
- Development of a testing procedure to evaluate microbial proliferation on bio-based earth materials;
- Study of the sensitivity of different materials to fungal growth.

3. Thesis layout

The present document is a collection of seven papers, already published (4) or submitted (3), which are incorporated in the various chapters. In order to meet the proposed objectives, this thesis is structured in four chapters, each one composed of a preamble, one or more articles and some concluding remarks.

The first chapter presents the state-of-the-art of earth materials containing bioresources with regard to the main engineering properties. First, the origin and characteristics of the natural fibers or aggregates are described and various treatments are reviewed. Then, the different earth construction techniques and compositions are presented. This chapter next focuses on how the bioresources influence not only the physical properties of the composites but also their mechanical, hygrothermal and durability properties.

The second chapter is dedicated to the description and characterization of the three main plant aggregates studied (barley straw, hemp shiv and corn cob). A study is carried out to evaluate the availability, in France, of agricultural and forestry by-products. Then, the physical properties of the plant aggregates are determined, and their chemical composition is characterized, as are their hygroscopic properties, through their water vapor sorption-desorption isotherms.

The third chapter aims to evaluate the use properties of the earth-based materials with the three main plant aggregates. It is composed of two articles. The first one deals with the mechanical properties, particularly compressive and flexural strength. The influence of various parameters is investigated, such as the straw length, the plant aggregate content, the compressive strength test procedure and the effects of treatment of the plant aggregates on their adhesion with the earth matrix. The second article works through the hygrothermal properties of the material. In this part, the same materials are investigated through their thermal properties and also some hygric properties, such as water vapor permeability, sorption-desorption isotherms or the theoretical Moisture Buffer Value.

Finally, the fourth chapter is composed of three papers and focuses on the durability properties. Three main aspects are studied: the resistance to weather or occupancy, with tests of erosion, abrasion or impact; the fire behavior and, finally, the resistance to fungal growth. Two other plant aggregates are used in the first part, cork granules and rice husks, but not all the formulations previously investigated are studied in each part. Corn cob granules and formulations containing 6% of plant matter are no longer considered.

Chapter 1

Literature review: bio-based earth materials

Preamble

Earth has already been investigated as a building material in some theses. Each of them proposed a specific literature review depending on the aim of the research. For example, Bruno's recent review (Bruno, 2016) focused on the physical properties of raw earth materials, their mechanical behavior, moisture buffering capacity and durability properties; Maskell's thesis (Maskell, 2013) aimed to develop stabilized extruded earth masonry units; and Flament's thesis (Flament, 2013) dealt with the physical characteristics of clay and earth stabilization with hydraulic binder or natural fibers. Bui's thesis (Bui, 2008) can also be quoted among others. Its review focused on the various earth construction techniques, specifically on rammed earth. One of the first theses about earth as a construction material, (Olivier, 1994) focused on manufacturing techniques and geotechnical tests. To avoid redundancy with this previous research, the literature review that follows concerns earth material with added bio-aggregates or fibers.

This chapter is mainly composed of an article published in Construction and Building Materials, issue of May 2016. This article is based on 50 main references to bio-based earth materials covering most techniques. The first section of the paper specifically reviews the bioresources used in the earth matrix. A general presentation is proposed, together with their recurrence, their physical, chemical and mechanical properties, and their durability. Some treatments are also investigated that are intended to improve some properties of the composite materials. They aim to enhance the mechanical properties of natural fibers or their adhesion to the earth matrix, or to decrease their water absorption. The second section of the paper presents the composition of the various composite materials found in the literature, with their manufacturing processes. The different earth construction techniques, such as bricks or plasters, are exposed. The influence of the plant aggregates or fibers on engineering properties is then studied. Among them, the effect on physical properties such as density, shrinkage, water absorption and sound insulation is firstly presented. The mechanical properties of the biomaterials are then investigated through compressive, tensile and flexural strengths and adhesion between earth plaster and the wall. Hygrothermal properties, particularly water vapor permeability, sorption-desorption capacity and thermal conductivity are also studied. Finally, durability properties are reviewed with the resistance to water, erosion, freezing-thawing and biodegradation.

Article A

(Article published in Construction and Building Materials the 15 May 2016)

Plant aggregates and fibers in earth construction materials: a review

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Abstract

Earth as a building material is increasingly being studied for its low environmental impact and its availability. Plant aggregates and fibers have been incorporated into the earth matrix in the aim of enhancing performance for thousands of years but scientific studies began quite recently. The present paper reviews the state of the art of research on the influence of these various natural and renewable resources in unfired earth materials such as compressed earth blocks, plasters, and extruded and stabilized blocks. This review, based on 50 major studies, includes characterization of the particles and treatments, and recapitulative tables of the material compositions, and the physical, mechanical, hygrothermal and durability performances of earth-based materials. A lack of references on hygroscopic and durability properties was observed. Future research orientations are thus suggested to promote and develop this type of sustainable material, which provides a solution for saving energy and natural resources.

Highlights

- Earth-based products with plant aggregates as a sustainable material are reviewed.
- Physicochemical and mechanical properties of plant aggregates or fibers are reviewed.
- Mechanical, hygrothermal and durability performances of the composites are reviewed.
- Further investigations are needed to promote these materials.

Keywords

Earthen construction materials, plant aggregates and fibers, characterization, performances, blocks, plasters.

1. Introduction

The impacts of buildings on the environment and on the health of their occupants have now become priority issues. Indoor air quality is considered as a major risk factor for human health and improving the environmental quality of buildings has become a major objective, which could be attained by improving their hygrothermal performance and by using safe construction materials with low impact. For many reasons (heritage, ecological, economic, proximity ...), construction materials that have been used by Human for thousands of years, such as earth and/or bio-based materials (wood, fiber and plant aggregates) are being reconsidered as pertinent materials. Earth is still the most widely used construction material in many countries in the world. Even today, around one third of human beings live in earthen houses and, in developing countries, this figure is more than one half [1]. In advanced economies, earth construction was abandoned in favor of concrete for several decades after the Second World War but earth is again becoming attractive nowadays because of its low environmental impact and because it is known to be a natural humidity regulator and to improve comfort inside buildings. It is usual to strengthen and/or to lighten the earthen construction materials by adding plant aggregates. This review results from the bibliographic survey realized prior to the BIOTERRA research project, which aims to characterize the mechanical and hygrothermal properties of building materials (blocks and plasters) composed of earth and bio-based aggregates. The project started in 2014 and is supported by the French National Research Agency. The consortium is composed of 9 French partners (5 laboratories, a technical center and three industrial partners).

The growing interest in traditional earth building has led to numerous publications during the past ten years, mainly on compressed earth blocks and rammed earth buildings, including two recent reviews. Pachego-Torgal and Jalali have written a review on the environmental benefits of earth for future eco-efficient construction [2] and Danso et al. have studied the enhancement of soil blocks in a quantitative review [3]. However, the paper by Danso et al. focused on the effect of binder (chemical or organic) and aggregate or fiber additions on the properties of earth blocks, and only a small part of it concerns plant or animal aggregates or fibers. Furthermore, the systematic use of chemical binders to stabilize earth blocks is open to debate [4].

The review presented here deals with the plant aggregates and fibers used in earth construction materials in general (blocks, earth plasters, rammed-earth, cob and wattle and daub). It is based on 50 published studies of earth-based composites with plant aggregates and also includes an analysis of 8 more works on some of the natural fibers found in the initial 50 studies. The review is separated into three parts. The first presents the plant aggregates used in earth construction materials (origins, characteristics and treatments used to improve performance). In the second part, the compositions and the manufacture of earth-based composites are presented. In this part, the subsections correspond to the technique used (earth blocks, earth plasters and a last part grouping rammed earth, cob, and wattle and daub). The third and last part of the paper is composed of 4 sections that present the effects of plant aggregates on the properties of earth-based composites. The four types of properties studied are: physical properties (density, shrinkage, water absorption and sound insulation), mechanical properties (compressive strength and elastic modulus, tensile and flexural strength, and adhesion between earth plasters and wall), hygrothermal properties (vapor permeability, sorption-desorption and thermal conductivity) and durability (resistance to water, resistance to wind-driven erosion, resistance to freezing/thawing, biodegradation and microorganism development).

2. Plant aggregates and fibers used in earth construction materials

2.1. Origins and characteristics of plant aggregates and fibers

Studies of many kinds of bio-aggregates according to the location, and hence the availability, of the materials can be found in the literature. The origin of these bio-aggregates can be plant or animal, but plant products are found in the majority of cases. Only two references concern studies of an animal aggregate, which was sheep wool. Thus, the term employed in this review to refer to bio-aggregates is plant aggregates or fibers as discussed further below. Some plant aggregates or fibers have been used in several studies, others are more original. It is nevertheless possible to group them into eight categories (presented by increasing order of use in the references): cereal straws, wood aggregates, bast fibers, palm tree fibers, waste and residues, leaf fibers, aquatic plant fibers and chips, and sheep wool. In that follows, general information about the origin and manufacture of each category is presented. As mentioned in the introduction, eight additional references, which deal with plant aggregates alone, have been included to complete the review. In order to distinguish these references, the rows of the tables concerning them are shaded.

Cereal straws (17/50)

This aggregate was studied in 16 of the 50 references. The straw used was from wheat, barley or oats, but this is not always specified. Straw is an agricultural by-product and is the part of cereal's stem rejected during the harvest. Wheat is an annual plant, ranking 3rd for global production after rice and corn. Its straw has a hollow, cylindrical structure. Barley, like oat, can be harvested once or twice a year. However, barley can grow in extreme climates, such as in the tropics or on mountains, whereas oat prefers cool temperate climates.

Wood aggregates (10/50)

These aggregates are always industrial waste from the wood processing industry (joiner's workshop) [5] or by-products of chemical pulping processes [6]. Wood is a ligneous plant used as fiber, chips, sawdust or fine branches. It is able to regulate hygrothermy and provide good thermal insulation [7].

Bast fibers (8/50)

Bast fibers are extracted from the outside of the stem of cultivated or even wild plants. They have high tensile strength [8] and good thermal insulation property [9]. The fibers are located in the phloem so they must be separated from the woody core by a mechanical and/or retting process. The use of fibers from hemp, flax, jute, kenaf and diss, a Mediterranean wild plant, is reported in the literature. Hemp hurds constitute the by-product of the hemp defibration process and correspond to the lignin-rich part of the stem. They have been used in two cases of study.

Palm tree fibers (7/50)

Palm trees grow in tropical and subtropical regions or in a warm temperate climate. In this review, three palm fibers are studied. Fibers from coconut husks, called coir, are particularly present in the Philippines, Indonesia, India and Brazil. Degradation of coir is quite slow due to its high lignin content [10]. Fibers from oil palm fruit bunches are also studied in two articles and come from Malaysia [11] or Ghana [12]. Lastly, only one reference deals with the use of date palm fibers (from southern Algeria) [13].

Waste and residues (7/50)

Some agribusiness wastes or by-products, e.g. from millet, cotton, tea, tobacco, cassava peels or grass, are considered to be no longer useful. They are stocked and abandoned, sometimes causing environmental problems or health hazards (for example, cassava peel degradation can develop hydrocyanic acid, which is very toxic for humans [14]). Using these low-cost, light materials in construction material is an interesting valorization.

Leaf fibers (5/50)

The fibers studied were extracted from the leaves of three different plants: sisal, banana and pineapple. All of them are tropical plants, which can be produced in Brazil, Indonesia or Eastern Africa [15] but also in some European Islands as Madeira and Azores.

Aquatic plant fibers and chips (4/50)

Some of the fibers studied came from aquatic plants, like the well-known seaweeds [16]. Typha (cattail) and phragmites (reed) are common plants used for wastewater treatment. Wastewater passes through wetlands in order to be treated before being released into the natural environment. This kind of aquatic plant can remove some pollutants, such as nitrogen or phosphorus, from the wastewater. They also provide a substrate for the growth of microorganisms able to degrade organic matter. In addition to this ecological function, they are harvested in a semi-natural wetland in Estonia to be used as a raw material for construction (insulation blocks or fiber-wool) [17].

Sheep wool (2/50)

Unlike cellulose-based fibers, sheep wool is an animal fiber and contains keratin. Protein fibers have poor resistance to alkalis, present in all concretes, which explains the small number of studies. In Aymerich et al. [18], wool was from black and white Sardinian sheep. The use of this wool is very limited in the textile industry, thus it is already used in thermal and acoustic insulation of buildings. Unprocessed wool from Scotland was studied in Galán-Marín et al. [19].

The plant aggregates and fibers were all incorporated into the earth matrix and most of the properties of the composites will be presented in the following parts of the review. In order to better understand these results, the detailed characteristics of the different plant aggregates and fibers studied in the literature are given below.

Physical, chemical and mechanical characteristics are presented in Table 1-1 and Table 1-2. In Table 1-1, dedicated to the physical characteristics, all the 50 references are considered, yielding 27 kinds of plant aggregates and fibers grouped in 8 families. The physical characteristics presented are: the length, the diameter, the apparent density, the absolute density, the water absorption and the thermal conductivity. Some references do not give any physical characteristics, as can be seen in this table. This was also the case for chemical and mechanical characteristics (composition, elastic modulus, and tensile strength) and Table 1-2 quotes only the references presenting these characteristics. It can also be noted that, of the 50 references used in this review, only 4 present both the chemical composition and mechanical characteristics (flax fibers [8], kenaf fibers [20], coir [21] and sisal fibers [22]). Moreover, little mention is made of the chemical and mechanical characteristics of plant aggregates and fibers in the references concerning earth material. In Table 1-2, partial information is provided by only 6 of the 50 references.

Table 1-1. Physical properties of plant materials used in earth construction materials

Type		Length (cm)	Diameter (mm)	Apparent density (kg.m^{-3})	Absolute density (kg.m^{-3})	Absorption (%)	Thermal conductivity (mW.m^{-1}.K^{-1})	Reference
Cereal straw	Wheat straw	1.5 – 2.5	0.5					[23]
		5		103.6			41.4 – 48.6	[24,25]
		5	3					[26]
		0 - 3		33	868	280 - 350		[27]
								[28]
	Barley straw	5		106.9				[24,25]
		1 - 6	1 - 4		2050	500 - 600		[29]
		0.5 - 2		47	870	400		[27]

	Oat straw	1 - 2						[30,31]
	Straw		2 - 5					[32]
		20 - 30						[33]
		5	3					[34]
		5						[35]
								[36-40]
Wood aggregates	Wood (shavings or fibers)	2						[5]
		2		111.4			35.3 – 53.9	[24,25]
		0.3 – 0.8						[41]
				50	440	240		[42]
			0.025-0.05					[6]
								[6,7,39,43,44]
Bast fibers	Hemp fiber	0.85 – 1.7						[45]
			0.035					[8]
					1500	80 - 105		[21]
	Hemp hurds	0.5 – 3.5	1 - 8			280		[46]
		2	2 - 5					[47]
	Flax fiber	7 – 8.5						[48]
			0.013					[8]
	Jute fiber	2 - 4	1		1700			[49]
					1460			[50]
	Kenaf fiber	3	0.13		1040	307		[20,51]
	Diss fiber	2						[52]
Palm tree fibers	Coir		2.38				78	[53]
		3.5 - 5	0.35					[54]
		5	0.27			100		[15]
					1177	145		[21]
								[55]
	Oil palm fiber	1						[11]
								[12]
	Date palm fiber	2 – 3.5	0.1 – 0.8	512 - 1089	1300 - 1350	97 - 203		[13]
Waste and residues	Cassava peel	5	5					[14]
	Millet residue				1164			[56,57]
	Cotton residue	0.01			500			[58]
	Tea residue							[59]
	Tobacco residue		0.025-0.05					[6]
	Grass		0.025-0.05					[6]
Leaf fibers	Sisal fiber	5	0.15			230		[15]
		4 - 5						[60]
		4	0.15					[47]
		7.2	0.3		1370			[22]

		45 - 160	0.004 – 0.012		1370	110		[50]
	Banana fiber	0.85 – 1.7						[45]
	Pineapple fiber	1						[11]
Aquatic plant	Phragmite	2	2					[17]
	Typha	2	2					[17]
								[31]
	Seaweed fiber	1						[16]
Wool	Sheep wool	1, 2, 3	0.035					[18]
								[19]

Table 1-1 shows that a great diversity of plant components can be used in earth blocks. It can be seen that their length varies greatly, from 0 to 30 cm, with a large majority of cases between 1 and 5 cm. This length has to be compared with the size of the sample. The longest particles, straw between 20 and 30 cm, were used in a cob wall, the size of which was 80 x 70 x 110 cm^3. This wall was, however, sawed into segments of 42 x 42 x 11.5 cm^3, which were used for the different experiments [33]. Particle lengths were sometimes chosen in relation with the specimen size to keep mixing uniform. For instance, fibers from pineapple leaves and oil palm fruit bunches in Chee-Ming [11] were 1/10 of the length of the block specimen. Sisal fibers in Sen and Reddy [50], having lengths between 45 and 160 cm are not taken into account because the article concerns the fiber alone, not incorporated into an earth matrix.

A first glance at the table shows that the variation in diameter is also considerable, from 0.004 to 8 mm. However, the diameter of fibers cannot be compared to that of shavings. It is also important to note that fibers form a bundle at first, but they are generally used separated from one another. Some of the reported values may be for fibers in bundle form, such as the jute fibers 1 mm in diameter in Güllü and Khudir [49]. Confusion between the two forms of fibers is often due to the difficulty of isolating an elementary fiber.

In order to distinguish the different types/families of plant particles (fibers, aggregates or straw), two geometrical dimensions are represented in Figure 1-1: the inverse of the aspect ratio (1/AR), which is the ratio of the length to the diameter, versus the diameter. It is noticeable that fibers present the smallest 1/AR and diameter and the aggregates have the highest 1/AR (higher than 0.10). Straw particle ARs are between the other two, but their diameter can be similar to those of fibers or aggregates.

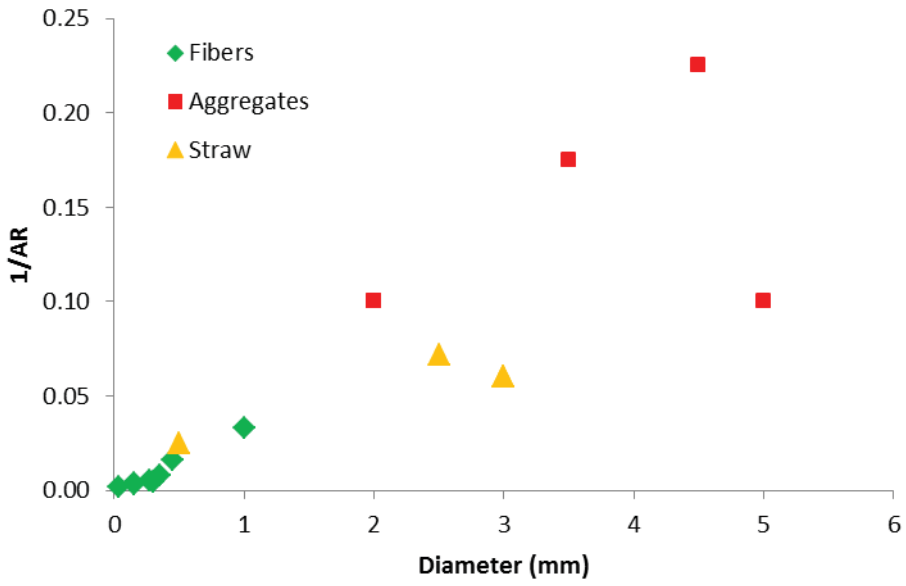

Figure 1-1. Aspect ratio of the particle versus its diameter

A huge diversity of plant aggregate and fiber shapes can therefore be added to an earth matrix, with distinct objectives. For instance, to reduce shrinkage cracking (especially in plasters), the use of plant particles in fiber form is preferable. In this case, the fiber length will play an important role in the non-propagation of cracks. However, when the aim is to reduce the weight of the composite material, and so increase thermal or acoustic insulation for example, the shape of the plant aggregate has a lower impact. The diameter of aggregates is larger than the one of fibers (Figure 1-1).

The apparent density can vary by a factor of three for the same plant particle. However, details of the procedures used to measure this property are not always provided in the literature, which could explain such differences. The definition of the density is quite confused: is it the bulk density of the particles or the apparent density of the particle itself? In the first case, knowledge of this characteristic does not seem very useful to predict the properties of the mixtures of earth and plant particles as the bulk arrangement of plant aggregates or fibers will be modified when they are introduced into the mixture. The only interest of such a characteristic could be linked to the transport of these plant aggregates or fibers but, in this case too, it is possible to compact them, thus changing this property again. It is also possible to observe significant differences in the absolute densities of a given plant particle (see the case of barley straw for example) but it is difficult to generalize this observation because this characteristic is rarely given for the plant aggregates and fibers studied.

For the majority of plant aggregates and fibers, the water absorption is significant (higher than 100%). This property is very interesting because it would provide composites with a high ability to regulate the humidity of the indoor air in the buildings. In the same way, the thermal conductivity of the plant aggregates and fibers is very low, ranging between 35 and 80 $mW.m^{-1}.K^{-1}$, and these aggregates could also be considered as insulating materials. Thus they could improve the thermal properties of earthen materials at the same time as their hygroscopic properties.

Table 1-2. Chemical and mechanical properties of plant particles used in earth construction materials

Type	Composition (%)			Elastic modulus (GPa)	Tensile strength (MPa)	Reference
	Cellulose	Lignin	Hemicellulose			
Hemp fiber	64	4	16			[9]
				21	1077	[8]
				34	900	[21]
Wood aggregates	50	16-33	7-29			[6]
Flax fiber (Harakeke)	61	8	27	21	805	[8]
	81	3	14			[61]
	60	3	16			[9]
Jute fiber	72	13	13			[61]
				10-30	400-800	[50]
Kenaf fiber	70	19	3	136	1000	[20]
Diss fiber					100	[52]
Coir	43	46	0.25			[53]
				3	150	[15]
	41	27	22		144	[61]
	21	47	12	10-40	73-505	[21]
Oil palm fiber	49	23	21			[61]
Date palm fiber				5	233	[13]
Sisal fiber				18	580	[15]
	65.8	9.9	12	15.5	472	[22]
				15	363	[50]
	73	11	13	15	347-378	[21]
Banana fiber	26	25	17			[21]

For the characteristics presented in Table 1-2, although some differences exist, the values obtained for a given plant aggregate or fiber are more homogeneous than those of Table 1-1. It is noteworthy that the only mechanical property previously studied in the literature is the tensile strength of some fibers, because fibers are used as flexural reinforcement. However, for some other applications, including plant aggregates such as wood shavings or hemp hurds, it could be relevant to assess the compressive behavior of plant particles. Hemp fibers have the highest elastic modulus and tensile strength. The other fibers could be ranked by decreasing tensile strength: flax, jute, sisal, date palm and coir. It is interesting to note that coir has the highest lignin content and the lowest tensile strength. Moreover, it seems that there is a link between the cellulose content and the mechanical characteristics of fibers, as suggested by Millogo et al. [20].

2.2. Aggregate and fiber treatments used for earth composites

Treatments could be useful to improve some characteristics and properties of natural aggregates and fibers for their use in earth materials. Alkaline treatment, acetylation, hydrothermal treatment and water-repellent coating were studied in ten references. In one case, aggregates were treated for health reasons: cassava peels were dried in the sun for three days to eliminate hydrocyanic acid (HCN), which is very toxic for humans [14]. The other impacts of the treatments are described below and summarized in Table 1-3.

2.2.1. Water absorption and dimensional variations of the aggregates and fibers

As shown in Table 1-1, natural aggregates and fibers can absorb significant amounts of water, often more than 100%. Their dimensions can also vary according to their moisture content, and vary greatly during drying of the sample. Plant aggregates and fibers are hydrophilic materials that absorb manufacturing water. This absorption by the particles can be avoided for good dimensional stability. The bigger the dimensional variation is, the lower is the binding with the matrix. Limiting water absorption is thus often desirable.

A water repellent can be applied to the aggregates or fibers. It may be enamel paint (spray or immersion), a mix of rosin and alcohol, bitumen or acrylic coating [48]. Coir and sisal fibers were treated with two kinds of bitumen coating called “cipla” and “piche” [15]. The “piche” repellent was more effective than the “cipla”. With this treatment, water absorption of sisal was about 30% lower than that of coir, whereas non-treated sisal was more absorbent (230% for sisal, only 100% for coir). It seems that sisal fibers were more porous than coir, so they could absorb the water-repellent more easily.

Other treatments, such as acetylation, were tested to change the morphology of the aggregates or fibers and thus to decrease their water absorption [22]. Acetylation is a chemical reaction that replaces OH-groups by others that are more voluminous and have less affinity with water.

Alkaline treatment was less effective than acetylation [22] because the fiber structure was more dense after it. The treatment engendered a decrease in the fiber diameter.

Three other effective treatments were tested on wood shavings in Ledhem et al. [42]: immersion in boiling water, in linseed oil and in boiling water with lime. During the immersion, the water-soluble substances were extracted. These substances can cause wood to swell. With the two treatments in boiling water, it appeared that the reduction of dimensional variation was greater than the water absorption. The best result was obtained with linseed oil, after which the water absorption by wood shavings was 42%, instead of

112% with boiling water treatment, 89% in boiling water with lime and 240% without treatment.

2.2.2. Mechanical properties of aggregates or fibers and composites

The effects of treatments on the mechanical properties of the fibers could be positive or negative. With a coating of enamel paint, fibers were more fragile than without treatment [48].

It was shown that acetylation could have both positive and negative effects. A decrease in tensile strength and elastic modulus was observed by Alvarez et al. [22] whereas Hill et al. [61] found different results. The chemical reaction improved tensile strength when it took place at 100°C but decreased it when performed at 120°C because, at this temperature, the fibers were damaged.

Three different alkaline treatments were tested in Efendy et al. [8] and all of them led to a decrease of up to a 30% in the tensile strength of harakeke and hemp fibers. This is explained by the decrease of the fiber diameters due to the treatment. Young's modulus of hemp fibers was increased with the three treatments but was not really modified in harakeke. The improvement for hemp may have been due to an increase in cellulose rigidity induced by the removal of non-cellulosic components. Flexural modulus was improved by 20% with a 48-h alkaline treatment at 25°C [22].

Boiling water treatment slightly improved the mechanical performances of the composite material according to Ledhem et al. [42] but decreased the compressive strength and slightly improved the flexural strength in Fertikh et al. [52]. Linseed oil treatments decreased mechanical properties, especially tensile strength [42], by around 11% for the mix with paraffin [54].

2.2.3. Aggregate and fiber adhesion to the matrix

Adhesion of the plant aggregate or fiber to the matrix can be evaluated by a tensile or flexural test: the adhesion is good if no aggregate or fiber debonding is observed, that is to say if the particle is broken at the end of the test [22]. Adhesion can also be estimated with SEM imaging: the rougher the texture of the fiber, the better its contact with the matrix [8].

Adhesion to the matrix was improved when the fibers were treated by acetylation [22] and treated fibers broke during the mechanical tests whereas the untreated ones slipped relative to the matrix. However, it was shown by Alvarez et al. [22] that a 1h of acetylation reaction improved the adhesion between fiber and matrix, whereas adhesion was decreased if the reaction lasted 24h or 48h.

Alkaline treatment was one of the most common among the references. Fibers were treated to improve adhesion to the matrix, which was one condition for obtaining good mechanical strength of the composite. Alkaline treatment separated fiber bundles in harakeke and hemp. Adhesion between fibers was due to pectin, hemicellulose and lignin, which were removed by the chemical reaction. After the treatment, the fibers were rougher and grooved, which explains the better adhesion to the matrix [8,22]. Another reason for alkaline treatment was to give a homogenous mix between the matrix and cotton waste. The cotton was soaked in a sodium hydrate solution (NaOH) to remove waxes and oil [58].

However, some treatments, such as immersion in boiling water or linseed oil, induced a decrease in adhesion. With immersion in boiling water, some spine fibers were removed, which led to lower fiber-matrix adhesion [52]. With the linseed oil treatment, the adhesion between fibers and a cement-clay matrix decreased as the treatment intensity increased [42].

2.2.4. Durability of the plant aggregates or fibers

Plant aggregates or fibers are organic materials, which are thus subject to natural decomposition that occurs through the centuries [35]. However, the definition of durability varies for different authors. For example, it was shown by Babu and Vasudevan [62] that coir fibers only lasted for 2 to 3 years without any treatment. Some authors tried to improve their durability by various processes.

Acetylation was applied to coir and oil palm fiber in Hill et al. [61]. It was observed that resistance to decay and humidity was improved with this treatment. Both the treated and untreated fibers were put in soil and a tensile test was performed every 4 weeks for 20 weeks. On untreated fibers, it was impossible to measure the tensile strength after 8 weeks because the fibers were too damaged. The tensile strength of treated fibers decreased with exposure, but was still about 80% of the initial value after 20 weeks.

Treatment was also applied to avoid mold development inside the matrix and thus avoid accelerated damage. Mold was observed inside the barley straw, certainly due to storage [27]. To eliminate the mold, fibers were immerged in a saline solution of NaCl and then in an aqueous solution of NaOH.

The immersion of coir in a mix of boiled linseed oil, paraffin and a solvent to coat the fibers was tested in an attempt to improve their durability [54]. According to the author, this kind of water repellent increased the lifetime by 8 to 12 years.

Table 1-3. Impacts of various treatments on the properties of plant aggregates and fibers (+ when the property is improved, - if not)

Treatment	Coating				Acety-lation	Alkaline	Thermal immersion		
	Enamel	Acrylic	Bitumen	Rosin-alcohol			Boiling water	Boiling water with lime	Linseed oil
Reference	[48]	[48]	[15,48]	[48]	[22,61]	[8,22,58]	[42,52]	[42]	[42,54]
Water absorption	+	+	+	+	+	negligible effect	+	+	+
Mechanical properties	-				+ 100°C - 120°C [61] - [22]	+ flexion[22] - tensile[8]	+ [42] - compression + flexion [52]		-
Adhesion					+	+	-		-
Durability					+	+			+

3. Compositions and manufacture of earth-based composites

The compositions of earth-based composites are summarized in the following tables (Table 1-4 to Table 1-8). They are grouped by construction techniques: blocks (adobes, compressed earth blocks, extruded blocks and stabilized blocks), plasters and monolithic walls (rammed earth, cob, wattle and daub). Properties of the soil used to make these materials are presented when available (Atterberg limits (liquid limit wL, plastic limit wP and plasticity index PI), dry density and particle size distribution). The weight percentages of the different types of plant aggregates and fibers are also presented with their respective lengths. Plant aggregate or fiber contents that were expressed by volume in the reference have been converted into weight contents (wt%) when possible (density known) to standardize the units of the data. Some fields were not presented in some articles, so the boxes have been left empty.

3.1. Earth blocks

3.1.1. Unstabilized adobes

Adobes are masonry elements, handmade and molded, and generally dried in the sun. This technique, still used in non-industrialized countries, is very ancient. It was used, for example, to build the city of Shibam in Yemen in the 15th century [1].

Table 1-4 presents the characteristics of the soils and plant materials used in adobes.

Table 1-4. Characteristics of soils and plant aggregates or fibers used in adobes

Ref.	Soils						Plant aggregates or fibers		
	wL (%)	wP (%)	PI (%)	Clay (%)	Silts (%)	Sand (%)	Type	Length (cm)	Content (wt%)
[32]	23	20	3	60% quartz, 18% illite, 22% vermiculite			straw, fine wood branches		0.23
[54]				9	18	73	Coir	3.5 or 5	0.8 - 3.2
[15]	30	24	6				sisal, coir	5	4
[35]				25			straw	5	0 - 1
[28]	47	16	31	49			wheat straw		1
[51]	38	20	18	25	30	45	hibiscus cannabinus	3 - 6	0.2 - 0.8
[34]	26	18	8	22	50	25	straw	5	0 - 1
[44]				10	5	85	sawdust, cow-dung, molasses		0 - 20 (by volume)
[26]				15	25	60	wheat straw	5	0.7 - 3.8

There are 9 references dealing with studies on adobes mixed with plant aggregates or fibers. For these references, the characteristics of the soil used are not always specified but when they are, strong differences appear: this is the case for Atterberg limits, where the Plasticity Index ranges from 3 to 31%. 7 studies give the mineralogical composition of the soil: in most cases, the percentage of clay corresponds to the particle size analysis (passing below 2 µm). Finally, in this type of material, the plant aggregate or fiber contents are quite low, ranging from 0.23 to 4% by mass, but this table shows the ability of adobes to be manufactured with large aggregates: most of them are around 5 cm.

3.1.2. Unstabilized Compressed Earth Blocks (CEB)

This type of masonry element is a descendant of adobe blocks. Blocks are compacted with a manual or mechanical press. Table 1-5 presents the characteristics of soils and plant materials used in CEB.

Table 1-5. Characteristics of soils and plant aggregates or fibers used in CEB

Ref.	Soils						Plant aggregates or fibers		
	w_L (%)	w_P (%)	PI (%)	Clay (%)	Silts (%)	Sand (%)	Type	Length (cm)	Content (wt%)
[18]	28	17	11	7	58	35	wool	1, 2 or 3	2 - 3
[56]				lateritic			millet		0 - 12.2
[57]				lateritic			millet		0 - 12.2
[29]	57	23	33				barley straw	1 - 6	0 - 3.5
	33	15	18						
	32	18	14						
	40	22	18						
[46]	33	21	12	19			hemp	0.5 - 3.5	15 - 22
[12]	35	24	11	11	-	-	palm		0 - 1.5

The number of studies of CEB containing plant aggregates or fibers (6 references) is much lower than for adobes. Contrary to what was observed previously with adobes, the characteristics of the soils studied seem a little less dispersed: the plasticity index varies from 11 to 33% and the clay content (based on the particle size distribution) from 7 to 19%. This may be related to the recommendations established in the French standard on CEB [63], which provides some reference values for the particle size distribution and the plasticity index of soils used for the manufacture of CEB. Finally, the most interesting thing in Table 1-5 is the high percentages of plant aggregates or fibers that could be added in CEB. In some studies, the authors added up to 22% by mass of hemp. Such additions were possible thanks to the CEB manufacturing technique, in which the blocks are pressed and the rheological behavior of the fresh material has little effect, in contrast to the case of extruded blocks, for example.

3.1.3. Unstabilized Extruded Earth Blocks (EEB)

Extruded blocks are manufactured with the earth in a plastic state. Generally produced in an industrial process, these blocks can present perforations and are dried in an oven (105°C [59]). Table 1-6 presents the characteristics of soils and plant materials used in CEB.

Table 1-6. Characteristics of soils and plant aggregates or fibers used in extruded blocks

Ref.	Soil				Plant aggregates or fibers		
	w_L (%)	w_P (%)	PI (%)	Clay (%)	Type	Length (cm)	Content (wt%)
[59]					tea		0 - 5
[6]					sawdust, tobacco, grass		0 - 10
[39]	Claytec brick 7002DF				straw, wood chips		0.2 - 0.8
[7]	48	22	26	20 – 40	wood fiber		

There are few references dealing with the use of plant aggregates or fibers in extruded blocks and those that exist give little data apart from the type and amount of plant aggregates or fibers added. The plant contents are often low, certainly because the process requires the mixture to be sufficiently flowable to pass through a machined die at the outlet of the extruder. Nevertheless, in one study, the content reached 10%. This may have been due to the small size of the aggregates but this characteristic was not given in the article.

3.1.4. Stabilized earth blocks

These blocks may be adobes, CEB or manufactured like concrete, by vibro-compaction or pouring into molds. A stabilizer (mineral binder, polymer, etc.) is added to the earth in order to improve the characteristics of the block, such as mechanical properties or durability (resistance to water). Some blocks were dried in the sun [16], others had water sprinkled on them during the curing process (exposed to sunshine for 2-3 weeks, and to air for 1 week) [55] and others were simply stored for 28 days before testing. Table 1-7 presents the characteristics of the soils and plant materials used in stabilized blocks and their manufacturing techniques.

Table 1-7. Characteristics of soils and plant aggregates or fibers used in stabilized blocks

Ref.	Soil						Stabilizer/treatment		Plant aggregates or fibers			Technique
	w_L (%)	w_P (%)	PI (%)	Clay (%)	Silts (%)	Sand (%)	Type	Content (wt%)	Type	Length (cm)	Content (wt%)	
[16]			3	0.5	51.5	48	beetroot (3/4) and tomato (1/4) polymer	10	seaweed	1	10	Adobe
[5]			12	alluvial aggregate			cement	20	wood aggregate	2	10 - 40	Pouring
[58]				limestone			cement	11	cotton		0 - 6	CEB
[36]				32	25	43	cement lime basaltic pumice gypsum	12.5 2.5 18.7 3.7	straw		2.7	Vibro-compaction
[37]				32	25	43	cement lime basaltic pumice gypsum	10 10 10 10				Vibro-compaction
[38]				32	25	43	cement basaltic pumice gypsum	15 15 15				Vibro-compaction
[41]				kaolinite			cement	25	wood	0.3 -	0 - 37.5	Pouring

									aggregate	0.8		
[11]	46	22	24				cement	0 - 15	pineapple , oil palm fruit bunch	1	0 - 0.75	CEB
[52]							lime		diss	2	6	
[19]	35	19	16	32	45	23	alginate	0 or 25	sheep wool		0 - 0.6	CEB
[49]	37	25	12				lime	0 - 10	jute	2 - 4	0 - 1	CEB
[53]	non-plastic			lateritic		12	cement	11	coir		0 - 2.2	CEB
[42]							cement	0 - 80	wood aggregate		0 - 29	Pouring
[45]	34	28	6	Bayeux quarry fines			cement lime	7 0 or 2	hemp, banana	0.8 - 1.7	0.6	CEB
[43]			15	lateritic			cement	8 or 10	sawdust		0 or 9	CEB
[14]	35	17	18				coal-ash	0 - 10	cassava peel	5	0 - 5	CEB
[55]				11	2	87	cement	7	coconut husk		0 or 1	CEB
[48]				3	20	77	cement	10	harakeke (flax)	7 or 8.5	0.6 or 0.8	Adobe
[13]	36	23	13				cement	0- 8	date palm	2 - 3.5	0 - 0.2	CEB

The references dealing with stabilized blocks containing plant aggregates or fibers are the most numerous (19 references). Several types of binders were used during these studies: cement (with or without mineral additions) was used in 13 out of 19 cases, lime in 5 cases and organic stabilizer in two (alginate, and beetroot and tomato polymer). The size of the plant aggregates or fibers used in stabilized blocks was comparable to that observed in other types of blocks (only flax fibers were a little bit longer (8.5 cm)) but the plant particle content in some blocks could be very significant, especially for wood aggregates: 40% in [5], 37.5% in [41] and 29% in [42]. Such high plant aggregate or fiber contents would certainly lead to significant problems of strength but these seem to have been solved by using high binder content (cement for the references on wood aggregates). However, the amounts of binder used in other references are often very high, which could raise questions on the environmental impact of such materials when cement and/or lime are used. It is important to note that the cement content in concrete blocks is below 7% (150 $kg.m^{-3}$) and these blocks are hollow. This means that the comparison with a solid earth block stabilized with cement is even more disadvantageous for the earth block regarding cement content. To date there are still few studies dealing with the use of natural organic stabilizers but this is certainly the most sustainable solution and should be developed in the future.

3.2. Earth plasters

Earth plasters are usually applied on masonry walls, wattle and daub or straw bale buildings to protect them. They are composed of clay, water and sometimes plant aggregates or fibers to avoid drying shrinkage but that is not absolutely necessary [64]. Here, none of the plasters studied were stabilized by a mineral binder, which could have improved resistance to rain and abrasion. Table 1-8 presents the characteristics of the soils and plant materials used in earth plasters.

Table 1-8. Characteristics of soils and plant aggregates or fibers used in earth plasters

Ref.	Soil						Plant aggregates or fibers		
	w_L (%)	w_P (%)	PI (%)	Clay (%)	Silts (%)	Sand (%)	Type	Length (cm)	Content (wt%)
[24,25]				31	22	47	barley and wheat straw (Sw), wood shavings (W)	5 (Sw), 2 (W)	0 - 16.7
[30]							oat	1 - 2	
[47]				26	32	42	sisal (S), hemp chaff (H)	4 (S), 2 (H)	0.5
[17]							wool and chips of typha, chips of phragmite	2	0 - 2
[31]							Oat straw, typha wool		0 - 80 (by volume)

The Atterberg limits of soils are not presented in the references dealing with the use of plant aggregates or fibers in earth plasters but, in general, these soils have a high level of plasticity that enables the plasters to be spread on the wall. The aggregates and fibers used for this type of material were short because of the small thickness applied. Moreover, in general, the particle content was low in order to obtain homogeneous mixtures that were easy to apply to the wall, except in reference [24], where a very high aggregate or fiber content was used.

3.3. Rammed earth, cob, wattle and daub

Other techniques exist, but are not currently studied, especially with plant aggregates or fibers. Rammed earth is a technique that consists in compacting moist earth within a formwork. Cob is a mixture of earth and straw assembled in layers to build a monolithic wall. Wattle and daub is a traditional construction technique with a wood structure.

These traditional earth construction techniques are very little studied in the literature and the few studies found were not very relevant. One concerned the 20-year durability of rammed earth [65] but the results for walls containing straw were not presented. Rammed earth was also studied in Miccoli et al. [33], where the mechanical behavior was compared with that of cob (which is made with 1-2% of straw 20-30 cm in length) and CEB. In Chabriac et al. [66], rammed earth and cob were studied, but their compositions were unknown. Two studies of wattle and daub were found. In Laurent's 1987 study [40], the straw weight content was between 0.5 and 22%. The other [60] studied "bahareque", but as a plaster. This technique, used in tropical countries, is similar to wattle and daub. It consists in applying a soil-fiber mixture to a wood or bamboo structure. The fiber used in the reference was sisal fiber, 4-5 cm in length, with a 1% weight content. The mixture was stabilized with cement or cactus pulp. Another way to build a monolithic wall with very lightweight material is the straw-clay technique. The material studied by Oudhof et al. [67] was composed of earth mixed with water and straw and then compressed in a mold by hand.

4. Effects of plant aggregates or fibers on the physical properties of earth-based composites

4.1. Density

This property is interesting because it can be correlated with many properties of the composite material, such as thermal or mechanical characteristics. As expected, the bulk densities of all the plant aggregates and fibers used in the references studied were lower than that of the soil. Increasing the particle content led to a decrease in the earth content and thus a decrease in the composite dry density [23,53,59]. For instance, a cotton residue content of 40% by volume engendered a 29% decrease in density [58]. Porosity also increased when millet content increased [56]. However, the density decrease was not significant with the addition of oil palm fruit bunch fiber [11], but this could be explained by the low proportion of fiber, less than 1% by weight.

Figure 1-2 recapitulates the dry bulk densities of the materials studied in the references according to their aggregate or fiber contents by weight. The values are classified according to the manufacturing technique. The global decrease of density with the increase of aggregate or fiber content is well illustrated, although wide dispersion of the values can be noted, especially for the lower particle contents. For example, for an aggregate or fiber content of 10%, the bulk density is between 1810 $kg.m^{-3}$ and 1010 $kg.m^{-3}$. Only one value does not follow the trend: a density of 800 $kg.m^{-3}$ relative to a particle content of 0.5% [39]. However, this value is easily explainable when the holes of these extruded blocks are taken into consideration.

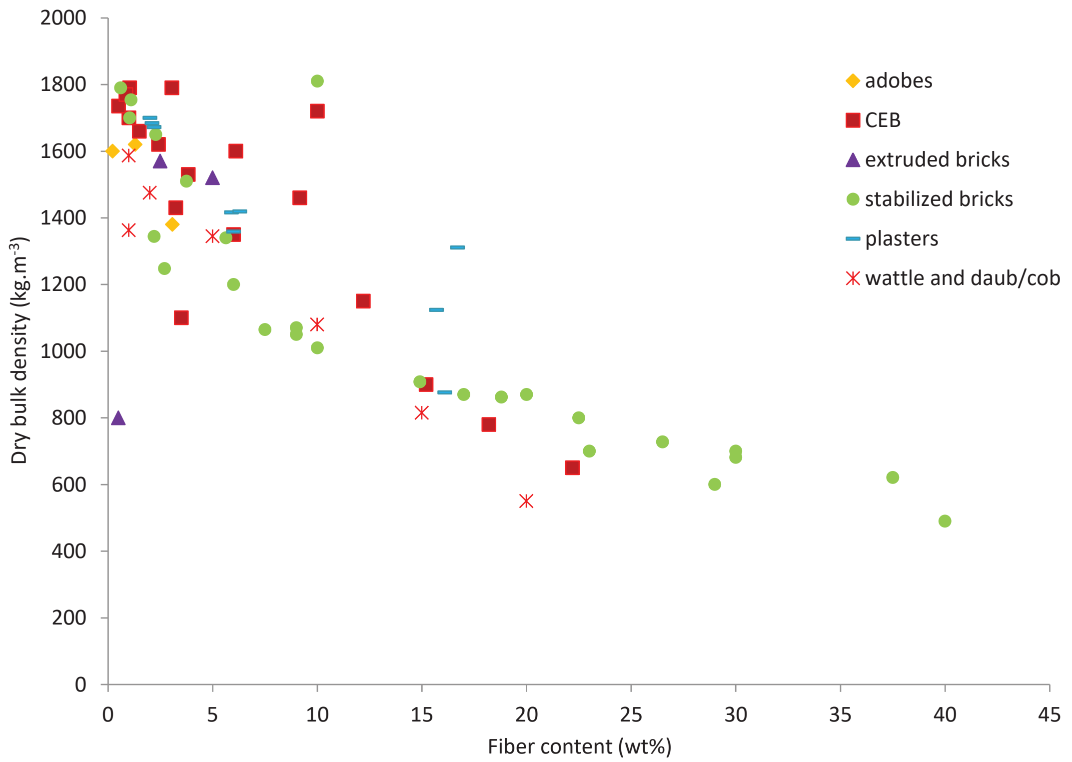

Figure 1-2. Comparison of the dry bulk density of the different manufacturing techniques according to the aggregate or fiber content values given in the literature.

4.2. Shrinkage

Shrinkage is a dimensional variation of the material caused by water evaporation just after manufacture. This volume decrease engenders internal stresses that can lead to shrinkage cracks.

In several studies [15,23,29,47], the addition of plant aggregates or fibers limited cracking as the particles opposed the deformation, thus limiting soil contraction and reinforcing the matrix, in particular for long fibers where adhesion between the fiber and the matrix was better [29]. In Ghavami et al. [15], the optimal length to minimize shrinkage was 5 cm in 4% weight content.

Shrinkage stabilization occurred earlier with the use of straw [29] because of the hollow structure of the stems, which allowed accelerated evaporation.

However, it was observed in Demir et al. [59] that increasing the tea waste content led to greater shrinkage. The authors explained this phenomenon by the increased amount of water needed in the manufacture of the composite, due to the absorbent nature of the aggregate. The use of more than 10% plant residue by weight is not recommended because of the excessive increase of shrinkage cracks engendered [6].

4.3. Water absorption

The influence of aggregates or fibers on composite water absorption was very rarely studied, that of stabilizers being much more investigated. Water absorption by the material can be measured by immersion or capillarity. Concerning plasters, absorption is not an indispensable criterion because they are assumed to be inside the building or to be protected from rain [30]. However, it is interesting to measure how they are affected by capillary rise that may occur from the soil. One clear result is that non-stabilized blocks do not resist immersion [11,16]. It is shown in Algin and Turgut [58] that the quantity of water absorbed was proportional to the cotton waste content. Increasing the cotton content by 40% (by volume) more than doubled the water absorption of the material (from 12.5% to 27.2%) but this value was acceptable in comparison to the other lightweight materials. It was also shown in Taallah et al. [13] that increasing fiber content to 0.2% led to an increase in water absorption and swelling. From these two articles [13,58], it could be generalized that addition of aggregates or fibers increases water absorption because of their high absorption capacity, as shown in Table 1-1. Moreover, in a study using another absorption test [60], it seemed that water absorption was accelerated by aggregates and fibers.

Water absorption by aggregates and fibers has an important effect on their adhesion with the matrix. Swelling of the particles, engendered by water absorption during the first 24 hours, pushes away the soil. When, after drying, the volume of the particles decreases, voids are created around them, as shown in Figure 1-3 [15,48].

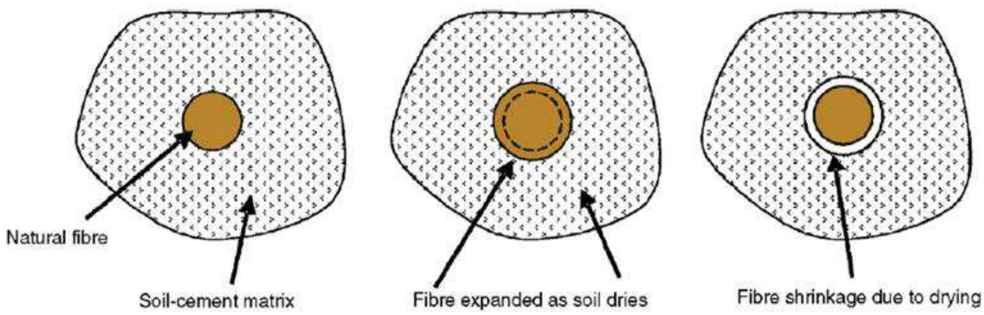

Figure 1-3. Effect of water absorption on aggregate or fiber adhesion with earth, from [48]

4.4. Sound insulation

Lightweight earth block (with straw for instance) is thought to give a good sound absorption coefficient [1]. However this property has been investigated in only one paper [38]. A measurement to characterize sound insulation of a wall in a small industrial building was performed by Binici et al. [38]. The sound absorption coefficients of three kinds of fibers and 3 stabilizers were compared by the impedance tube method using a sound frequency between 100 and 1800 Hz. Sound absorption coefficients increased with frequency (the opposite of what is observed in concrete and fired bricks). These coefficients were higher with straw than with earth alone, but lower with straw than with plastic fibers or polystyrene. Sound insulation was greater with basaltic pumice than with cement and gypsum.

5. Effects of plant aggregates and fibers on the mechanical properties of earth-based composites

These properties are widely studied in all the references, but it is not easy to compare the values measured in a table because each parameter could change: test procedure, sample dimension, type of aggregate or fiber, composition… Therefore, only general trends are described below.

5.1. Compressive strength and elastic modulus

The effect of plant particle additions on compressive strength differed from author to author. In 8 references [6,11,12,19,29,36,51,59], compressive strength was improved by adding aggregates or fibers under different conditions. For example, [29] showed that the strength of the most clayey earth (between 28 and 40% of clay) was improved by 10 to 20% by adding 1.5% of barley straw. For [59], the strength increase was due to the composition of the residues used, which contained other components in addition to cellulose. The addition of pineapple and palm fibers studied in Chee-Ming [11] improved the strength only when the cement weight content was above 15%. Two fibers lengths and different proportions were studied in Millogo et al. [51]. The optimum determined was a fiber content of 0.4%, which increased the strength by around 16% for short fibers (3 cm) and 8% for the longest fibers (6 cm). This increase of compressive strength was correlated with the characteristics of the plant used. The fiber studied here, Hibiscus Cannabinus (or kenaf), showed the highest tensile strength of all the aggregates and fibers investigated (Table 1-2). Millogo et al. explained that the incorporation of fibers of kenaf reduced the propagation of cracks in the blocks, through the good adherence of fibers to the clay matrix (shown on SEM micrographs), and therefore improved their mechanical properties. Compressive strength was even improved by 37% by sheep wool and was doubled if the wool was combined with alginate [19].

Four other references [7,13,35,45] did not report any influence for plant aggregate or fiber additions. However, this may be explicable by the low aggregate or fiber content (below 1% by weight in the 4 cases).

The compressive strength decreased in 11 references [5,15,23,26,29,34,35,41,42,53,58]. It was found by Algin and Turgut [58] that the dry compressive strength was inversely proportional to the cotton content: a decrease of 71% was reached for an addition of 7% of cotton. When plant particles were added, the dry density was lower and the composite material was more porous [5,15]. Some models linking mechanical properties with porosity were adapted for clay-cement-wood materials and compared with experimental results [41]. Compressive strength decreased with increasing porosity, i.e. increasing wood content. Bal'shin's and Hasselman's models [41] were very close to the experimental results. Moreover, compressive strength was lower with aggregates or fibers because of the weak adhesion between particles and clay matrix [35,53]. The aggregates or fibers could slip easily, reducing the homogeneity of the composite material.

However, no generalization could be made; the influence of aggregates and fibers depended on the type of particle, the soil composition and the testing method. For a given study, using the same soil and the same testing procedures, the addition of palm fibers induced an increase of compressive strength while the addition of pineapple fibers resulted in a decrease [11].

In all cases, ductility was greatly improved with aggregates and fibers, increasing proportionally to their content. There were more cracks but they were less deep [29,35]. This occurred because aggregates, e.g. straw, had a bridging effect with the clay matrix and were more compressible, generating high residual strength. During the rupture of the sample, no particle breaking was observed, only a loss of adhesion with the matrix shown by the debonding of the aggregates. The deformation reached at failure varied considerably depending on the particle type and content: 24% with 0.75% of straw [34], 10% with 4% of coir [15], 13% with 5% of cassava peels [14]. In one case [54], samples containing more than 1.8% of coir fibers never reached failure. Because of their proximity, the fibers could be expected to behave as a mesh, which would lead to the recompression of the earth. When the specimens contained more than 1.8% of fibers, a strain limit of 18% was chosen in order to determine a compressive strength. In Flament [46], the deformation was also large (between 20 and 25%), thus the compressive strength was determined at strains of 1.5 and 7.5% (values inspired by [68]).

The procedure used for the measurement of compressive strength could strongly influence the results because of the specific behavior of earth when it was confined in the compressive strength test [69,70]. The confinement was more marked for earth materials, because of their ductility, and the presence of plant aggregates or fibers, which increased this ductility, increased the effect of confinement during the test. Moreover, because of the high ductility of earth materials having a high plant aggregate or fiber content, there is a problem in the definition of the rupture criteria because, as mentioned before, in some cases of study, the samples never reached failure. For the moment (and as far as the authors know), no standard takes this specific problem into account.

Young's modulus decreased when natural aggregate or fiber content increased. For example, the highest modulus was 211 MPa for earth alone but it fell to between 100 and 150 MPa with an addition of up to 1% of straw [35]. This Young's modulus reduction was related to the compressive strength by various authors [11,20,27,54]. An empirical model to determine Young's modulus using compressive strength is described in Piattoni et al. [34]. Three theoretical models (Voigt, Reuss and Hill) were there compared and showed good correlation with the experimental results. Other empirical relationships are proposed by Al Rim et al. [5]. The static modulus was calculated from stress-strain graphs and the dynamic modulus, higher, was measured using ultrasonic waves.

5.2. Tensile and flexural strength

Some direct and indirect (Brazilian test) tensile tests were performed. As for compressive strength, tensile strengths varied markedly depending on the reference.

In 5 cases of study, the addition of natural aggregates or fibers decreased the tensile strength [13,26,42,43,54]. For instance, the addition of 0.2% of oil palm fibers led to a decrease of tensile strength of approximately 20% [13]. This could be attributed to the heterogeneity of the material and the fragility and low tensile resistance of the fiber, as can be seen in Table 1-2. It also could be explained by the decrease of the mineral matrix when it was replaced by sawdust [42] or the weakness of the adhesion between the fibers and the matrix even when there was only 0.6% of fibers [26]. To justify the decrease in tensile strength, [54] assumed that the material was less compacted with the fibers, thus soil particles were less close to each other and cohesion and friction forces were less pronounced.

In 4 other cases of study, tensile strengths were improved by natural aggregate or fiber additions [23,42,45,51]. Tensile strength was improved by 30% with the addition of 1% of wheat straw, which was the particle content leading to the highest dry density [23]. Tensile strength seemed to be influenced by the fiber length for Hibiscus Cannabinus contents of around 0.2% [51]. Ductility was also improved with the addition of fibers, which are able to self-deform a lot, especially when their length increased [51]. Blocks could store elastic energy and were more resistant to crack propagation, an interesting property in case of earthquake [45]. The behavior of the tensile failure was described by Mesbah et al. [71] as a two stage failure: the first one corresponding to the failure of the clay matrix and the second one to that of the aggregate or fiber mesh. Tensile strength was also dependent on the type of particle, its tensile strength and its roughness, which allowed better adhesion to the matrix [27].

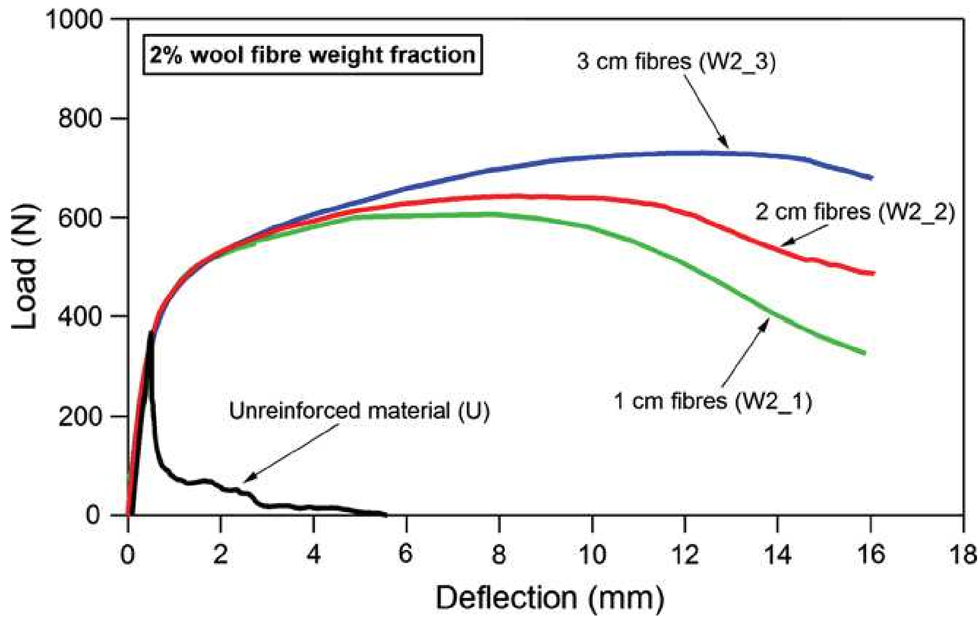

Figure 1-4. Example of load-deflection curves of unreinforced and reinforced specimens tested by flexion [18]

In 6 references out of 8 [5,18,19,29,46,52], flexural strength was improved by the addition of plant (or sheep wool) aggregates. For instance, flexural strength increased by 30% with an addition of 25% of sheep wool [19]. Crack propagation was limited by the presence of fibers, which led to a bridging effect with the clay and increased the friction at the interface between the fibers and the matrix [18,19]. This was observed specifically for soil containing a lot of clay and for the longest fibers [29]. The link with the length could be explained by a higher contact surface with the clay matrix, and thus better anchorage of the fibers [18]. A relation between flexural and compressive strengths (R_f and R_c respectively) was found by Al Rim et al. [5], which was $R_f=k(R_c)^n$. Ductility was also greatly improved by fibers, due to their high tensile strength [15]. The behavior of samples containing wool fibers is given in Figure 1-4 [18]. Fibered samples had an elevated residual strength and more, but smaller, cracks [29]. Deformation was only 3.5% for an earth sample whereas it was about 20% for an earth sample with diss [52]. In only two cases was a decrease of flexural strength found (with the addition of cotton waste [58] and cassava peels [14]).

5.3. Adhesion between coating and wall

The mechanical strength of earth plasters was measured in [47] and [30] by testing the adhesion between the plaster and the wall. Different coating formulations were studied to obtain the best adhesion. The strength was determined by applying a mass that was increased by 0.5 kg every 30 seconds. No standardized test exists. Shear resistance improved with an increasing clay content until shrinkage cracks became too severe. The optimal clay content was around 6%. Shrinkage cracks were reduced by adding sisal fibers or, to a lesser extent, by adding hemp hurds, but there were too few results to observe a significant effect on adhesion with the matrix. However, shear strength depended on the wall on which the plaster was applied. For example, the plaster adhered twice as strongly when the wall was made of rammed earth rather than cob [47].

6. Hygrothermal properties

6.1. Water vapor permeability

The water vapor permeability property has been very little studied for bio-based earth materials. [30] and [67] are the only references in which measurement of the permeability of a fibered earth material is described. In Faria et al. [30] a water vapor resistance factor μ of 8 was found for the wet cup method but the effect of aggregates or fibers was not investigated. In the case of straw-clay mixture [67], which contained much more plant aggregate and was lighter, the water vapor resistance factor was lower: around 3 with the wet cup method and around 5 with the dry cup. However, it was demonstrated that earth had a higher permeability than most building materials [72], although the addition of a stabilizer could change this behavior. In the German standard on earth blocks [73], the use of a standard range between 5 and 10 is suggested, which is in accordance with the values measured on non-fibered earth blocks [74–76].

6.2. Sorption-desorption

Porous materials can absorb a certain quantity of the humidity contained in the ambient air and desorb more or less the same quantity. Unfired earth is known for its high capacity to balance air humidity; it is a good water buffer. Moisture absorption is 50 times higher for unbaked blocks than for bricks fired at high temperature [1]. However, only four references that focused on plasters studied this property on earth with plant aggregate or fiber, [17,25,30,31]. This sorption and desorption property can be considered as static or dynamic. For the static property, it has been shown that earth plaster moisture content increases when the ambient relative humidity increases and decreases when ambient temperature increases, but with a smaller effect. Plasters reinforced by barley or wheat straw and wood shavings show a higher absorption rate (up to 6.5% for barley straw) than earth alone (up to 1.7%) [25]. Sorption isotherms were also studied on different straws not combined with earth by Bouasker et al. [27] and showed similar behavior for sorption. For desorption, the decrease in water content of barley straw occurred at a lower relative humidity than for the other straws.

However, the moisture content of a plaster is in permanent dynamic equilibrium with the environment. A sudden increment of relative humidity showed a dynamic Moisture Buffer Value (MBV) that rose from 50% to 80% with fibered plasters in [17,31]. According to [31], plasters containing 2% of typha chips absorbed 38 $g.m^{-2}$ of water whereas the same plaster without typha absorbed 30 $g.m^{-2}$ and a gypsum wallboard absorbed only 10 $g.m^{-2}$. The plasters studied by [31] belong to the higher adsorption class defined in the German Standard DIN 18947 [77], with a value higher than $60 g.m^{-2}$ after 12 hours, but no differences were observed between the different type and proportion of aggregates or fibers. All the humidity absorbed was desorbed by the samples after 12h [17,30]. Nevertheless, the moisture sorption was lower with 2% of typha wool than without, and it was this aggregate which gave the higher density reduction. All the natural aggregates and fibers presented by Minke [1] permitted an improvement of the water absorption by the material, which increased with the particle weight content. 2% coconut fibers were the most efficient, followed by 2% cellulose fibers, 3% sawdust, 1% coconut fibers, 2% wheat straw and 3% cement (higher humidity sorption after 15 hours). But, generally speaking, humidity regulation is very rapid due to the high permeability of earth material [76].

6.3. Thermal conductivity

Heating and cooling energy can be saved by using material with low thermal conductivity for building. Thermal properties were studied in 13 of the 50 references; it is a common property for building material. Small buildings made of fibered earth blocks were 53.3% cooler in summer than buildings using basaltic pumice blocks and 41.5% warmer in winter [37]. The earthen material allowed 69% savings of heating energy in winter and 57% savings of cooling energy in summer.

It has frequently been shown [5,24,42,51,53,78,79] that an increase in aggregate or fiber content leads to a decrease of thermal conductivity. For instance, the value decreased from 0.24 to 0.008 $W.m^{-1}.K^{-1}$ for a rise in the proportion of wood shavings from 10 to 50% [5]. This evolution could be linked with the composite density and porosity, and a linear relation between thermal conductivity (λ) and density (d) was found in [5]: $\lambda = 0.228d-0.006$. Earlier, the empirical model determined by [40] was: $\lambda_{dry} = 0.103.10^{0.517d}$. The decrease in thermal conductivity was quite small above a wood content of 20% but the swelling due to water absorption continued to increase (a lot from 10%) [42]. Thus, limiting wood content to 20% was an interesting choice to reduce density and thermal conductivity, while avoiding excessive dimensional variations. The increase of porosity and consequently a lower density led to a decrease in thermal conductivity [41]. Thermal conductivities found in the references according to the dry density of the material are recapitulated in Figure 1-5. It can be seen that plant aggregates and fibers, by lightening the composites, led to a decrease in thermal conductivity. However, density is not the only parameter that influences thermal conductivity and a disparity of the conductivity values can be observed for the same density. The type of plant aggregate or fiber, the manufacturing technique or the testing machine can also play a role. Tortuosity has also been found to rise with the wood shaving content and a theoretical model has been created to correlate this physical characteristic with thermal conductivity [41].

Moreover, it was found that thermal conductivity decreased when particle length increased. A better volume occupancy was induced by the longer fibers [51]. The effect of fiber treatment on the thermal properties of fibered earth composites was also studied by Ledhem et al. [42]. Thermal conductivity was not modified by boiling water treatment but increased with linseed oil treatment. This rise was expected because of the higher thermal conductivity of oil in comparison with air and the higher density of the composite.

Thermal insulation decreased a lot when water content increased a little [9,43,56,57]. This can be explained by the fact that air in the pores was replaced by water, which is a better heat conductor than air [43]. Models have been proposed to link water and millet content to thermal conductivity [57] and to thermal capacity [56].

Thermal inertia is one of the principal advantages of earth material. Resistance to changes of temperature increases when thermal diffusivity decreases. The diffusivity was around 4.10^{-7} $m^2.s^{-1}$ for earth material [39,43], whereas it was around 12.10^{-7} $m^2.s^{-1}$ for concrete. However, thermal inertia was not improved by the addition of wood aggregates, and [43] showed that diffusivity was slightly higher with wood than without.

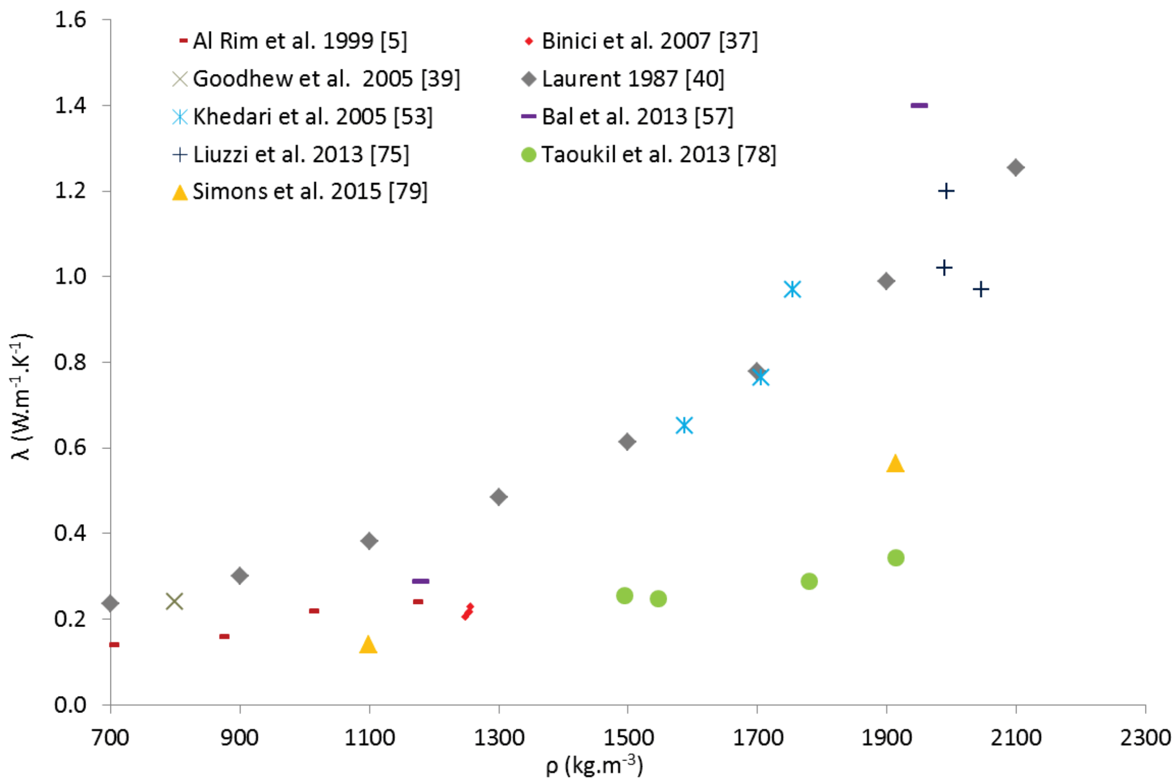

Figure 1-5. Thermal conductivity according to dry density from the literature

7. Durability of the composites

Vernacular heritage is very important all over the world and especially in France, where many villages are composed of earth constructions that were built more than 150 years ago [32]. Earth construction could therefore be considered as durable but, when it is not stabilized with a binder, earth can be very sensitive to environmental factors: meteorological and biological agents (microorganisms) and intrusion by organisms such as insects. Thus the durability of earth composites should be investigated. Few studies have focused on this topic but some authors [5,6,29,42,43] are conscious of the importance of performing such tests and verifications.

7.1. Resistance to water (rain)

Some tests were carried out in several studies but always in different ways. Durability norms created for conventional masonry cannot be applied to unfired earth. Hence, existing procedures had to be adapted. A test called “Bulletin 5 Spray” was adapted by [55]. A spray of water under pressure was applied for 1 hour at a distance of 50 cm from the sample and at pressures of 2.07 and 4.13 MPa. The depth of erosion was measured. The sample was considered to satisfy the water resistance criterion when the speed of erosion was less than 1 mm/min. A water dripping test called the “Geelong test” was performed on adobes in Achenza and Fenu [16]. The blocks were placed at an angle of 30° to the horizontal plane and received water for 30 minutes from a distance of 40 cm above the surface. The water dripping test proposed by [29] lasted 2 hours with the nozzle situated 18 cm from the sample surface and at a pressure of 1 bar. The water erosion test used [60] consisted in exposing a limited surface of a sample to a water jet at a pressure of 1.4 bar for 1 hour, again at 18 cm (Figure 1-6).

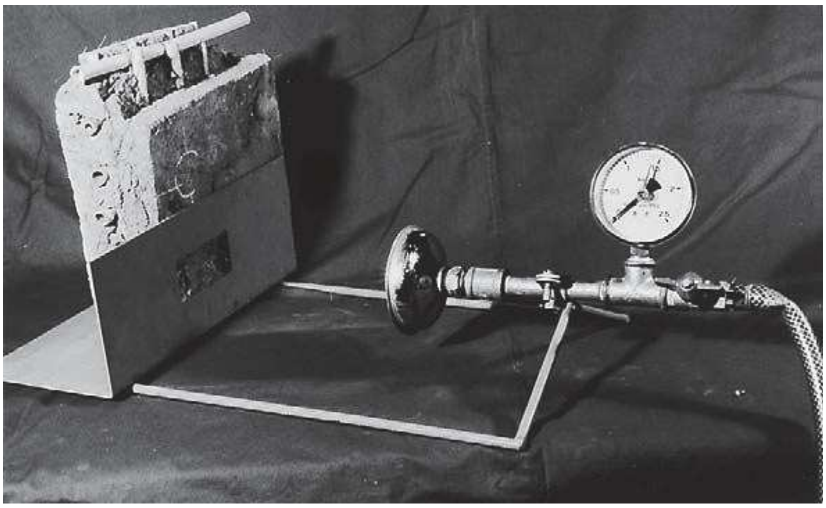

Figure 1-6. Erosion test equipment [60]

Earth containing natural polymer or cement was more resistant to water erosion than the other composites [16,29,60]. For example, samples composed of only earth were totally eroded in 13 minutes, but those containing cement or cactus pulp, resisted 1 hour of “raining” [60]. Only one study compared resistance according to the presence or absence of fibers [55]. All the samples satisfied the maximum criterion of 1 mm/min; soil-cement blocks had a negligible erosion rate whereas the soil-cement blocks with fibers had an erosion rate close to the limit. Using coir in that kind of blocks considerably decreased the durability against water. However, adding aggregates or fibers could be essential for block properties, thus more research is necessary to improve durability properties, by modifying the aggregate or fiber and cement proportions for example.

7.2. Resistance to wind-driven erosion and to abrasion

Wind-driven erosion and abrasion, which are induced by friction, lead to a loss of mass of the material. This has to be limited to avoid recurrent maintenance. This property has been more studied for plasters (3 references) than for blocks (1 reference). Different methods have been tested to measure it: rubbing with an abrasive paper for 20 cycles [60], with different polyethylene brushes [30] or with a metallic brush [51]. Superficial cohesion was also measured with adhesive tape [30] by weighting it before and after testing. It was shown by Hamard et al. [47] that, the higher the clay content, the better the resistance to abrasion. Concerning aggregates or fibers, their presence did not influence resistance to abrasion for the plaster or for stabilizer, according to [60]. However, [51] showed that the abrasion coefficient decreased for hemp fiber contents between 0% and 0.4% by weight, then increase until the fiber content was 0.8%. Fiber length also had an influence: the coefficient was better for fiber lengths of 6 cm than 3 cm. Adhesion between fibers and matrix is better for longer fibers, which explains the better resistance to abrasion.

7.3. Freezing-thawing strength

The ability of unfired earth and plant aggregates or fibers to resist freezing-thawing was studied by [49], but the material was considered as a soil and not as a building material. However, the results are presented here because they are interesting and applicable to earth building material. Only two articles have studied this property for earth blocks [80] and stabilized earth blocks [81], so much remains to be investigated.

Compression strength was measured before and after some freezing-thawing cycles. The higher the jute fiber weight content was, the better were the results (before or after cycles). The strength decreased with the number of cycles when the jute fiber content was between 0 and 0.50%. Beyond 0.50% of jute, the compressive strength increased, except for the third freeze-thaw cycle. The increase in strength was due to the tensile reinforcement brought by the fibers. Even if the soil was subjected to a compressive force, some strains were produced by tensile forces. [49]

7.4. Biodegradation and microorganism development

All natural aggregates and fibers are biodegradable, as experience has shown, and are sensitive to biological attacks [50]. Over the centuries, organic material decomposes, e.g. ancient Roman adobe blocks have disappeared without trace [35] and only some prints persist of other old adobes [82]. This kind of degradation is often caused by microorganisms. Molds were observed on the sample by Flament [46] 10 days after its manufacture and fungi were observed on three earth mortars containing hemp fibers out of the four groups of binders in [83]. In this latter case, the biological growth decreased with the increase of binder and there was no proliferation at all with the hydrated air-lime mortars. Microbiological deterioration can also begin during storage [9] and it may be necessary to add a treatment before mixing the particles with the earth. Some small insects can live in earth based materials if they contain a lot of organic material such as straw or wood. However, it was the wattle and daub technique that presented the highest risk because there were more holes in it (not compacted) [1]. One example of insect degradation is given by [84] with Centris Muralis bees (Figure 1-7). By building their nests, these bees caused huge erosion similar to that caused by weather. It was shown that adobes with high fiber contents were less damaged than others.

To counter such biodegradation, some authors tested treatments (cf. subsection on aggregate and fiber treatment). The Bioterra project will study microorganism proliferation in addition to the mechanical and hygrothermal properties, which seem to be correlated. Sorption-desorption properties can have an impact on the durability of a composite material. A high relative humidity (above 70%) can encourage microbial activity inside the material and thus accelerate biodegradation of the plant material. The project should determine the species that can grow into the material and maybe limit their proliferation.

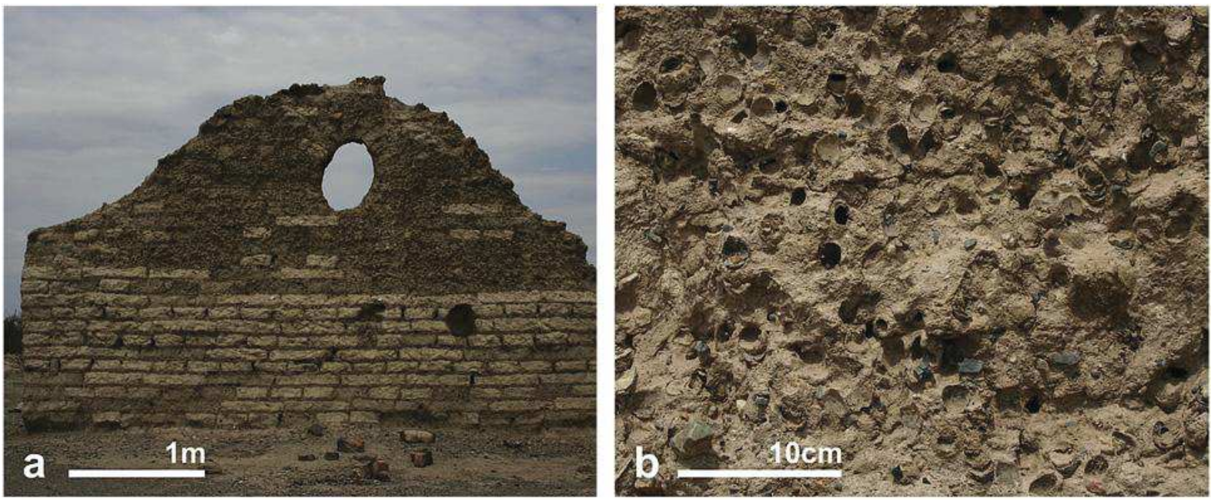

Figure 1-7. (a) Adobe wall damaged by biodeterioration. (b) Detail of superficially deteriorated area [84]

8. Conclusion

Based on a review of plant aggregates and fibers incorporated into an earth matrix, the following conclusions can be drawn:

- The first concerns materials and plant aggregates or fibers. Various types of aggregates or fibers are used in various proportions, and have different shapes, chemical compositions, capacities for water absorption and mechanical properties. Aggregate and fiber treatments are sometimes used to improve characteristics of the composites, e.g. by decreasing water absorption (dimensional variation), or increasing tensile strength (alkaline treatment and acetylation with some conditions), adhesion with the matrix and durability. Six different techniques have been studied, but CEB and stabilized blocks appear most frequently, mostly with cement.

- Concerning physical properties, a decrease of density is observed with the increase in aggregate or fiber content, as is a reduction of shrinkage cracks. Water absorption is increased with the addition of aggregates or fibers, because of their high capacity of absorption, but there have been very few studies. Sound insulation is better with the presence of plant aggregates or fibers, but this conclusion is based on a single existing study.

- Many studies have investigated mechanical properties. The effect of aggregate or fiber on compressive strength depends on the type of plant aggregate or fiber, the particle geometry, and the soil composition. It is generally improved with the addition of cement or another binder. Ductility is always improved with increased plant aggregate or fiber content, while Young's modulus is decreased. Tensile and flexural strengths depend on the shape of the plant particles: they are particularly improved when fibers are used.

- The water vapor permeability of soil is very high, but the influence of plant aggregates or fibers on this property has rarely been studied. A high capacity of the earth to balance air humidity can be noted, which seems to be increased by the addition of plant aggregates or fibers but, again, there are too few studies for a general conclusion to be drawn. Thermal conductivity decreases with the addition of plant aggregates or fibers and this is directly linked with the density of the material.

- Finally, few references deal with durability. Resistance to erosion and abrasion is not really influenced by the presence of plant particles or binder. The behavior of composite materials in compression after freezing-thawing cycles seems to be better with the addition of jute fiber, but only one study has investigated that topic. Concerning biodegradation, molds seem to be observed more often when plant aggregates or fibers are present, but it has been shown that fibers play a role of protection against bees. More work is needed on this vast topic.

Investigations are still needed to complete databases about earth composites with plant aggregates or fibers, especially concerning hygrothermal properties and durability. The main advantage of adding plant aggregates or fibers to earth materials is to improve their thermal insulation or, in other words, to save energy. Other properties of earth materials, such as their ductility or their capacity to balance air humidity, are often improved, whereas compressive strength can be decreased. An optimization of the plant aggregate or fiber content and shape is thus needed in order to obtain the most efficient material. Further experiments are still necessary, including on extruded blocks, which seem to be the easiest material to produce industrially.

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Concluding remarks and thesis methodology

The above literature review focuses on the effects of various bio-aggregates and fibers on engineering properties. It shows the advantages and drawbacks of adding such materials to an earth matrix. The numerous studies about the mechanical properties allow conclusions to be drawn on the reduction of shrinkage, the increase of ductility and, in the case of fibers, the increase in flexural strength. According to the large number of thermal studies, bioresource additions also engender a decrease in the bulk density leading to a reduction of thermal conductivity. However, there are not many studies about the effect of bio-aggregates and fibers on hygric or durability properties, which precludes the drawing of any conclusion. No general tendency can thus be found with bioresources as diverse as shown in the article. Although earth has been used as a building material for centuries, which has led to considerable feedback about its durability, very few scientific studies exist. For example, no study investigates the fire resistance of an earth-based material. Although earth is a non-combustible material, the addition of a high proportion of lignocellulosic resources can raise questions. It is stated in the standard DIN 4102-1 (German Standard, 1977) that, below a density of 1700 $kg.m^{-3}$, an earth material containing straw is no longer considered as non-combustible. Some research concerning low density earth materials would hence be interesting. A similar reasoning can be applied to the resistance to biodegradation. The addition of plant resources, sources of nutrients for microorganisms, can also raise major health issues (Sick Building Syndrome).

This literature review shows the difficulty of comparing earth-based materials containing different bioresources. The challenge lies in the fact that multiple bioresources with distinct chemical compositions, mechanical performances or shapes exist. Moreover, other factors concerning the variability of the earth used, the content of bioresources added into the earth matrix or the testing procedures are also of significant importance. The most striking example might be the mechanical performance. Many parameters vary from one study to another: the manufacturing process, the formulation of the material, the geometry of the specimens, the load or deflection rates of the press, the confinement of the specimen (with friction with the press or not)… There is indeed a lack of international standards to regulate such tests, especially in the case of ductile materials such as bio-based materials.

Consequently, one of the objectives of this thesis is to propose experimental procedures able to be compared with the largest number of bibliographic data. Moreover, this literature study highlights that none of the bioresources used in an earth matrix are studied for all the properties of the composite. For the majority, tests are focused on one or two properties. This thesis will thus study a composite material for all its main engineering properties: mechanical, hygrothermal and durability. The earth material studied is a brick, whether it is compressed or extruded. A single earth matrix, provided by a Bioterra project partner (Carrières du Boulonnais), and five different plant aggregates are investigated: barley straw, hemp shiv, corn cob, rice husk and cork particles. The objective is to study the effects of various plant aggregates on the multiphysical performances of a single earth.

Table 1-9. Experimental tests performed on each kind of material

<table>
<tr><th rowspan="2">Chapter</th><th rowspan="2">Article</th><th>Raw materials</th><th>Earth</th><th colspan="2">Straw</th><th colspan="2">Hemp shiv</th><th colspan="2">Corn cob</th><th>Cork</th><th>Rice husk</th></tr>
<tr><th>Formulation</th><th>FWAS</th><th>S3</th><th>S6</th><th>H3</th><th>H6</th><th>CC3</th><th>CC6</th><th>C3</th><th>RH3</th></tr>
<tr><td rowspan="8">2</td><td rowspan="8">B</td><td colspan="10">Raw material properties</td></tr>
<tr><td>SEM</td><td></td><td colspan="2">X</td><td colspan="2">X</td><td colspan="2">X</td><td></td><td></td></tr>
<tr><td>Granulometry</td><td>X</td><td colspan="2">X</td><td colspan="2">X</td><td colspan="2">X</td><td></td><td></td></tr>
<tr><td>Bulk density</td><td></td><td colspan="2">X</td><td colspan="2">X</td><td colspan="2">X</td><td></td><td></td></tr>
<tr><td>Thermal conductivity</td><td></td><td colspan="2">X</td><td colspan="2">X</td><td colspan="2">X</td><td></td><td></td></tr>
<tr><td>Water absorption</td><td></td><td colspan="2">X</td><td colspan="2">X</td><td colspan="2">X</td><td></td><td></td></tr>
<tr><td>Chemical and/or mineralogical characterization</td><td>X</td><td colspan="2">X</td><td colspan="2">X</td><td colspan="2">X</td><td></td><td></td></tr>
<tr><td>Sorption isotherms</td><td>X</td><td colspan="2">X</td><td colspan="2">X</td><td colspan="2">X</td><td></td><td></td></tr>
<tr><td rowspan="7">3</td><td rowspan="3">C</td><td colspan="10">Mechanical properties</td></tr>
<tr><td>Compressive strength</td><td>X</td><td>X</td><td>X</td><td>X</td><td>X</td><td>X</td><td>X</td><td></td><td></td></tr>
<tr><td>Flexural strength</td><td>X</td><td>X</td><td></td><td>X</td><td></td><td></td><td></td><td></td><td></td></tr>
<tr><td rowspan="4">D</td><td colspan="10">Hygrothermal performances</td></tr>
<tr><td>Thermal conductivity</td><td>X</td><td>X</td><td>X</td><td>X</td><td>X</td><td>X</td><td>X</td><td></td><td></td></tr>
<tr><td>Water vapor permeability</td><td>X</td><td>X</td><td>X</td><td>X</td><td>X</td><td>X</td><td>X</td><td></td><td></td></tr>
<tr><td>Sorption isotherms</td><td>X</td><td>X</td><td>X</td><td>X</td><td>X</td><td>X</td><td>X</td><td></td><td></td></tr>
<tr><td rowspan="9">4</td><td rowspan="5">E</td><td colspan="10">Durability properties</td></tr>
<tr><td>Dry abrasion resistance</td><td>X</td><td>X</td><td></td><td>X</td><td></td><td></td><td></td><td>X</td><td>X</td></tr>
<tr><td>Erosion resistance</td><td>X</td><td>X</td><td></td><td>X</td><td></td><td></td><td></td><td>X</td><td>X</td></tr>
<tr><td>Water absorption</td><td>X</td><td>X</td><td></td><td>X</td><td></td><td></td><td></td><td>X</td><td>X</td></tr>
<tr><td>Impact resistance</td><td>X</td><td>X</td><td></td><td>X</td><td></td><td></td><td></td><td>X</td><td>X</td></tr>
<tr><td>F</td><td>Fire resistance</td><td>X</td><td>X</td><td></td><td>X</td><td></td><td></td><td></td><td></td><td></td></tr>
<tr><td rowspan="3">G</td><td colspan="10">Fungal growth</td></tr>
<tr><td>At 30°C</td><td>X</td><td>X</td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></tr>
<tr><td>At 20°C</td><td>X</td><td>X</td><td></td><td>X</td><td></td><td></td><td></td><td></td><td></td></tr>
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In order to facilitate the overall understanding of the thesis organization, Table 1-9 summarizes the various experimental tests performed according to the raw material or the formulation. Because of time and organization restrictions (stay in other laboratories), not all tests were conducted on each material. The formulations studied are the following: FWAS, a material made of earth alone; S3 and S6 composites containing 3 and 6% of straw, respectively; H3 and H6 containing 3 and 6% of hemp shiv; CC3 and CC6 containing 3 and 6% of corn cob; C3 containing 3% of cork granules and RH3 containing 3% of rice husk. Barley straw and hemp shiv, with a weight content of 3%, are the only aggregates studied for all the properties. As will be seen later, straw is largely available and promising, and hemp shiv is already quite well known thanks to studies on hemp concrete, which will allow some comparisons to be made. Moreover, the content of 3% is favored because it appeared, after a preliminary study, that these formulations were more easily extrudable.

There is a huge diversity of plant aggregates or fibers that can be used for building applications. However, a specific bioresource has to be chosen wisely. For example, banana fibers or wheat straw are not available everywhere or in the same geographical locations. It is naturally ecologically more interesting to valorize local resources. Hence, an investigation about the availability of biomass in France is undertaken to determine the resources that are potentially interesting to study in this country. Moreover, to understand the properties of a composite, it is important to correctly characterize the bioresource used. Such a study could also facilitate the design of the composites according to the intended purpose: nature of the matrix (earth or other mineral binder), plant aggregate content, particle size distribution etc. These different aspects will be discussed in chapter 2, concerning barley straw, hemp shiv and corn cob.

Chapter 2

Availability and characterization of three bioresources

Preamble

Bio-based materials are composed of plant, or sometimes animal, resources and, most often, of a matrix. This chapter focuses on the bio-aggregates potentially used in construction materials generally, not only earth-based materials. The bio-aggregates can be added to a hydraulic, pozzolanic, air lime, organic or gypsum binder, or just used as loose-fill insulation material. The bioresources studied in this thesis are plant resources, also called lignocellulosic resources. They might be by-products of agriculture, the agri-food industry or forestry, for example.

The first part of this work aims to establish a list of lignocellulosic resources produced in France. There is also a need to assess their availability since they are primarily destined for human consumption (including animal feed and litter). Their use as building materials is not common yet as research in this field is quite recent. As presented in the previous chapter, few bio-aggregates used in earth-based materials have been studied at different levels (chemical, physical, mechanical and hygric). Hemp shiv is probably the most studied plant aggregate, thanks to much research on hemp concrete. The second part of this article is thus the evaluation of the different characteristics of bioresources that it is essential to know if their potential as bio-aggregates in construction is to be assessed. First appraised are: the physical properties, with a microscopic description; the particle size distribution by image analysis; and the determination of bulk density, thermal conductivity and water absorption. The chemical composition and the sorption-desorption capacity are then determined. These properties will give a better understanding of the performances of the composite materials. No standardized method exists to test this kind of material. Hence, the present characterization mainly follows the methodology proposed by RILEM for bio-aggregates (Amziane et al., 2017), which was essentially tested on hemp shiv. The application of this methodology to other types of lignocellulosic resources is also discussed.

The three main agro-resources used in the present work, namely barley straw, hemp shiv and corn cob, are chosen according to several parameters. First of all, they are selected for their local availability, then for their presence in materials already studied (like hemp shiv) and in traditional materials (like straw in wattle and daub), or for their originality (in the case of corn cob).

This work (Article B) was published in Waste and Biomass Valorization in April 2017. Rice husk and cork aggregates are not studied in this paper but they are used for durability tests. A short state-of-the-art concerning these plant aggregates is thus given in Chapter 4 and some characteristics are determined according to the same methodology as presented here.

Article B

(Article published in Waste and Biomass Valorization in April 2017)

Characterization of barley straw, hemp shiv and corn cob as resources for bioaggregate based building materials

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Abstract

Expanding the use of low-environmental impact materials in the field of building materials is a major aim in a context of sustainable development. These alternative materials should be non-polluting, eventually recycled, and locally available. Bioresources are already used in some building materials but few studies have investigated their relevance in such applications. The aim of this paper is to evaluate the suitability of three kinds of vegetal aggregates: barley straw, hemp shiv and corn cob. The availability of these bioresources, extracted from a French database, is discussed, as are their physical properties and chemical compositions. Their microstructure is described with SEM images and their particle size distributions are provided through image analysis. Sorption-desorption isotherms are measured by a Dynamic Vapour Sorption system. Bulk density, thermal conductivity and water absorption are also quantified. The results highlight a tubular structure for the three different aggregates, with low bulk density and thermal conductivity (0.044, 0.051 and 0.096 $W.m^{-1}.K^{-1}$ respectively for straw, hemp shiv and corn cob) and high water absorption, especially for barley straw and hemp shiv (414 and 380% vs. 123% for corn cob). Their hygric regulation capacity is also sufficiently good, with a water sorption of between 20 and 26% at 95% of relative humidity. These plant aggregates could therefore be used as additions in an earth matrix, or a hydraulic, pozzolanic, air lime or gypsum binder, or just as loose-fill insulation material. However, future research should focus on their resistance to fire and bacterial growth to validate this approach.

Keywords

Barley straw, hemp shiv, corn cob, bio-based building material, availability, properties

1. Introduction

The impacts of buildings on environment, and especially on energy consumption and CO_2 emissions have now become priority issues. Energy use in buildings generates about 40% of the EU's total final energy consumption and 36% of its CO_2 emissions [1]. Innovation or changes are thus necessary to decrease buildings' environmental impact and improve their energy efficiency. Currently, in France, a huge proportion of non-renewable materials is used in the construction industry and large amounts of waste are produced (around 50 million tons per year, while municipal solid waste is around 30 million tons per year [2]).

On the way to a sustainable future, eco-friendly building materials could be part of the solution. These materials would allow consumption and pollution to be reduced during the production process and also during their whole service life and their end-of-life. In that context, bio-based building materials present the advantage of using plant resources that have absorbed CO_2 through photosynthesis and can thus reduce the material's environmental impact by sequestering CO_2 for at least the life-time of the construction [3]. Bio-sourced materials and the building sector have been identified by the French Ministry of Ecology, Sustainable Development and Energy (Commissioner-General for Sustainable Development) as one of the 18 "green" sectors with a high potential for economic development in the future. In order to produce these low carbon materials, renewable resources, such as by-products from agriculture or forests, are needed. An example that is being increasingly studied is a bio-based earth material. Unfired earth bricks are fibred with distinct renewable resources such as kenaf fibre [4], straw [5], wood chips [6] or wool fibre [7]. Plant concretes, which combine bioaggregates with a pozzolanic, lime-based or cement matrix, are also a good alternative. Numerous studies on hemp concrete currently exist [8-11] and there are also some concerning sunflower [12] or lavender [13] concrete. Although bio-resources are renewable, they also need to be cultivated at locations close to where they are implemented so as to avoid unnecessary transportation and its related environmental impacts. For that reason, the present paper focuses on estimating the potential availability of such resources in the case of France, where by-products and the availability of agricultural land are increasingly being studied, especially with a view to the use of bio-fuels [14-16]. However, these studies provide information about quantities of available, still-unused by-products which could be employed as building materials. A similar study has already been carried out by Palumbo et al. [17] for insulating materials. Their study focused on the main resources available in Europe and especially in Spain, i.e. cereals and sunflower. Biomass is more and more in demand. In the past, it was already used but population growth engendered an increase in food needs. Studies are now being conducted to avoid biomass usage conflicts between priority sectors and others. The former concern human consumption, including animal feed and litter, whereas the latter are related to industry (biomaterials, bio-based chemistry) and energy, which are considered as lower priority by France Agrimer [18]. This study will focus only on plant-sourced biomass.

Plant particle characteristics, which are very specific to the raw material, are studied and compared in the present paper. These characteristics have to be taken into account in the development and the characterization of further composite materials. Three plant aggregates were studied in this work because they were readily available and presented important morphological differences: barley straw, hemp shiv and corn cob. Straw is currently one of the most commonly studied plant aggregates, and is the subject of one third of the 50 references reviewed by Laborel-Préneron et al. [19]. This resource, very common in the plant world, is also present in our cultural heritage [20], [21]. It can come from wheat [22-24], barley [22], [25], oats [26] or other cereals. Hemp shiv constitutes a resource that has received considerable attention in France, which is the greatest producer of hemp in Europe with more than 50% of the total European production [27]. This plant aggregate is especially studied for use in hemp concrete [28], [29] but also as a bio-composite material with earth [30], [31]. Corn cob is an original resource that has been studied only once with an earth matrix, but not crushed [32]. It was studied by Verdier et al. in a pozzolanic matrix [33], and corn pith, which is softer, was studied by Palumbo as an insulating material in an alginate matrix [34].

The use of bio-based aggregates in building materials is becoming increasingly widespread. It is worth noting that various plant aggregates are available in the world and could be used for building construction. However at the present time, no international standardized method exists for characterizing such materials, as it already exists for mineral aggregates. The new standard could define testing protocols for the characterization of bio-aggregates and also the restrictions applying to each application in building materials. This lack of recognized procedures led the RILEM BBM Technical Committee to work on recommendations concerning protocols for bio-aggregate characterization, mainly on hemp shiv [35].

Applying these recommendations to other plant aggregates is a way to check the validity of the method. The objective of this study is thus to characterize and observe the differences of three available agro-resources by following the RILEM recommendations in terms of bulk density, thermal conductivity, water absorption and particle size analysis. Complementary characteristics proposed by other authors are also studied: availability in France, microscopic description, chemical composition and sorption-desorption capacity. This whole methodology could allow the differences between the characteristics of these three plant particles to be highlighted and their potential for future applications in building materials to be assessed.

2. Materials and methods

2.1. Materials

2.1.1. Barley straw

Straw is an agricultural by-product and is the part of cereal stems rejected during the harvest. Barley is harvested once or twice a year. It is the third most cultivated cereal in France with 10 million tons per year [18]. The straw studied here (Figure 2-1), already chopped, was supplied by the CalyClay company (Drôme, France), which is specialized in services for straw and earth construction.

2.1.2. Hemp shiv

France was the first producer of hemp in 2013 [36]. Hemp shiv is the by-product of the hemp defibration process and corresponds to the lignin-rich part of the stem (Figure 2-1). It was provided by the Agrofibre company in Cazères (Haute-Garonne, France).

2.1.3. Corn cob

Maize is the second most cultivated cereal in France, with around 15 million tons per year [18]. Corn cob is the central part of the ear of maize, cleared of grain and crushed. The "woody" part (in red in Figure 2-1), which is also the hardest part, was studied here. This corn cob, already calibrated, was provided by the Eurocob company in Maubourguet (Hautes-Pyrénées, France).

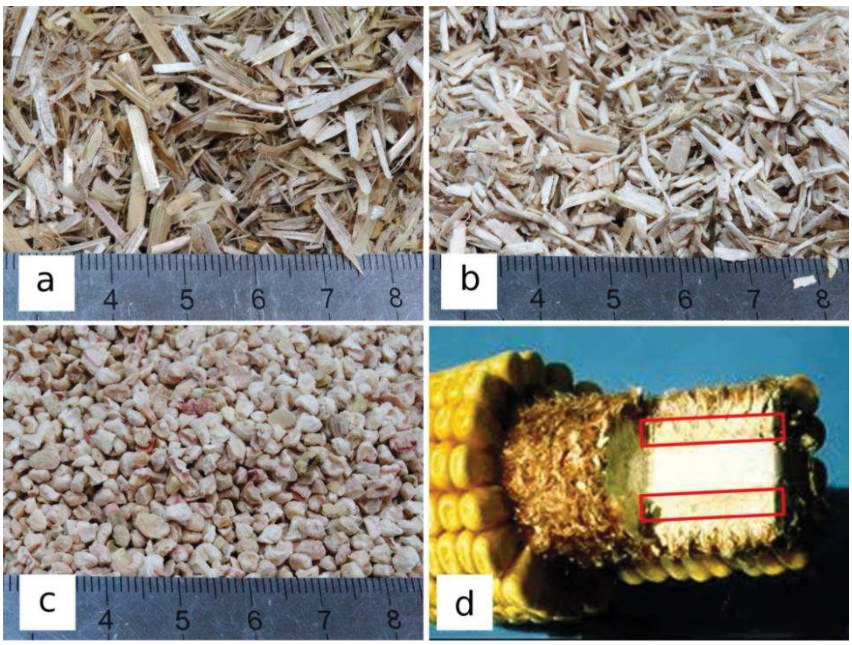


Figure 2-1. Raw materials (a) Barley straw, (b) Hemp shiv, (c) Corn cob and (d) Part of corn studied (in red)

2.2. Availability of agricultural and forestry by-products

The availability of resources was estimated for the case of metropolitan France and specifically for biomass from agriculture, the agri-food sector, industrial crops and forestry residues. Most data providing both the yearly total and the available production of crop by-products come from a national authority, France Agrimer [18], which monitors products and biomass from agriculture and the sea. The data presented in this study are from 2015 and some values were completed by data from 2013-2014 in Agreste [37], provided by the French Ministère de l'Agriculture, de l'Agroalimentaire et de la Forêt. In these two references, data were collected from various economic bodies such as Ademe (Agence de l'Environnement et de la Maîtrise de l'Energie), IGN (Institut National de l'Information Géographique et Forestière) and the FAO (Food and Agriculture Organization of the United Nations). Nevertheless, the collected information should be interpreted cautiously, as a resource may not have been fully counted.

Some data, specifically for industrial crops, were expressed only as quantity produced, and were therefore converted into quantity available by applying the unused biomass factor found in Jölli and Giljum [38]. Furthermore, data corresponding to forestry biomass were given by volume per year (Mm^3/y). In order to be compared with the other resources, volume was converted into mass assuming a density of 0.88 t/m^3, which is an average for various wood species from an FCBA memento [39]. No recent data were available concerning fruit production, except for those for wine and cider production. Agri-food industry by-products will be thus slightly undervalued.

Twenty-four distinct resources were documented in these two references (France Agrimer and Agreste), such as soft wheat, sunflower and beetroot. It was decided to group them into 5 families: cereals, oilseed crops, industrial crops, agri-food industry residues and wood residues.

The method for determining the available by-products (Ba) was based on the following equation 1, greatly inspired by the work of Palumbo et al. [17]

$$Ba = Bt - Bn - Bu \tag{1}$$

where Bt is the total by-products (Mt/y), Bn is the non-harvestable by-products (Mt/y) (necessary to maintain soil fertility or inaccessible) and Bu corresponds to by-products allocated for other uses (Mt/y) such as litter, animal feed or energy (only in the case of wood). All quantities of by-products were expressed by mass (dry basis) over one year and they were determined from areas and farm yield. This equation is recapitulated by Figure 2-2.

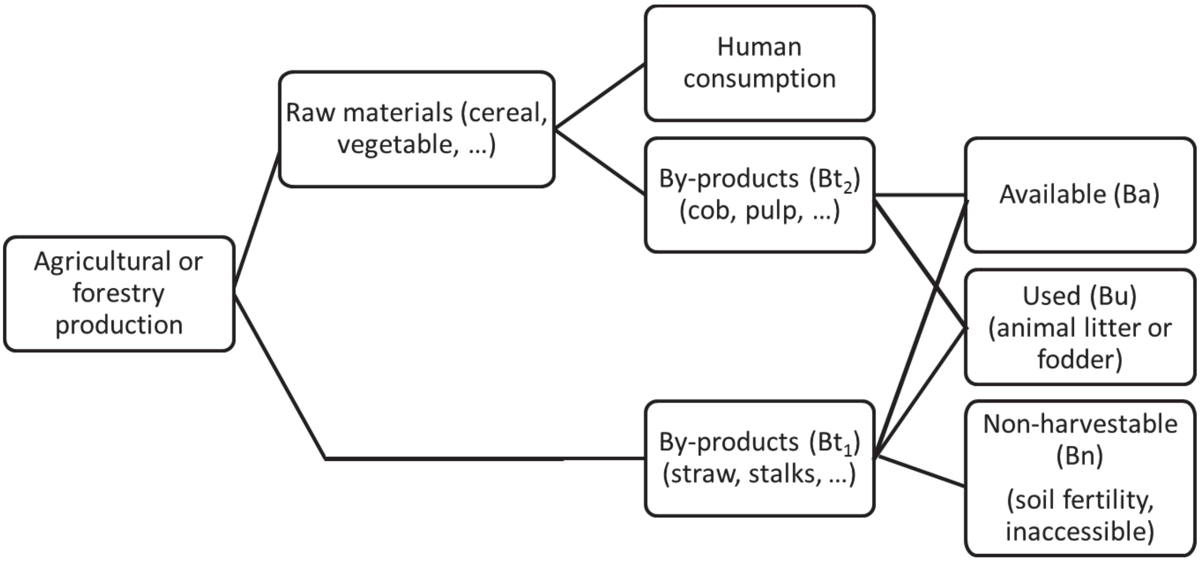


Figure 2-2. Recapitulative scheme of utilization of by-products

For example, for oleaginous plants, it was considered that the minimum return to the soil to maintain agronomic potential was 50% of the straw produced [18].

2.3. Physical properties of plant aggregates

Most of the physical plant aggregate characterization tests were based on a current work of the RILEM TC 236-BBM because there is no standardized method for this kind of material. To evaluate the validity of the results and analyse the dispersion of the results, these protocols recommend calculating a coefficient of variation. This coefficient corresponds to the ratio between the standard deviation and the mean value. The mean value is considered to be representative if the coefficient is lower than 5%. The methods are explained in the following sections.

2.3.1. Microscopic description (SEM images)

Porous structure and morphology were analysed visually with a JEOL - JSM-6380 LV Scanning Electron Microscope (SEM). The particles were glued onto a metallic support and then covered with a thin layer of evaporated gold before observation. These microstructural investigations were carried out with a 15 kV accelerating voltage in high vacuum mode.

2.3.2. Particle size analysis

A particle size analysis was performed by image analysis using ImageJ software. This increasingly used method [40-42] is particularly interesting in the case of non-spherical particles. Size distribution and morphology (width and length) were determined using this method whereas traditional mechanical sieving analysis would have given only the width. However, this method is only achievable for small quantities of particles.

First, the particles were sieved at 500 µm to remove dust. The plant aggregates were then homogenized before being distributed into small boxes. The particles were scanned on a black background in order to obtain better contrast for the ImageJ analysis (Figure 2-3). All the particles were then grouped into a single table to plot representative curves. In total, more than 7000 particles were analysed for each type of plant aggregate which corresponds to a mass of 6.7 g of straw, 13.6 g of hemp shiv or 71.0 g of corn cob. This is more than the mass recommended by the RILEM TC 236-BBM, of between 3 and 6 g, or the minimum number of 1000 particles suggested by [43]. Sampling quality is a key point for the representativeness of the results.

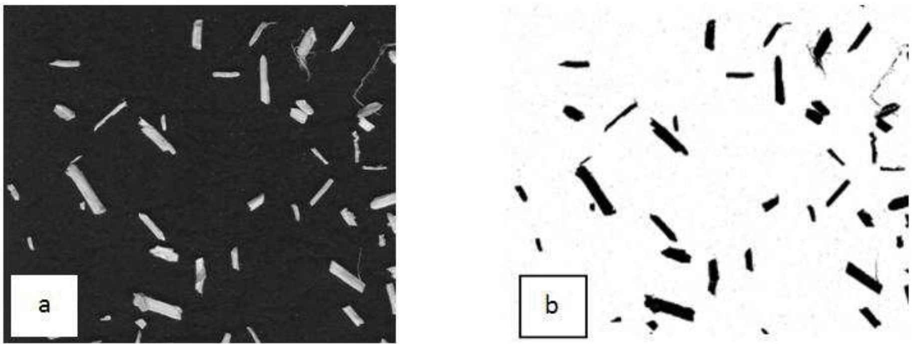


Figure 2-3. Particle image processing for straw: (a) Scan in shades of grey, (b) Image processing

This analysis gave the geometrical parameters of the particles: the major and minor axis lengths (Major and Minor respectively), and the Equivalent Area Diameter (EAD), based on a particle of circular cross section and calculated with equation 2:

$$EAD = \sqrt{\frac{4 * A}{\pi}} \quad (2)$$

with A the cross sectional area of the particle (m²). It also gave the aspect ratio (AR) which is the length to width ratio, or Major to Minor ratio. If the value is close to 1, the particle is almost circular or square; the more AR differs from 1, the more elongated is the particle.

2.3.3. Bulk density

Three specimens of each plant aggregate were dried at 60°C until the weight became constant (weight variation of less than 0.1% between two weighings 24 h apart). Each specimen was put in a cylindrical mould 12 cm in diameter and 24 cm high. The mould dimensions were chosen in accordance with the RILEM work. The RILEM TC observed that the mould size used to measure bulk density had very little effect on the density as long as the height was at least twice the diameter and the diameter was at least 10 cm (big enough compared to the particle size). The quantity of material was selected by a quartering procedure and adjusted to be half the volume of the mould. The mould was then upended 10 times before the final level was marked with a cardboard disk. The volume occupied by the particles was measured from the weight of the corresponding volume of water and the bulk density (ρ_b, $kg.m^{-3}$) was calculated using equation 3:

$$\rho_b = \frac{m_d}{m_w} \times \rho_w \tag{3}$$

where m_d is the mass of dry particles (kg), m_w is the mass of water (kg) and ρ_w is the density of water (1000 $kg.m^{-3}$).

The bulk density of a given type of plant aggregate was taken as the average value measured on three samples.

2.3.4. Thermal conductivity

Three specimens of each plant aggregate were dried at 60°C until the weight became constant (weight variation less than 0.1% between two weighings 24 h apart). The particles were put in a PVC box of dimensions 15x15x5 cm^3, thermal conductivity 0.11934 $W.m^{-1}.K^{-1}$ and thickness 1.6 mm. According to the recommendations of the RILEM TC 236-BBM, the dry density of the sample for thermal conductivity measurement was checked and adjusted (by shaking) to the same value measured during the bulk density test. For this last test, the procedure recommends upending the cylindrical mold ten times. The measurements were made with a hot plate apparatus (λ-meter EP 500) and were performed at 25°C. The specimen was located between the two plates of the apparatus, one hot and the other one cold, with a temperature difference of ΔT=10 K. A steady state was assumed to have been reached when the change in conductivity was less than 1% in 60 minutes. The apparent thermal conductivity of the plant aggregates within the PVC box (λ_{app} in $W.m^{-1}.K^{-1}$) was calculated at the steady state with the following equation:

$$\lambda_{app} = \frac{Q.e_t}{\Delta T.S} \tag{4}$$

where Q is the heat input (W), e_t the total thickness (m) and S the cross section of the specimen (m^2). Knowing the dimensions and thermal conductivity of the PVC box, the thermal conductivities of the plant aggregates were then deduced. A transfer by conductivity through the plant aggregates and the box was assumed.

The specimens were weighed at the end of the test to measure the water uptake during the measurement.

2.3.5. Water absorption

Water absorption during 1 minute, 15 minutes, 4 hours and 48 hours was measured on 3 specimens of each of the 3 plant aggregates. Each sample was dried at 60°C until the weight became constant (weight variation less than 0.1% between two weighings 24h apart). Specimens were put into permeable nets for which the water absorption was negligible.

Their mass, of 25 g for straw, 20 g for hemp shiv and 50 g for corn cob, depended on the available volume of the nets. It is assumed that representativeness was ensured by careful mixing and quartering the particles. After water immersion, the samples were drained for 1 minute in their nets with a salad spinner and then weighed to determine their water absorption (%) according to the following equation:

$$w(t) = \frac{m(t) - m_0}{m_0} \times 100 \tag{5}$$

where m(t) is the wet mass after spinning (kg) and m_0 is the dry mass (kg).

2.4. Chemical characterization

Before the tests, samples were crushed to a grain size of less than 1 mm and dried at 105°C for at least 12 h. The main chemical compounds were measured by the Eurofins company using the Van Soest method, according to standard NF V18-122 [44]. This test provides 3 results: NDF (Neutral Detergent Fibre), corresponding to the total fibre; ADF (Acid Detergent Fibre), which contains mainly cellulose and lignin; and ADL (Acid Detergent Lignin), corresponding to the lignin. Measurement uncertainties were 10% for NDF and ADF and 15% for ADL. Cellulose and hemicellulose were thus calculated by the subtractions ADF-ADL and NDF-ADF, respectively.

The following two tests were carried out in triplicate in the laboratory. To determine the proportion of water-soluble components, around 1 g of dried material (m_i, kg) and 100 mL of distilled water were introduced into a flask and boiled for 1 h in a heating system with return flow. The mixture was then separated by filtration on a sintered-glass filter. The flask and the filter containing the sample were dried at 105°C and weighed to obtain the mass of aggregates after boiling (m_f, kg) by deducting the tare weights t_1 (flask) and t_2 (filter). The water-soluble content (WS) was determined according to equation 6:

$$WS\ (\%) = \frac{m_i - m_f}{m_i} \times 100 \tag{6}$$

To determine the mineral matter content, around 1 g of dried material (m_i) was heated at 550°C for 7 h in a crucible of tare weight t. After cooling in a desiccator, the ash was weighed in the crucible (m_f). Mineral matter content (MM) was calculated with equation 7:

$$MM\ (\%) = \frac{m_f - t}{m_i} \times 100 \tag{7}$$

The coefficients of variation were between 10 and 18% and between 2 and 8% for extractive and ash contents respectively.

2.5. Sorption-desorption isotherms

The sorption-desorption property is necessary to model the buffering effect of a material and has great influence on its resistance to the proliferation of micro-organisms [34]. The sorption-desorption isotherms of the 3 plant aggregates were evaluated by the Dynamic Vapour Sorption (DVS) method. Temperature and relative humidity were regulated by the device (Surface Measurement Systems, London, UK). The uncertainties of the device are ± 0.01 mg for the microbalance, ± 0.2°C for the PT100 thermometer and ± 0.5% for the dew point sensor. The specimen, suspended by a micro-balance, was weighed every 60 seconds. Two specimens of each plant aggregate were tested and were assumed to be representative of these very hygroscopic materials [45-47]. The mass of the samples tested was very low, between 13 and 65 mg, due to the volume available in the sample holder. The samples were composed of 10 to 20 plant particles. However, Bui et al. [47] have shown that a sample of 20 mg of cut straw shows quite a good representativeness on DVS measurements. Before testing, the specimen was dried for 2 h at 50°C (using dry N_2 gas) in the DVS device. The test was carried out at 23°C, which is the temperature specified in the standard for the saturated salt solution method, NF EN ISO 12571 [48]. Relative humidity was regulated in successive stages from 0 to 95% by steps of 10%, except for the last stage, which was equal to 5%. For each step, the specimen was considered to have reached moisture balance if $dm/dt < 5.10^{-4}$ $\%.min^{-1}$ over a ten-minute period [49] or in a maximum time interval of 360 minutes (twice this time for the last three steps). Figure 2-4 shows an example of straw sorption-desorption behaviour with this programme. It can be seen that, at high relative humidity, the change of step was triggered by the time criterion, which resulted in a slight underestimation of the moisture content for the last three steps.

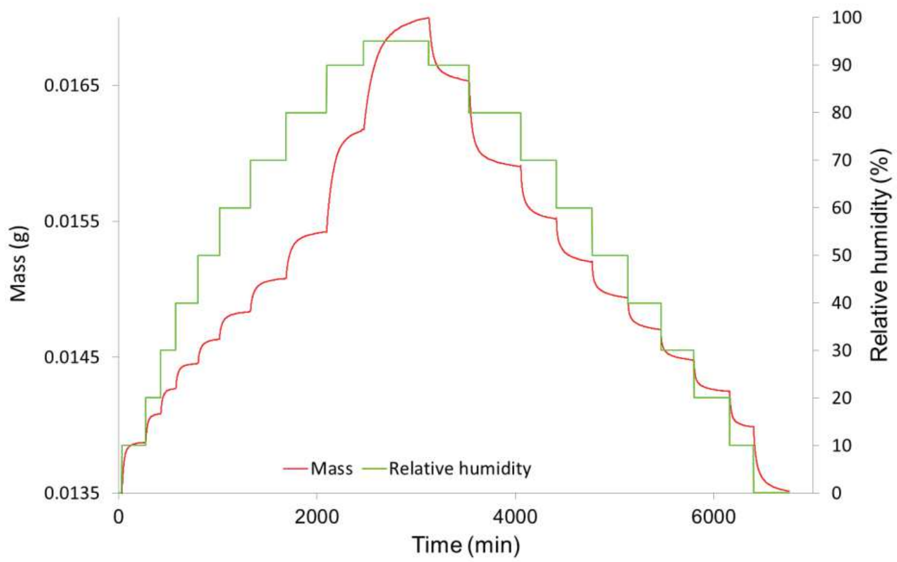


Figure 2-4. Typical variation of mass and relative humidity for the DVS (here sorption-desorption of barley straw)

3. Results and discussion

3.1. Availability of by-products

Figure 2-5 represents the quantities of by-product produced (Bt) and available (Ba) for the 5 families mentioned in the Material and Methods section. Production is greatest for the cereal straw family with 85 Mt/y, while the most available by-product is wood residue with 33 Mt/y. Nevertheless, the availability of cereal straw is 7 Mt/y.

The families of the three plant particles of the present study are framed in orange: barley straw, hemp shiv and corn cob, belonging respectively to cereal straws, industrial crops and agri-food industry by-products.

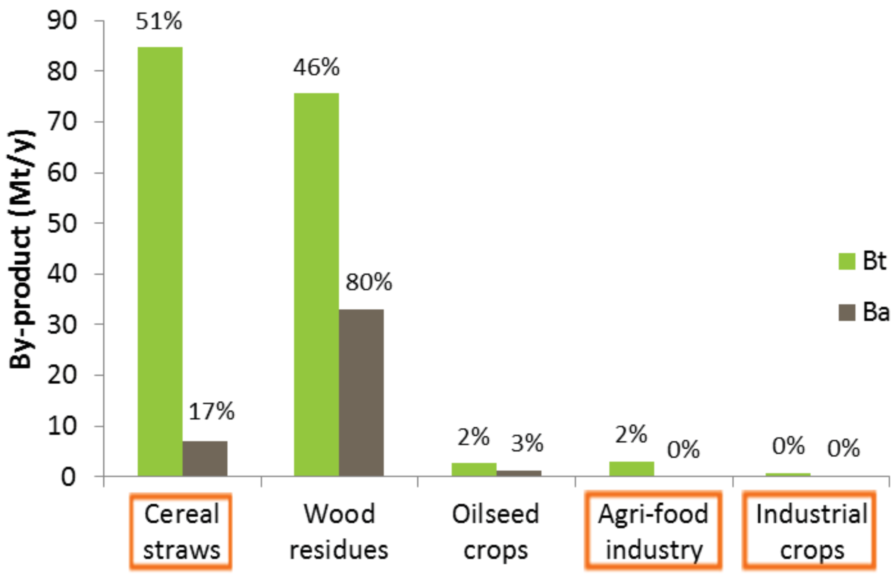


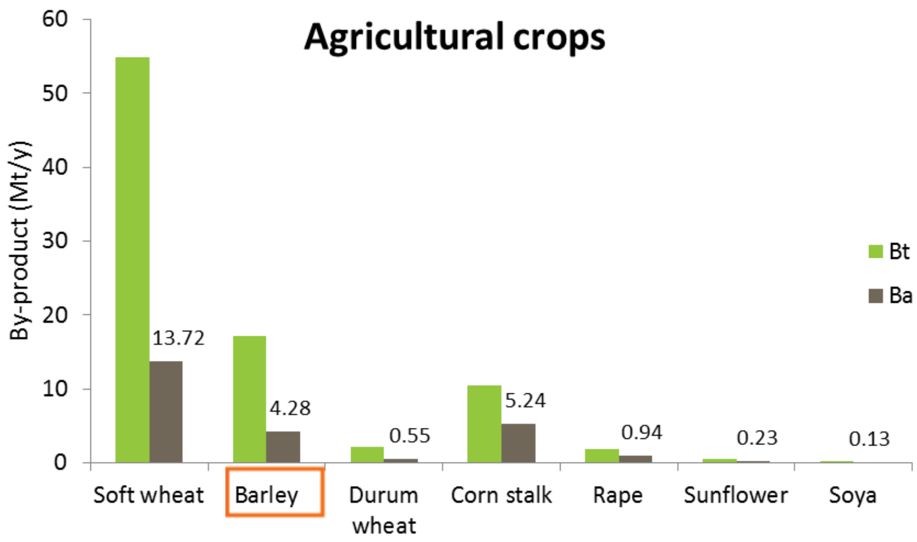
Figure 2-5. Production and availability of different by-product families

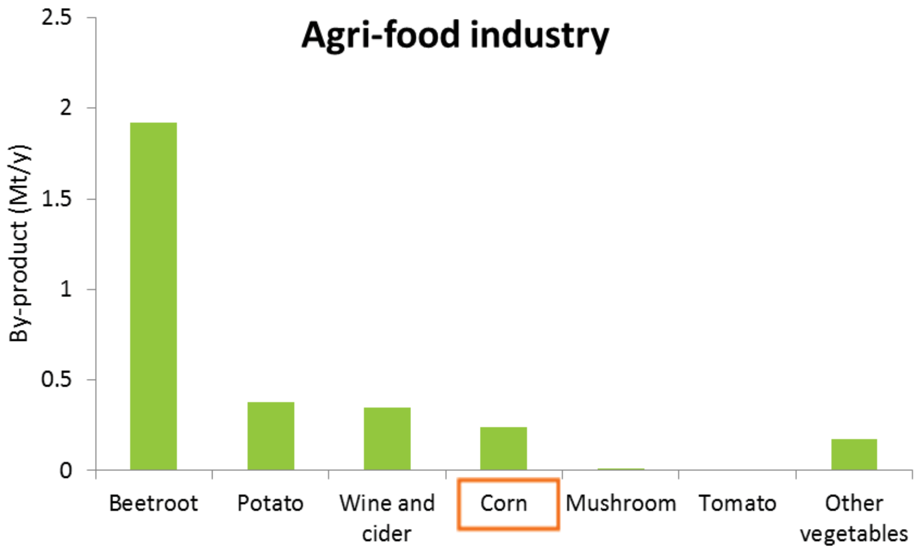
The quantities of by-products produced are represented in Figure 2-6 for the 3 families (agricultural crops, agri-food industry and industrial crops). Production is presented rather than availability, because available resources are not reported by France Agrimer [18] or others for the agri-food industry and industrial by-products. The majority of agri-food industry by-products are used for animal fodder.

Cereal straw and stalks and oleaginous crops are included in agricultural crops. Barley straw makes up the second highest quantity produced in this category and the third most available crop by-product, with 4.3 Mt/y, after soft wheat straw and maize stalk. Much more cereal straw is produced than oleaginous straw (rape, sunflower and soya).

Corn cob is a by-product of the agri-food industry. It is included in corn by-product, along with leaf and damaged grain. The quantity of corn by-product produced is around 0.24 Mt/y, which makes it the fourth category produced in this family.

Hemp is the second most-produced industrial crop by-product after flax, with around 17 thousand tons. This value is much lower than those of the other plant aggregates selected, but it is a plant by-product that has already been the subject of a number of studies for building materials. France is the biggest European hemp producer [27], and some professional rules exist [50]. This has led to the development of construction or rehabilitation of buildings with hemp concrete in the past 10 years.





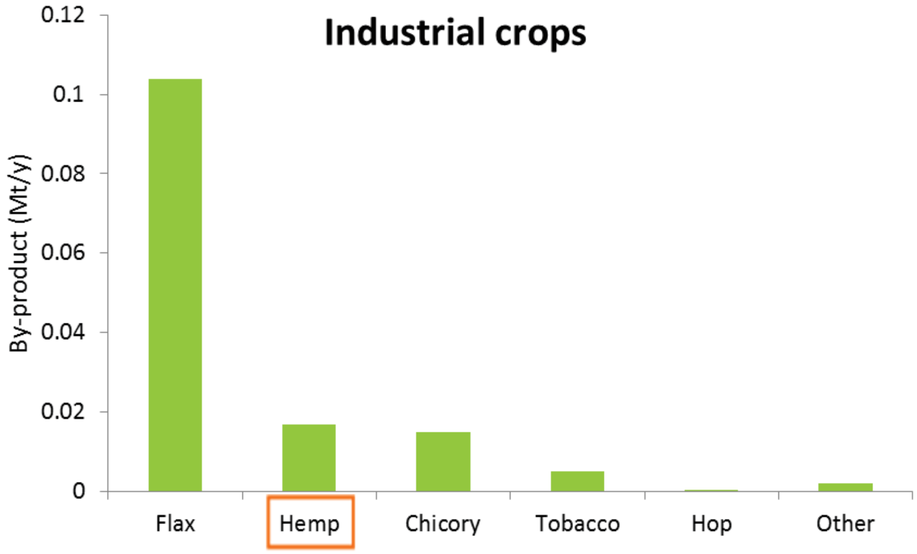


Figure 2-6. Quantities of by-products from agricultural crops, agri-food industry and industrial crops

In France, various types of by-products are available, in different proportions. Cereal straws are the greatest resource available. It is thus interesting to study barley straw in order to develop new bio-composites promoting this kind of resource. Maize and hemp do not provide the greatest quantities of by-products of their respective families. However, they both have huge potential: maize is the cereal most produced in the world [36] and France is the biggest hemp producer in Europe [27]. Forthcoming studies should focus on bioresources with widespread availability, such as wood residues, which represent 80% of the by-products available; flax, which yields around five times as much by-product as hemp; or beetroot residue, which constitutes the largest by-product by weight from agro-industry.

3.2. Physical properties

3.2.1. Microscopic description

The morphology and porous structure of the plant particles are illustrated by the SEM images of Figure 2-7.

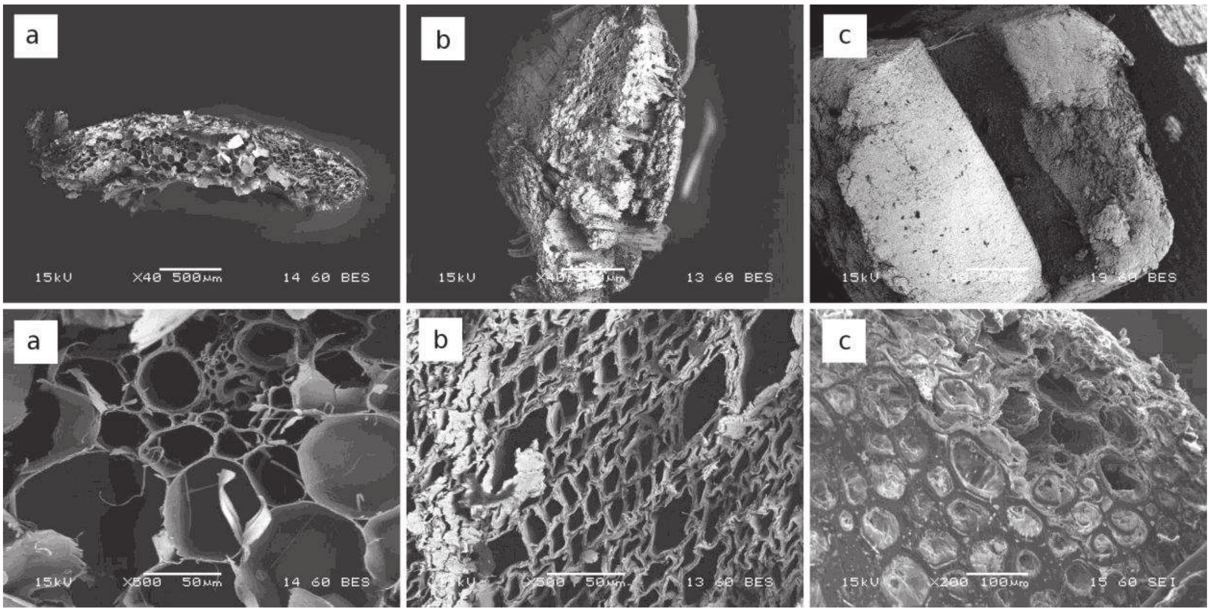


Figure 2-7. SEM images of: (a) straw, (b) hemp shiv and (c) corn cob at low (first line) and high (second line) magnification factors

These SEM images clearly show a tubular microstructure for the three materials. However, some differences can be observed. The pores of the straw, from 2 to 100 µm, are multi-scale and the cell walls are very thin (maximum thickness of 2 µm). Hemp shiv pores range from 5 to 40 µm and the particles present regular cell walls 4 µm thick on average. Concerning corn cob, the diameter of the pores is between 20 and 80 µm with a thick cell wall of up to 45 µm. The fact that the parenchyma is thicker for corn cob than for hemp shiv or straw indicates a lower macro-porosity and thus a higher density of the plant particle. According to Pinto et al. [51], this could lead to a strength capacity comparable to that of soft woods.

3.2.2. Particle size analysis

The particle size distribution of plant aggregates seems to have an influence on the mechanical performance of bio-based building materials. For example, Millogo et al. have shown that the compressive strength of an earth-based composite with short pieces of straw (3 cm) is higher than that of similar composite with longer pieces (6 cm) [4]. Conversely, in the case of hemp concrete, it has been shown that the coarser the hemp shiv is, the higher is the mechanical performance at 28 days [11], [52]. Danso et al. [53] have studied the effect of fibre aspect ratio on mechanical strength in an earth matrix. Compressive and tensile strengths improved for coconut, oil palm and bagasse fibres when the major axes increased.

In this study, the morphological characteristics of straw, hemp shiv and corn cob were compared. Dust content, determined by sieving at 500 µm, was about 7.2%, 2.1% and 0.3% respectively for the straw, hemp shiv and corn cob. The grading curves are presented in Figure 2-8. Averages and standard deviations of the major and minor axes, EAD or aspect ratio were calculated from equations 8 and 9 [40] and are presented in Table 2-1.

$$E_{am}(x) = \frac{\sum A_i\,(x_i)}{\sum A_i} \quad (8)$$

$$Sd_{am}(x) = \sqrt{\frac{\sum A_i(x_i - E_{am}(x))^2}{\sum A_i}} \quad (9)$$

where $E_{am}(x)$ is the arithmetic mean of the dimension x (Major, Minor, EAD or AR), A_i is the projected area of each particle detected (mm²), x_i is the dimension of each particle detected and $Sd_{am}(x)$ is the associated standard deviation.

Table 2-1. Arithmetic means and dispersions of plant aggregate dimensions

Dimension	Barley straw	Hemp shiv	Corn cob
Major (mm)	7.6 ± 4.4	5.6 ± 2.6	3.6 ± 0.8
Minor (mm)	2.3 ± 1.5	2.0 ± 1.2	2.6 ± 0.4
EAD (mm)	4.0 ± 2.1	3.3 ± 1.6	3.1 ± 0.5
AR	4.1 ± 3.7	3.3 ± 2.0	1.4 ± 0.4

The standard deviations associated with the mean dimensions of barley straw and hemp shiv are higher than for the corn cob. This is due to the marked heterogeneity of these particles, which are obtained by mechanical grinding without any specific calibration process. In contrast, the standard deviation associated with the mean values of the corn cob morphological parameters is quite low since this aggregate is made up of a single granular fraction obtained after an industrial process of calibration.

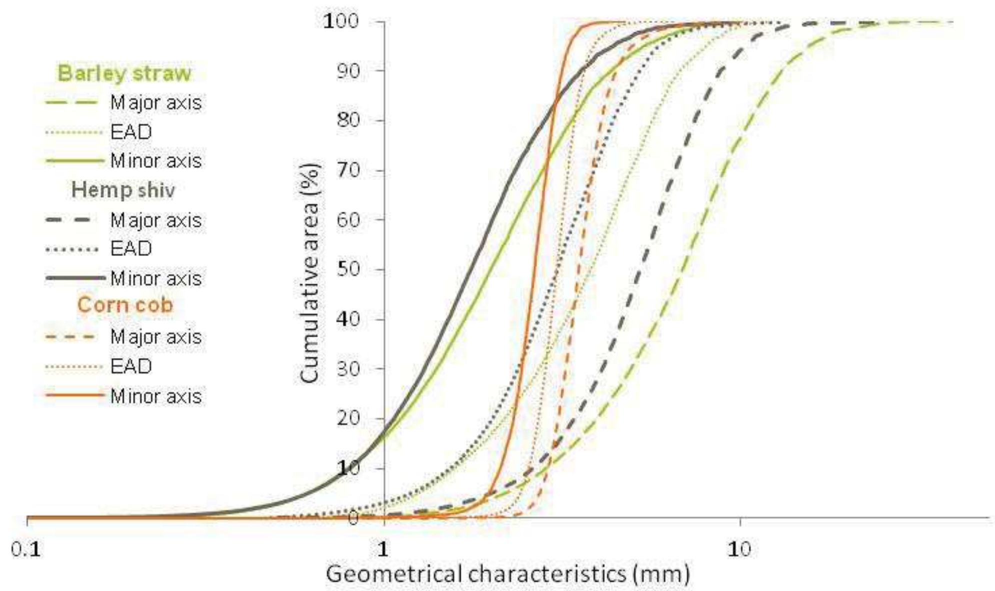


Figure 2-8. Grading curves of the plant aggregates as a function of the cumulative area

It can be observed in Figure 2-8 that the corn cob spindle (between the major and minor axes) is the smallest and the steepest. This means that the major and minor axes are of similar length and the size of the particles is fairly regular. This result is understandable because corn cob is a calibrated material (by the Eurocob company). Grading curves are quite similar between straw and hemp shiv. The size of straw particles is just slightly larger than the size of hemp shiv particles.

Proportions of AR in cumulative area are represented in Figure 2-9.

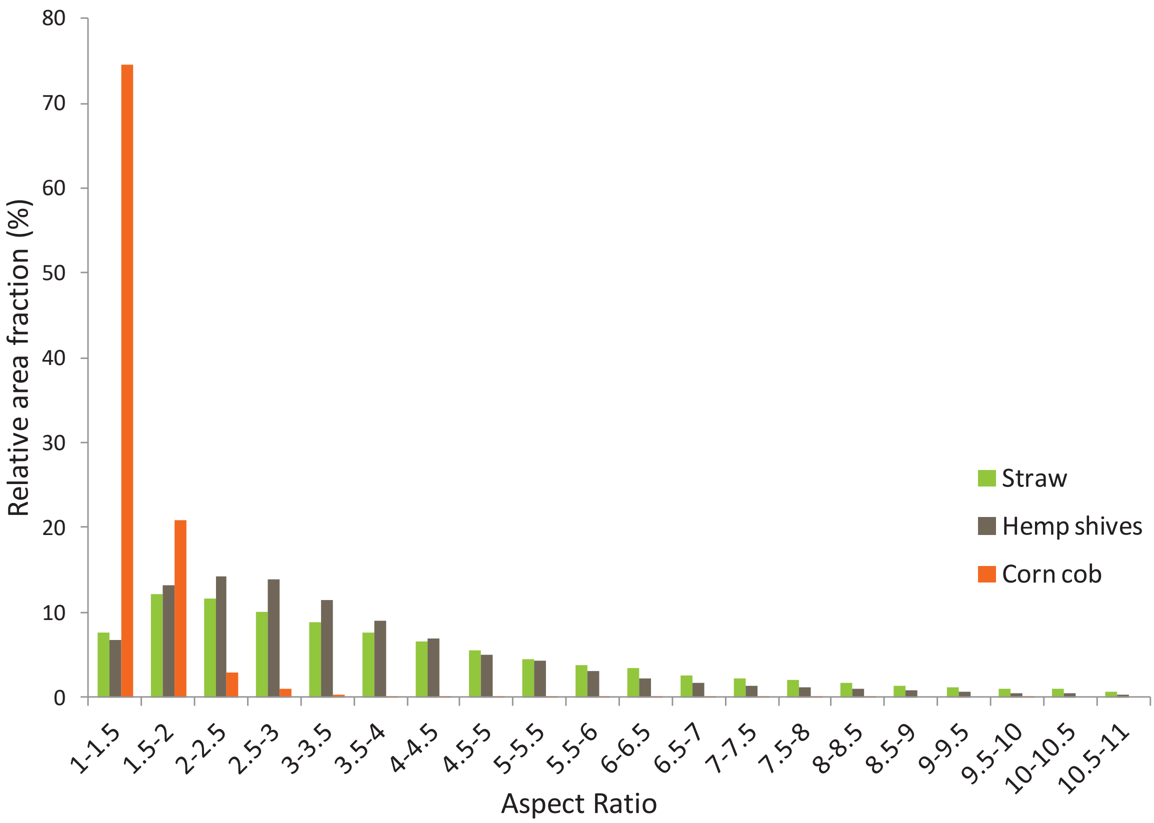


Figure 2-9. Relative area distribution as a function of aspect ratio

It can be noted that the AR of corn cob is very close to 1, meaning that corn cob particles present a nearly circular cross section. Straw and hemp shiv are more elongated (especially straw) with more variation among particles.

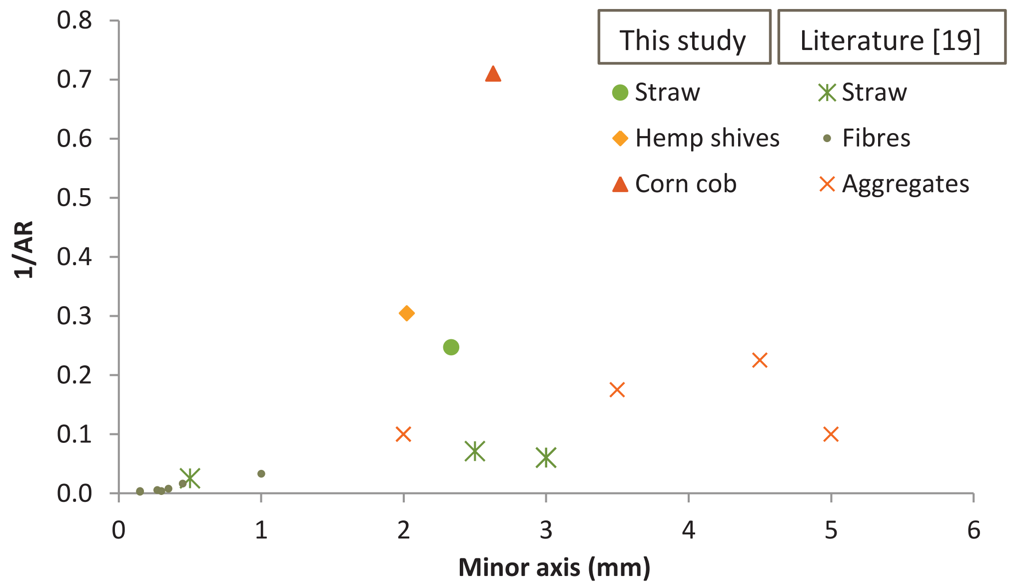


Figure 2-10. Reciprocal of the aspect ratio as a function of average minor axis

In Figure 2-10, the reciprocal of the AR is represented as a function of the average minor axis, in order to be compared with some values found in the literature. The minor axis of this study was considered to be equivalent to the diameter of the literature. These values from the literature, quoted in a review by Laborel-Préneron et al. [19], were classified by type of particle: fibres, aggregates and straw. Their AR was lower than that of the aggregates studied here, although their minor axes were of the same order of magnitude as in the particles of this study. For the straw, this can be explained by the shortness of our particles even though the diameter was similar to those in the literature. Hemp shiv was more crushed and finer than in the literature and corn cob was almost spherical, which is not very common for plant aggregates.

Optimizing the particle size distribution could be an interesting lever to improve the compactness of the particle arrangement of bio-based building materials, as is common for basic concretes. The selection of specific particle fractions or the combination of particles with different morphologies could lead to an enhancement of their mechanical performances.

3.2.3. Bulk density and thermal conductivity

Using insulating materials in buildings is a way to save energy. Adding some vegetal particles into existing building materials such as earth or concrete is a means of reducing their density and thus improving their thermal insulation [6], [54]. Such particles can also be applied in buildings as loose-fill insulation. Bulk density (Table 2-2) and thermal conductivity (Table 2-3) are presented together, because thermal conductivity is highly dependent on the density [19].

Table 2-2. Bulk densities ($kg.m^{-3}$) of the plant aggregates studied

Plant aggregate	Barley straw	Hemp shiv	Corn cob
This study	57 ± 1	153 ± 2	497 ± 14
Literature	47 [55] 116 [56]	110.9 [9] 148.3 [57] 130 [29]	450 [58] 495 [57]

Table 2-2 presents the average bulk density of each plant aggregate with the standard deviation and some values from the literature. The values from this study are close to the values from literature, except for one value for barley straw. This difference may be due the length of the straw or the method used to measure it. Barley straw has the lowest bulk density, followed by hemp shiv and then corn cob, which has the highest. The coefficients of variation are 2.0, 1.6 and 2.8% respectively for straw, hemp shiv and corn cob. This value is lower than 5% which suggests good representativeness of the different samples.

Table 2-3. Thermal conductivity ($W.m^{-1}.K^{-1}$) of the plant aggregates studied

Plant aggregate	Barley straw	Hemp shiv	Corn cob
This study	0.044 ± 0.001	0.051 ± 0.002	0.096 ± 0.001
Literature	0.035-0.054 [22]	0.058 [9] 0.055 [28] 0.057 [42]	0.099 [57]

Table 2-3 shows that the thermal conductivity values measured were similar to those found in the literature. The coefficients of variation are 1.4, 3.9 and 1.1% respectively for straw, hemp shiv and corn cob. This value is lower than 5% which suggests good representativeness of the different samples. Moreover, the water uptake during the test was lower than 0.2% for each specimen. The moisture content, influencing thermal conductivity, can thus be neglected.

Figure 2-11 compares the thermal conductivity of various plant aggregates from this study and the literature in function of their bulk densities. The relation plotted is linear: the lower the bulk density is, the lower is the thermal conductivity. Thermal conductivity depends mostly on the bulk density. However, there are small variations even within a given particle type. They can be explained by the difference in moisture content or in the testing method. The type of vegetal does not seem to influence the thermal conductivity significantly, but more results would be needed to confirm that hypothesis.

The barley straw and hemp shiv characterized in this study present bulk densities and thermal conductivities similar to those of loose-fill insulation materials such as cellulose wadding or glass fibre (quoted in Goodhew and Griffiths [59]). Maskell et al. [60] characterized other natural loose-fill insulation materials: wool, hemp fibre and wood fibre.

Such lightweight aggregates could be used in great quantities in an earth matrix as was done by Labat et al. [5] with an earth clay material. The density was 241 kg.m^{-3} for a thermal conductivity of 0.071 W.m^{-1}.K^{-1}, much lower than for a traditional earth material, for which the corresponding values are between 1940 and 2007 kg.m^{-3} and 0.47 and 0.59 W.m^{-1}.K^{-1} [49]. As far as hemp concrete is concerned, it presents a thermal conductivity between 0.06 and 0.19 W.m^{-1}.K^{-1} for a dry density between 200 and 840 kg.m^{-3} [29].

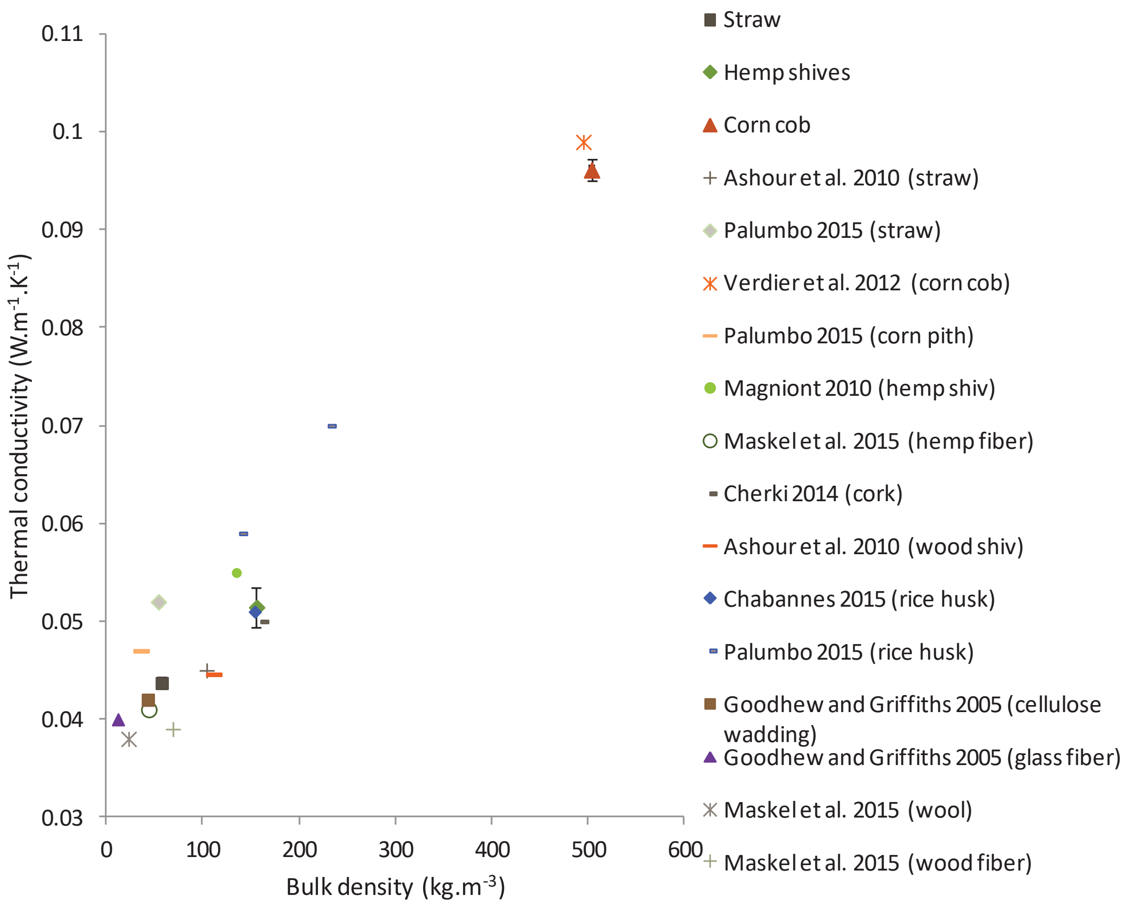


Figure 2-11. Comparison of thermal conductivity values measured experimentally for straw, hemp shiv and corn cob and values found in the literature

These materials, especially barley straw and hemp shiv, could thus be used either as lightweight aggregates to improve the thermal insulation properties of an earth or mineral matrix, or as loose-fill insulation materials. A material is considered to be a thermal insulator when the thermal conductivity is lower than 0.065 W.m^{-1}.K^{-1} [61]. However, the thermal conductivity found for these agro-resources was measured for particles in the dry state and it is known that thermal conductivity increases with relative humidity [62]. The round robin tests of RILEM TC 236 BBM [35] revealed an increase of the thermal conductivity by 12.9% between the dry state and a relative humidity of 50%. If that increase is applied to the values measured for the dried plant aggregates, thermal conductivities become 0.049 $W.m^{-1}.K^{-1}$ for straw, 0.058 $W.m^{-1}.K^{-1}$ for hemp shiv and 0.109 $W.m^{-1}.K^{-1}$ for corn cob. In the case of straw and hemp shiv, these estimated values are still lower than the value expected for an insulation material.

3.2.4. Water absorption

Within a hydraulic matrix, plant aggregate water absorption is an important formulation parameter as competition can occur between the particle absorption and the matrix hydration [9], leading to potential problems. Consequently, the amount of mixing water has to be largely overestimated. This implies a very long drying time, hardly compatible with the current rate of building. Water absorption was evaluated for up to 48 h for the three kinds of aggregates (Table 2-4).

Table 2-4. Absorption capacity (%) after 48 h of immersion

Plant aggregate	Barley straw	Hemp shiv	Corn cob
This study	414 ± 4	380 ± 11	123 ± 2
Literature	500-600 [25] 400 [55]	280 [30] 301 [9] 406 [42]	90 [58] ~150 [57]

Maximum absorption was 414% for straw followed by 380% for hemp shiv and 123% for corn cob in the present work. Calculating the coefficients of variation gave 1.1% for barley straw, 2.8% for hemp shiv and 1.3% for corn cob. These values are lower than 5%, which indicates good representativeness. These retention capacities seemed to be consistent with the literature, which presents nevertheless a high range of water absorption. This high variability could be due to the diversity of the plant particle itself or to the test method used. Only the value from [9] was obtained with the same protocol. Absorbent paper was used in references [30], [57] and the method used was not specified in the other cases. The low water absorption of corn cob could be interesting because, according to Bouhicha et al. [25], a high retention capacity is not good for adhesion to a soil matrix. During the manufacturing process, the swelling of the particles engendered by water absorption in the first 24 hours pushes the soil away. Then, when the composite material dries, shrinkage creates voids around the particles at the interface with the soil [63].

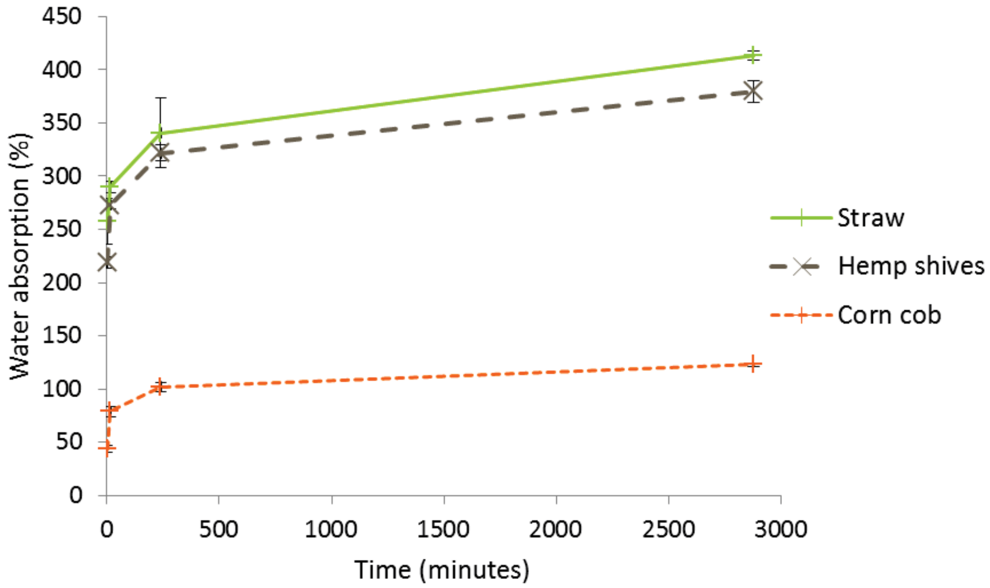


Figure 2-12. Water absorption as a function of time

The kinetics of this absorption is presented in Figure 2-12. It is very fast for these plant aggregates: absorption by straw and hemp shiv at 1 minute is close to 60% of the final absorption (48 h) and 36% for corn cob. These curves can thus be considered in two parts: the first one, with fast kinetics, represents an absorption by capillarity forces in the pores filled by free water [12], [55]. The second one corresponds to a diffusion mechanism based on Fick's law in the micro-pores and water bonding through openings (20-40 nm) in the plant cell walls [64].

Water absorption occurs through the multi-scale porosity of the particles, especially for stems such as straw and hemp shiv, which transport the sap in the plant. This water content seems to be linked with the internal porosity of the material [41]. On the SEM pictures (Figure 2-7), high internal porosity can be seen for straw and hemp shiv. Corn cob is less porous and the water absorption is calculated from the particle mass, so the volume of water absorbed might not be very different from that found for straw and hemp shiv.

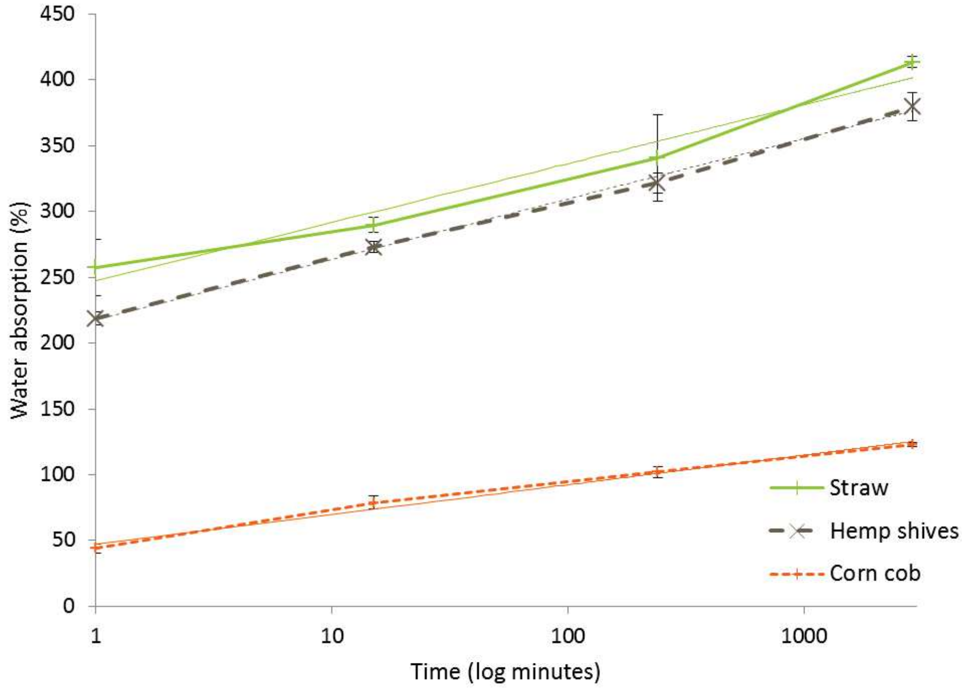


Figure 2-13. Water absorption as a function of logarithm of time

This absorption can be represented as a function of logarithm of time (Figure 2-13), where the curves of the equation:

$$W = IRA + K_1 \times Log(t) \quad (10)$$

proposed by Nozahic and Amziane [12], are linear. The Initial Rate of Absorption (IRA, %) is the absorption relative to an immersion of 1 minute, representing the adsorption of external water on the surface of the particles [65]. K_1 represents the diffusion rate in the cells (%.(log min)$^{-1}$), the second part of the curve. Both values, IRA and K_1, are recapitulated in Table 2-5, with R^2, the correlation coefficient between the experimental values and the straight line.

Table 2-5. Water absorption parameters

Plant aggregate	Barley straw	Hemp shiv	Corn cob
IRA[a]	247	218	48
K_1[b]	19.4	19.9	9.8
R^2[c]	0.9632	0.9972	0.9874

[a] IRA is the Initial Rate of Absorption; [b] K_1 represents the diffusion rate in the cells; [c] R^2 is the correlation coefficient

K_1 and IRA, to a lesser extent, of barley straw and hemp shiv are very close to each other. However, the logarithmic representation of barley straw water absorption is less linear (R^2 of 0.96). The coefficient K_1 of corn cob is lower than the other two, meaning that the water diffusion rate in the cells is quite low.

As previously commented, due to the high water absorption of barley straw and hemp shiv, the amount of mixing water required should be increased if these particles are added to a hydraulic binder [66]. This can lead to a problematic increase of the drying time after demoulding [9]. However, different treatments could be applied to reduce the water absorption and drying time. Some treatments can also increase the bonding between the plant particles and the matrix. Nozahic and Amziane [12] studied three different treatments to improve the adhesion of sunflower aggregates to a mineral matrix: pre-wetting by an alkaline solution, a linseed oil coating, and a paraffin wax coating, which showed the best efficiency. Other plant aggregate treatments were studied in the case of an earth matrix, such as acetylation, rosin-alcohol coating or thermal immersion in boiling water, and are summarized in Laborel-Préneron et al. [19].

3.3. Chemical characterization

The chemical characterization of plant aggregates is important in the case of bio-composites with hydraulic binder as chemical composition can influence their properties or those of the composites, such as setting time or hydration mechanisms.

Lignocellulosic raw materials are composed of three main components: cellulose, hemicellulose and lignin. There are also other chemical species present such as pectin, extractives and ash. Cellulose, a polymer containing various alcoholic hydroxyl groups, can significantly affect the mechanical performance of the fibres [67]. Hemicellulose is a highly hydrophilic component, easily hydrolysed by acids and soluble in dilute alkali solutions. This could thus influence the water absorption of the plant particle and affect its durability in an alkaline mineral matrix, such as cement or lime, and the bonding mechanisms at the interface in these composite materials [68]. Lignin is a polymer able to protect the stem of the plant from chemical or physical aggressions, notably from most microbial attacks. The nature and the amount of lignin thus affect the durability and the biodegradability of the distinct vegetal materials [67]. A variety of functions is attributed to pectins, including mechanical properties, cell-cell adhesion, wall porosity and binding of ions [69]. An affinity between pectin and cations exists and could affect the setting mechanisms of mineral binders (Portland cement, lime, etc.) used in bioaggregate-based composites.

These interactions can take place at different times. At early age, they can disturb the setting and hardening mechanisms of mineral binders. Hemp shiv [70], hemp fibres [71], wood particles [72-75], cereal straw [76], [77] (cited in [78]), arhar stalks [79], sugar cane bagasse [80] and coir particles [81], among others, have been shown to negatively impact the setting and early hardening of cement paste. Wood particles can also be associated with a plaster matrix (calcium sulfate hemihydrate). Boustingorry et al. [82] emphasized that poplar and forest pine extracts clearly delayed the hydration of hemihydrate. Finally, the setting of a pozzolanic binder (mix of lime and metakaolin) has been shown to be affected by the presence of lavender stalks [13]. In the hardened state, they can modify the properties of the composite. Poor cohesion has been observed between plant aggregates and the mineral matrix, associated with a powdering of the binder [70]. In the long term, they can influence the durability of the material through mineralization of the plant aggregates by cement hydration products [83]. This engenders a loss of ductility of the fibres, which is a disadvantage for plant fibre reinforced concrete, which has to work in flexion. However, it might be an advantage for bioaggregate-based composites, leading to a continuous enhancement of compressive strength [84].

The chemical composition of the three plant aggregates is presented in Table 2-6 and compared with some other values from the literature. Values are expressed in percentage of dry mass of the plant material. The column "Extractives" refers to the water-soluble content (this study), the sum of pectin, wax, fat and protein, or the content obtained with Soxhlet extraction (literature).

Table 2-6. Chemical composition of the 3 plant aggregates

Aggregate	Reference	Lignin (%)	Cellulose (%)	Hemicellulose (%)	Extractives (%)	Ash (%)
Barley straw	This study	5.5	37.7	26.7	14.4	12.3
	[85]	15.8	37.6	34.9	-	-
	[86]	15	46	23	-	-
Hemp shiv	This study	17.2	50.3	17.9	5.9	2.1
	[87]	18	52	9	-	2
	[88]	22.1	37.7	26.8	8.9	-
	[70]	21.8-23.3	45.6-49.2	17.8-21	5.1-6.2	2.6-3.7
	[89], [90]	28	48	12	10	2
	[91]	28	44	18	10	2
	[92]	23	44	25	4	1.2
Corn cob	This study	6.6	41.4	40.7	6.9	1.4
	[58]	6.8	47.1	37.3	-	1.2
	[93]	6.7-13.9	32.5-45.6	39.8	-	-
	[68]	14.7	48.1	37.2	7	-

Among the three types of particles studied, barley straw showed the highest amount of extractives (14.4%) and ash (12.3%). The extractive contents of hemp shiv and corn cob were 5.2 and 6.9%, respectively. The ash content of these two aggregates was also lower, at 2.1 and 1.4%. It is important to take the extractives content into account because it is the main cause of interactions with the hydraulic binder.

These chemical compositions are represented in a ternary diagram showing lignin, cellulose and hemicellulose (Figure 2-14).The values are normalized to 100%, as in [68]. The comparison of the composition of particles is facilitated by this presentation. The plant aggregates used in this study are circled in red.

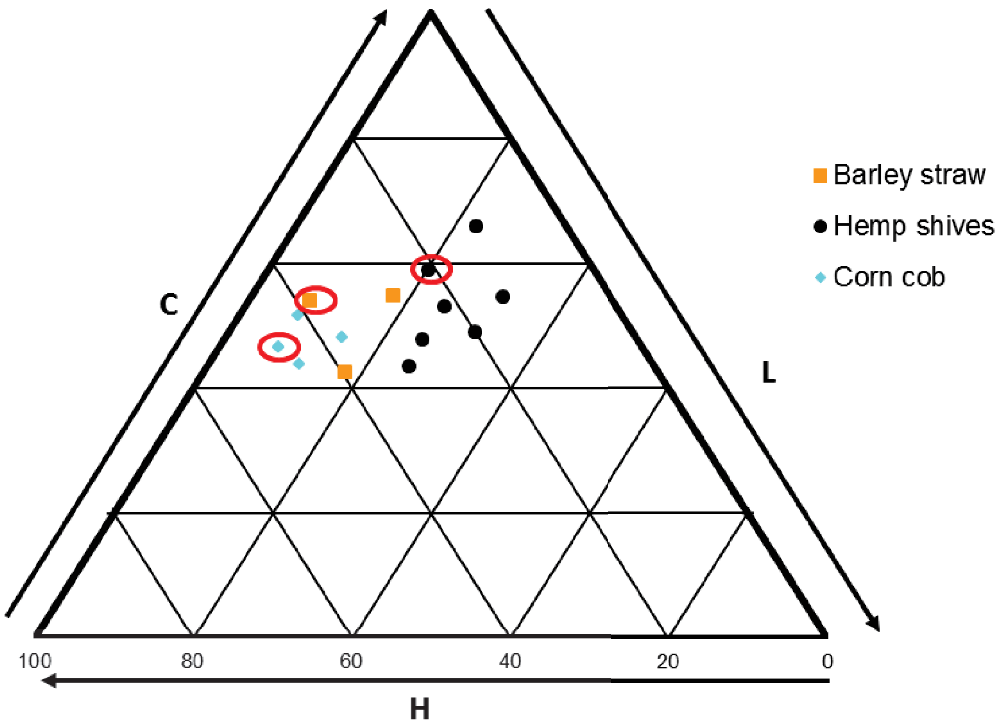


Figure 2-14. Chemical composition of the 3 plant aggregates

All the bioaggregates were rich in cellulose, which several authors link to the mechanical performance of the aggregate [67], [94], [95]. Corn cob was the aggregate presenting the highest hemicellulose content, a component easily dissolved by alkaline attack. It is thus the least usable in an alkaline mineral matrix. The lignin content was higher for hemp shiv. This aggregate could thus be the most durable of the three types.

Values for a type of aggregate are quite scattered. These variations may be due to the measurement method used but may also be connected with differences in the maturity of the stems, the season of harvest, the variety or, in the case of hemp, the retting process [96].

3.4. Sorption-desorption isotherms (DVS)

Figure 2-15 presents the sorption-desorption isotherms of the 3 plant aggregates.

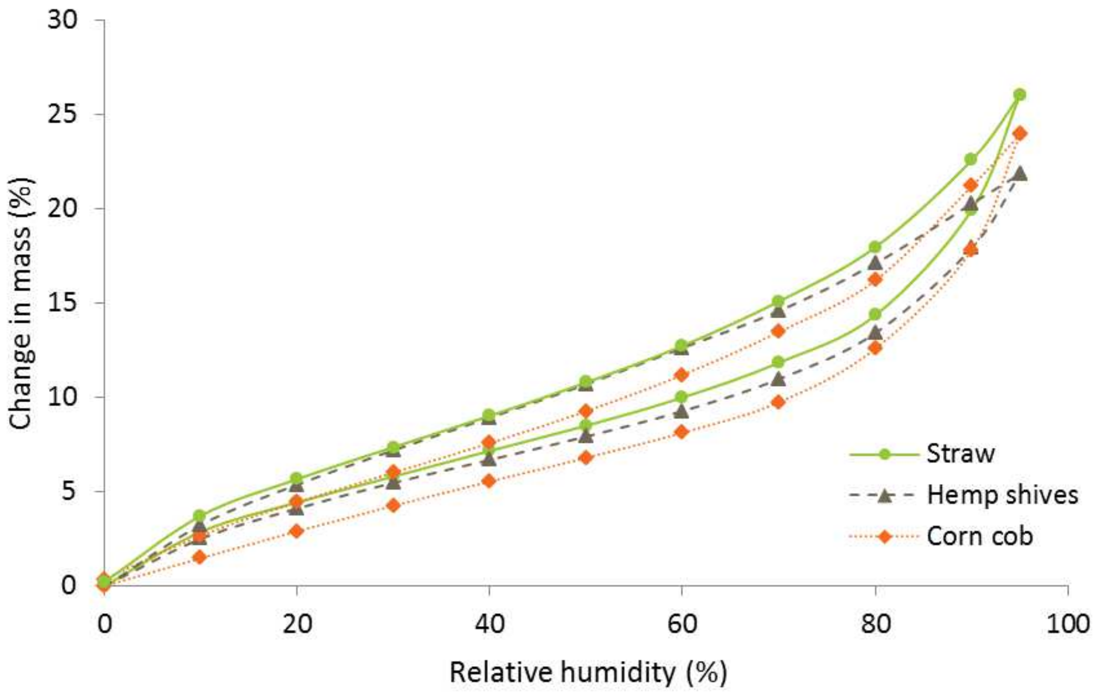


Figure 2-15. DVS water vapour sorption isotherm of the 3 plant aggregates at 23°C

The shape of the sorption curves is similar for all the particles and corresponds to a sigmoidal isotherm, Type II according to the IUPAC (International Union of Pure and Applied Chemistry) classification. This result is very common for cellulosic and lignocellulosic materials [34]. Although the results are similar for the three aggregate types, straw has a slightly higher sorption value at high relative humidity (but below 90% of relative humidity), followed by hemp shiv and corn cob.

Hysteresis (Figure 2-16) represents the gap between the sorption and desorption isotherms (moisture content in desorption is higher than in sorption). This phenomenon is commonly explained by capillary condensation, the ink-bottle effect and the contact angle difference between adsorption and desorption [97], [98]. This phenomenon also seems to be influenced by the lignin content of the natural fibres [98]. The hysteresis values, ranging from 0.1 to 3.8%, increase with relative humidity except for the last 1 or 2 steps. They are comparable for the three plant particles.

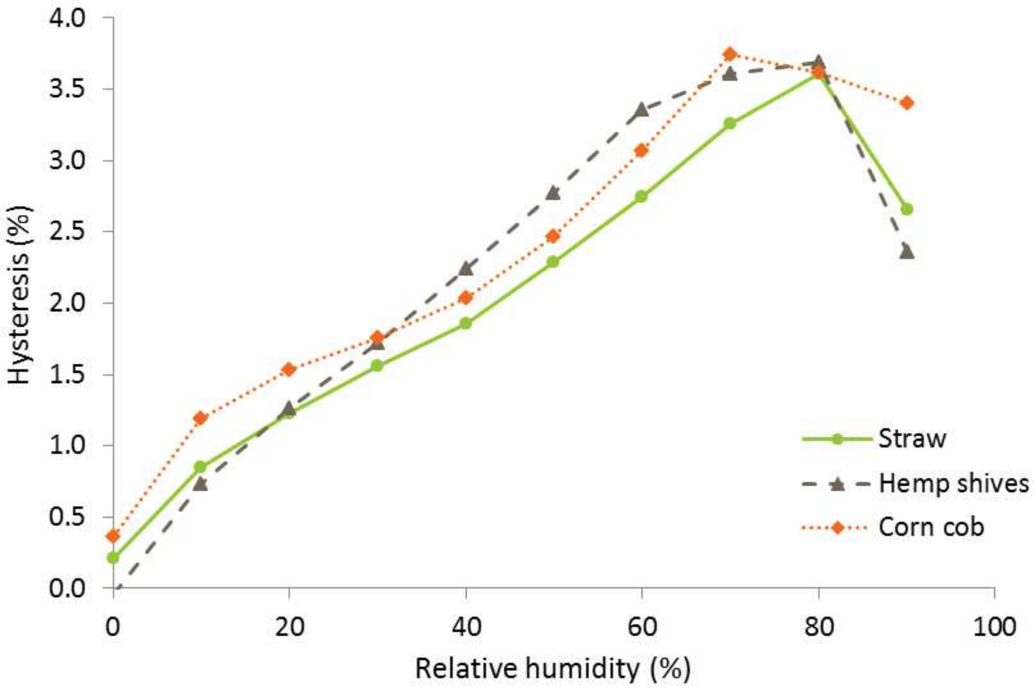


Figure 2-16. Hysteresis calculated from the sorption and desorption isotherms of the 3 plant aggregates at 23°C

These plant aggregates, whose sorption values reach 20 to 26% at 95% of relative humidity, could be used as aggregates in a matrix to increase the sorption capacity of the material. A high sorption capacity could induce good moisture buffering if the kinetics of sorption-desorption proved to be fast. This would improve the balance of the indoor air humidity, making it more comfortable for the occupant [99]. Complementary tests to study the kinetics of the sorption and desorption of each materials would be necessary to conclude on the subject of moisture buffering. This improvement has already been studied by Ashour et al. [100] with straw added to an earth plaster. The sorption capacity improved from 1.7% for earth alone to 6.5% with the addition of 75% of straw by volume.

At 80% RH, the sorption capacity of a lime plaster studied by Černý et al. 2006 [101] was lower than 1% and those of pozzolanic lime plasters were up to 4%. The sorption capacity of the plant aggregates of this study was between 12 and 14%. Their addition to that kind of mineral matrix could increase the sorption capacity of the composite material.

4. Conclusion

In this paper, three potential resources for bio-based building materials were characterized: barley straw, hemp shiv and corn cob. Their availability in France, physical properties, chemical composition and hygric properties were investigated. This overall methodology is mostly adapted from the RILEM method for characterizing bio-based aggregates. Although the round robin test was performed for hemp shiv, it can be adapted to other plant particles:

the mass of the samples should be modified according to the bulk density of the particle type. However, image analysis may be less useful in the case of spherical particles, such as corn cob, which can be characterized by the faster sieving method, as is done for mineral aggregate. The study of the availability of plants was deliberately restricted to France, as the use of local resources allows the environmental impact to be limited. Nevertheless, this kind of investigation should be carried out to evaluate the potential each time an agro-resource is considered for the development of a new bio-based material.

The main results are summarized below:

- Barley straw is a resource having good availability, with more than 4 million tons available each year. Corn cob and hemp shiv also present an interesting potential with 240000 and 17000 tons of by-products available each year.
- The microstructure of these three aggregates is composed of tubular pores, but with a different cell wall thickness for each type.
- Bulk density, highly dependent on these microstructures, is around 60, 150 and 500 $kg.m^{-3}$ respectively for the straw, hemp shiv and corn cob.
- Thermal conductivity is, like bulk density, the lowest for straw, followed by hemp shiv and then corn cob.
- The morphology of the particles is quite similar for straw and hemp shiv (the straw being slightly more elongated) whereas corn cob aggregate is more spherical. That property could lead to different qualities of adhesion in case of use in a matrix.
- Water absorption is very high for straw and hemp shiv (414% and 380% respectively after 48h of immersion) but it is much lower for corn cob, with only 123% of water absorption.
- The chemical composition of the bioaggregates is rich in cellulose. Corn cob is rich in hemicellulose, hemp shiv has the highest lignin content (17%) and straw presents the highest extractive and ash content (around 25%).
- Sorption-desorption isotherms are similar for the three types of particles.

Some conclusions can thus be drawn for each particle type. Barley straw is the most available agro-resource of this study and the best thermal insulator thanks to its low bulk density. A major drawback is its high water absorption which would be prejudicial in case of introduction of this straw into a pozzolanic or hydraulic matrix. It would induce the need for a surplus of water leading to a longer drying time. However, there are some possibilities for treating the particles to reduce this absorption and avoid its negative effects on adhesion or setting time. Straw also presents the highest aspect ratio, which seems to be a positive factor in terms of mechanical strength in an earth matrix [53]. Hemp shiv appears to be the most suitable plant aggregate for use within a hydraulic matrix thanks to its lowest hemicellulose and extractive contents. The thermal conductivity of both straw and hemp shiv is compatible with their use as loose-fill insulation. As far as corn cob is concerned, its use might be interesting because of its low water absorption, which would avoid the earth to be expanded. However, its high hemicellulose content, sensitive to alkaline attack, limits its use in a pozzolanic or hydraulic matrix for example.

All three plant aggregates could be used in an earth matrix, where there is no risk of interaction. The high vapour sorption capacity of these lignocellulosic materials could be useful to improve the capacity of earth or mineral matrices to store moisture. Concerning morphological parameters, it has been shown that mechanical strength is higher when coarser particles are incorporated [52]. However, complementary studies will be necessary to correlate morphological parameters or size distribution of the plant particles with the performances of the composites. These bioaggregates show promise for the development of bio-based building materials if they are used in an appropriate matrix. More research is needed in order to study other parameters such as mechanical strength, fire resistance or microbial growth resistance. Other available resources should also be investigated to develop new building materials, with beetroot for instance.

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Concluding remarks

This chapter presents the availability of the agro-resources and forestry by-products in France, with regard to data extracted from the literature. This study was done with the objective of valorizing the use of bio-resources in buildings materials. The three raw materials chosen (namely barley straw, hemp shiv and corn cob) were particularly investigated. Most of the lignocellulosic resources available come from stems: barley straw is the part of cereal stems rejected during the harvest and hemp shiv is the by-product of the hemp defibration process and corresponds to the lignin-rich part of the stem. This is not the case for corn cob, however, which is the central part of the ear of maize, cleared of grain and crushed. The various characteristics of these three lignocellulosic resources are then studied through experimental tests. To determine the physical properties of the plant aggregates, the RILEM methodology (Amziane et al., 2017) could also be applied to straw and corn cob to some extent, but some modifications had to be made with respect to the hemp shiv methodology. For example, their bulk densities being quite different, the mass of the test specimens was adapted.

The results found in this chapter are summarized in Table 2-7, which highlights the main differences between the three plant aggregates.

Table 2-7. Summary of the main results on the three lignocellulosic resources

Aggregate	Availability (Mt/y)	AR	Bulk density ($kg.m^{-3}$)	Thermal conductivity ($W.m^{-1}.K^{-1}$)	Water absorption (%)	Lignin-cellulose-hemicellulose (%)	Sorption capacity 50-95%
Barley straw	4.28	4.1	57	0.044	414	5.5-37.7-26.7	8.5-26.0
Hemp shiv	< 0.24	3.3	153	0.051	380	17.2-50.3-17.9	7.9-21.8
Corn cob	< 0.017	1.4	497	0.096	123	6.6-41.4-40.7	6.8-23.9

In France, barley straw is much more readily available than hemp shiv and corn cob. Straw (from whatever cereal) is a lignocellulosic resource with huge potential for valorization. Hemp and corn are also produced in great quantities and could provide a significant amount of by-products. Concerning physical properties, barley straw has the lowest bulk density and thus the lowest thermal conductivity. The other two particle types also present interesting properties with respect to thermal insulation. The sorption capacities of these plant aggregates are similar and quite significant. Consequently, moisture storage might be important in a composite material containing these aggregates. The hygrothermal properties of the bio-based composites studied will be discussed later in Chapter 4. The use of the particles in specified matrices is also discussed, in particular because of the water absorption and the chemical composition of the lignocellulosic resources. The three types of particles seem eligible for addition into an earth matrix. Concerning the geometry of the plant aggregates, the aspect ratio (AR) is very different between corn cob and the other two particles. Corn cob particles have a rounded shape whereas barley straw and hemp shiv are more elongated. The shape and particle size distribution of the aggregates might influence mechanical properties; this will be investigated in the next chapter. The high water absorption of these materials may also have a negative effect on the particle adhesion with the matrix and thus on the mechanical properties of the composite. A treatment intended to improve the adhesion of the bio-aggregate with the matrix is presented in the following chapter.

Chapter 3

Use properties: mechanical and hygrothermal performances

Preamble

This third chapter focuses on the use properties of various earth-based composites. It is composed of two articles. The three agro-resources previously characterized, namely barley straw, hemp shiv and corn cob, are introduced into an earth matrix in proportions of 0, 3 and 6% by weight. Here, the three plant aggregates are compared concerning their influence on mechanical properties (Article C) or on hygrothermal properties (Article D). Chapter 2 has shown the diversity of the plant aggregates used. Their shape, in particular, can influence mechanical properties, as already shown by (Danso et al., 2015). Moreover, their porosity described from the SEM images, their bulk density or their sorption-desorption capacity can influence hygrothermal properties.

Article C, about mechanical properties, was accepted in Journal of Materials in Civil Engineering in June 2017. As it is the first chapter of this thesis using earth, this article characterizes the material through its particle size distribution, its Atterberg limits and its chemical and mineralogical composition. One of the main advantages of adding plant aggregates to an earth matrix is that they improve the ductility of the composite (Laborel-Préneron et al., 2016). The objective of this paper is thus to quantify the decrease or increase in compressive or flexural strength and the influence on ductility. Two manufacturing processes are used in this study. The specimens intended for compressive strength tests are compressed while those for the flexural tests are extruded. The extrusion technique makes it easier to manufacture the parallelepipedic specimens needed for testing by flexion. Moreover, its efficiency means it is also an interesting manufacturing process for an industrialized application.

The first part of the article deals with the compressive strength. In the literature review (Chapter 1), no consensus could be found about the influence of a bio-aggregate on compressive strength. Performance depended on many parameters, such as the methodology used, the plant aggregate contents and the adhesion between the aggregates and the matrix. Three agro-resources are tested here. The influence of their shape on the mechanical strength will thus be discussed. Moreover, two protocols are tested to evaluate the influence of the friction between the material and the press. To avoid confinement and reduce friction, Teflon capping is added between the material and the press.

The second part concerns the flexural strength of the extruded specimens. Only straw and hemp shiv, which are elongated, are studied in flexural tests as they are of greater interest than corn cob, too round, which showed poor behavior during the compressive tests. Two lengths of straw are also compared to investigate the influence of the aspect ratio on flexural strength. As seen in Chapter 1, the treatment of the bio-aggregates used in an earth matrix improve some of their properties, notably the adhesion with the matrix. Two surfactants used as plant aggregate treatments are thus tested for this purpose. The behavior of the composites is described through measurements of the apparent stiffness and also the energy absorbed during the test.

Article D, about hygrothermal properties, was submitted to Energy and Buildings in July 2017. As seen in Chapter 1, the other main advantage of adding plant aggregates to an earth matrix is to lighten the material in order to make it more thermally insulating. Several studies have investigated the effects of the addition of plant aggregates and fibers on thermal conductivity. However, few data exist in the literature concerning hygric properties. The objective of this article is to determine the benefits and possible disadvantages of the addition of barley straw, hemp shiv and corn cob as far as hygrothermal properties are concerned. Here, the tested samples are manufactured only by compression.

First, a steady-state thermal property is assessed by measuring the thermal conductivity. This appraisal is carried out with a hot plate apparatus. Then the water vapor permeability is evaluated with the wet cup method in order to highlight the effects of capillary condensation in an earth material. In fact, an apparent water vapor permeability is measured, which corresponds to both vapor and liquid transport. The other steady-state hygric property measured is the sorption-desorption isotherms. These represent the capacity of the material to store water vapor at a constant temperature according to a certain relative humidity. Measurements are made on bulk materials alone and on monolithic samples with two methods. The well-known and standardized method of saturated salt solutions has the main advantage of allowing a large number of samples to be tested at once. However, the duration of the test can be quite long depending on the size of the samples. A more recent method is also used, which involves apparatus called the Dynamic Vapor Sorption (DVS) device. Before being developed for building materials, this method was mainly used in the pharmaceutical field and the agri-food industry at the beginning. A single test is very much faster than a test with the saturated salt solution method but only one very small sample can be tested during one run. The whole cycle of sorption and desorption is fully automated, from the change of relative humidity to the weighing. The results obtained with the two methods will be compared and discussed. Finally, a dynamic hygric property is determined. The theoretical Moisture Buffer Value, called MBV_{ideal}, is calculated from the water vapor permeability and the sorption-desorption isotherms measured. It represents the ability of the material to regulate the moisture when relative humidity fluctuates.

Article C

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Effect of plant aggregates on mechanical properties of earth bricks

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Abstract

A building material is mainly characterized by its mechanical performance, which provides proof of its quality. However, the measurement of the compressive or flexural strength of an earth-based material with plant aggregates, which is very ductile, is not fully standardised. The objective of this study is to determine the compressive and flexural strengths of a composite made of earth and 0%, 3% or 6% of barley straw, hemp shiv or corn cob. Given the manufacturing processes available, cylindrical compressed specimens were studied in compression whereas extruded specimens were studied in flexion. Two protocols were tested for compressive strength measurements: one with direct contact between the specimen and the press, and the other with reduced friction. The test with reduced friction engendered a huge decrease of the stress and a slight decrease of the strain. For both compressive and flexural strengths, the specimens made of earth alone were the most resistant, followed by composites containing straw. The influence of two different treatments applied to the straw is also discussed.

Keywords

Mechanical properties, earth blocks, straw, hemp shiv, corn cob, extrusion

1. Introduction

The building sector is currently innovating in order to use more environmentally friendly materials and to ensure the comfort of users. To this end, it is developing new ecological materials (such as lightweight concrete (Chabannes et al., 2014; Magniont, 2010), or concrete using wastes (Palankar et al., 2015)) but it is also looking into older, traditional ways, focusing on materials such as earth, stone or wood.

Nowadays, around 30% of the world's population still lives in earth shelters, especially in developing countries (Minke, 2006). Earth is a local resource that is available in abundance and presents many other advantages. This material has low environmental impact because of its recyclability, the little energy needed for the transformation process, the minimal transport required and its energy efficiency. Moreover, it is able to regulate indoor moisture and to improve the comfort of the building's users (Islam and Iwashita, 2006; Minke, 2006).

However, earth material presents some weaknesses, such as low mechanical strength, brittleness, hygroscopic shrinkage and limited durability with respect to water (Aymerich et al., 2012; Islam and Iwashita, 2006). In order to reduce these drawbacks, some authors have studied the effect of adding stabilizers such as hydraulic binders and artificial or natural fibres or aggregates (Danso et al., 2015a; Laborel-Préneron et al., 2016). The enhancement of soil blocks by stabilizers was reviewed by Danso et al. (Danso et al., 2015a), especially concerning mechanical and water absorption properties. The interest of adding plant aggregates was also highlighted by Laborel-Préneron et al. (Laborel-Préneron et al., 2016). Based on empirical knowledge, the use of natural fibres and excrement has always helped to improve the properties of earth for building (Chazelles et al., 2011; Millogo et al., 2016). Such additions are now being increasingly studied within an earth matrix because of their apparently huge potential to improve thermal insulation (Bal et al., 2013) and ductility (Mostafa and Uddin, 2015) among other properties.

The present paper deals only with the mechanical properties of earth blocks containing plant aggregates. These properties are indeed essential if the material under study is to be used for construction purposes. They will determine whether it can be used in a load bearing structure or not. However, the mechanical requirements vary from one standard to another, as do the testing procedures, which makes the characterization of this kind of material difficult. In the literature, many studies focus on the influence of plant fibres or aggregates on compressive strength. Twenty-three references investigating compressive strength on this kind of materials are cited in (Laborel-Préneron et al., 2016). Several studies have observed an increase of compressive strength with increasing proportions of plant aggregates such as tea residue (Demir, 2006), sawdust, tobacco residue or grass (Demir, 2008) or cassava peel (Villamizar et al., 2012). However, others have found a decrease in strength: Algin et al. showed a 71% compressive strength decrease with the addition of 7% of cotton waste (Algin and Turgut, 2008), and a decrease was also observed with straw (Mohamed, 2013) or coconut fibres (Khedari et al., 2005). In some cases, the effect of fibre length was studied. According to Millogo et al. (Millogo et al., 2014, 2015), the compressive strength of the earth composite increased by as much as 16% with the addition of short Hibiscus Cannabinus fibres (3 cm) but decreased with long ones (6 cm), except for a content of 0.4%. An influence of the aspect ratio was also observed by Danso et al. (Danso et al., 2015b) for coconut, bagasse and oil palm fibre, but with an increase of compressive strength as the length of the added fibres increased. None of these studies on earth material with plant aggregates deal with the influence of the testing protocol. However, Morel et al. (Morel et al., 2007) reviewed the various existing protocols for compressive strength testing of blocks of earth alone. Aubert et al. (Aubert et al., 2013, 2015) have discussed the testing of extruded earth blocks, considering the influence of: aspect ratio, confinement (capping with Teflon or not), anisotropy and the mortar joint between two half blocks, on the compressive strength measurement.

Several references focus on the flexural strength of these materials. An increase in flexural strength is observed in most of the studies with an addition of plant aggregates, e.g. Bouhicha et al. (Bouhicha et al., 2005) with barley straw or Aymerich et al. (Aymerich et al., 2012) with wool fibres, but others have observed a decrease, e.g. Villamizar et al. with cassava peels (Villamizar et al., 2012). In all cases, ductility is greatly improved, as the fibre bridging of microcracks prevents them from expanding (Galán-Marín et al., 2010; Mattone, 2005; Mostafa and Uddin, 2015; Segetin et al., 2007).

A few, relatively recent, works have investigated the energy absorbed and the mode of failure (Aymerich et al., 2016; Islam and Iwashita, 2006; Lenci et al., 2012; Martins et al., 2014). In flexion, failure usually occurs by fibre gradually slipping from the matrix, leading to both pull out and breaking of the fibres (Mostafa and Uddin, 2015). Some authors have treated the fibres in an attempt to improve the adhesion between the fibre and the matrix and thus enhance the flexural or tensile strength. Some encouraging results have been obtained, notably with acetylation, depending on the temperature of the chemical reaction (Hill et al., 1998), or alkaline treatments on sisal fibres (Alvarez and Vázquez, 2006) or banana fibres (Mostafa and Uddin, 2015). However, a linseed oil treatment used by (Ledhem et al., 2000) on wood shavings gave less promising results, with a decrease in strength, especially in traction.

The material studied in this paper is a bio-composite composed of earth and three different plant aggregates: barley straw, hemp shiv and corn cob. Composite specimens were manufactured according two processes: compression or extrusion. After characterization of the earth used, the influence of plant aggregate content on compressive strength and flexural strength was analysed. Compressed specimens were tested in compression following two protocols, one with friction and the other using a system to reduce friction. The extruded specimens were tested in flexion. The effects of various treatments on barley straw and the effect of its aspect ratio were investigated with this test. The fracture energy developed during the test was also calculated.

2. Materials and methods

2.1. Raw materials

2.1.1. Earth

Earth used in this study was composed of quarry fines from aggregate washing processes (FWAS). These fines, smaller than 0.1 mm, were generated by the washing of limestone aggregates produced for the chemical or concrete industry. The sludge created was left to dry in sedimentation basins and was then reduced to powder to be used in different applications.

2.1.2. Plant aggregates

Three types of plant aggregates were used in this study: barley straw (two different lengths), hemp shiv and corn cob. Barley Straw (S) is the part of cereal's stem rejected during the harvest. Hemp shiv (H) is the by-product of the hemp defibration process and corresponds to the lignin-rich part of the stem. Corn Cob (CC) is the central part of the ear of corn cleared of grain and crushed. The hardest part was studied here. The physical and chemical characteristics of these plant aggregates were determined in (Laborel-Préneron et al., 2017) and are recapitulated in Table 3-1. However, the previous characterization was done only for the shortest straw (S_{short}), of average length 8 mm. In the present study, the longest straw (S_{long}), of average length 15 mm, was used only for the flexural strength test.

Table 3-1. Physicochemical properties of the plant aggregates

Material	**Barley straw**	**Hemp shiv**	**Corn cob**
Designation	S_{short}	H	CC
Bulk density (kg.m^{-3})	57.4 ± 1.2	153.0 ± 2.4	496.8 ± 14.0
Water absorption (%)	414 ± 4	380 ± 11	123 ± 2
Diameter* (mm)	2.33 ± 1.52	2.02 ± 1.23	2.63 ± 0.43
Thermal conductivity (W.m^{-1}.K^{-1})	0.046 ± 0.001	0.053 ± 0.002	0.097 ± 0.001
Chemical composition			
Lignin (%)	5.5	17.2	6.6
Cellulose (%)	37.7	50.3	41.4
Hemicellulose (%)	26.7	17.9	40.7

* Corresponding to average minor axis by image analysis

2.1.3. Surfactant additives

In agriculture, the efficiency of crop protection products can be improved by the addition of surfactants that increase the absorption of the product on the plants. These additives can optimize spreading and reduce negative effects such as drift and run off. In this study, the objective of using these surfactants was to increase the adhesion between the plant aggregates and the earth matrix by reducing the surface tension of the mixing water. Two types of surfactant additives were tested: A1, which was soya lecithin based, and A2, which was latex based. They were both applied to the long straw only.

To apply the surfactants to the straw, the particles were immersed for 1 hour in the additive, diluted at the rate recommended by the supplier: 0.5% of the water volume for A1 and 0.1% for A2. The straw was then sieved to remove extra water before being dried at 30°C in an oven.

2.2. Physical, chemical and mineralogical characterization of FWAS

2.2.1. Particle size distribution and Atterberg limits

The size distribution was determined by sedimentation after wet sieving at 80 µm, according to standard NF P94-057 (AFNOR, 1992). The geotechnical characteristics were evaluated using the Atterberg limits, according to standard NF P 94-051 (AFNOR, 1993).

2.2.2. Chemical and mineralogical composition

X-ray diffraction on a sample crushed to a size of less than 80 µm was carried out with a Siemens D5000 powder X-ray diffractometer equipped with a monochromator having a Ka (λ=1.789Å) cobalt anticathode. Thermal mineralogical characterization was also performed by thermal gravimetric analysis (TGA) of a crushed sample (< 80 µm) heated to 1050°C at a constant rate of $10°C.min^{-1}$. Major oxide composition was evaluated on the basis of macroelemental analysis performed by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) on crushed samples smaller than 80 µm. The mineral composition was determined from X-ray diffraction results and the chemical composition.

2.3. Manufacturing process

Specimens for the different tests were prepared by two manufacturing processes: one using static compression and the other using extrusion. The proportion of plant aggregates was expressed by dry weight content, according to formula (1):

$$Ag.content = \frac{m_{Ag}}{m_{Ag} + m_{FWAS}} \times 100 \quad (1)$$

where Ag. content is the plant aggregate content in %, m_{Ag} is the dry mass of plant aggregates and m_{FWAS} is the dry mass of earth.

2.3.1. Compressed specimens

Various mixtures were prepared to make the specimens: FWAS only and mixtures containing one of the plant aggregates in a proportion of 3% or 6%. The water contents of the mixtures were determined by the Proctor test, and then rounded up because, according to Minke (Minke, 2006), this is a minimum value for manufacturing compressed earth bricks. Table 3-2 recapitulates the different mixture proportions and the dry densities of the specimens obtained (average of six specimens). As expected, the water content of the dry mass needed to make the mixtures increased when the plant aggregate content increased. It was higher for straw than for hemp and corn cob because straw particles have a higher water absorption coefficient than the other two aggregates (414% vs. 380% for hemp and 123% for corn cob) (Laborel-Préneron et al., 2017).

Table 3-2. Mixture proportions and Proctor density of compressed specimens

Reference	FWAS	S3	S6	H3	H6	CC3	CC6
Plant aggregate	-	Short straw	Short straw	Hemp	Hemp	Corn cob	Corn cob
Plant aggregate content (%)	0	3	6	3	6	3	6
Water content (%)	14	19	21	17	20	16	16
Dry density ($kg.m^{-3}$)	1988 ± 9	1520 ± 1	1195 ± 169	1553 ± 69	1190 ± 44	1877 ± 2	1704 ± 71

To manufacture the specimens, the earth and plant aggregate fractions were poured into a blender and mixed by hand. Then, water was added and the materials were mixed mechanically until a homogeneous mix was obtained. The raw materials were mixed the day before moulding.

Cylindrical specimens 5 cm in diameter and 5 cm high (Φ5H5), intended for compressive strength tests, were manufactured by double static compression at the Proctor density. After demoulding, the height of the specimens containing barley straw and hemp shiv increased significantly due to the high compressibility of the plant aggregates. This increase reached 10% of the height for an addition of 6% of hemp shiv, for example. This expansion led to the formation of distributed cracks, specifically in the case of an addition of 6% of straw (Figure 3-1). The specimens were first dried at 40°C for 24 hours, then the temperature was increased by 0.1°C/min to 100°C and kept at 100°C until the weight became constant (weight variation less than 0.1% between two weighings 24 hours apart). This rise in temperature was carried out slowly to keep shrinking homogeneous and to avoid mechanical stresses. The specimens were then stored in a room regulated at 20°C and 50% relative humidity (RH) and were tested when they were in equilibrium with the environment (about one week later).

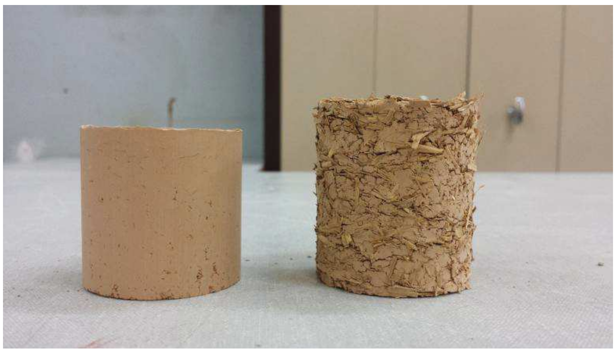

Figure 3-1. Compressed specimens of FWAS (left) and S6 (right)

2.3.2. Extruded specimens

Six types of specimens were prepared: specimens made with FWAS only (i), and specimens made with 3% of short straw (ii), long straw (iii), hemp shiv (iv), long straw treated with the A1 surfactant (v), or long straw treated with the A2 surfactant (vi). Corn cob was not tested in extrusion because of the poor distribution of the particles observed in a preliminary trial and its low strength in compression. To manufacture these specimens, earth and plant aggregate fractions were poured into a blender and were mixed by hand. Then, the materials were mixed mechanically in the blender and water was added progressively until the consistency of the mixture was sufficiently homogeneous and plastic to be extruded. The details of the mixes are recapitulated in Table 3-3.

Table 3-3. Mixture proportions of extruded specimens

Reference	FWAS	$S3_{short}$	$S3_{long}$	H3	S_{A1}	S_{A2}
Plant aggregate	-	Short straw	Long straw	Hemp	Long straw	Long straw
Plant aggregate content (%)	0	3	3	3	3	3
Water content (%)	20	24	26	25	24	25
Surfactant	-	-	-	-	A1	A2
Dry density ($kg.m^{-3}$)	1982 ± 8	1781 ± 10	1734 ± 20	1712 ± 11	1784 ± 10	1782 ± 12

The specimens were manufactured with a medium sized laboratory extruder. The mixture was extruded under vacuum through a 7 cm x 3.5 cm die (Figure 3-2). The specimens were difficult to cut in the fresh state because of the presence of plant particles. They were therefore air-dried until the weight become constant (weight variation of less than 0.1% between two weighings 24 hours apart) and then cut to a length of 18 cm with a circular saw. Treatment of the straw did not modify the dry density of the composites. However, when the two manufacturing processes were compared (Table 3-2 and Table 3-3), an increase of density was observed for the extruded specimens containing plant aggregates. It was due to the extrusion of the material under vacuum, which reduced porosity, and to the extrusion pressure due to the worm screw.

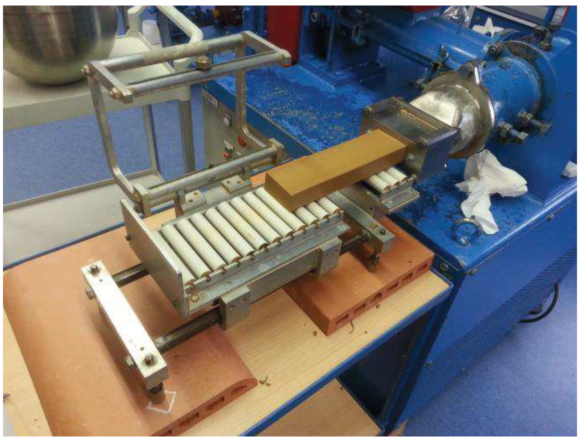

Figure 3-2. Vacuum extruder

2.4. Compressive strength test

The compressive strength tests on the Φ5H5 specimens were performed using a 100 kN capacity hydraulic press. The load was applied at a constant deflection rate of 3 $mm.min^{-1}$. This speed was chosen as an intermediate value between the 1.2 $mm.min^{-1}$ specified in the French standard XP P 13-901 (AFNOR, 2001) (intended for compressed earth blocks) and the 5 $mm.min^{-1}$ used by Cerezo (Cerezo, 2005) (intended for hemp concrete). Three specimens of each mixture were tested in two different tests: one test with the specimen in direct contact with the steel plates (generating friction) and the other including a system avoiding friction (Figure 3-3) as described by Olivier et al. (Olivier et al., 1997). In the latter case, a 2-mm-thick piece of Teflon and a thin neoprene sheet - with a drop of oil between the layers - were placed between the earth specimen and the steel (neoprene in contact with the specimen, and Teflon in contact with the steel). Teflon was used because of its low friction coefficient and neoprene because of its high mechanical resistance. Displacements and loads were measured in each case. The apparent stiffness of each specimen was then calculated from the linear part of the stress-strain curve.

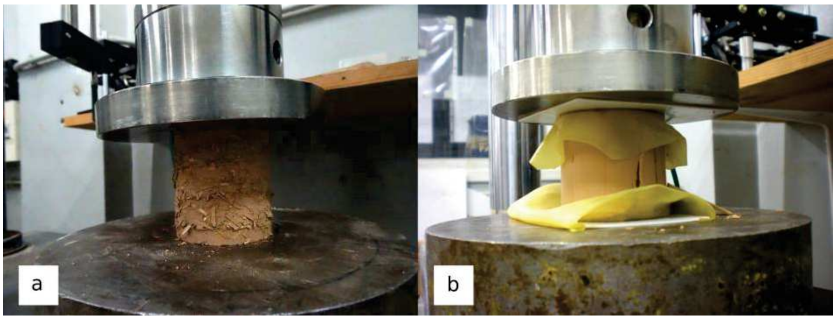

Figure 3-3. Compressive test method: (a) with friction and (b) with reduced friction

2.5. Flexural strength test

The flexural strength tests on the extruded specimens were performed using a 100 kN capacity hydraulic press with a 10 kN sensor. The load was applied at a constant deflection rate of 1 $mm.min^{-1}$ as was done by Aymerich et al. (Aymerich et al., 2012). The samples were loaded under three point loading conditions with the lower supports placed 10 cm apart, corresponding to the value given in the French standard NF EN 196-1 intended for cements (AFNOR, 2006). Measurements were made in triplicate.

This test was carried out in order to study the effect of the plant aggregate addition on ductility. According to the literature, this kind of addition has a marked effect on ductility (Aymerich et al., 2012; Bouhicha et al., 2005; Galán-Marín et al., 2010; Ghavami et al., 1999). Deflection was measured from bottom to top on an aluminium platelet glued in the middle of the sample, as can be seen on Figure 3-4. The test was stopped for a deflection close to 3 mm, the limit of the sensor stroke.

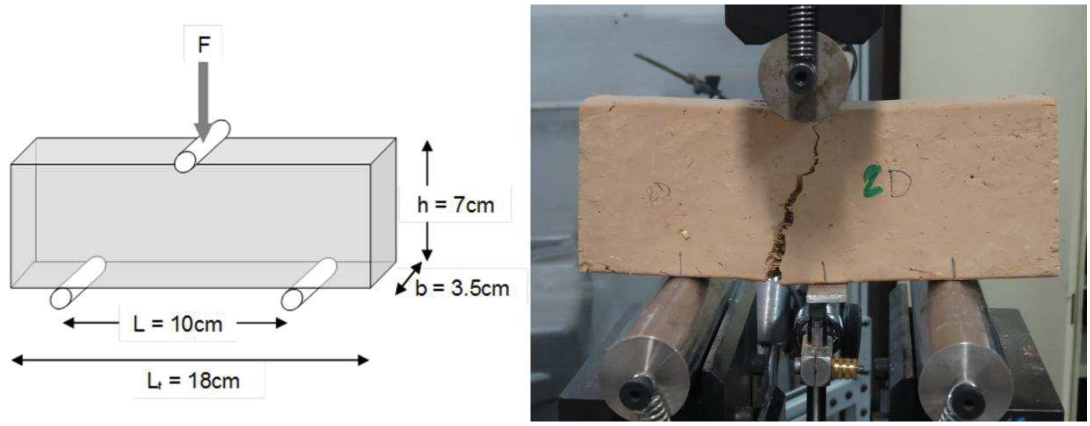

Figure 3-4. Flexural test set up

In order to compare the flexural strength with values reported in the literature or values from standards, the bending stress was calculated only at failure with the beam theory. Considering that classical hypotheses of solid mechanics applied and that the section was not cracked until peak load (elastic part of the curve) (Lenci et al., 2012; Mostafa and Uddin, 2015), the stress σ (MPa) was calculated from the following expression (2):

$$\sigma = \frac{3FL}{2bh^2} \tag{2}$$

with F the maximum load at failure (N), L the distance between the supports (mm), b the width (mm) and h the height (mm) of the sample.

To determine the effect of the plant aggregate on the behaviour at failure and post-peak, the fracture energy (G_f) was calculated. It provided information about the amount of energy absorbed when the specimen was broken into two parts. It was represented by the area under the load-displacement curve divided by the projected fracture area (Guinea et al., 1992). Usually, it is measured on notched samples (Aymerich et al., 2012, 2016; Guinea et al., 1992), so the whole load-displacement curve is considered. However, as the samples did not have a notch, the area was taken into account from the failure point and to a deflection of 3 mm. The fracture energy was calculated from the expression (3):

$$G_f = \frac{\int_{\delta_f}^{\delta_{3mm}} F(\delta)}{S} \tag{3}$$

where δ_f is the deflection at failure (m), F is the load (N) and S is the initial section (m²).

The apparent stiffness of each specimen was then calculated from the linear part of the stress-strain curve.

3. Results and discussion

3.1. Characterization of earth

3.1.1. Particle size distribution and Atterberg limits

The particle size distribution is presented in Figure 3-5 and compared with the size distribution recommended for compressed earth blocks (CEB) in the XP 13-901 standard (AFNOR, 2001).

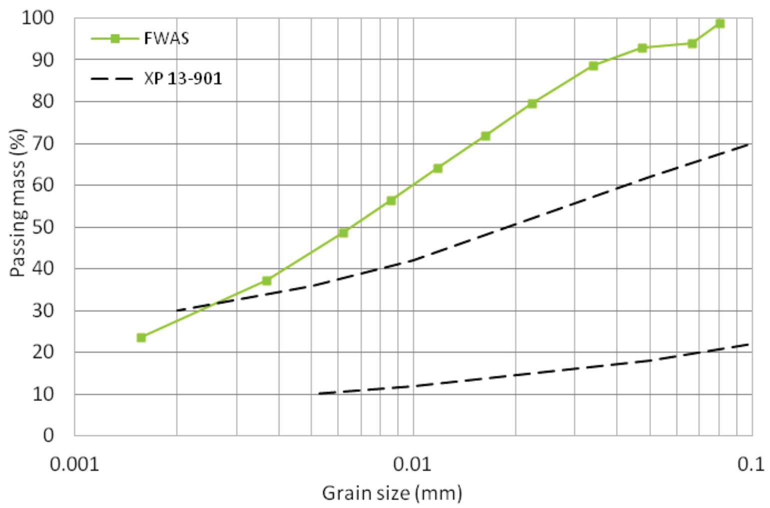

Figure 3-5. Comparative grain size distribution curve for earth: FWAS and standard

The earth was extremely fine: 99% of the particles were smaller than 80 µm and the average particle size (D50) determined using the pipette analysis was 6.5 µm. The curve did not fit the limits recommended by the standard, the passing mass being higher for each grain size. Atterberg limits were equal to 30% for the liquid limit, 21% for the plastic limit and 9% for the plasticity index. The plasticity of this material was not located in the spindle of the diagram recommended by the XP 13-901 standard (AFNOR, 2001). However, even though the size distribution and Atterberg limits did not meet the recommended criteria, it was already shown that it was possible to manufacture CEBs with a huge variety of earths (Aubert et al., 2015; Laborel-Préneron et al., 2016).

3.1.2. Chemical and mineralogical composition

The X-ray diffractogram is presented on Figure 3-6. This diagram reveals the large presence of calcite ($CaCO_3$) and shows the presence of other minerals in smaller quantities: kaolinite ($Al_2Si_2O_5(OH)_4$), quartz (SiO_2), illite ($KAl_2(AlSi_3)O_{10}(OH)_2$), goethite ($FeO(OH)$) and dolomite ($CaMg(CO_3)_2$).

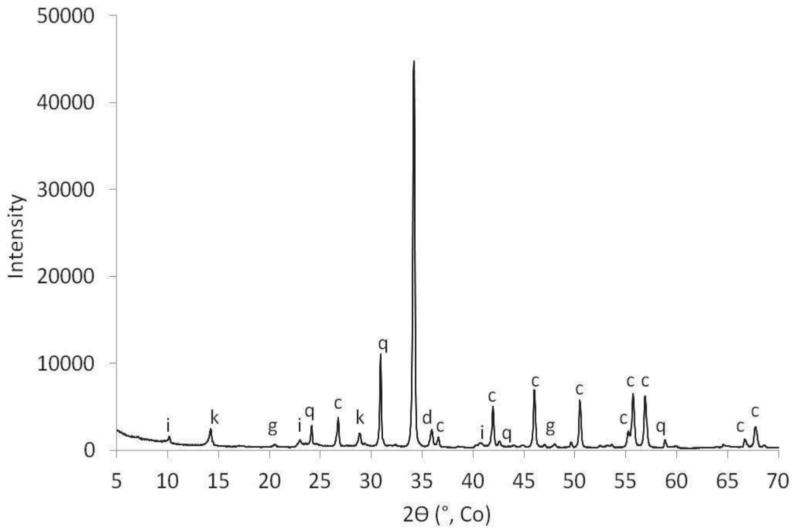

Figure 3-6. X-ray diffraction pattern of the earth. (c) calcite, (d) dolomite, (g) goethite, (i) illite, (k) kaolinite, (q) quartz

To complete its mineralogical characterization, the earth was subjected to thermal gravimetric analysis, the results of which are presented in Figure 3-7. The loss of mass around 105°C was due to the evaporation of water and that around 540°C indicated the removal of the constituting water, which led to the transformation of kaolinite into metakaolinite (Kornmann and Lafaurie, 2005). The greatest loss of weight, of about 25%, occurred around 910°C and was due to the decarbonation of calcite and dolomite.

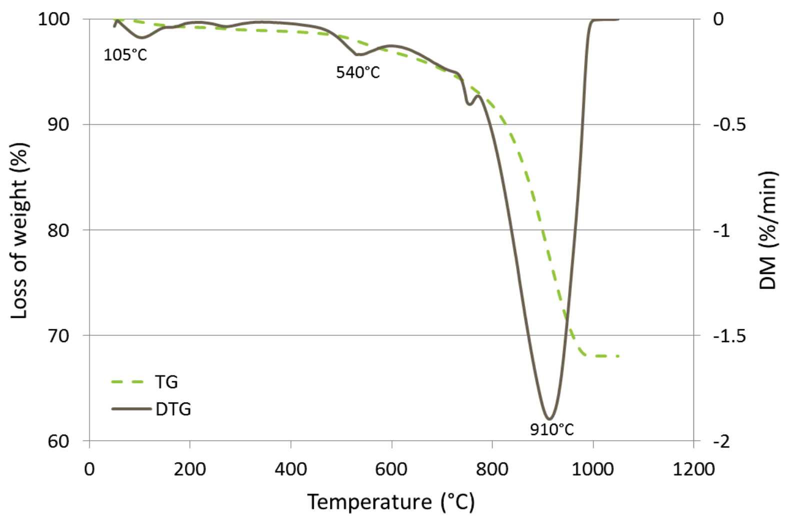

Figure 3-7. TG and DTG (Derivative Thermo-Gravimetric) curves of the earth

The chemical composition of the earth is given in Table 3-4, where a large amount of calcium is noticeable. Using the chemical composition and the mineral characterization, it is possible to estimate the proportion of each mineral. The fines used were thus composed for 60% of calcite, 11% of kaolinite, 11% of illite, 10% of quartz, 6% of dolomite and 3% of goethite.

Table 3-4. Chemical composition of the earth (LOI: Loss on Ignition)

Oxides	SiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O_5	LOI
%	18.73	7.47	2.39	0.03	1.27	35.27	0.09	0.90	0.39	0.09	31.92

3.2. Compressive strength measured on compressed specimens

The average over three specimens of dry density, maximum compressive strength and other mechanical properties of each mixture and for each testing protocol are reported in Table 3-5. The compressive strength value required by the New-Zealand Earth Building standard NZS 4298 (NZS 4298) is 1.3 MPa. This value was reached for all the mixtures of this study, for both protocols, except for CC6 tested with reduced friction (0.9 MPa).

Table 3-5. Measured mechanical properties of the materials: dry density (ρ_{dry}) average compressive strength (σ_c), average ultimate strain (ε_c), average compressive strength at 1.5% strain ($\sigma_{c,1.5\%}$) and average experimental apparent stiffness (E_c)

Testing protocol	Reference	ρ_{dry} (kg.m^{-3})	σ_c (MPa)	ε_c (%)	$\sigma_{c,1.5\%}$ (MPa)	E_c (MPa)
With friction	FWAS	1995 ± 0	4.0 ± 0.4	1.3 ± 0.1	4.0 ± 0.4	439 ± 54
	S3	1519 ± 1	3.3 ± 0.2	7.8 ± 0.6	0.7 ± 0.1	62 ± 3
	S6	1315 ± 27	3.8 ± 0.3	19.9 ± 1.1	0.4 ± 0.0	31 ± 1
	H3	1603 ± 57	2.4 ± 0.2	4.8 ± 0.3	0.7 ± 0.1	75 ± 8
	H6	1221 ± 70	1.8 ± 0.2	10.7 ± 3.1	0.4 ± 0.1	26 ± 3
	CC3	1878 ± 1	3.2 ± 0.2	2.4 ± 0.2	2.1 ± 0.3	217 ± 45
	CC6	1754 ± 13	1.8 ± 0.6	2.5 ± 0.5	1.3 ± 0.6	102 ± 69
Reduced friction (RF)	FWAS	1982 ± 10	3.9 ± 0.9	1.0 ± 0.1	3.9 ± 0.9	564 ± 161
	S3	1520 ± 1	2.1 ± 0.2	5.6 ± 0.5	0.6 ± 0.0	43 ± 5
	S6	1075 ± 30	3.6 ± 0.2	17.2 ± 1.5	0.3 ± 0.0	25 ± 0
	H3	1504 ± 54	1.6 ± 0.1	3.6 ± 0.3	0.7 ± 0.1	51 ± 5
	H6	1159 ± 41	1.5 ± 0.1	7.5 ± 1.1	0.4 ± 0.0	22 ± 1
	CC3	1876 ± 1	1.3 ± 0.1	1.2 ± 0.1	1.3 ± 0.1	136 ± 40
	CC6	1654 ± 53	1.0 ± 0.1	1.8 ± 0.2	0.9 ± 0.0	69 ± 9

3.2.1. Influence of plant aggregate type and content on the bulk density

A number of authors have shown that bulk density is influenced by the addition of plant aggregates (Algin and Turgut, 2008; Demir, 2008; Khedari et al., 2005). The bulk density of each mixture used here is plotted on Figure 3-8. As expected, bulk density decreased as the aggregate content increased for the three kinds of plant aggregates. However, some differences between the mixtures with straw or hemp and the mixture with corn cob can be noted. Bulk density was higher for the mixtures with corn cob than for those with straw or hemp. This difference may have been due to the huge variability of the particle bulk densities: 497 kg.m^{-3} for corn cob against 57 kg.m^{-3} and 153 kg.m^{-3} for straw and hemp shiv, respectively (Laborel-Préneron et al., 2017).

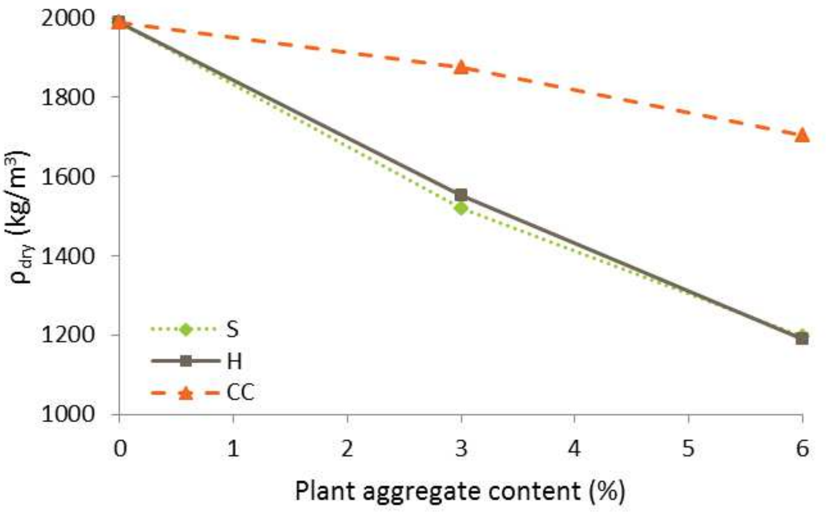

Figure 3-8. Bulk density as a function of the plant aggregate content

3.2.2. Influence of the testing protocol on compressive strength

Usually, only the compressive test with friction is performed ((Galán-Marín et al., 2010; Mohamed, 2013; Villamizar et al., 2012)) and follows standards ASTM D2166 (ASTM D 2166, 2004), XP P 13-901 (AFNOR, 2001) or TS EN 772-1 (TS EN 772-1, 2002). However some authors have also measured the strength with lower friction (using a layer of sand and a transparent film on either side of the sample (Piattoni et al., 2011; Quagliarini and Lenci, 2010) or with Teflon (Aubert et al., 2016)) to obtain the "real" compressive strength. Both methods, with friction and with Teflon reducing friction, were carried out to see what difference was actually observed. The results are shown in Figure 3-9.

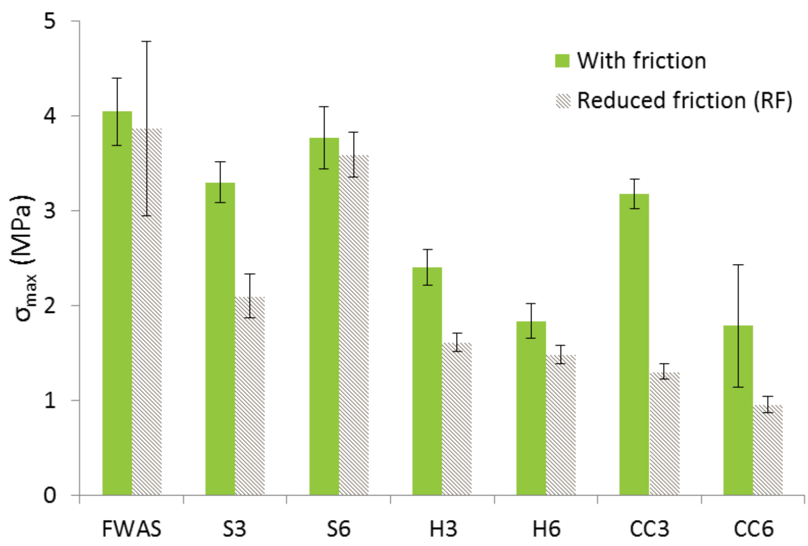

Figure 3-9. Compressive strength of the mixtures according to the testing protocol

For each composition, the compressive strength measured in the tests with friction was greater than that found in tests with reduced friction because of the confinement (transverse displacements not allowed at the ends of the specimens). In the case of earth alone (FWAS) and S6 specimens, the decrease in strength between the protocols was only about 3 to 5%, whereas it reached between 28% (C6) and 59% (CC3) for the other mixtures. In some cases (FWAS or CC6), standard deviation was quite high. In the case of CC6, for example, this large variability of the results was due to one specimen having significantly higher strength than the other two - probably because of the heterogeneity of the material (Aubert et al., 2016), with a poor distribution of the corn cob granules. The results of H3, H6, CC3 and CC6 measured with reduced friction are very close; it is thus difficult to establish the highest strength with this protocol.

Unlike Aubert et al.'s finding (Aubert et al., 2016) that the strength decreased by only 10% with the use of Teflon capping, the choice of the method was observed to significantly affect the strength value measured here. It is thus important to choose the most adequate method. In order to allow comparisons among samples, and with the literature, only the values obtained with friction were kept here. This protocol was also easier to set up and more similar to the behaviour of a brick within a wall, with friction between the bricks.

3.2.3. Effect of the plant aggregates on compressive strength

The compressive strengths of the specimens are summarized in Figure 3-10 for the different plant aggregate types and contents when the protocol with friction at the interface between the specimen and the press was employed.

The compressive strength of the specimen composed of earth alone is higher than that of all the others, which is in accordance with the density values of the various specimens. Its average strength of 4.0 MPa is higher than the typical value for CEBs which is, according to Morel et al. (Morel et al., 2007), between 2 and 3 MPa. Furthermore, a decrease in compressive strength is noticeable when hemp shiv and corn cob contents increase. The values are 2.4 and 1.8 MPa for H3 and H6, and 3.2 and 1.8 MPa for CC3 and CC6 specimens. This reduction, linked to the incorporation of particles with low compressive strength and stiffness, can be correlated to the decrease in bulk density observed with the addition of plant aggregate (Al Rim et al., 1999; Ghavami et al., 1999). In the case of barley straw, the average strengths are 3.3 MPa and 3.8 MPa for S3 and S6 respectively. The ultimate compressive strength of S6 specimens is thus higher than that of S3 specimens. This can be explained by a consolidation phenomenon due to the high compressibility of the straw that allows its porosity to decrease as strain increases. This phenomenon is not observed for H6 or CC6 specimens because of the lower ductility of hemp and corn aggregates. This difference could also be due to the different shapes of the particles, straw being more elongated than hemp shiv (Laborel-Préneron et al., 2017). This kind of result was also observed by Millogo et al. (Millogo et al., 2014) for the longest fibres but for smaller quantities (less than 1%). This observation was explained by the limitation of crack opening by the fibres.

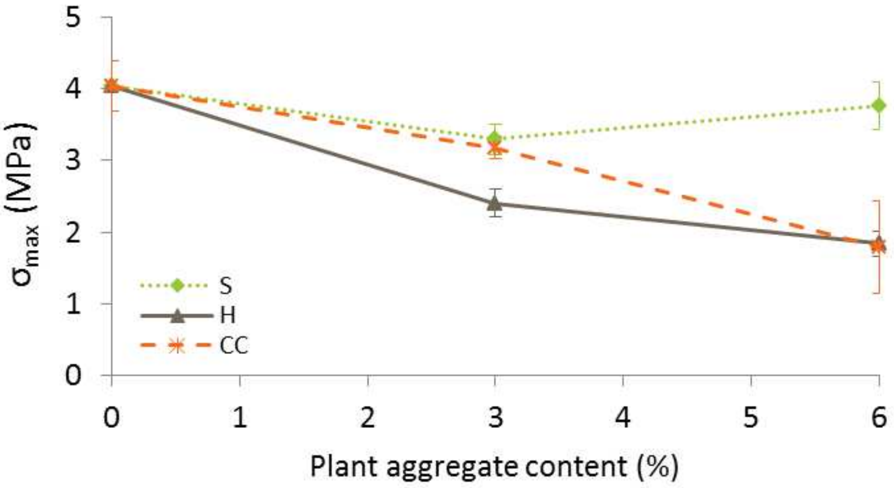

Figure 3-10. Results for compressive strength test with friction

3.2.4. Effect of the plant aggregates on ductility

Figure 3-11 shows the stress-strain curves of all the specimens. It can be noted that FWAS specimens show brittle failure whereas the ultimate strain is high for the specimens containing plant aggregates, especially those with 6%. Their peak strain is, on average, 19.9, 10.7 and 2.5% for S6, H6 and CC6, respectively, whereas it is only 1.3% for FWAS. Although these specimens are weaker than FWAS specimens, they are also more ductile, with a larger zone of plasticity. Ductility of the composite is thus increased by the addition of plant aggregates. However, in calculating building structures, such deformations of the material cannot be tolerated.

In order to make comparisons among the materials and to maintain a strain level compatible with the intended use, we chose to limit the strain to 1.5% and to keep the corresponding compressive strength value, as described by Cerezo (Cerezo, 2005) for hemp concrete. The maximum compressive strength was kept in cases when the failure occurred before 1.5% strain (which only concerned FWAS specimens).

These values are compared with the values at failure in Figure 3-12. For a given deformation, compressive strength is higher for FWAS specimens. The values are far below the maximum compressive strength and do not reach 1 MPa for the specimens with straw or hemp shiv whereas the compressive strength is above the limit of 1.3 MPa in the case of corn cob. In the cases where the materials do not have the strength required to be used as bearing structures, they can be used as infill material in a wood structure or as a partition wall, for instance.

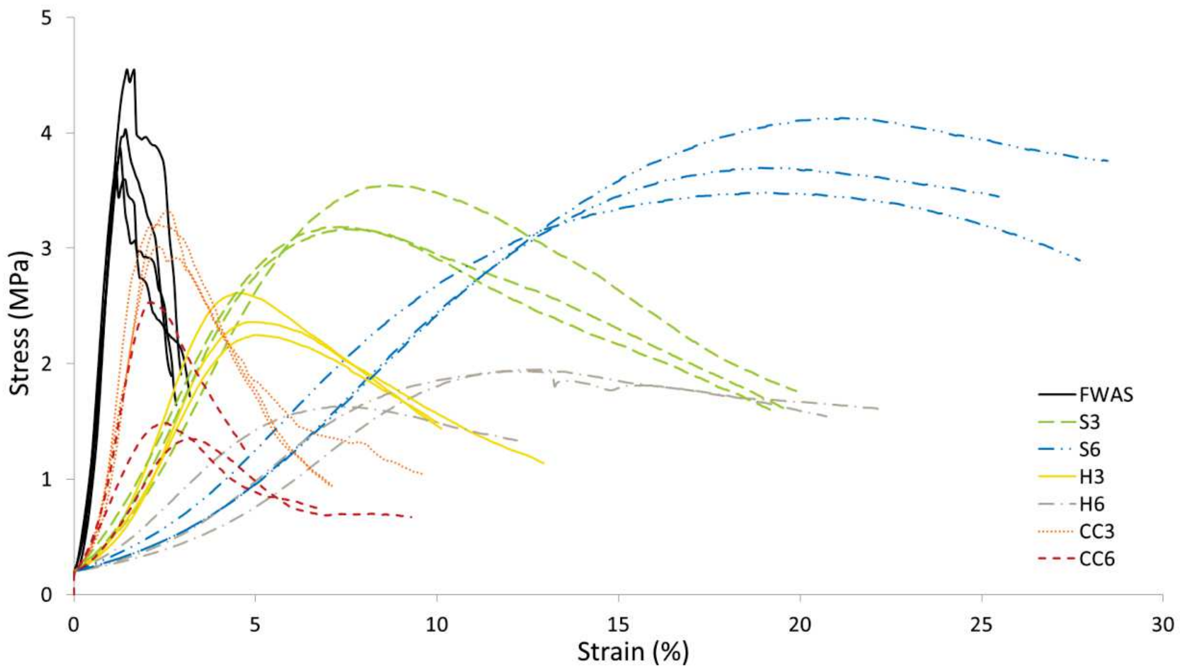

Figure 3-11. Strain-stress diagram for all the specimens

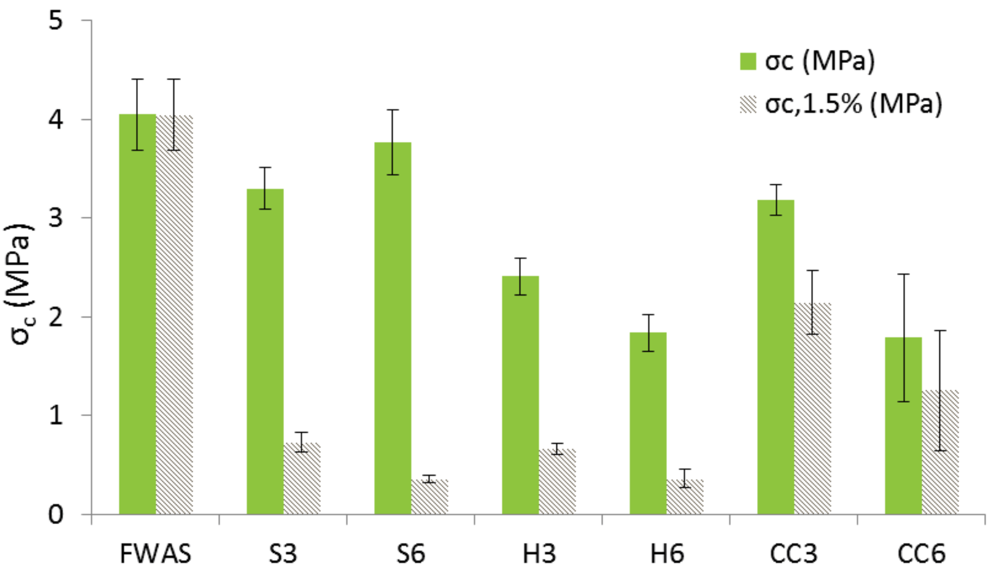

Figure 3-12. Maximum compressive strength (σ_c) and compressive strength at 1.5% strain ($\sigma_{c,1.5\%}$)

3.2.5. Influence of the testing protocol on apparent stiffness

Apparent stiffness was obtained from compressive strength tests and is recapitulated in Figure 3-13, according to the testing protocol. Friction does not seem to have any great influence on the stiffness, which is of the same order of magnitude for both situations (with quite large standard deviations). The most striking result visible in the figure is that the apparent stiffness of FWAS specimens is the highest (around 500 MPa).

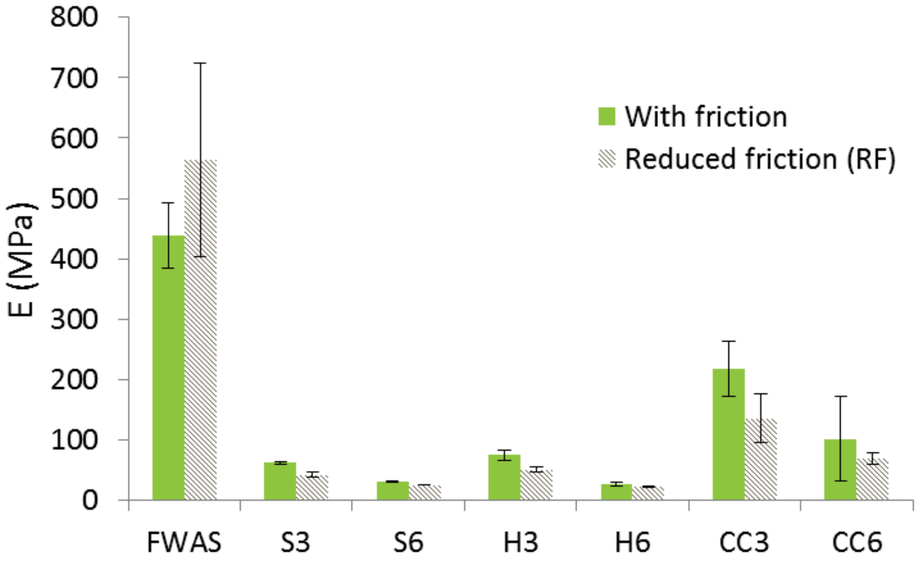

Figure 3-13. Apparent stiffness (E) of the materials for both protocols

3.2.6. Effect of the plant aggregates on apparent stiffness

The apparent stiffness obtained from the tests with friction is represented according to the plant aggregate content in Figure 3-14. The apparent stiffness of the specimens containing 3% of barley straw, hemp shiv and corn cob is respectively 62, 75 and 217 MPa. For an addition of 6%, the stiffness is 31, 26 and 102 MPa respectively for barley straw, hemp shiv and corn cob. Specimens made with straw and with hemp shiv showed very similar stiffness for a given content. With an apparent stiffness of 439 MPa, FWAS specimens had the highest stiffness. For an increase of each plant aggregate content, there was a decrease in the apparent stiffness. This result can be explained by the high compressibility of the plant particles (Cerezo, 2005) and is in agreement with various references (Al Rim et al., 1999; Chan, 2011; Piattoni et al., 2011; Quagliarini and Lenci, 2010) stating that the straw addition controls the plastic behaviour of the specimen through a lower homogeneity of the mixture. This decrease of apparent stiffness could be linked with the density of the specimens as shown in Figure 3-15. An empiric exponential correlation between apparent stiffness and dry density is found: $E = 0.3184 \exp\left(0.0035\rho_{dry}\right)$ with ρ_{dry} in $kg.m^{-3}$. Such a relation has already been proposed by Al Rim et al. for earth specimens with wood aggregates (Al Rim et al., 1999), but it was $E = 1127d^{3.142}$ with d the density of the dry material relative to the density of water.

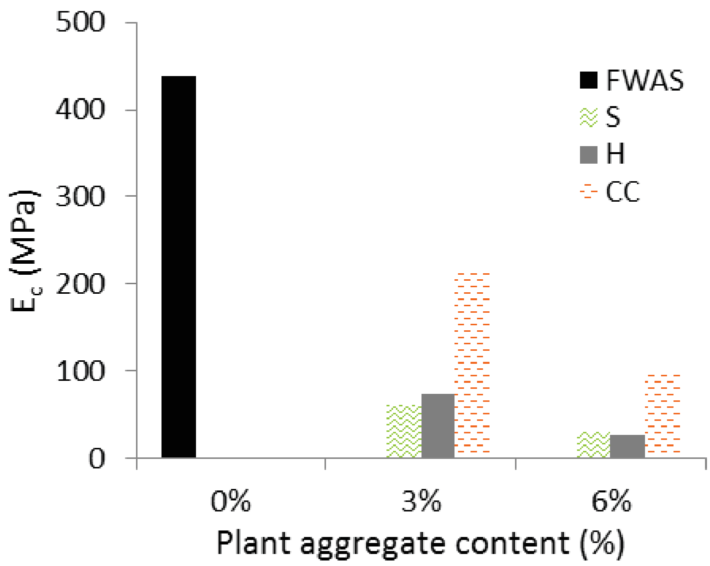

Figure 3-14. Apparent stiffness (E_c) from compressive test as a function of the plant aggregate content

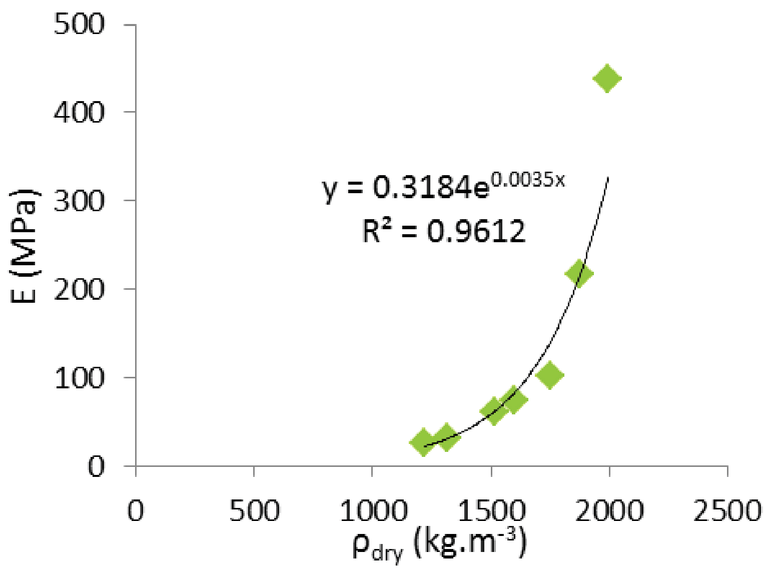

Figure 3-15. Apparent stiffness (E) as a function of the density

3.3. Flexural strength measured on extruded specimens

The average of dry density, the maximum flexural strength and other mechanical parameters are reported in Table 3-6. The minimum flexural tensile stress required by the Masonry Standards Joint Committee (MSJC) (Masonry Standards Joint Committee (MSJC), 2008) (quoted in (Villamizar et al., 2012)) for clay and concrete blocks is 0.21 MPa. Another value, of 0.65 MPa, is required by the British Standard BS 6073 (BS 6073, 1981) (quoted in (Algin and Turgut, 2008)) for building materials to be used in structural applications. All the flexural strengths of the specimens tested in this study are above these two minimum requirements, the lowest strength being 1.34 MPa, found for H3 specimens.

Table 3-6. Average experimental mechanical properties: dry density (ρ_{dry}), flexural strength (σ_f), ultimate strain (ε_f), experimental apparent stiffness (E_f) and fracture energy (G_f)

Type	ρ_{dry} (kg.m^{-3})	F (N)	σ_f (MPa)	ε_f (%)	E_f (MPa)	G_f (J.m^{-2})
FWAS	1982 ± 8	2053 ± 120	1.88 ± 0.10	0.22 ± 0.02	856 ± 57	7 ± 0
$S3_{short}$	1781 ± 10	1900 ± 123	1.80 ± 0.13	0.44 ± 0.11	475 ± 49	296 ± 50
$S3_{long}$	1734 ± 20	1776 ± 135	1.69 ± 0.14	0.55 ± 0.14	385 ± 29	484 ± 41
H3	1712 ± 11	1453 ± 86	1.34 ± 0.08	0.31 ± 0.04	577 ± 72	157 ± 9
S_{A1}	1784 ± 10	1798 ± 163	1.69 ± 0.16	0.49 ± 0.03	442 ± 72	462 ± 11
S_{A2}	1782 ± 12	1824 ± 183	1.73 ± 0.10	0.36 ± 0.08	508 ± 67	538 ± 81

3.3.1. Effect of the plant aggregate type on the flexural strength

The maximum flexural strengths of the different mixtures are represented in Figure 3-16 (a). FWAS has the highest flexural strength, followed by $S3_{short}$, $S3_{long}$ and H3, with values of 2053, 1900, 1776 and 1453 N respectively. This result could be correlated with the respective densities: the lower the density, the lower the flexural strength.

It can be seen that the flexural strength is higher for the specimens with short straw than for those with long straw. This result is contrary to the findings of some other authors (Danso et al., 2015b; Mostafa and Uddin, 2015), who stated that an increase of the fibre length increased the embedded length and thus the adhesion area, leading to an improvement in flexural strength. This result could be explained by the fact that, for the same straw content, there are more particles in a mix with short straw than with long straw, engendering a better distribution of the particles in the matrix. Another explanation could be the existence of a length limit, depending on the specimen size, above which the difficulty of dispersion offsets the positive effect of the reinforcement. Moreover, after being extruded under vacuum, the specimens with short straw presented a better visual surface quality than the specimens with long straw. This could lead to a better adhesion between the earth and the straw, explaining the higher resistance.

Although most cases in the literature show an increase in flexural strength with an addition of plant aggregate ((Al Rim et al., 1999; Aymerich et al., 2012; Bouhicha et al., 2005; Galán-Marín et al., 2010)), an adverse effect (decrease of flexural strength) was found by Villamizar et al. (Villamizar et al., 2012) with an addition of cassava peels and by Algin and Turgut (Algin and Turgut, 2008) with an addition of cotton wastes. This could be due to the heterogeneity of the material or the weakness of the adhesion between the particles and the matrix (Yetgin et al., 2008).

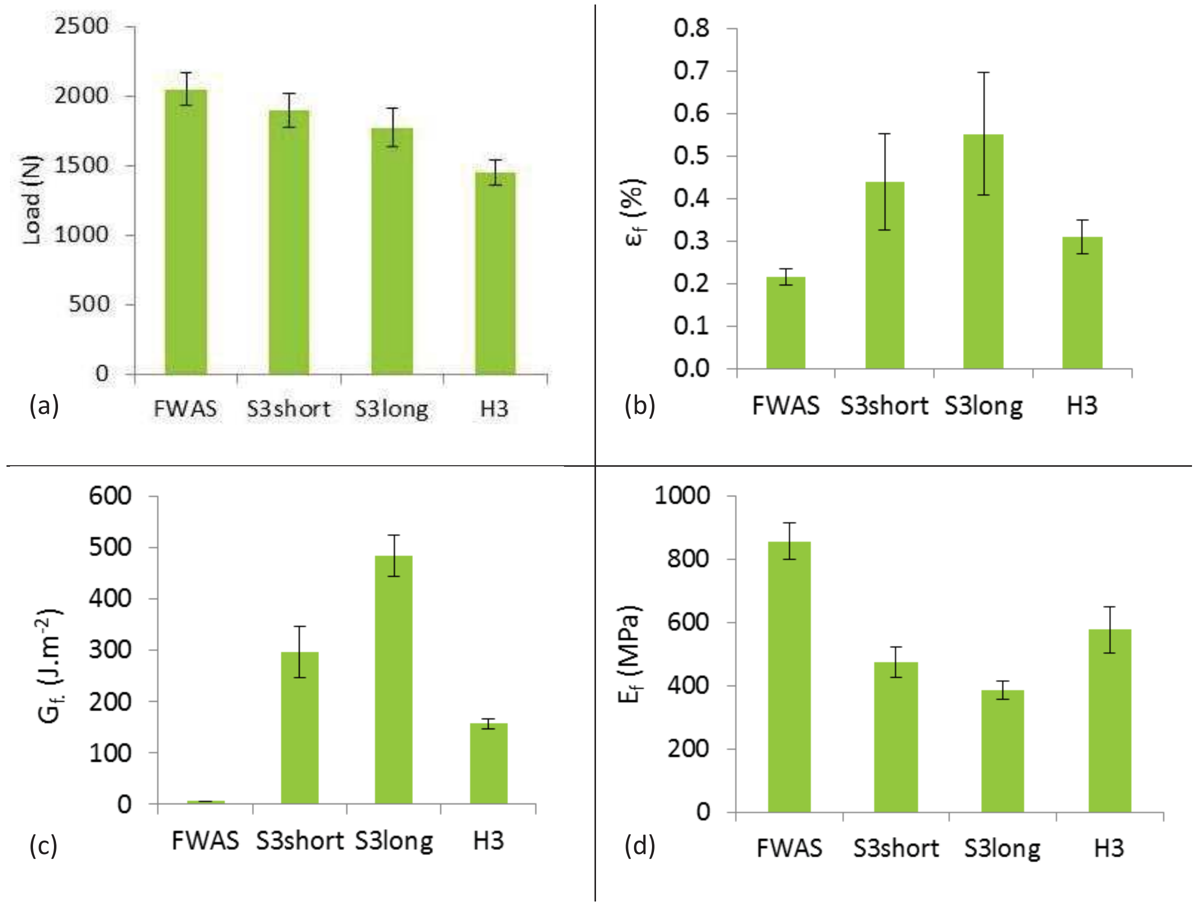

Figure 3-16. Influence of the plant aggregate on flexural behaviour: (a) Flexural load-carrying capacity, (b) Peak strain (ε_f), (c) Average fracture energy (G_f) and (d) apparent stiffness (E_f)

3.3.2. Effect of the plant aggregate type on ductility and apparent stiffness

At the end of the tests, reinforced specimens were not totally split into two parts (Figure 3-4) and extra manual force was necessary to separate them, whereas FWAS specimens were divided into two parts. Figure 3-17 presents typical load-deflection curves obtained during the bending test for the different samples. These curves clearly show that the addition of plant aggregates increases the ductility, increasing the deflection at failure and giving some residual strength.

Peak strain is represented in Figure 3-16 (b). As mentioned above, the strain is increased with the addition of plant aggregates, especially for long particles. The strain was 0.44 and 0.55% respectively for short and long straw. The lower value in the case of hemp shiv (0.31%) could be attributed to a morphological effect: the particle is indeed less elongated and so does not have as much surface area in contact with the earth matrix as the straw particle.

Figure 3-16 (c) represents the average fracture energy for each composition. The value is close to 0 J.m^{-2} for the FWAS specimens whereas the fracture energy of the other specimens is higher: 296, 484 and 157 J.m^{-2} respectively for S3$_{short}$, S3$_{long}$ and H3. The addition of plant aggregates allows a huge increase in fracture energy. The energy absorbed increases when the length of the fibre increases; it is 39% higher with long than short straw, meaning that its residual strength is greater. This result shows that the fracture response of materials reinforced with plant aggregates or fibres is governed by mechanisms of toughening such as fibre bridging and fibre pull-out (Aymerich et al., 2016). These effects occur only for sufficient crack opening.

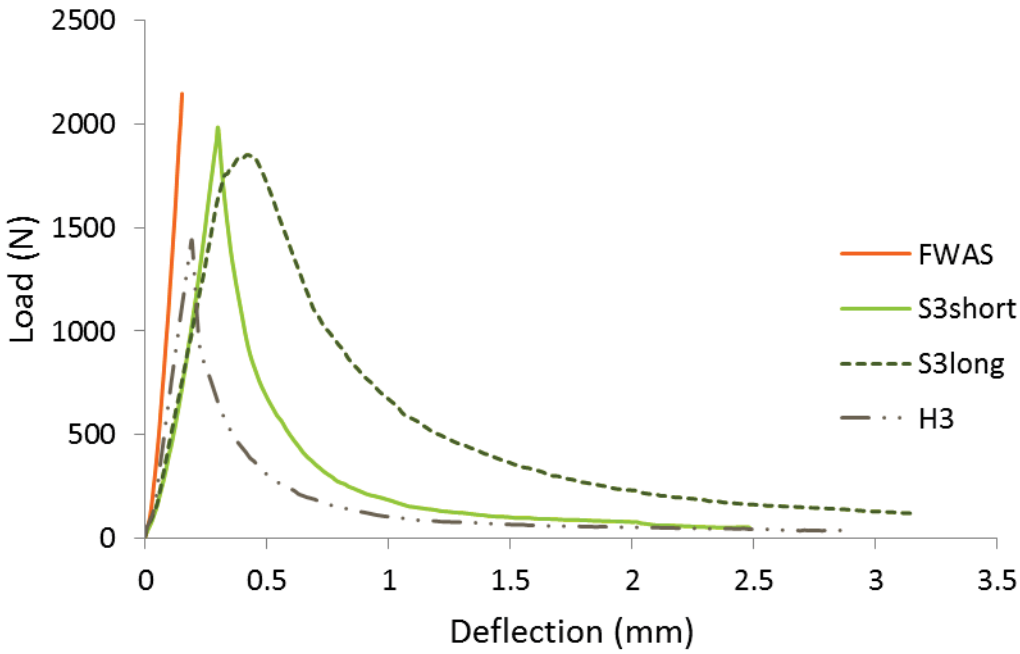

Figure 3-17. Typical load-deflection curves

The experimental values of apparent stiffness are presented in Figure 3-16 (d). As for the apparent stiffness from the compressive test, they seem to decrease with the addition of plant aggregates.

This result is partially correlated with the literature. Although the flexural apparent stiffness of an earth material with wood aggregates increased with between 10 and 20% of addition, it decreased above 20% (Al Rim et al., 1999).

3.3.3. Effect of the surfactant on the flexural strength, post-peak behaviour and apparent stiffness

The behaviour under flexion of the mixtures with untreated and treated long straw is represented on Figure 3-18. The increase in flexural load capacity between S_{A2} and $S3_{long}$ is only about 3% and the standard deviations are high. The surfactant has no effect on the flexural strength.

Peak strain is represented in Figure 3-18 (b). The strain decreases with the addition of a surfactant, especially the A2 additive. Strain is 0.55, 0.49 and 0.36% for $S3_{long}$, S_{A1} and S_{A2} respectively.

Figure 3-18 (c) represents also the average fracture energy for the untreated and treated compositions. $S3_{long}$ and S_{A1} present similar values, of 482 and 462 $J.m^{-2}$, respectively but a small increase of 10% can be noticed for S_{A2}, with a fracture energy of 538 $J.m^{-2}$.

Experimental values of apparent stiffness are presented in the Figure 3-18 (d). The apparent stiffness is higher for the treated specimens, with values of 442 and 508 MPa for S_{A1} and S_{A2}, respectively, whereas it is only 385 MPa for $S3_{long}$. The A2 additive again seems to be the more efficient of the two surfactants tested here, giving an increase in stiffness of about 24%.

Even though the flexural strength is not increased by the straw treatment, the stiffness of the material seems to be increased and the adhesion between the straw and the matrix should also be improved. Surfactant A2 seems to have a greater effect than A1. However, this is a preliminary study, which needs to be pursued further, in particular to optimize various parameters such as the dilution ratio, application method and drying temperature.

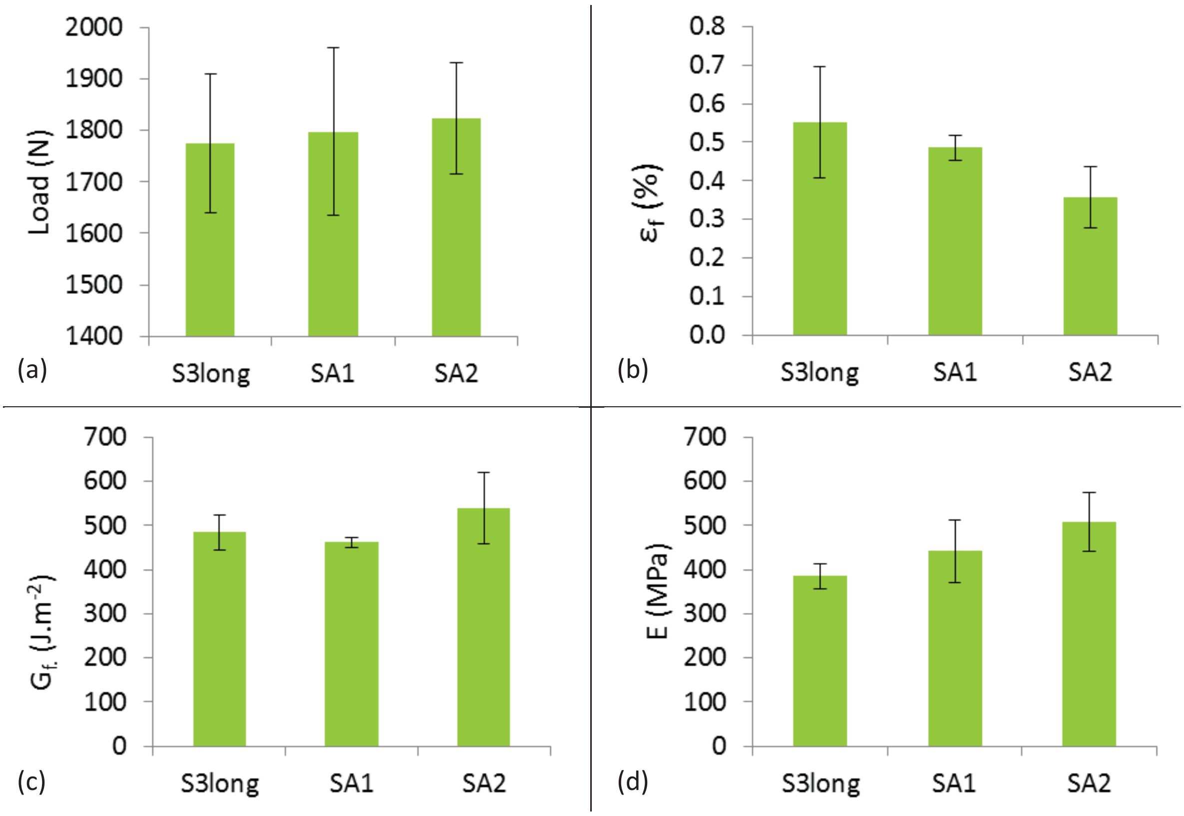

Figure 3-18. Influence of the surfactants on flexural behaviour: (a) Flexural load-carrying capacity, (b) Peak strain (ε_f), (c) Average fracture energy (G_f) and (d) apparent stiffness (E)

4. Conclusion

The mechanical properties of compressed and extruded earth-based specimens were tested. These two ways of manufacturing led to different densities for the same formulation. However, compressive and flexural measurements were independent. Several main conclusions can be drawn concerning the influence of the various parameters such as the plant aggregate type, the protocol of the test or possible treatment. Concerning the compressive tests, the measurement with reduced friction gave a lower compressive strength and peak strain. This method gives a more "realistic" resistance, but is still little used in the literature. At rupture, strength was higher in the case of straw addition, followed by hemp shiv and corn cob additions. For both compressive and flexural tests, the addition of plant aggregates decreased the strength but improved the ductility of the material, decreasing the apparent stiffness. Concerning flexural strength, a better resistance was observed for short than for long straw, but a higher strain was noted for the longest straw. Strain at peak was lower when the straw was treated with the A2 additive, but with the flexural strength was the same.

Various works have shown the diversity of mechanical tests existing for earth bricks and these should be harmonized by means of more investigation and standardization. However, the testing of bio-based earth materials should not be forgotten as their behaviour is much more ductile and cannot be tested in the same way. Although the treatment with surfactants did not improve the mechanical strength of the composites, it did cause a slight decrease in the strain at rupture. More investigation is thus required to optimize its effect on strength, in particular concerning the treatment process (the dilution rate and details of the straw treatment method). In this work, compressed specimens were used to study the influence of plant aggregates and surfactant additives during compression tests whereas extruded specimens were tested in flexion. However, in further work it would be interesting to determine whether or not the adhesion between earth and fibre differs according to the process used: compression or extrusion.

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Article D

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Effect of plant aggregates on the hygrothermal properties of unfired earth bricks

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Abstract

Earth is a sustainable building material thanks to its low embodied energy. Several other key strengths can also be mentioned, such as its good hygric properties. This parameter is an important point as comfort and indoor air quality have become major issues. Nowadays, the energy efficiency of a construction during its service life is also crucial. The thermal insulation provided by an earth brick is, however, not very high. Therefore, some plant aggregates were added to lighten the material and thus decrease its thermal conductivity. The effect of such an addition on the hygric properties was also investigated. The hygrothermal properties of seven formulations made of earth with 0, 3 or 6% by weight content of barley straw, hemp shiv or corn cob were assessed. The properties determined were thermal conductivity, water vapour permeability measured with the wet cup method, and sorption-desorption isotherms with the saturated salt solution and DVS methods. The theoretical MBV was also calculated. The study showed a large decrease in thermal conductivity when a large volume of plant aggregates was added. Concerning water vapour permeability, as earth is a very permeable material, the addition of plant aggregates did not improve this property. The sorption capacity of bio-based earth materials was slightly increased in comparison with earth alone. Finally, the calculated MBV showed the excellent buffering capacity of this kind of material, with and without plant aggregates.

Keywords

Earth bricks, hygrothermal properties, thermal conductivity, permeability, sorption isotherms, MBV

1. Introduction

Research is currently focusing on the reduction of energy consumption and the improvement of hygrothermal comfort during the construction phase and the service life of buildings. Earth is a construction material that meets this major challenge. With its numerous assets such as its availability, recyclability and the low energy required for the transformation process and transportation, earth is increasingly being studied. One of its main qualities is its ability to regulate indoor moisture and improve the comfort of the building's users thanks to its hygroscopic properties [1–4]. These properties are also very important for human health. Poor moisture regulation can engender fungal growth leading to several diseases such as allergies or rhinitis [5,6].

Any porous material construction exchanges water vapour with its environment [7]. It has been shown that earth, specifically, is able to store a considerable amount of moisture through the phenomena of surface adsorption (monolayer or multilayer) and capillary condensation [8]. Some authors believe that earth materials can be considered as natural air-conditioners [9]. The hygric regulation through moisture transport and storage can be quantified by measuring the water vapour permeability and the sorption-desorption isotherms, for example. These two tests are performed under stationary conditions and are useful to give data for Heat, Air and Moisture (HAM) models [7,10]. They also allow a theoretical Moisture Buffer Value (MBV_{ideal}, a dynamic hygric property) to be calculated [4,8,11].

Earth can store heat but is not a good thermal insulator [1]. The addition of plant aggregates to improve thermal insulation of the material is thus the subject of more and more studies [12–16]. Their effect is mainly evaluated by measuring the thermal conductivity. It has been extensively demonstrated that an increase in plant aggregate content leads to a decrease in thermal conductivity [12,14,15,17]. For example, the thermal conductivity of an earthen plaster containing about 16% by weight of barley straw was reduced by about 56% in comparison with a specimen made only of earth (0.154 and 0.350 $W.m^{-1}.K^{-2}$ respectively) [18]. It has also been shown that thermal conductivity depends on the moisture content of the material tested [14,19–21] and on its porosity [19,22]. However, hygric properties of composite materials and their efficiency to regulate the indoor climate remain to be proved and few papers have studied this aspect.

The sorption-desorption isotherm curves describe the Equilibrium Moisture Content (EMC) of a material when it is exposed to a given Relative Humidity (RH). Different methods exist to evaluate the sorption capacity of a material. In the standardized method of Saturated Salt Solutions (SSS) described in NF EN ISO 12571 [23], the various RHs are regulated by saturated salt solutions. The main advantage of this technique is that various samples can be tested at the same time and there is no size limit. Another method is Dynamic Vapour Sorption (DVS), in which RH is regulated by a nitrogen flow. A small sample is weighed continuously by a microbalance. This technique was first used in the pharmaceutical field and the agri-food industry but is now being increasingly used to assess the sorption capacity of building materials [7,24–27]. However, it is controversial because the size of the sample is very small and building materials tend to be heterogeneous [4,28]. These two methods were quantitatively compared by Bui et al. [7]. Both methods showed comparable results for the sorption capacity of barley straw but low correspondence between SSS and DVS techniques was found on corn flakes [27].

The present paper deals with the influence of the nature of plant aggregates and their content on the hygrothermal properties of earth materials. Three plant aggregates, barley straw, hemp shiv and corn cob, were added to an earth matrix to obtain weight contents of 0 to 6%. This experimental study presents the results of the thermal conductivity measured together with two hygric properties: water vapour permeability and EMC, with an assessment of the sorption-desorption isotherms. The sorption-desorption curves were plotted from measurements made with both methods, SSS and DVS. The two methods were qualitatively compared in order to determine whether small samples could be representative of heterogeneous materials. Finally, the MBV_{ideal} was calculated from the two steady-state hygric properties measured (permeability and sorption capacity) in order to evaluate the impact of plant aggregates on the moisture buffering capacity of the composites.

2. Materials and methods

2.1. Raw materials

The materials used in this study were earth, as the main matrix, and three plant aggregates: barley straw, hemp shiv and corn cob. The earth was waste generated by the washing of limestone aggregates produced for the concrete industry, called quarry fines from aggregate washing processing (FWAS). The sludge created is left to dry in sedimentation basins and is then reduced to powder and used. These fines were composed of calcite (60%), kaolinite (11%), illite (11%), quartz (10%), dolomite (6%) and goethite (3%). FWAS were extremely fine: 99% of the particles were below 80 µm and the average particle size (D50) determined using pipette analysis was 6.5 µm. Some physical properties of plant aggregates, determined in a previous study, are recapitulated in Table 3-7.

Table 3-7. Physical properties of the plant aggregates

Material	Barley straw	Hemp shiv	Corn cob
Designation	S	H	CC
Bulk density ($kg.m^{-3}$)	57 ± 1	153 ± 2	497 ± 14
Water absorption (%)	414 ± 4	380 ± 11	123 ± 2
Diameter* (mm)	2.3 ± 1.5	2.0 ± 1.2	2.6 ± 0.4
Thermal conductivity ($W.m^{-1}.K^{-1}$)	0.044 ± 0.001	0.051 ± 0.002	0.096 ± 0.001

* Corresponding to average minor axis by image analysis

2.2. Manufacturing process

The hygrothermal properties were assessed on seven different formulations: a reference material made of FWAS only and composite materials made with 3% or 6% by weight content of one of the plant aggregates. The manufacturing process and the main mechanical properties have been described in a previous article [29]. Table 3-8 recapitulates the different proportions and mechanical properties of the blocks studied.

Table 3-8. Composition and properties of the specimens

Block formulation	Proportion of plant aggregate (%)	w_{OPN} (%)	R_c (MPa)
FWAS	0	14	4.0 ± 0.4
S3	3	19	3.3 ± 0.2
H3	3	17	2.4 ± 0.2
CC3	3	16	3.2 ± 0.2
S6	6	21	3.8 ± 0.3
H6	6	20	1.8 ± 0.2
CC6	6	16	1.8 ± 0.6

2.3. Thermal conductivity

Thermal conductivity properties were assessed on three 150x150x50 mm^3 rectangular prisms for each composition. The measurements were carried out with an EP500 guarded hot plate apparatus. Before testing, the specimens were dried at 100°C and placed in a desiccator to cool. They were wrapped in a thin plastic film to avoid any humidity uptake during the measurement. The test was performed at 25°C with a difference of temperature of 10 K between the two plates. Steady state was assumed to have been reached when the change in conductivity was less than 1% in 60 minutes. To calculate the thermal conductivity, one-dimensional heat transfer by steady state conduction was assumed (Equation 1):

$$\lambda = \frac{Q.e}{\Delta T.S} \qquad (1)$$

where Q is the heat input (W), e the thickness of the specimen (m), and S its cross section (m^2).

2.4. Water vapour permeability

Measurements were realized on three cylindrical specimens 5 cm in diameter and 5 cm high (Φ5H5) for each composition. The wet cup method was applied according to standard NF EN ISO 12572 [30]. The wet cup was chosen to highlight the effects of capillary transfer in an earth material. Water vapour and liquid transfers occurred simultaneously through the sample during the wet cup test. The assessed property thus corresponded to an apparent water vapour permeability. Before testing, specimens were kept at 20°C and 50% RH. The humidity of the wet cup was regulated at 86% by means of a saturated salt solution of potassium chloride. This cup, with the specimen on its top, was placed in a chamber regulated at 23°C and 50% RH. The gradient of relative humidity created an outgoing flow of water vapour. The specimens were surrounded by an adhesive waterproof aluminium tape on the lateral face. They were then placed on a plastic support to avoid contact with the saline solution. The whole setup was finally sealed by a mix of 60% beeswax and 40% paraffin. The arrangements were weighed regularly until a steady-state vapour flux was established. The transmission rate of water vapour through the sample (G in $kg.s^{-1}$) was determined by linear regression excluding the initial, non-linear, phase.

The water vapour permeability (δ) and the water vapour diffusion resistance factor (μ) were then determined for the seven compositions. The water vapour permeability ($kg.m^{-1}.s^{-1}.Pa^{-1}$) was calculated with the following equation (2) which takes the resistance of the air-layer between the base of the sample and the saturated salt solution into account [30]:

$$\delta = \frac{e}{\frac{A.\,\Delta \mathrm{p}}{G} - \frac{e_a}{\delta_a}} \tag{2}$$

with e (m) the thickness of the sample, A (m^2) the exposed surface area of the sample, Δp (Pa) the vapour pressure difference across the sample, e_a (m) the thickness of the air layer between the sample and the salt solution and δ_a the water vapour permeability of the air ($kg.m^{-1}.s^{-1}.Pa^{-1}$).

The vapour pressure difference was calculated using equation (3) [30]:

$$\Delta p = (RH_2 - RH_1) \times 610.5 \times e^{\frac{17.269 \times \theta}{237.3+\theta}} \tag{3}$$

with RH_1 and RH_2 (%) the relative humidity inside and outside the cup, respectively, and ϴ the temperature (°C).

The water vapour permeability of the air was determined using equation (4) [31]:

$$\delta_a = \frac{2.3056 \times 10^{-5}.\, p_0}{R \times T \times p}\left(\frac{T}{273}\right)^{1.81} \tag{4}$$

with p_0=1013.25 (hPa), the standard atmospheric pressure, R=462 ($N.M.kg^{-1}.K^{-1}$) the gas constant for water vapour, T (K) the temperature and p (hPa) the atmospheric pressure.

The water vapour diffusion resistance factor was finally calculated with equation (5):

$$\mu = \frac{\delta_a}{\delta} \tag{5}$$

2.5. Sorption and desorption isotherms

The relation between the water content of a material and the relative humidity of the environment at constant temperature can be represented by its sorption-desorption isotherm. The sorption-desorption property is necessary to model the buffering effect of a material and has great influence on its resistance to the proliferation of micro-organisms [28].

2.5.1. *Saturated salt solution method*

The saturated salt solution method consists of exposing a material to various defined relative humidity levels regulated by saturated salt solutions at a constant temperature. The water content of the material is then determined gravimetrically. The detailed experimental procedure is presented in the standard NF EN ISO 12571 [23]. The samples were exposed to six conditions controlled by six different salts. Their theoretical values of relative humidity and the associated uncertainties are presented in Table 3-9. The choice of the salts was made according to their availability in the laboratory and the recommendations of the standard, which requires at least five salt solutions in the range of 9% to 93% of relative humidity.

The test was assessed on broken monolithic samples (Φ5H5-specimens), of mass between 7 and 16 g, and on loose particles (plant and earth), of mass between 0.5 and 5 g. The masses differed depending on the bulk density of the material. Approximately the same volume was tested each time. The test was performed in triplicate. The samples were first dried at 50°C, as done in [7,25], until constant mass was reached (weight variation of less than 0.1% between two weighings 24 h apart). The samples were then put in a sealed ventilated box to be successively exposed to the different relative humidity steps. The relative humidity was increased in steps from around 8% to 95% to determine the sorption curve. It was then decreased from 94% to 8% in order to trace the desorption curve. The moisture content was calculated at equilibrium, when the mass variation was less than 0.1% between three consecutive weighings.

Table 3-9. Equilibrium relative humidity of the different saturated salt solutions at 20°C

Relative humidity (%)	Salt
7.98 ± 1.9 [1]	Sodium hydroxide (NaOH)
22.51 ± 0.32[1]	Potassium acetate (CH_3COOK)
43.16 ± 0.33[1]	Potassium carbonate (K_2CO_3)
65.5[2]	Ammonium nitrate (NH_4NO_3)
75.47 ± 0.14[1]	Sodium chloride (NaCl)
94.62 ± 0.66[1]	Potassium nitrate (KNO_3)

[1] According to NF EN ISO 12571 [23]

[2] According to Winston and Bates [32]

2.5.2. *DVS method*

The sorption-desorption isotherms of the different materials were also evaluated by the Dynamic Vapour Sorption (DVS) method. Temperature and relative humidity are the two parameters regulated by the device (Surface Measurement Systems, London, UK). The RH inside the hermetic cell is regulated by means of a dry gas (nitrogen). The schematic system is represented in Figure 3-19. The specimen, suspended by a micro-balance, was weighed every 60 seconds. The test was assessed on monolithic broken samples (Φ5H5-specimens), of mass between 0.4 and 1.4 g, and on loose particles (plant and earth), of mass between 30 and 90 mg. The masses were very low because of the small sample holder volume (Figure 3-20). Two specimens of each formulation were tested and were assumed to be representative of these hygroscopic materials [4,33].

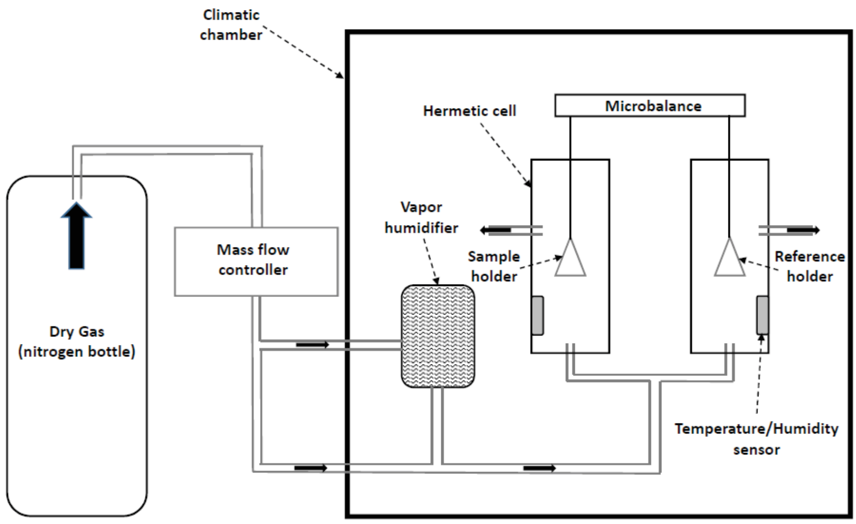

Figure 3-19. Schematic layout of the DVS system [7]

Figure 3-20.Sample holder in its opened cell (here CC3 sample)

Before testing, the specimen was dried at 50°C in the DVS device for 2h. The test was carried out at 23°C, which is the same temperature as the one presented in the standard of the saturated salt solution method, EN NF ISO 12571 [23]. Relative humidity was regulated in successive stages from 0 to 95% by steps of 10% RH, except for the last stage, of 5% RH. For each step, moisture balance was considered to be reached in the specimen if the mass variation over the time variation (dm/dt) was less than 5.10^{-4} $\%.min^{-1}$ over a ten-minute period [25] or in a maximum time interval of 360 minutes (twice this time for the last three steps). The water content was calculated as the ratio of the water contained in the material to the mass of the sample (weight over weight). Figure 3-21 is an example of sorption-desorption behaviour with this programme for an S6 specimen.

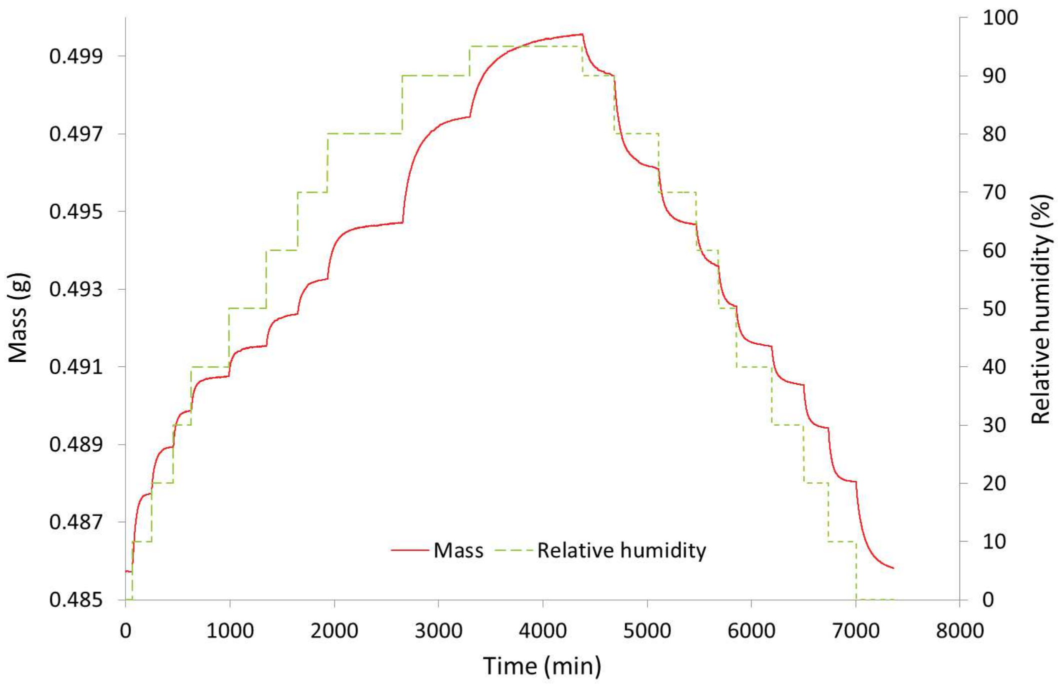

Figure 3-21. Typical variation of mass with relative humidity steps for the DVS (here sorption-desorption of an S6 specimen)

2.6. Prediction of the Moisture Buffer Value from the steady state properties

This paper deals with the measurement of the steady-state hygrothermal properties of earthen materials. However, the moisture buffering capacity of this type of material is often asserted [34]. Thus, it was chosen to calculate a theoretical Moisture Buffer Value, called MBV_{ideal}, by means of calculations presented in the literature. A good correlation between the experimental and the theoretical values was found in [4]. The calculations were thus made with the equations proposed by the Nordtest report [11].

First, the moisture effusivity b_m (kg.m^{-2}.Pa^{-1}.s$^{-1/2}$) was determined with equation (6):

$$b_m = \sqrt{\frac{\delta . \rho_d . \xi}{p_{sat}}} \tag{6}$$

with δ (kg.m^{-1}.s^{-1}.Pa^{-1}) the water vapour permeability measured with the wet cup, ρ_d (kg.m^{-3}) the dry density, p_{sat} (Pa) the water vapour saturation pressure at Θ=23°C, and ξ the moisture capacity (kg.kg^{-1}). The water vapour saturation pressure was calculated with equation (7):

$$p_{sat} = 610.5. e^{\left(\frac{17.269.\theta}{237.3+\theta}\right)} \tag{7}$$

The moisture capacity was calculated from the results of the sorption experiment (with SSS and DVS). It corresponds to the slope of the sorption curve between 33 and 75%, which is assumed linear, calculated according to equation (8) using a linear correlation:

$$\xi = \frac{\partial u}{\partial \phi} \tag{8}$$

with u ($kg.kg^{-1}$) the moisture content and Φ (without unit) the RH.

The MBV_{ideal} was finally calculated from equation (9), relative to 8h/16h cycles:

$$MBV_{ideal} = 0.00568.p_{sat}.b_m.\sqrt{t_p} \quad (9)$$

where t_p (s) is the time period, corresponding to 24h.

3. Results and discussion

3.1. Thermal conductivity

As has been widely reported in the literature, the main interest of adding a bio-resource in an earth matrix is to improve the thermal insulation behaviour of the material [8,13,35]. The thermal conductivity of the seven materials was measured on three samples of each formulation, in a dried state. The effect of the plant aggregate content on thermal conductivity is shown in Figure 3-22.

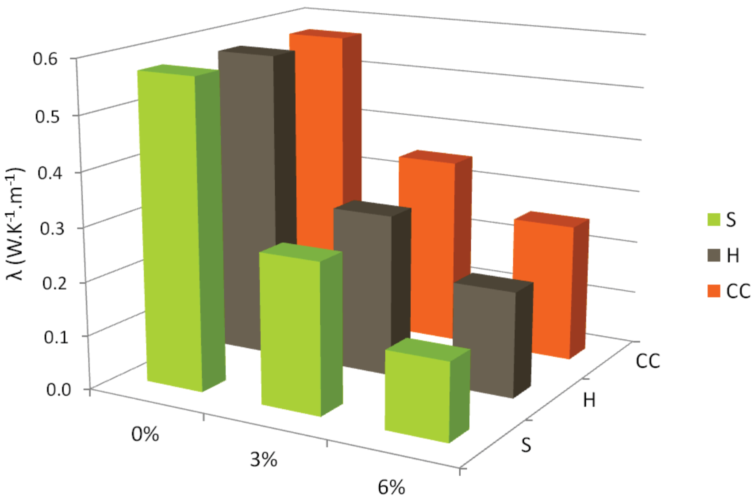

Figure 3-22. Thermal conductivity of the different materials

The values of the thermal conductivity ranged from 0.57 $W.m^{-1}.K^{-1}$ for FWAS specimens to 0.14 $W.m^{-1}.K^{-1}$ for S6 specimens. These results show that an addition of plant aggregates in an earth matrix decreases the thermal conductivity of the material. The most efficient plant aggregate for improving the thermal insulation of the material seems to be straw. The addition of 6% of straw decreased the thermal conductivity by 75% in comparison with an FWAS specimen whereas the decrease was only 55% in the case of an addition of 6% of corn cob. This is in accordance with previous results on the bulk density of plant aggregates (Table 3-7) and of the composites tested here (Table 3-10).

Although a constant mass proportion of the different plant aggregates was used in the manufacture of the material, the resulting volume proportion was different for each nature of plant aggregate. The volume proportion (V) of the plant aggregates contained in the different composites can be calculated knowing the density of the FWAS specimen and the density and mass proportion of plant aggregate for each composite. The results are presented in Table 3-10, together with the dry density (ρ_d), the thermal conductivity (λ) and the mass proportion (M) of the different mixes.

Table 3-10. Physical description of the different formulations

Reference	ρ_d (kg.m^{-3})	λ (W.m^{-1}.K^{-1})	M (%mass.)	V (%vol.)
FWAS	1891 ± 54	0.57 ± 0.03	0	0
S3	1537 ± 5	0.28 ± 0.02	3	21
H3	1519 ± 38	0.30 ± 0.01	3	22
CC3	1671 ± 21	0.35 ± 0.02	3	14
S6	1100 ± 49	0.14 ± 0.01	6	45
H6	1271 ± 16	0.20 ± 0.01	6	37
CC6	1565 ± 18	0.26 ± 0.01	6	22

The volume proportion difference is clear for the composites containing 6% of plant aggregates. There is indeed a volume of straw of 45% whereas the volume of hemp shiv is only 37% and corn cob only 22%.

Concerning thermal properties, the higher the plant aggregate content is, the lower is the thermal conductivity. This can be explained by the associated decrease of bulk density due to the intra-granular porosity of the plant particle. Thermal conductivity values of the composites studied are plotted versus their dry density in Figure 3-23. Other values of earth materials with bio-aggregates, from the literature, have been added for comparison. All the values from the literature came from experiments, except for those of Laurent [36] and Al Rim et al. [12], which came from theoretical models.

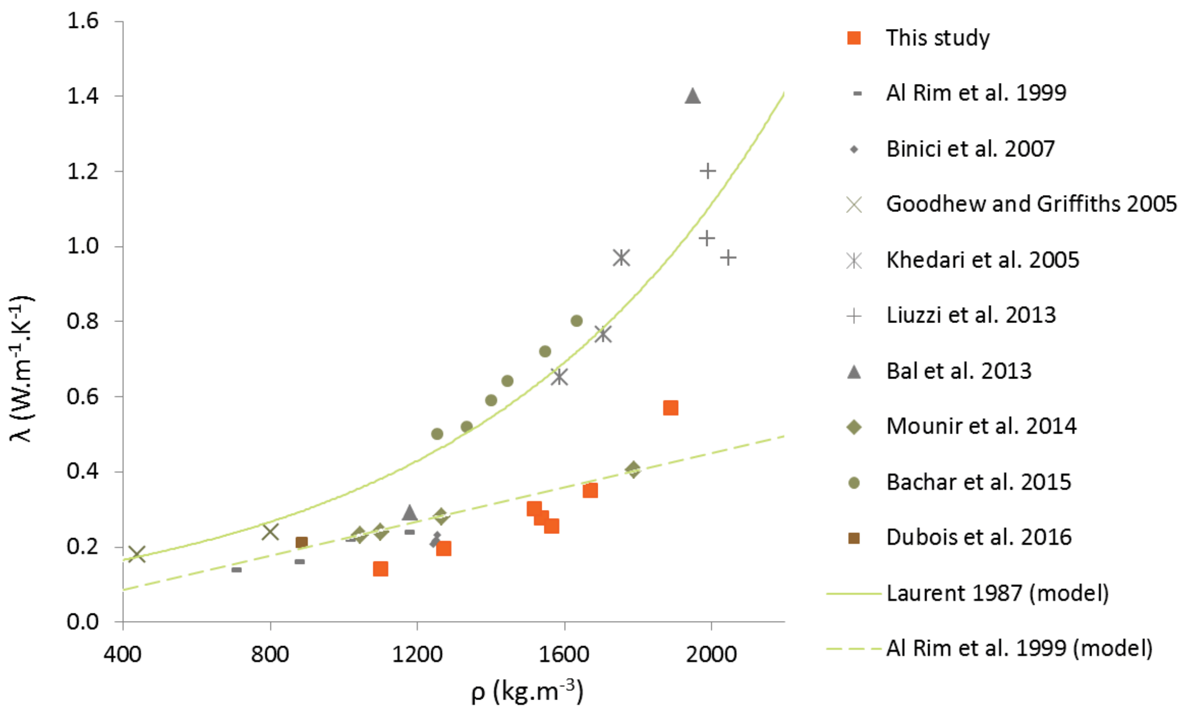

Figure 3-23. Comparison of thermal conductivity values found experimentally (in this study and in the literature) and theoretical values (from the literature) as a function of density

All the materials from the literature presented in Figure 3-23 are earth-based. They can be stabilized with lime [3], with plant particles such as coconut fibres [37] or straw [36], or with plant particles and cement [17]. Even though the results all came from the same type of material, the values present a large range of variation. For example, it can be seen on Figure 3-23 that, for a dry density of around 1500 $kg.m^{-3}$, the thermal conductivity values range between 0.3 $W.m^{-1}.K^{-1}$ (this study) and 0.7 $W.m^{-1}.K^{-1}$ [35]. In the same way, for a dry density of around 2000 $kg.m^{-3}$, the thermal conductivity values range between 0.6 $W.m^{-1}.K^{-1}$ (this study) and 1.4 $W.m^{-1}.K^{-1}$ [14]. The measured values of the present study are thus situated in the lower part of the measured thermal conductivities. It can be observed that the values of the present study are of the same order of magnitude as [17,38] and quite close to the values of [12], but significantly lower than the other references.

However, a correlation can be made between the thermal conductivity and the density of the materials. Several relations have already been developed. Laurent [36] has developed a relation for wattle and daub expressed as follows (16):

$$\lambda = 0.103 \times 10^{0.517\rho} \tag{16}$$

A linear relation (17) was determined for clay-cement-wood composites by Al Rim et al. [12]:

$$\lambda = 0.228\rho - 0.006 \tag{17}$$

where, in both cases, λ is the thermal conductivity of a material in the dry state and ρ is the dry density.

These two relations, plotted on Figure 3-23, are quite different but seem to follow the two tendencies of the thermal conductivities. Laurent's relation gives a higher thermal conductivity than Al Rim's. The results of the present study are more correlated with Al Rim's.

3.2. Water vapour permeability

The water vapour permeability was measured for each formulation using the wet cup method. The assemblies were weighed daily. Using the measurements and the calculation presented in standard NF EN ISO 12572, the water vapour permeability (δ) and the water vapour diffusion resistance factor (μ) were deduced for each composite specimen. The average values calculated from the different formulations are presented in Table 3-11. The water vapour diffusion resistance factor is generally used in comparisons. All the average water vapour diffusion resistance factors are close: between 4.8 and 7.0. The values for earth alone (FWAS specimens) are similar to those of earth bricks studied by Cagnon et al. [25], for which μ-values were between 3 and 7. They are slightly lower than other values of the literature, ranging between 5.5 and 8.2 (also measured with the wet cup) [4] or between 8 and 11 [3].

The values for the bio-composites can be compared with the earth render containing oat fibres studied by Faria et al. [39], which was around 8. They can also be compared with the water vapour permeability measured on cob (construction technique using earth with straw) by [5]. The μ-values were between 6.5 and 9.8. This result is quite close to the permeability of S6 and H6, which were respectively 7.0 and 6.1.

Table 3-11. Water vapour permeability of the materials

Reference	$\delta \times 10^{-11}$ ($kg.m^{-1}.s^{-1}.Pa^{-1}$)	μ
FWAS	4.0 ± 0.3	4.9 ± 0.3
S3	3.8 ± 0.3	5.2 ± 0.4
H3	4.1 ± 0.5	4.8 ± 0.6
CC3	3.8 ± 0.5	5.2 ± 0.7
S6	2.8 ± 0.1	7.0 ± 0.3
H6	3.2 ± 0.1	6.1 ± 0.2
CC6	3.9 ± 0.5	5.1 ± 0.7

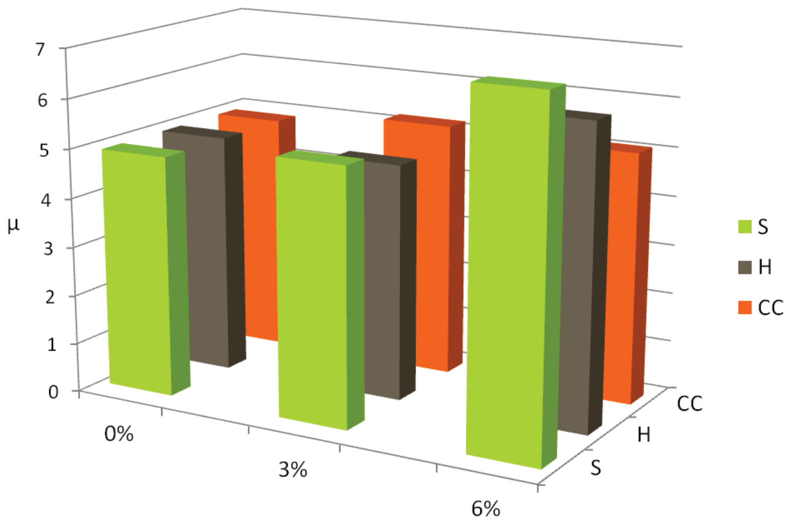

Figure 3-24. Water vapour diffusion resistance factor (µ) according to the plant aggregate content

The µ-values with their standard deviations are shown in Figure 3-24. The results presented here do not bring out significant influence of the addition of 3% of plant aggregates on the permeability of the material. The same comment can be made about the composite including 6% of corn cob. In contrast, when the mixes with 6% of straw or hemp shiv are considered, an increase of µ is observed. Thus, even though the S6 and H6 composites present lower density due to the macroporosity of the plant aggregates, their apparent water vapour permeability is limited in comparison with the FWAS specimen. Liquid transfer in smaller pores has a major impact on the macroscopic water transport in clay material as shown by Fouchal et al. [9]. Their study on extruded earth bricks underlines the fact that, from 50%RH, capillary condensation occurs in the pores that are smaller than 0.1 µm [9]; another study set a limit at 50 nm [40]. Moreover, it has also been shown that the intra-aggregate pore size in an earth material compacted to the Proctor density is between 10 and 50 nm [41].

The decrease of water vapour permeability in wet conditions due to the inclusion of straw or hemp shiv could then be explained by the reduction of capillary diffusion within the earth matrix. Indeed, the large volume content of these two plant aggregates (see Table 3-10) reduces the connectivity of the capillary porous network in comparison with FWAS or corn cob samples. This is consistent with the results of Fouchal et al. [9] showing that an earth material was more permeable than wood. Moreover on clay brick, a µ-value in dry conditions was found to be 1.5 to 3.8 times greater than in wet conditions [25].

Considering the water vapour permeability, the inclusion of plant particles in an earth matrix does not seem beneficial. Nonetheless, the present results underline once again the large water vapour permeability of earth brick in comparison with the reference values of other load bearing building materials according to the 2012 French Thermal Regulations [42]. The permeability of earth bricks is comparable to that of porous construction materials such as wood concrete or gypsum (lower than 10) [25].

3.3. Sorption isotherms

3.3.1. Saturated salt solution method

The saturated salt solution (SSS) method is very time consuming. Three to four weighings per week were performed for around one year for all the sorption-desorption steps. Equilibrium needs to be reached at the end of a given RH. However, the equilibrium was not completely reached at 94% of RH because of potential microbial growth [7]. During a preliminary test, mould growth was observed on the plant aggregates after two weeks at this step. The exposure to this last stage was thus stopped after two weeks, even if equilibrium was not reached.

Bulk materials

The sorption-desorption isotherm curves of the bulk materials are presented in Figure 3-25. The water content represented in the graph corresponds to a mass percentage (ratio of the mass of water to the mass of material). As already noted by other authors [5,28,43] for bio-based materials, the curves presented here have a sigmoidal shape. According to the IUPAC classification [44], which determines six types of curves, these curves belong to Type II. There is a strong increase in water content at high RH because this is the zone of the curve where capillary condensation occurs [25,44]. The curve is close to a vertical asymptote and the representativeness can thus be questionable at these RH.

In Figure 3-25, similar behaviour can be noted between the three plant aggregates up to 75%, although the sorption capacity of the straw is slightly higher than the other two in this range. At 94% RH, the corn cob has a moisture content that is slightly higher than that of the straw and much higher than that of hemp shiv. However, with the high standard deviations at 94% (particularly for the straw), no significant difference can be highlighted between straw and corn cob. The water content of the plant aggregates at 94% RH is thus between 21 and 26%, whereas it is only around 2% for the powdered FWAS. To compare with other natural fibres, at 60% RH for example, the three plant aggregates have a water content of approximately 8.5%. At the same RH, the natural fibres tested by Hill et al. [24] presented quite a similar sorption capacity, between 7.5 and 10% for flax, coir, jute, Sitka spruce or hemp fibres, the exception being cotton fibres, which showed a lower sorption of 5.5%. According to their conclusions, the sorption capacity increases with the OH accessibility and the lignin content of the fibres.

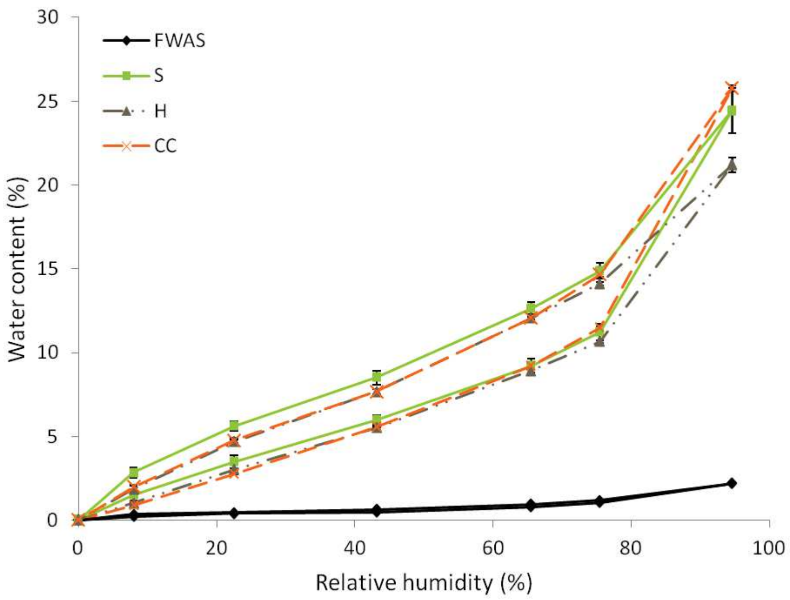

Figure 3-25. Sorption-desorption isotherms (at 20°C) of the bulk materials

The hysteresis is calculated by subtracting the water content of the sample during the drying isotherm from the water content during the wetting isotherm. This is a typical phenomenon for cellulosic and lignocellulosic materials and is explained by the ink-bottle effect (interconnected pore spaces) [5,43]. The hysteresis values of the different raw materials are presented in Figure 3-26. For the three plant aggregates, it can be observed that the hysteresis value increases with the RH. This is also the case for FWAS between 22 and 65%. The values are much higher for the plant aggregates (between 1 and 3.5%) than for the earth (lower than 0.2%). According to Hill et al. [24], this result was to be expected because the hysteresis is greater for materials with high lignin content than for materials with low or no lignin content.

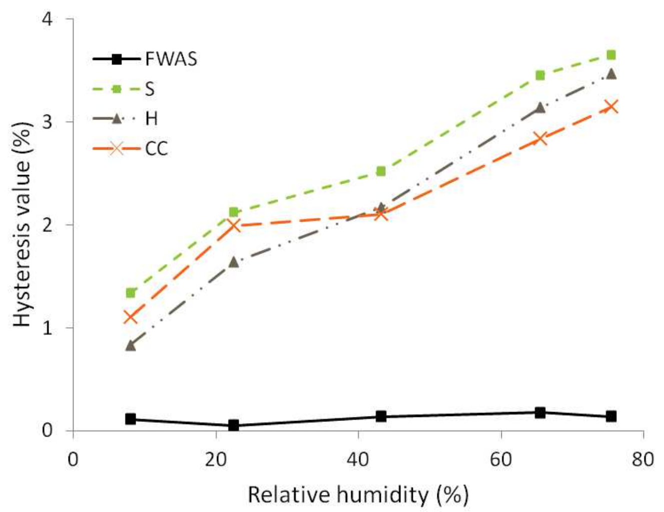

Figure 3-26. Isotherm hysteresis of the raw materials (SSS)

Monolithic materials

Composite and monolithic materials were also tested with the SSS method. Their sorption-desorption isotherm curves are shown in Figure 3-27. In the same way as in the raw materials, the moisture content increases with relative humidity following the usual Type II curve [44].

The sorption capacity is higher in the case of an addition of 6% of plant aggregates than 3% or no addition (FWAS specimens). This is consistent with the moisture sorption capacity of bulk materials presented in Figure 3-25. Plant particles present higher porosity and their inclusion in an earth matrix consequently increases the moisture sorption capacity of the composites. There is no significant difference of sorption capacity for the same addition of content. As was seen with the sorption capacity of bulk plant aggregates, the type of plant does not have any real influence (Figure 3-25). At high RH, the sorption capacity of FWAS specimens is 2.2%. It is 2.7% and 3.2% on average for additions of 3% and 6% of plant aggregate, respectively. Consequently, an addition of 3% of plant aggregates increases the sorption capacity of the earth by about 21% and an addition of 6% increases it by about 46%. The good sorption capacity of the plant particles improves the lower capacity of the earth, because of the increase in porosity [3]. However, it must be stressed that EMC was expressed in $kg.kg^{-1}$ (Fig. 9). For a typical wall, made of the same volume of bricks, the difference of adsorption between the various mixes would be less marked since the density of the mixes including the plant aggregates would be reduced in comparison with the FWAS alone.

When compared with the literature results, the sorption capacity of FWAS is similar to the one found by Ashour et al. [45] for an earth plaster (EMC of 1.8% at 95% RH), but differs from other literature values. Cagnon et al. [25] assessed the sorption capacity of five unfired bricks, whose water content was between 4 and 5.5% at high RH. McGregor et al. [4] also studied a clay material and found an EMC of 4% at around 95% RH. The water sorption of cob was assessed in [5]. The water content was around 5.5% at high RH, but the straw content is unknown. In the case of a straw-clay mixture [46], where the straw content was very high in comparison to the clay content, the water content was around 12% at 93% RH. Thus, the sorption capacity of the FWAS seems to be lower than that of the earth reviewed. A link has been made between the sorption capacity and the nature of the clay mineral to explain that difference. Kaolinite seems less reactive than montmorillonite or illite [3,25,47]. Moreover, the clay content of the FWAS is low (<20%) compared to the calcite content.

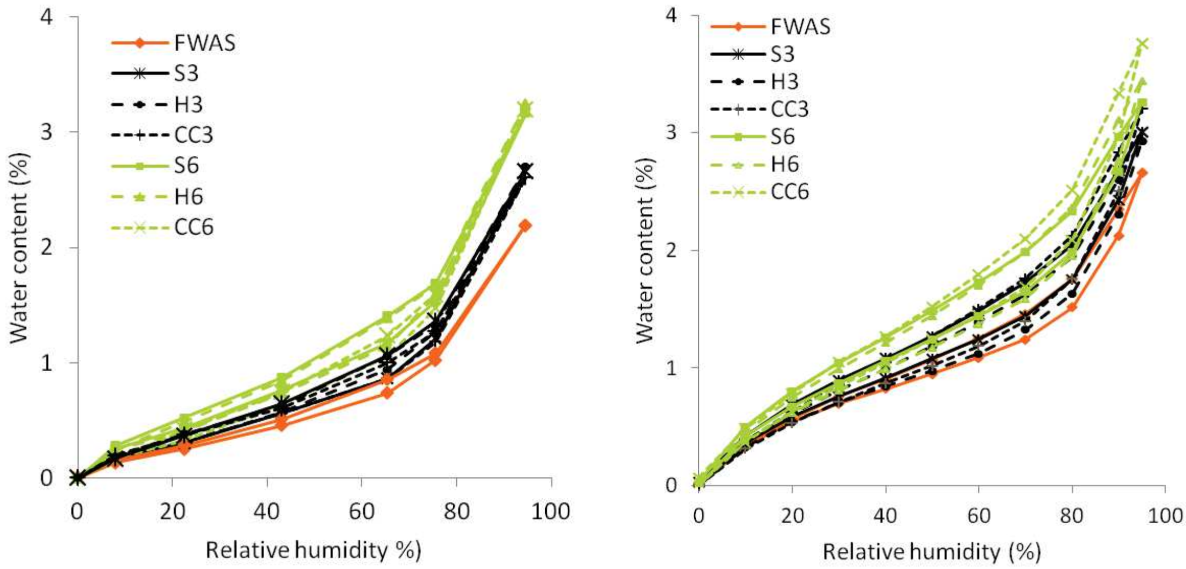

Figure 3-27. Sorption-desorption isotherms of all the formulations (SSS left, DVS right)

The isotherm hysteresis of each material is shown in Figure 3-28. For all the materials, the hysteresis value increases with the RH (except for the last point at around 75%). The hysteresis of earth is the lowest while the hysteresis of composite materials with straw is one of the highest. The values are lower than in Cagnon et al. [25] where the hysteresis values can reach 0.7%. In comparison, the hysteresis values of concrete are much greater [48], around 7%. In the case of hemp concrete, hysteresis values ranged between 1 and 5% [10], which is an expected result because hemp concrete contains more lignocellulosic materials than the composites of the present study.

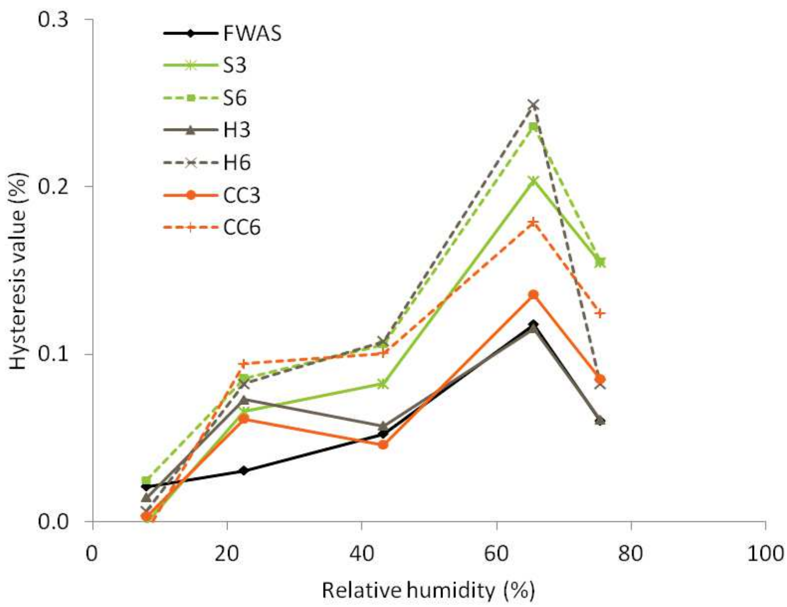

Figure 3-28. Isotherm hysteresis of the monolithic materials (SSS)

The sorption-desorption curves of powdered samples and monolithic samples of FWAS are compared in Figure 3-29. At low RH, the bulk sample presents a higher sorption capacity than the monolithic sample. However from 50% RH, the two curves are very similar, with the same water content at 94% RH. This difference at low RH might be due to the difference of specific surface area. For powdered samples, the surface area available for monomolecular layer sorption is larger than for monolithic samples.

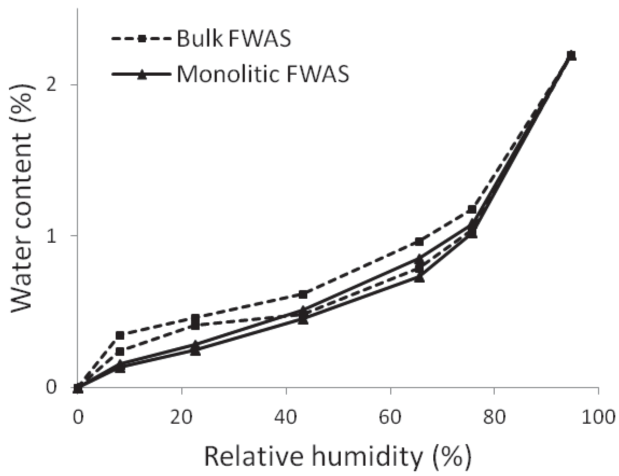

Figure 3-29. Sorption-desorption isotherms (SSS) of bulk and monolithic FWAS samples

It should be noted that the SSS method can present some uncertainties in the regulation of RH. The saturated salt solutions are highly sensitive to small variations of temperature. For example, a rapid increase from 20°C to 21°C can cause a drop of an NaCl solution from 75% to 71% [49]. The test was carried out in an air-conditioned room, but small variations of temperature could occur depending on the outside climate or the presence of people in the room.

3.3.2. DVS method

The test lasted about 7 days for each sample. Two samples per formulation were tested and the total duration was thus about 100 days. The results of the DVS sorption-desorption isotherm curves for monolithic samples are shown in Figure 3-27. The results, quite similar to those obtained with the SSS method, are not commented in detail.

For the sake of clarity, the standard deviations of the tests are not represented. However, the standard deviation of S6 specimens was very large. With the small volume of the sample tested and the heterogeneity of the composite material, there is obviously a problem of representativeness. In the case of CC3 and CC6 formulations, the samples were weighed to obtain a corn cob content of 3% or 6%. Even if the samples were monolithic, the corn cob particles presented poor adhesion with the earth matrix, allowing separate weighing of the corn cob and the earth. The standard deviations of the composites with corn cob were thus lower than for the straw composites.

3.3.3. Comparison between DVS and SSS methods

The two methods to plot sorption-desorption isotherm curves can be compared. The curves were similar for the different materials, so only one is presented here for the bulk materials (S sample) and two materials for the monolithic samples (FWAS and a bio-composite, CC6) in Figure 3-30.

For each material, the curves obtained with the two techniques show a similar shape. It can, however, be observed that the sorption capacity is always higher when measured with the DVS technique. This observation was already made in [25], where it was compared for clay bricks and in [28] for the case of bio-based insulating materials. This difference was just slightly higher in [7] for barley straw. The difference between the two techniques was only of the same order of magnitude as the uncertainties. This can be explained by the definition of the dry state according to the method used. When dried in an oven, the sample can contain residual humidity [50] and its dry mass might be overestimated [7]. Thus the sorption capacity would seem lower with the SSS technique. Even if the initial samples did not have exactly the same moisture content, the loss of water during the drying was quite different according to the method used. For example, the loss of water was approximately 0.2% for a monolithic FWAS sample dried in the oven whereas it was around 0.8% in the case of drying with the DVS device.

A "correction" of the initial dry mass M_0 was applied in Figure 3-30 (d) in order to evaluate the impact of a supposed overestimation of the dry mass with the SSS technique. With a decrease of 0.45% of the dry mass of the monolithic CC6 samples, the curves obtained with SSS and DVS methods are quite closely superposed. A significant difference remains for the point at 8% RH, which could be due to the duration of the test as the stabilization time with the SSS technique was much longer than with the DVS technique, especially for low RH. Further tests will be performed in order to evaluate the impact of the stabilization criteria for the DVS technique on the EMC.

A difference of hysteresis can also be seen between the two methods. The hysteresis obtained with the DVS method is higher than that obtained with the SSS method. This might still be due to a kinetics issue: although the sample was bigger in the case of the SSS method, it was exposed to a certain RH for at least two weeks, against 6 hours (for RH lower than 70%) with the DVS method.

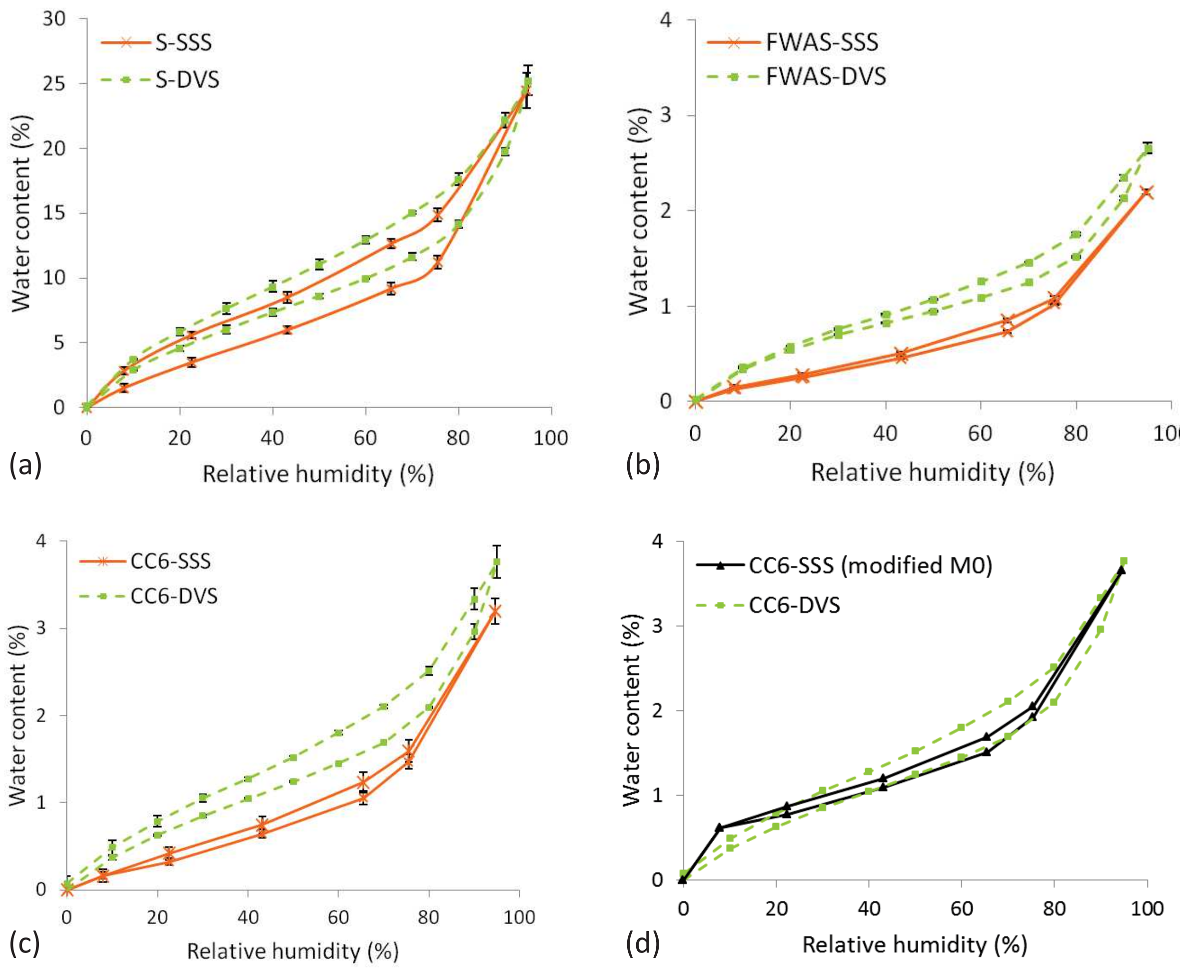

Figure 3-30. Comparison of the isotherm curves obtained with SSS and DVS methods: (a) bulk straw, (b) monolithic FWAS sample, (c) monolithic CC6 sample and (d) monolithic CC6 sample with a modified "dried" M_0 of 0.45% more

3.4. Prediction of the Moisture Buffer Value from the steady state properties

Although it is only a theoretical value, the MBV calculated is useful to compare the various formulations of the present study. The MBV_{ideal} of each material is presented in Table 3-12 and Figure 3-31.

Table 3-12. Calculated dynamic properties

Reference	ξ_1 from SSS (kg.kg^{-1})	ξ_2 from DVS (kg.kg^{-1})	b_m (kg.m^{-2}.Pa^{-1}.s$^{-1/2}$) with ξ_1	b_m (kg.m^{-2}.Pa^{-1}.s$^{-1/2}$) with ξ_2	MBV_{ideal} (g.m^{-2}.%RH^{-1}) with ξ_1	MBV_{ideal} (g.m^{-2}.%RH^{-1}) with ξ_2
FWAS	0.0157	0.0154	6.49E-07	6.42E-07	3.0	3.0
S3	0.0182	0.0188	6.12E-07	6.22E-07	2.9	2.9
H3	0.0196	0.0174	6.62E-07	6.24E-07	3.1	2.9
CC3	0.0176	0.0195	6.32E-07	6.66E-07	3.0	3.1
S6	0.0219	0.0211	4.89E-07	4.81E-07	2.3	2.3
H6	0.0238	0.0216	5.86E-07	5.57E-07	2.7	2.6
CC6	0.0232	0.0235	7.09E-07	7.14E-07	3.3	3.3

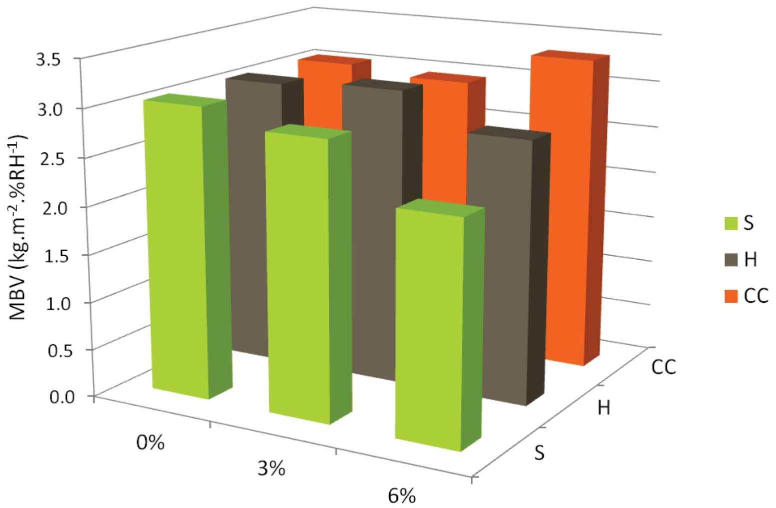

Figure 3-31. MBV_{ideal} according to the material

The moisture buffering capacity of the materials studied here are between 2.3 and 3.3. A very small difference of value (maximum 0.2) can be observed between the calculations based on the SSS or the DVS experiment. For all the formulations, the MBV_{ideal} is above 2. This means, according to the Nordtest classification [11], that the materials present an excellent moisture buffering capacity.

No significant effect can be observed for an addition of 3% of plant aggregates in comparison to FWAS specimens. However, the addition of 6% engenders a decrease of the MBV_{ideal} for the addition of straw or hemp shiv, linked to the influence of these plant aggregates on the water permeability, which appears to be the major factor. The increase of the value of MBV in the case of corn cob is the result of the higher density of this composite, even if the sorption (in mass) of corn cob is close to that of the other two bio-resources.

4. Conclusion

This paper has focused on the hygrothermal characterization of earthen materials containing several types of plant aggregates, namely barley straw, hemp shiv and corn cob. The experimental tests have shown that the addition of plant aggregates in an earth matrix improves the thermal behaviour of the material by decreasing the thermal conductivity. For example, the addition of 6% by weight of straw, corresponding to a volume of around 45%, decreased the thermal conductivity by 75% with respect to the earth material without any plant aggregate. However, the thermal conductivity is not low enough for the developed products to be considered as insulating [51].

Concerning water vapour permeability, the addition of a large volume of plant aggregates, such as 6% by weight of straw or hemp shiv (corresponding to volumes of 45 and 37% respectively), limits the moisture transport slightly. However, for the other formulations, no effect on that property was observed.

The moisture storage capacity of the bulk plant aggregates is quite high in comparison to that of earth. However, the water content is expressed as the amount of water per unit mass of material. Thus, the final product presents only a slight improvement of the sorption capacity, due to the low plant matter mass. Two methods were used to assess the sorption-desorption isotherms: the saturated salt solution method and the Dynamic Vapour Sorption method. The DVS method gave higher values of sorption capacity, which might have been partly due to the definition of the dry state.

Finally, the theoretical Moisture Buffering Value was calculated. This hygric property is interesting by its dynamic nature, which gives information about the indoor moisture buffering when there are RH fluctuations. Although there are some differences in the isotherms between the SSS and DVS methods, no difference is observed for the MBV_{ideal} calculated with the two methods as the slope of the curve is taken into account, not the sorption capacity.

Further studies could be carried out to measure the real MBV and compare the values obtained with the calculated ones. Moreover, a complementary test of the MBV would be the measurement of the heat storage capacity and the restitution of heat. This would enable the thermal inertia of the material to be quantified, which is of major interest for bioclimatic constructions. Finally, it would be interesting to estimate the theoretical sorption capacity by calculation using the values corresponding to the raw materials. This would avoid the problem of poor representativeness due to the small size of the sample and would save time by reducing the number of tests. However, the problem remains of the influence of the aggregate on the structure of the earth matrix at the interface between matrix and plant aggregate. This phenomenon has already been studied in the concrete field, but not yet in the case of unfired earth.

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Concluding remarks

Use properties of earth-based composites containing barley straw, hemp shiv or corn cob are determined in this chapter.

The mechanical behavior of the materials is firstly assessed (Article C). Compressive strength is measured on compressed specimens while flexural strength is measured on extruded specimens. One important result is the ability of an earthen material incorporating plant aggregates to be extruded. Extrusion is possible up to a content of 6% by weight, but it is easier with only 3%. This 3% content was thus kept for the current work on extruded specimens. The main general result is that the addition of plant aggregates decreases the compressive and the flexural strength. However, some observations can be made. Concerning compressive strength, the two protocols compared give two, totally different, strength values. The test with reduced friction (with Teflon capping) gives much lower strengths than the common test with friction, particularly for bio-based materials. The "real strength value" is discussed. Furthermore, a ductile material (the case of bio-based materials) undergoes considerable strain during a compressive test. This strain is sometimes too great to be tolerated in building construction. Comparisons are therefore proposed for the compressive strength corresponding to a certain strain, i.e. 1.5%, as has already been done in some studies for hemp concrete (Cerezo, 2005). For the more ductile composites, with straw and hemp shiv, a very low strength corresponding to a strain of 1.5% is measured. The flexural tests indicate a better flexural strength with straw than hemp shiv. Short straw fragments give a better flexural strength than long ones, whereas long straw fragments give a higher strain than short ones. The resistance of the bio-composites was not influenced by the surfactants tested in this study, but the A2 surfactant slightly decreased the strain at rupture. These results highlight the need for standardized procedures in order to be able to compare the material results among different studies.

Hygrothermal properties are studied in Article D, in particular the measurement of the thermal conductivity and a study of moisture storage and transport (sorption capacity and water vapor permeability). The effect of the addition of plant aggregates is more obvious on thermal conductivity. The higher the plant aggregate content is, the lower is the thermal conductivity, which is highly dependent on the density, or more specifically on the volume content of the plant matter. The values of water vapor permeability obtained show that all the formulations are very permeable to moisture transport, although a small decrease is observed for a plant aggregate content of 6%. Thus this high permeability result seems to be mainly due to the earth characteristic. Concerning the sorption-desorption isotherms, the storage capacity of the material is slightly increased by the plant addition. A comparison of the two methods used, saturated salt solutions and DVS, shows higher equilibrium curves in the case of measurement by DVS. However, the curves present the same shape and tendency. Finally, a theoretical value of the moisture buffering of the different formulations is calculated. The moisture buffering is excellent for all the materials, particularly for the samples containing 6% of corn cob.

Regarding both mechanical and hygrothermal properties, the earth containing 6% of barley straw seems to present the best compromise: mechanical strength that is decreased only slightly and very good thermal insulation. However, its moisture buffering capacity is slightly decreased. The behavior of composites containing corn cob is only of little interest. Thus, this agro-resource will not be studied in the next chapter. The use properties of these materials having been characterized, the durability properties and safety of the occupant will be investigated in Chapter 4. This study will be mainly performed on materials containing only 3% of plant aggregates because this is the content that gives the more easily extrudable and mechanically resistant material (except for straw).

Chapter 4

Durability properties

Preamble

In the previous chapter, the use properties of earthen bio-composites were assessed. As part of the development process of a building material, characterizing its durability is the logical next step. Durability is the ability of a material to withstand wear and tear by continuing to perform its function without significant deterioration over some time. A construction material therefore has to be able to resist the various potential risks occurring in a building (weather, impact, fire, microorganisms, etc.). Thus, the different durability aspects have to be verified to ensure the safety and comfort of the occupants. For example, in case of fire, the material has to be resistant enough to prevent fire from spreading and to maintain the stability of the construction long enough for the occupants to be evacuated. Another example would be the pathogenic effect of the material on the occupants, which can generate some diseases. This is called the Sick Building Syndrome. The materials thus have to avoid damaging the indoor air quality, e.g. by resisting fungal growth, which might be highly pathogenic.

The objective of the present chapter is to characterize the material behavior using some durability tests. The literature review proposed in Chapter 1 shows that few papers focus on the durability properties of earthen bio-composites. Although earth construction seems to be durable, with the various examples of the vernacular heritage in France and all over the world, unstabilized earth is quite sensitive to environmental factors (weather, occupants, micro-organisms). The resistance of an earth material to liquid water is particularly low, and the addition of plant fibers seems to decrease its resistance even more (Obonyo et al., 2010). However, this is only one reference, and investigations concerning other durability properties are needed. Moreover, the incorporation of organic matter, containing cellulose, raises questions about the behavior of the composite with respect to fire or their resistance to molds. This last point is the main objective of the Bioterra project, which aims to reconfirm the healthy nature of earth, even with plant matter incorporated.

This chapter is divided into three parts, each being a submitted article or a published conference paper. The first part (Article E) focuses on durability tests corresponding to weather or occupants' actions. The second (Article F) concerns the fire behavior of the bio-composite materials. Finally, the third part (Article G) deals with bio-durability and more specifically, with the study of fungal growth. In this chapter, only two of the three bioresources previously studied are tested: straw and hemp shiv, as Chapter 3 has shown the little interest of adding corn cob into an earth matrix, particularly concerning the mechanical strength.

Article E has been submitted to Materials and Structures (in June 2017). Several tests are assessed here concerning durability in the presence of liquid water, abrasion and impact, simulating rain, wind or occupants' activities. All the experiments presented in this article were conducted in a laboratory of NOVA University of Lisbon, Portugal, during a two- month visit. This mobility was financially supported by a grant for international mobility of PhD students from the doctoral school MEGeP, obtained after a call for applications. The main interest of this study is the development of test procedures, since no international standard exists for this type of material. Two tests to assess the water resistance of the material are presented: the erosion test, or Geelong test, and the low pressure water absorption test, with Karsten tubes. The resistances to dry abrasion and to impact are also appraised. The composite materials studied here contain only 3% of plant aggregates. Barley straw and hemp shiv are kept as bioresources because of their quite good results in Chapter 3. Furthermore, two other kinds of agricultural by-products are tested for possible valorization: cork granules and rice husk. Portugal is the greatest producer of cork worldwide and is also a large rice husk producing country. A brief review of the literature on these two additional resources is presented in the introduction of the paper.

Article F is a conference paper for the ICBBM-ECOGRAFI conference which took place in June 2017 in Clermont-Ferrand, France. The study aims to characterize the fire behavior of earth materials with plant aggregates. Although earth is a non-combustible material, the addition of cellulose matter can raise questions. Moreover, according to the German standard DIN 4102-1, an earth material containing straw is still non-combustible if the dry density is higher than 1700 $kg.m^{-3}$. This was not the case for the samples with either 3% or 6% of plant aggregates. Hence, checks were carried out to assess their fire behavior. For this purpose, collaboration was initiated with the Fire Laboratory of the Polytechnic University of Catalonia, in Barcelona, Spain. This laboratory is composed of 4 people plus 2 additional collaborators. It is equipped with a dozen machines for testing fire behavior at laboratory scale. The visit was funded by a grant from University Paul Sabatier Toulouse (ATUPS), also obtained after a call for applications, for a research mission in a foreign laboratory. During this stay, some laboratory tests concerning the reaction and resistance of the materials to fire were carried out. First, fire reaction was assessed through a Pyrolysis Combustion Flow Calorimetry test and the ignition-extinction test. Fire behavior was then evaluated using two criteria: stability, with the compressive test, and insulation, with the measurement of thermal conductivity and the temperature of the unexposed face. For practical reasons, these tests were performed at laboratory scale although the standards require tests at actual size. The materials tested in this study were only the composites containing straw and hemp shiv, at a mass content of 3% and 6%.

The last paper, Article G, has been submitted to Building and Environment and deals with fungal growth. This aspect is one of the main objectives of the Bioterra project, where different partners are involved. A current PhD thesis, related to two laboratories, the “Laboratoire de Génie Chimique” (LGC) and the “Laboratoire de Recherche en Sciences Végétales” (LRSV), aims to develop new technologies for the sampling and identification of microbial strains. Another objective is the characterization of the microflora of the earthen materials investigated in the present work, and also of earth materials from inhabited heritage buildings. The last objective of this microbiology thesis is to develop a positive flora for microbial control methods, in collaboration with Agronutrition SAS. A preliminary study, co-authored with researchers from the microbiology laboratories (LGC and LRSV) has already been published in Matériaux et Techniques (see Annex).

As part of the present work, article G, also in collaboration with these two other laboratories, investigates the impact of environmental parameters on fungal growth. Mold proliferation engenders degradation of the material, but also, and more critically, poor indoor air quality. Few recent studies focus on this issue of fungal growth on building materials. Therefore, the major part of the work for this article was performed to adapt and develop tests suitable for a laboratory of materials. For this part, a trainee contributed to the implementation of the methodology. Fungal growth was then assessed on samples containing 0 and 3% by weight of straw or hemp according to several climate conditions. Microscopic observations helped to describe the intensity and the kinetics of the growth depending on the substrate.

Article E

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Effect of plant aggregates on the durability of unfired earth blocks

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Abstract

A growing number of studies are focusing on the properties of earth-based building materials and, in particular, on their mechanical and hygrothermal properties. However, the durability of this type of material is a major concern, especially for authorities and owners, and knowledge of the various aspects of durability is essential to anticipate maintenance and to sustain certain performance levels. In this study, the durability of earth blocks containing 3% of barley straw, hemp shiv, cork granules or rice husk is investigated. The addition of these plant aggregates engenders a decrease of stiffness, estimated by ultrasound velocity measurements. The aggregates show better resistance to impact and to erosion in tests simulating rain impact but water absorption under low pressure is increased by such additions and dry abrasion resistance is decreased.

Keywords

Durability, compressed earth block, straw, hemp shiv, cork, rice husk

1. Introduction

Earth building materials are increasingly being recognized for their huge availability, their low environmental impact and their capacity to regulate indoor climate [1]. However, they present major drawbacks, such as low resistance to liquid water or poor ductility and shrinkage. Some of these aspects can be partially improved by the addition of plant aggregates [2]. These resources are renewable, are by-products of agriculture or industry most of the time, and are available in huge amounts. Their use is thus environmentally friendly. Several studies have dealt with bio-sourced earth blocks or plasters [3-5] but few of them focus on durability [6].

In France, cereal straw is the most common vegetal by-product, with an availability of 7 Mt/y [7], and a number of studies have dealt with straw as an addition in an earth matrix. It can be observed that 17 references of the 50 reviewed by Laborel-Préneron et al. [8] concern straw incorporated in an earth matrix. However, most of these studies focus on mechanical [9-14] or thermal properties [15-16]. Bouhicha et al. [5], using barley straw, looked at the mechanical properties and also the durability of earthen composite material. Some water shower tests were carried out to observe the impact of various straw treatments (water-repellent coatings). Cement renders with lime or polymers were more efficient than lime or earthen renders.

The mechanical, thermal and durability properties of hemp shives in lightweight concrete applications have recently received attention [17-20] but studies of hemp shives in an earth matrix are rare. Hamard et al. [21] focused on the case of an earth plaster with hemp chaff or sisal, studying its mechanical properties, shrinkage and bond with the wall. Gomes et al. [22] characterized earth-based mortars containing 1 part clayish earth and 3 parts sand, by volume, for rammed earth repair. The addition of fibers (5% wt. of earth) decreased linear and volumetric shrinkage and slowed the drying but, after the drying test, gave rise to undesirable biological growth, except when air lime was added as a stabilizer. Flament et al. [23] studied the fresh state and mechanical properties of hemp-clay tiles. Another type of hemp shiv composite, with a sapropel matrix, was studied by Balciunas et al. [24]. The usual properties of these composites, such as thermal conductivity, compressive strength, acoustic insulation or water vapor diffusion resistance factor, were investigated together with water absorption, which gives information about durability with respect to rain, for example.

Portugal and France are 2 of the 5 main rice producers in Europe. With this industry, they both produce rice husk, which is the protective coat of the grain. This agro-industrial by-product is often considered as waste, burnt for its high calorific value to produce energy, or mixed with the soil. It is known to be rot-proof and resistant to insects. It appears that adding unground rice husk into unfired earth bricks has never been studied, although rice husk has been investigated in several other building material applications, mainly to create lighter bricks that can improve thermal insulation. Unground rice husk [25-26] or rice husk ash [27] has been added to fired bricks to increase their porosity. In the clay-sand-rice husk ash material developed by Rahman [27], the rice husk ash was observed to increase the compressive strength. The ash is obtained by incineration, which is harmful for the environment if carried out in the fields, but the controlled burning of rice husk can be a source calorific energy production. Lightweight concrete with rice husk has been explored in a few studies [28-30]. Chabannes et al. [28] developed a bio-based concrete in the same way as for hemp concrete, with a lime-based binder. The thermal and mechanical properties of rice husk concrete and hemp concrete are comparable but hemp-based composites present slightly higher performances. An application of rice husk ash in unfired earth material was studied by Muntohar [31], who investigated the addition of rice husk ash and lime in compressed stabilized earth bricks. This enhanced the soil bearing capacity, the optimal ratio of lime to rice husk ash being 1:1.

Portugal is the world's number one producer of cork. Cork is a renewable resource which is harvested from the tree every 9 to 12 years [32]. Cork granules are an industrial waste from manufacturing or forest maintenance, with few industrial utilizations [33]. As a building material, cork is well known for its thermal and acoustic properties and its hydrophobicity [34]. In the literature, few studies of building materials with earth and cork granules are to be found. Maaloufa et al. [35] and Mounir et al. [36] investigated the thermal properties of composites based on clay and cork granules. A stabilized earth concrete was also studied by Bachar et al. [37]. A decrease of mechanical strength and an increase of thermal insulation were noted with the addition of cork. Other materials including a binder have also been studied, such as cork-gypsum composites [33,34] for false ceilings or partition walls, insulating material based on cork and cement [38-40] or cement-cork mortars [41] for thermal bridge correction. In all cases, thermal insulation was improved by the addition of cork, as was the ability to regulate moisture in the studies of Maalouf et al. [39] and Moreira et al. [40].

The present study investigated the durability properties of bio-based earth products. The main interest of this paper is the presentation of several test procedures for assessing the durability of such materials. The major drawback of earth materials is their poor water resistance. Few specific standardized procedures currently exist on this matter: the German Standard DIN 18945 [42] and the New-Zealand Standard 4298 [43]. The German Standard presents three water resistance tests. The immersion test, consisting in immersing an earth material for 10 minutes, is not really representative of a real environment of the material, except if flooding occurs, and was not performed. The capillary absorption test, where the material is laid on a very hydrophilic material, itself partially immersed in water, is only of modest interest. Finally, the contact test, consisting in placing the specimen on a wet compress for 24h, is more a test to evaluate the behavior of the material during the manufacturing period (with the application of wet plastering or rendering mortar) than a durability test. The New-Zealand Standard also presents three water resistance tests: the wet/dry appraisal test, the erosion test by spraying, and the Geelong test. The latter, less aggressive, was performed in this investigation. Some durability tests from the literature were also undertaken, such as low pressure water absorption with Karsten tubes or impact resistance test using the Martinet-Baronnie apparatus. In this study, 3% weight content of barley straw, hemp shiv, cork granules and rice husk were incorporated into earth blocks in order to improve the durability properties, especially the water resistance. The influence of the type of plant aggregate on ultrasound velocity, dry abrasion resistance, low pressure water absorption, erosion resistance and impact resistance were thus analyzed.

2. Materials and methods

2.1. Materials

2.1.1. Raw materials

Quarry Fines from Washing Aggregate Sludge (FWAS) were used as the earth. These fines are wastes generated by the washing of limestone aggregates produced for the concrete industry. The sludge created is left to dry in sedimentation basins. It is then reduced to powder and used. The properties of the earth, determined in a previous study [44], are given in Table 4-1.

Four plant aggregates are studied in this work: barley straw, hemp shiv, cork granules and rice husk. Some of their physical properties are recapitulated in Table 4-1. The barley straw and hemp shiv properties were determined in a previous study [45] while cork granules and rice husk properties were measured for the present study, using the same procedures.

Table 4-1. Physical and mineralogical properties of earth

Earth (FWAS) properties	
Atterberg limits	
w_L (%)	30
w_P (%)	21
PI (%)	9
D50[a] (µm)	6.5
Mineralogical composition	
Calcite (%)	63
Dolomite (%)	3
Kaolinite (%)	11
Quartz (%)	10
Illite (%)	9
Goethite (%)	3

Plant aggregates properties				
Material	**Barley straw**	**Hemp shiv**	**Cork**	**Rice husk**
Designation	S	H	C	RH
Bulk density ($kg.m^{-3}$)	57.4 ± 1.2	153.0 ± 2.4	184.9 ± 6.5	97.2 ± 1.1
Water absorption (%)	414 ± 4	380 ± 11	200 ± 8	198 ± 7
Diameter[b] (mm)	2.33 ± 1.52	2.02 ± 1.23	1.60 ± 0.83	2.09 ± 0.72
Thermal conductivity ($W.m^{-1}.K^{-1}$)	0.044 ± 0.001	0.051 ± 0.002	0.045 ± 0.003	0.047 ± 0.001

[a] Diameter by sedimentation

[b] Corresponding to average minor axis by image analysis

2.1.2. Blocks

To carry out the various tests, five different mixtures were prepared to produce testing blocks: reference blocks made with FWAS only and blocks each containing 3% by weight of one of the 4 plant aggregates. This 3% content of plant aggregates was chosen because it had been shown in a previous study that it was possible to manufacture blocks by extrusion with this content, which could be interesting for the industrialization of the process [46]. The water contents of the formulations were determined by the Proctor test (w_{OPN}). To manufacture these blocks, earth and plant aggregate fractions were poured into a blender and mixed by hand before adding water. The raw materials were mixed the day before molding.

Rectangular prismatic blocks 150x150x50 mm^3 and cylindrical specimens 5 cm in diameter and 5 cm high were manufactured by static compression at the Proctor density. The cylindrical specimens were intended only for compressive strength tests. Once demolded, the cork blocks showed dimensional instability, leading to cracks. The other types of blocks were in a good state, with just a slight swelling for the blocks with the addition of plant aggregates. The blocks were first dried at 40°C for 24 hours, then the temperature was increased slowly to 100°C and kept at 100°C until the weight became constant (weight variation less than 0.1% between two weighings 24 hours apart). The blocks were then stored in a room regulated at 20°C and 65% relative humidity (RH) and were tested from the moment when they were in equilibrium with the environment (about one week later). Table 4-2 recapitulates the different proportions and properties of the blocks studied. Bulk density (ρ_d) and compressive strength (σ_c) of FWAS, S3 and H3 were measured in a previous study [46]. The values for C3 and RH3 specimens are new results based on the same experimental procedures as used for the others. The thermal conductivity values were obtained on samples at equilibrium at 20°C and 65% RH with a heat transfer analyzer. The compressive strength of FWAS specimens was the highest, followed by S3, RH3, H3 and finally C3 specimens. The resistance value for C3 specimens was significantly lower than for the other composites. This result can be directly linked to the cracks that appeared after demolding and generated poor cohesion of the specimens. These cracks can be explained by the resilience of cork granules, which regained their shape when the compression stopped, and their hydrophobic nature, preventing the wet soil from adhering to them. Thermal conductivity was directly linked with the dry density.

Table 4-2. Proportions and water content, density, compressive strength and thermal conductivity of the blocks

Block formulation	FWAS	S3	H3	C3	RH3
Proportion of plant aggregate (%)	0	3	3	3	3
w_{OPN} (%)	14	19	17	17	17
ρ_d (kg.m^{-3})	1995 ± 0	1519 ± 1	1603 ± 57	1314 ± 21	1769 ± 21
σ_c (MPa)	4.0 ± 0.4	3.3 ± 0.2	2.4 ± 0.2	0.9 ± 0.0	3.1 ± 0.1
λ (W.m^{-1}.K^{-1})	1.1 ± 0.04	0.36 ± 0.02	0.45 ± 0.02	0.26 ± 0.04	0.60 ± 0.05

2.2. Ultrasound velocity

This non-destructive technology is useful to estimate several characteristics of a material, such as homogeneity, dynamic elastic modulus and presence of cracks.

Ultrasound velocity was obtained using a Pundit Lab-Proceq device, and tested three times for each of the three blocks of the same type. The measurements were performed by the direct method, meaning that the two probes (transmitter and receiver) were facing each other on either side of the block. Eight measurements were carried out on each sample, four for each direction under consideration (perpendicular to side A and sides B), following Figure 4-1. Toothpaste, a viscoelastic material, was used to ensure good contacts between the transducers and the block. The device gave the time of propagation, and the velocity was deduced from that value and the distance between the transducers.

The dynamic modulus can be calculated with equation (1):

$$E = \frac{\rho V^2(1+\upsilon)(1-2\upsilon)}{(1-\upsilon)} \quad (1)$$

where ρ is the density in $kg.m^{-3}$, V is the ultrasound velocity in $m.s^{-1}$ and ν is the Poisson's coefficient. However, this coefficient was not measured in this study. The results were thus only discussed with respect to the ultrasound velocity linked with the porosity or the dry density of the material. This approach has already been used by Aubert and Gasc-Barbier [47] to evaluate the effects of freezing-thawing cycles on clayey soil blocks.

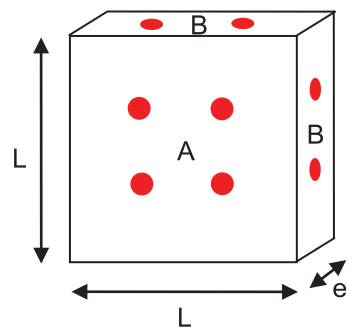

Figure 4-1. Positioning of test points for ultrasound velocity test

2.3. Dry abrasion resistance

This test was carried out on different earth building elements such as plasters [6], bricks [48] or bahareque (technique similar to wattle and daub) [49]. It is useful for assessing the surface resistance to mechanical erosion due to the repetitive friction of the occupants' transit or to solid particles blown by the wind.

Dry abrasion resistance was evaluated according to the German Standard DIN 18947 [50] for unstabilized plastering mortars, by measuring the quantity of material removed from the specimens after 20 rotations with a hard plastic brush. The 65 mm diameter brush was pressed against the surface of the specimen by a mass of 2 kg (Figure 4-2). Three tests were carried out on each of the three blocks per formulation. A coefficient of abrasion in $g.cm^{-2}$ was then calculated with equation (2), according to Millogo et al. [48]:

$$C_a = \frac{m_1 - m_2}{S} \quad (2)$$

where m_1 and m_2 are the masses before and after brushing, in g, and S is the brushed area, in cm^2.

Figure 4-2. Dry abrasion test

2.4. Low pressure water absorption

This test was performed to measure the water absorption of a porous wall under low pressure, which was taken to simulate the action of rain combined with wind. The Karsten tube penetration test [51] was performed using a glass tube filled with water and sealed to the sample with water-resistant plasticine (Figure 4-3). The tube was graduated to measure the volume of water that penetrated into the material and the time of absorption was measured (EN 16302 [52]). Vertical tubes were sealed on the horizontal surface of the blocks because of the difficulty of sealing the tubes on vertical surfaces of the materials under study. Two measurements were made at different points of each of the three blocks tested per formulation. This test was also carried out three times on a fired brick, in order to compare the results with those of the unfired earth blocks.

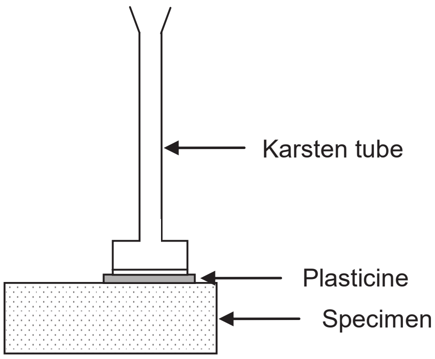

Figure 4-3. Test of water absorption under low pressure

Absorption times were very different according to the mixture tested. It was thus chosen to measure the time taken for each 0.5 ml of water to be absorbed. A water absorption coefficient (C_A in $g.m^{-2}.s^{-1}$) was also calculated between a water uptake of 3 ml and 2 ml following equation (3):

$$C_A = \frac{m_{3ml} - m_{2ml}}{A \times (t_{3ml} - t_{2ml})} \tag{3}$$

where m_{3ml} and m_{2ml} are the masses corresponding to water uptakes of 3 ml and 2 ml, A is the contact area between the Karsten tube and the earthen blocks in m² and t_{3ml} and t_{2ml} are the times in s corresponding to an uptake of 3 ml and 2 ml of water, respectively. This equation was adapted from Stazi et al. [53] and Hendrickx [54] to suit the water absorption rate of certain of the materials used here.

2.5. Erosion resistance

The wet erosion test was performed to simulate rain droplets. It was carried out according the New-Zealand Standard NZS 4298 [43], intended for unstabilized adobes and pressed blocks, based on the Geelong method. 100 ml of water was allowed to drip from a height of 400 mm onto the sample, which was inclined at 27° (Figure 4-4). The duration of the test was between 15 and 25 minutes (adapted from the standard, from 20 to 60 min) and the pit depth was measured. An erodibility index, between 3 and 5 (fail), was deduced from this value according to [43]. With a pit depth between 5 and 10 mm, the erosion class was 3 and the material was considered as erosive. A pit depth between 10 and 15 mm corresponded to class 4 and the material was considered as very erosive. Finally, if the pit depth was greater than 15 mm, the class was 5 and the material failed the test.

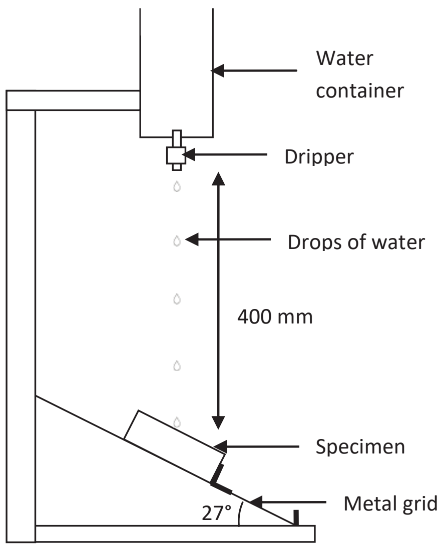

Figure 4-4. Geelong erosion test

2.6. Sphere impact test

Vertical walls of buildings may be subjected to different kinds of impacts such as a stone thrown at the wall or an impact from people or furniture. To evaluate that resistance, the Martinet-Baronnie impact apparatus was used and the test was carried out on the blocks that had already been tested by all the previous tests (ultrasound, thermal conductivity, dry abrasion, erosion and low pressure absorption). This test was inspired by ISO 7892 with a small hard impact body [55], currently used for testing ETICS following EN 13497 [56] and also for renders [57]. The device consists of a spherical metallic mass of 500 g on a metallic stalk that is attached to the wall. It is dropped from the horizontal position onto the sample located vertically underneath (Figure 4-5) in contact with a very rigid wall. The energy created by the test is 3 joules. The number of impacts was between 1 and 4, depending on the impact resistance (cracks). The test was performed on 3 or 4 specimens.

The diameter of the impact point was measured, as was the cracking and general behavior of the blocks.

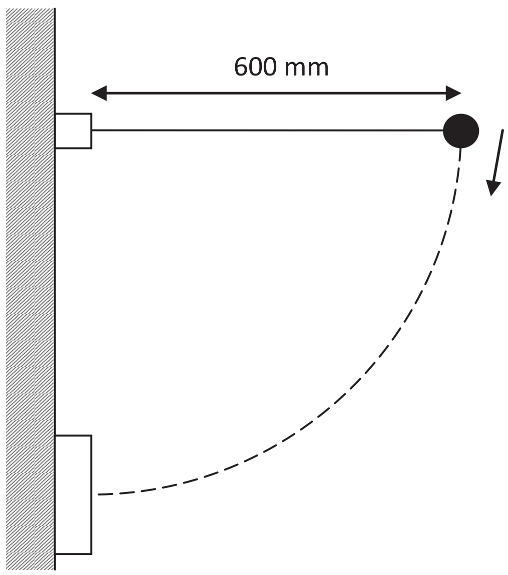

Figure 4-5. Impact resistance test

3. Results and discussion

3.1. Ultrasound velocity

The time of ultrasound propagation was measured on the 2 directions of the blocks. However, the 50x150 mm^2 surfaces of the C3 blocks were not as good as the other surfaces and the signal of the face A was already very low, which made measurements difficult in the longest dimension (sides B). Propagation between sides B of FWAS specimens was only measured for one block because the other blocks were damaged on these sides during demolding. Ultrasound velocity values are presented in Figure 4-6.

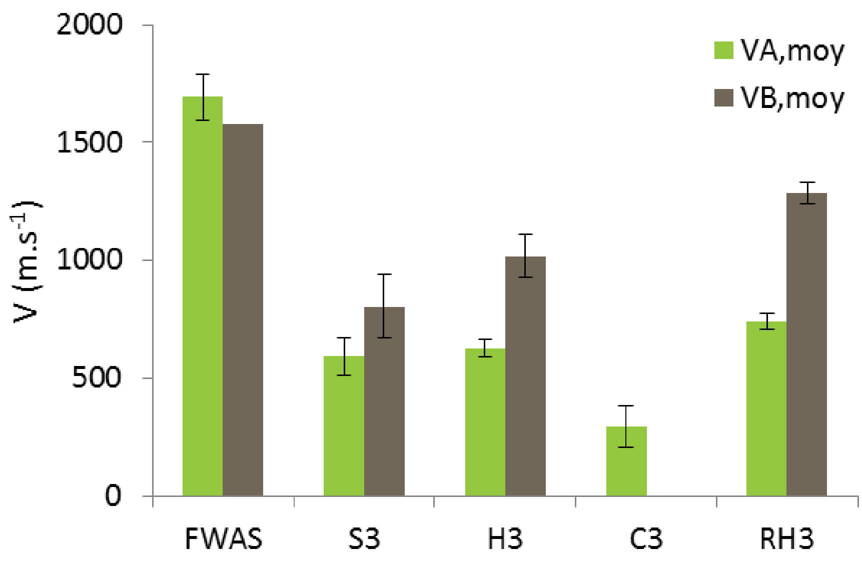

Figure 4-6. Ultrasound velocity of the blocks according to the two directions A and B

Velocity differed slightly with the direction of propagation: perpendicular to the molding pressure (side A) or parallel to the molding pressure (sides B) (Figure 4-6). Based on the most representative results of S3, H3 and RH3, ultrasound velocity was higher when it was measured on sides B. A decrease in ultrasound velocity was also observed with the addition of plant aggregates. This was due to the increase in porosity and thus the decrease in mechanical strength [58]. The low ultrasound velocity of C3 specimens could also be linked to their poor mechanical behavior. In comparison with a FWAS block, the decrease in velocity measured between sides A was 65%, 63%, 83% and 56% for S3, H3, C3 and RH, respectively. Thus, ultrasound velocity decreased with the density. For FWAS specimens, velocity was higher than for the rammed earth studied by Faria et al. [59], where it was 928 $m.s^{-1}$, but the specimens with plant aggregates, except C3, presented velocities close to that of the lime-earth render studied in Faria's paper (between 551 and 775 $m.s^{-1}$).

This non-destructive test is interesting in a durability approach as it characterizes some degradation inside the material. For example, ultrasound velocity measurements were used by Barbera et al. [60] to assess the durability of stone, specifically during accelerated aging tests.

3.2. Dry abrasion resistance

The results of abrasion are presented in Figure 4-7. The smaller the coefficient of erosion is, the better is the durability of the block.

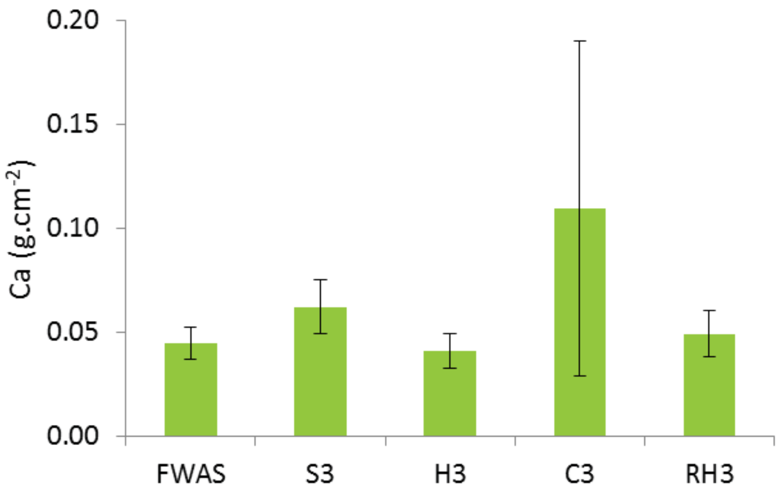

Figure 4-7. Dry abrasion coefficients of the blocks

Differences of dry abrasion resistances between the various formulations are not particularly marked except for C3 specimens, which are much less resistant. FWAS and H3 blocks are the mixtures with the lowest abrasion coefficient (0.04±0.01 g.cm^{-2}) followed by RH3 with 0.05±0.01 g.cm^{-2}, S3 with 0.06±0.01 g.cm^{-2} and C3 with 0.11±0.08 g.cm^{-2}. The standard deviation of C3 blocks is very high because the blocks were not all in the same initial state; some of them were damaged. The elevated value of the abrasion coefficient of C3 blocks can be explained by the rounded shape of the cork particles and their hydrophobic nature, both of which lead to poor cohesion of the mixture. The S3 abrasion coefficient is also quite high because of the length of the straw particles; they are longer than hemp shives or rice husks and thus allow more earth to be removed.

Usually, abrasion results for different mixtures are compared according to the quantity of earth removed during the test [49,61] and the material is classified according to the standard [50], but this applies only to the case of mortars. Here, it was chosen to express the result as a coefficient depending on the surface under test in order to compare the results with others from the literature. The results of this study are slightly higher than the results from the study by Millogo et al. [48] for earth blocks, which are between 0.015 and 0.04 g.cm^{-2}. However, their maximum fiber content was 0.8% by weight, which can explain the difference. Moreover, it is important to note that abrasion coefficients can also be influenced by the hardness of the brush, which makes it difficult to compare results from different laboratories [61].

3.3. Low pressure water absorption

Low pressure absorption test results are presented in Figure 4-8. This graph shows the water absorbed per unit of surface (ml.mm^{-2}) as a function of time (min).

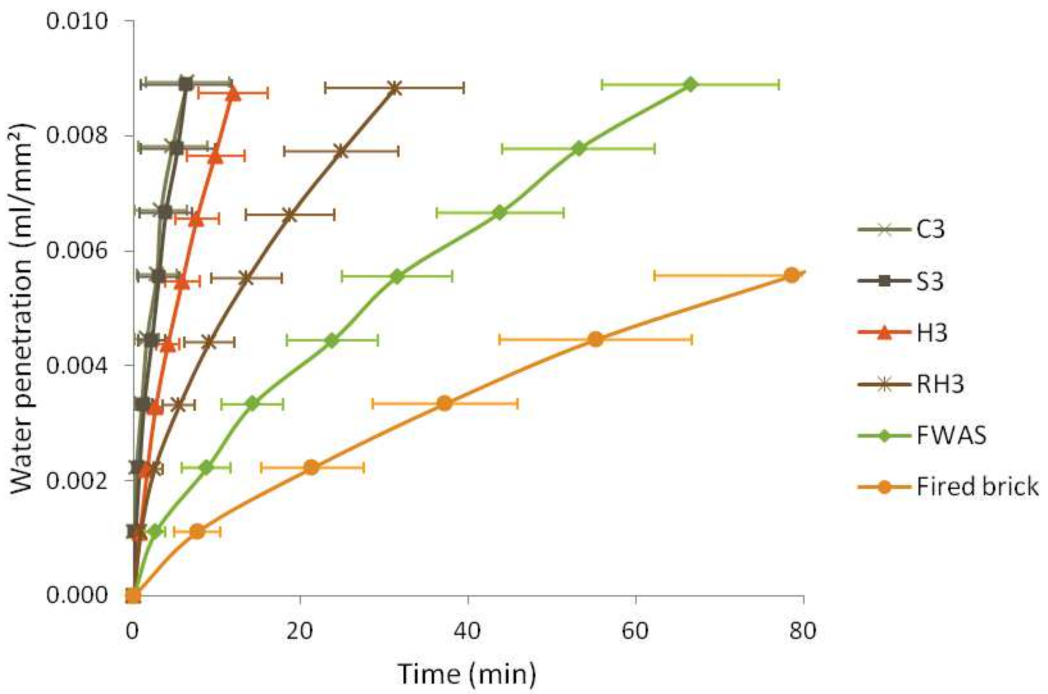

Figure 4-8. Absorption curve and absorption coefficient under low pressure of the blocks

The first striking result of Figure 4-8 is that absorption velocities are very different according to the materials tested. The time to absorb the same volume of water is much shorter for bio-sourced blocks than for the earth alone blocks and fired bricks. The total amount of water, 4 ml, is absorbed in 6.4±5.4, 6.5±5.0, 11.9±4.2, 31.2±8.3, 66.5±10.5 and 152.0±19.5 minutes for S3, C3, H3, RH3, FWAS and the fired bricks respectively.

The increase in absorption rate, or liquid water permeability, of bio-sourced blocks is due to the presence of the plant aggregates and their high absorption capacity [49]. This absorption rate, corresponding to the slope of the curves and expressed by the absorption coefficient C_A, varies considerably among the mixtures (Table 4-3): from 0.79 $g.m^{-2}.s^{-1}$ for fired bricks to 22.03 $g.m^{-2}.s^{-1}$ for C3. C_A can then be generally related to the density of the composite (Table 4-2). The lower the density, the higher the water absorption capacity. Nevertheless, the hydrophobicity of cork is highlighted by these measurements: the absorption capacity of C3 and S3 mixes is equivalent, although C3 is significantly less dense than S3. With a water absorption coefficient of 3.82 $g.m^{-2}.s^{-1}$, RH3 is the earthen block with plant aggregates that is most interesting in terms of durability to liquid water. Its coefficient is quite close to that of FWAS, which is 1.85 $g.m^{-2}.s^{-1}$.

These results can be compared with some absorption coefficients obtained by Stazi et al. [53]. A coefficient of 2.33 $g.m^{-2}.s^{-1}$ was obtained for a basic plaster with barley straw, 1.59 $g.m^{-2}.s^{-1}$, and 0.50 $g.m^{-2}.s^{-1}$ for a rammed earth wall. Nevertheless, in that study, the barley straw content was much lower than the 3% by mass used for plant aggregates in the present study, which could explain the huge difference between the absorption rates of this plaster and of the S3 blocks. The absorption rate of the FWAS blocks is quite close to that of the basic plaster of Stazi et al. [53]; some differences can be due to the difference in clay content, for example. A high clay content can reduce liquid water permeability [53]. Moreover, the type of clay could also influence the water permeability. Another comparison can be made with a study by Faria et al. [59]: the absorption observed in the present study is lower than for the rammed earth of Faria's study, which absorbed around 15 mL of water in 5 minutes. Here, the maximum for S3 was 4 mL in 6 minutes. However, the tests were performed with the Karsten tubes applied horizontally and vertically, respectively, and that may also have introduced differences.

At the end of the test, each part of the blocks in direct contact with water, except for fired bricks, had changed into mud. This behavior has already been observed by Mattone et al. [49] from the sixth minute of the test.

Table 4-3. Absorption coefficient C_A for each formulation

References	C_A ($g.m^{-2}.s^{-1}$)
FWAS	1.85
S3	21.98
H3	10.44
C3	22.03
RH3	3.82
Fired brick	0.79

3.4. Erosion resistance

The Geelong test is proposed especially for adobe blocks possibly containing straw. It is thus a suitable method to assess the resistance to wet erosion of the various blocks. As it is an aggressive test, it simulates accidental circumstances occurring mainly when the earth blocks are not rendered.

The results of the erosion test are shown in Figure 4-9.

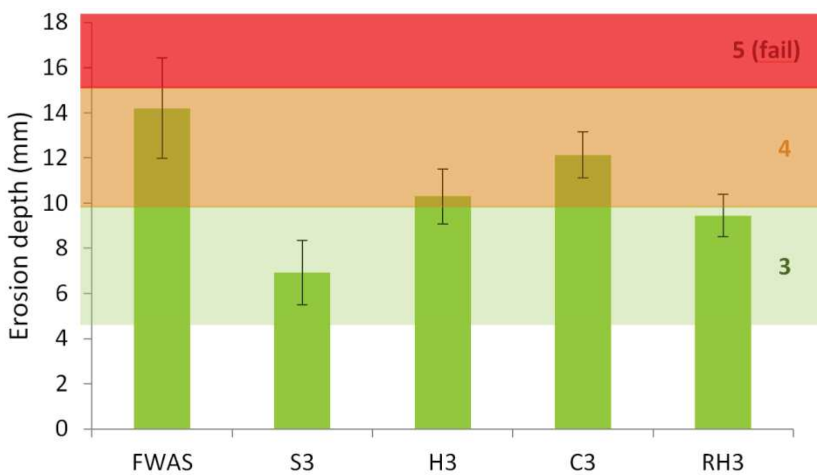

Figure 4-9. Erosion depth of the blocks after the Geelong test

In Figure 4-9, the erosion depth is presented for each mixture and the erodibility index has been added so that the class of erosion of the blocks can be seen directly. The least erosive block is S3, with a pit depth of 6.9±4 mm, followed by RH3 with 9.4±0.9 mm, H3 with 10.3±1.2 mm, C3 with 12.1±1.0 mm and FWAS with 14.2±2.2 mm. According to the erodibility index, S3 and RH3 specimens are class 3, which is erosive according to Frencham [62] (cited in [63]), and H3, C3 and FWAS blocks are class 4, very erosive. According to the New Zealand standard [43], all these formulations are suitable for adobe blocks for building constructions.

For each test performed, all the water dripped was absorbed by the block. The higher resistance with an addition of plant aggregates was also observed by Ashour and Wu [64] with barley straw, wheat straw and wood shavings. According to Ashour and Wu [64], the improvement of the resistance could be attributed to a uniform distribution of water by the particles. Erosion depth was also measured by Bui et al. [65] on walls exposed to natural weather. For unstabilized earth walls, the average depth of erosion was 6.4 mm. This is below the values of this study, but it was experimented in the south-east of France, where rainfall is low.

However, this test is appropriate if the rainfall is less than 500 mm per year [63]. In France, rainfall is between 800 and 900 mm per year on average, which is considerably higher. In that case, Frencham [62] recommends considering the category of erosion to be one or two classes higher. A protective coating can also be envisaged.

3.5. Sphere impact test

This test was very quick and easy to perform. The impact diameters on the blocks tested are presented in Figure 4-10. However, the qualitative behavior and aspect of the blocks after the test also give very interesting information. The occurrence of cracking, observed visually, denotes a lower deformability, which is negative, while the capacity to recover shape indicates resilience, which is very positive. Pictures of the blocks after the test are shown in Figure 4-11. C3 results are not presented here because diameter was non-measurable and the samples were too damaged (crumbled). Nevertheless, higher resilience, in comparison with other blocks, could be observed in the surface of C3 blocks after the strong impact.

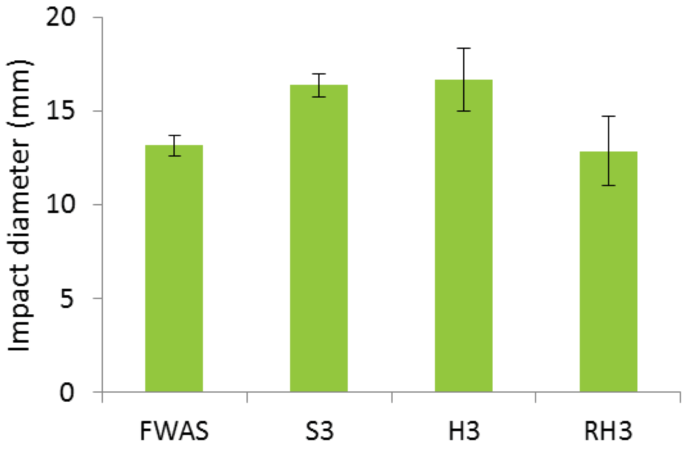

Figure 4-10. Impact diameter on the blocks

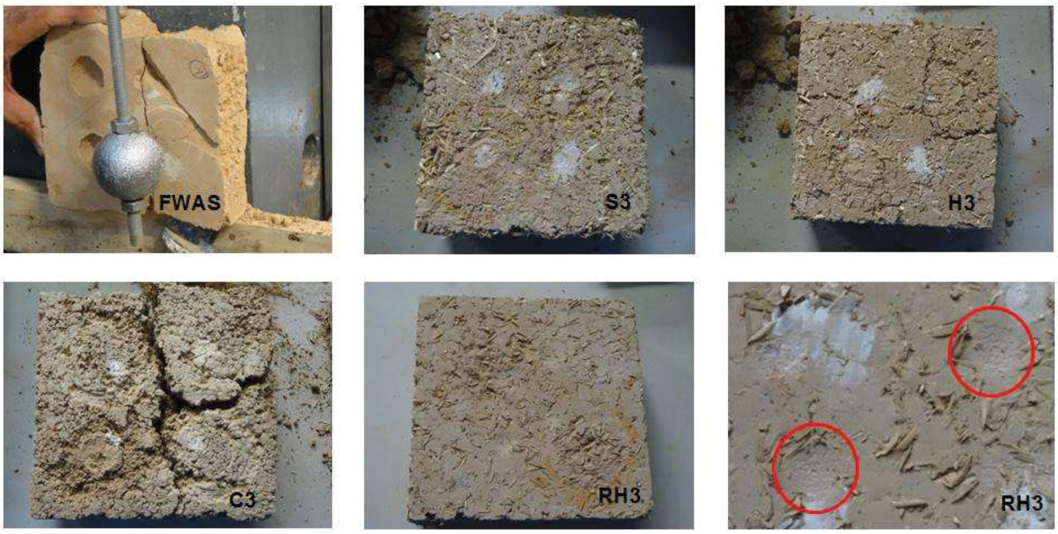

Figure 4-11. Blocks after sphere impact test

As can be seen in Figure 4-10, impact diameters were 13.2±0.5, 16.4±0.6, 16.7±1.7 and 12.9±1.8 mm respectively for FWAS, S3, H3 and RH3 blocks. RH3 blocks, with the lowest impact diameter, were the most resistant material, but with some deformability: there were no cracks, even after 3 or 4 impacts on the same sample. S3 specimens had the second highest diameter but the impact was hardly discernible and there were only small cracks, except for a bigger one in one block. H3 specimens showed a huge contrast in behavior after impact: one sample was totally broken, another one had a big crack and the last one had only a small crack. The worst resistance was shown by FWAS specimens, which were very brittle. Each time, the sample was cracked or broken after the first and only impact.

It is difficult to compare the results of this test with other results because, in the literature, superficial hardness is usually measured with a durometer or a sclerometer [59,66].

3.6. Discussion

Four plant aggregates were tested as components of earth blocks: barley straw, hemp shiv, cork granules and rice husk. Their influence on ultrasound velocity and durability properties was studied. The main results are synthesized in a radar chart (Figure 4-12) that indicates which formulation is the most interesting (100%) or least advantageous (0%) according to a certain property: ultrasound velocity, dry abrasion, water absorption, erosion depth or impact resistance. The intermediate outcome, which is a percentage, was calculated proportionally to the best and the worst value. For ease of understanding, the ranking of C3 specimens has been removed from the diagram. This was the worst formulation in each category except for erosion depth, where the FWAS formulation was the worst, followed by C3 (ranking 29%). For the impact behavior, the values are arbitrary, depending only on the material behavior and not on the diameter of the impact. The material behavior after an impact is a more defining factor.

The properties presented in the chart can be distinguished as follows: ultrasound velocity represents a use property whereas the other four belong to the durability properties.

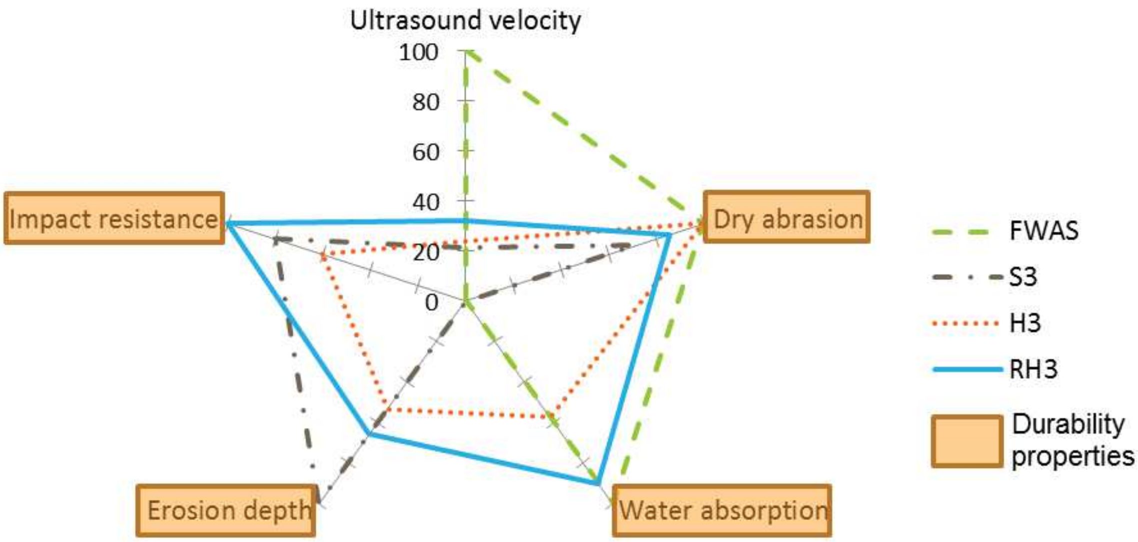

Figure 4-12. Diagram comparing the various formulations qualitatively according to the property

Choosing one formulation rather than another requires a compromise among the different properties. The strengths and weaknesses of the different block formulations are summarized below.

- Blocks without plant aggregates (FWAS) present the highest ultrasound velocity, or stiffness, but this property leads to very low impact resistance: blocks are easily breakable. This formulation is the best for water absorption resistance (Karsten tubes) and for dry abrasion resistance (equal to H3 specimens). However, it should be noted that dry abrasion results are very close to each other for all the materials (bearing in mind that Figure 4-12 does not take the uncertainties into account). FWAS specimens are the least resistant to erosion (Geelong test).

- Blocks with barley straw (S3) are resistant to erosion and to impact. Their dry abrasion resistance is less than that of FWAS, H3 and RH3 but is still competitive. However, their stiffness and water absorption resistances are very low.
- Blocks with hemp shiv (H3) present a behavior quite close to S3's, with a slightly better dry abrasion resistance, better resistance to water absorption but lower resistance to erosion.
- Blocks with rice husk (RH3) present the best compromise. Their behavior is very good with respect to each durability property although stiffness is much lower than for blocks made of earth alone. RH3 stiffness is, however, the highest of the blocks with plant aggregates.
- For the tested properties, the worst overall behavior is reached by the blocks with cork granules (C3). There is not enough cohesion of the material and the samples are very brittle. Cork should not be used in an earth block for a building application unless the formulation or manufacturing process is optimized. A binder could be added, as was done, for example, by Bachar et al. [37] and Brás et al. [41] with cement. A surface treatment of the cork could also be considered in order to decrease the hydrophobicity of this material and improve its adhesion with the earth matrix.

The relevance of some tests can also be discussed. For example, the Karsten tubes would be more representative of rainfall if the test was performed on a vertical sample (equivalent to a wall). However, the tube with the sample is delicate to seal, in particular with samples containing plant aggregates. The ultrasound velocity test, used to evaluate the stiffness or the damage caused by freezing-thawing cycles in some studies, is easy to perform and has the advantage of being non-destructive. However, no correlation could be found in the present investigation between the measured velocity and the durability properties.

Other drawbacks of the tests can be pointed out. The analysis of the impact resistance test could be improved: the impact diameter, quite difficult to measure, is also not sufficient to describe the behavior after impact. The impact behavior alone is very subjective. Concerning dry abrasion resistance, the test is quite useful for comparing results within one study but it becomes complicated to obtain a "real" value, for comparison with those of other studies. As mentioned in the results section, the material removed during the test depends on the hardness of the brush. Nevertheless, impact resistance, dry abrasion and erosion resistance tests are very representative of real potential conditions.

4. Conclusion

The number of studies published about unfired earth products with plant aggregates or fibers is increasing year by year. They focus mainly on mechanical properties and, to a lesser extent, on hygrothermal properties. However, once the use properties are defined, it is of major importance to investigate the durability of the material. Thus, the present study was intended to characterize some durability properties of earth blocks containing one of the following plant aggregates: barley straw, hemp shiv, cork particles and rice husk.

From a durability point of view, the tests have shown compatibility between earth and straw, hemp shiv or rice husk. However, the formulation containing rice husk (RH3) seems to be the most interesting: it presents a good result for each durability property tested; it is thus the best compromise. Deeper knowledge of rice husk composite, especially concerning its mechanical resistance, fracture behavior or hygric buffering ability would allow an interesting sustainable product to be developed.

The aim of the study was to investigate various test procedures to assess the durability of bio-based earth materials. As discussed before, the tests presented here are sometimes difficult to adapt to bio-based materials, such as the sealing of the Karsten tubes, and the relevance is not obvious for all of them. Other existing tests could be performed on earth materials. For example, the German Standard proposes different protocols for earth blocks in DIN 18945 [42]: the capillary ascent of water, the immersion test and the contact test. The tests proposed in DIN 18945 are interesting because they could be used on unstabilized earth blocks; it would also be interesting to test these procedures on earth blocks containing plant aggregates to study the relevance of these tests with such materials and to use other tests to evaluate the effect of plant aggregates on the resistance of earth blocks to water. These new tests could be usefully compared with those described in this paper. Another perspective that would merit investigation is the effect of the plant aggregate content and its casting.

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Fire behavior of bio-based earth products for sustainable buildings

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Abstract

Construction is one of the most polluting industrial sectors. For this reason, developing sustainable building materials is a world-wide interest. Earth as a building material is thus increasingly studied because of its low environmental impact and its ability to regulate indoor moisture and to improve occupant's comfort. Moreover, earth is a well-known non-combustible material. Recent studies deal with unfired earth bricks with plant aggregates incorporated to the earth matrix in order to lighten and improve various properties of the composite materials. However, these vegetal additions, of combustible nature, raise questions about the fire-behavior of the bio-based material. To our knowledge, the fire-behavior of this kind of materials has been little investigated in the literature.

The present paper constitutes a preliminary study about the fire reaction of earth bricks containing 0%, 3% and 6% weight content of barley straw or hemp shiv. A pyrolysis combustion flow calorimetry (PCFC) was done to predict the fire-behavior of the plant aggregates. Other tests were performed on the composites to determine their flammability, their thermal insulation and their mechanical strength after high temperature exposure.

The following conclusions were reached. The PCFC test has shown a peak of heat release rate around 350°C for the plant aggregates, which corresponds to the degradation of the cellulose. The ignition-extinction test has proved that the material is still non-flammable even with vegetal additions. Thermal conductivity of the composites decreased when the experienced temperature increased (until 800°C) due to the higher porosity. The higher the plant aggregates content, the more fire resistant was the material, meaning that the rise of temperature was delayed. Concerning mechanical performance, a strength drop was observed for composites around 400°C before a slight increase until 800°C thanks to the firing of the earth.

Keywords

Bio-based building material; Earth bricks; Fire resistance; Barley straw; Hemp shiv

1. Introduction

Sustainable construction is a major concern. In the building sector, a growing interest has been observed during the last decades within ecological materials, namely renewable, energy efficient or healthy.

Earth-based material is therefore part of a sustainable solution. This material, increasingly studied, is abundantly available, recyclable and requires little energy for the transformation process [Minke, 2006]. It is moreover able to regulate indoor moisture and presents a good thermal inertia. Some properties such as thermal insulation or mechanical strength can however be improved by adding some plant fibers or aggregates in the earth matrix [Laborel-Préneron, 2016].

Developing a « new » material requires to characterize its basic properties. The verification of the safety and comfort of the building's user is also necessary. Some hazards may indeed occur on a building that can lead to reconsider these two criteria. It is the case, for instance, in a fire situation. The fire risk is high in a dwelling with various factors triggering such as electricity networks, chimney fires or lightning strike. Consequences can be severe, notably in case of collapse, if the resistance to fire of the materials was not adapted.

The material is suitable for building construction if it prevents fire from spreading and if the stability of the construction is ensuring enough time to potentially evacuate the occupants. Fire safety regulations vary from country to country, but they are generally based on international standards such as ASTM E119 [ASTM, 2000] or ISO 834 [ISO, 2014]. In Europe, the standard Eurocode 6 - EN 1996-1-2 [CEN, 2005] focuses on the fire design of masonry structures. For testing fire resistance, the European standards EN1363-1, EN 1364-1 and EN 1365-1 [CEN, 1999a, 1999b, 2012] are used.

These regulations request that the tests to assess the load-bearing capacity are realized at actual size (on walls for example). Still, this preliminary study is realized at small scale to estimate the fire behavior of this type of material, very poorly studied in the literature. The earth, as a mineral material, is a non-combustible material. However, adding straw or other vegetal particles, which combust readily, can raise some issues. According to the German Standard DIN 4102-1 [German Standard, 1977], even with some straw content, as long as the earth-based material is higher than 1700 $kg.m^{-3}$, the material is still considered as non-combustible. The present study focusing on adding high vegetal aggregates content in the sample, it is thus important to evaluate the fire behavior of such materials, which bulk densities are lower than 1700 $kg.m^{-3}$.

Few studies exist on the fire behavior of bio-based earth materials. Buson et al. showed a good fire resistance of walls made of compressed earth blocks of soil-cement and cellulose pulp derived from the recycling of cement sacks [Buson, 2013]. The characterization of straw and earth to fire was also realized by [Apte, 2008]. The effect of high heat sources on two straw bales rendered by earth or lime of 45 and 43 mm thick respectively was studied. When the material was subjected to a heat of 30 $kW.m^{-2}$ during 30 minutes, it resulted in little damage to the render layer and no combustion of the straw bale. However, when subjected to 50 $kW.m^{-2}$ during 40 minutes, the combustion of the straw was initiated 24 hours after the end of the exposure regime. The render has played the role of a barrier to oxygen transfer into the straw.

Fire behavior studies of bio-composites often focus on insulated materials, mostly composed of organic matter, such as rice husk with alginate for example [Palumbo, 2015, 2017]. It also deals with polymer composites with natural fibers. The effect of a fire retardant on a bio-composite of polybutylene succinate with natural fibers was studied in [Dorez, 2013]. The fibers decreased the thermal stability but the increase in mass residue led to the creation of a char barrier. Fire behavior of another type of bio-based material, made of cellulose derivatives and starch matrix and sisal fibers was investigated by [Alvarez, 2004]. It seemed that the addition of fibers did not influence a lot the fire resistance of the material.

In this research, various tests were realized in order to evaluate the fire reaction and fire resistance of an earth-based material containing 0%, 3% or 6% by mass content of straw or hemp shiv. First, small-scale flammability tests were carried out on the plant aggregates only. Other thermal degradation tests were realized on the bio-composites to evaluate their flammability, their residual thermal insulation and compressive strength after a high temperature exposure. These last two tests are part of the fire resistance characterization, composed of three criteria (stability, insulation, sealing) while the two firsts define the fire reaction, indicating the way the material behaves as a combustible.

2. Material and methods

2.1. Materials and manufacturing

Fines from limestone aggregate washing process (FWAS) were used for this investigation. These fines have a high proportion of limestone (around 60%) and only around 20% of clay. Barley straw and hemp shiv were also tested in the earth matrix in different proportions: 3% and 6% by dry mass. The manufacturing water content was determined with the Proctor test. Formulations of the different types of samples are recapitulated in Table 4-4.

To manufacture the specimens, earth and plant aggregate fractions were poured into a blender and mixed by hand. Water was then added and the materials were mixed mechanically in the blender until a homogeneous mix was obtained. The raw materials were mixed the day before molding.

The prismatic samples of 15 x 15 x 5 cm^3 were manufactured by static compression. They were then dried at 40°C for 24 hours followed by an increase in temperature until 100°C at a rate of 0.1°$C.min^{-1}$. This temperature was finally kept constant until the weight became constant (weight variation less than 0.1% between two weighings 24 hours apart).

For purposes of the different tests, the samples were cut with a circular saw into different sizes:

- 7.5 x 7.5 x 5 cm^3 for ignition time and extinguishability and thermal conductivity tests
- 5 cm cubes intended for compressive strength test
- 15 x 10 x 5 cm^3 for the fire resistance test

Table 4-4. Mixture proportions and density of the samples

Ref.	FWAS	S3	S6	H3	H6
Aggregate	-	Straw	Straw	Hemp	Hemp
Aggregate content (%)	0	3	6	3	6
Water content (%)	14	19	21	17	20
Dry density ($kg.m^{-3}$)	1988	1520	1195	1553	1190

2.2. Pyrolysis Combustion Flow Calorimetry

Small-scale flammability was characterized with a Pyrolysis Combustion Flow Calorimeter (PCFC) from Fire Testing Technology. Some fire reaction properties were predicted thanks to this device. In that experiment, thermal analysis was combined with oxygen consumption calorimetry. The temperature range was between 100°C and 750°C and was increased at a rate of $1°C.min^{-1}$. Products from the anaerobic thermal degradation completed in a nitrogen atmosphere were mixed with a 20 $cm^3.min^{-1}$ stream of oxygen prior to entering the combustion furnace at 900°C. Heat release rate (HRR) was then determined from the oxygen consumption. The measurement was conducted on two specimens of each type of plant particle which mass was around 5 mg (balance at one-hundredth milligram).

2.3. Ignition time and extinguishability

This fire reaction test, based on the Spanish standard UNE 23-725 [AENOR, 1990] and its French counterpart NF P92-505 [AFNOR, 1995], highlights the flammability of the material. The test protocol involves placing the sample below a radiator that act as a source of heat of around 500 W. The radiator has to be removed if the sample undergoes an ignition and replaced when the flame is extinguished. The parameters determined are the first ignition time, the number of ignitions and the average value of combustion extent during the first 5 min of assay. In this case, only one test was performed on each formulation.

2.4. Thermal conductivity

This test provides information about the insulation aspect of the material fire resistance. Specimens were heated up at various temperatures until 800°C following the temperature curves of Figure 4-13. Their residual thermal conductivity was measured with a thermal properties analyzer Quickline-TM 30 Anter Corporation, which uses a dynamic method of measurement. Thermal conductivity values were measured the day after their exposure to high temperatures. The size of the specimens tested in that study was 7.5 x 7.5 x 5 cm^3.

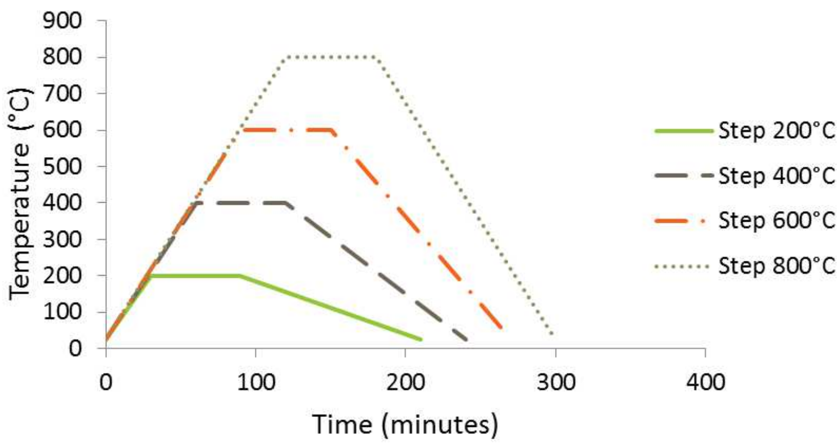

Figure 4-13. Temperature curves

2.5. Compressive strength

Measuring compressive strength allows determining the fire resistance of a material and more specifically its stability. Compressive strength of 5 cm cubic specimens was measured after high temperature exposure. The four temperature steps are illustrated in Figure 4-13. This test was carried out by means of a hydraulic press Wyhehan Farrance Ltd. 5000 kg the day after exposure. The load was applied at a constant deflecting rate of 3 $mm.min^{-1}$. Two to three specimens for each formulation were tested.

2.6. Temperature of the unexposed face test

This test is part of the fire resistance characterization and identifies the capacity of a material to prevent heat spreading. One face of a 15 x 10 x 5 cm^3 sample was exposed to high temperatures. This face was directly submitted to an increase in temperature: 2 hours of increase until 800°C then 2 hours stable at this temperature. The other face, unexposed, is stuck out of 1.3 cm in a room at 25°C. The temperature was measured by a thermocouple inside the oven and 2 other thermocouples were in direct contact with the middle of the unexposed face. Lateral sides of the sample outside the oven were thermally insulated with mineral wool (Figure 4-14). The heat flow is thus considered one-dimensional.

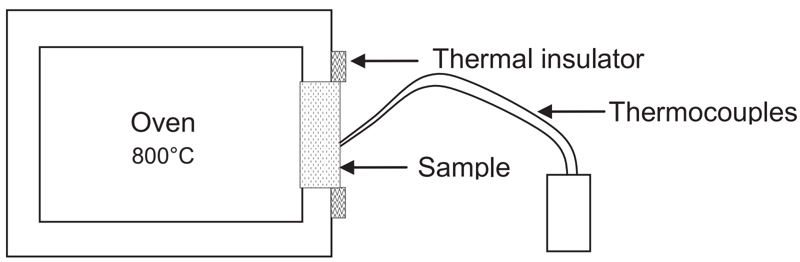

Figure 4-14. Temperature of the unexposed face

3. Results and discussion

3.1. Pyrolysis Combustion Flow Calorimetry

Two tests for each type of plant aggregate were realized: barley straw (Straw1 and Straw2) and hemp shiv (Hemp1 and Hemp2). The Heat Release Rate (HRR) is plotted as a function of the temperature in Figure 4-15. A single peak of heat release is observed around 350°C for each vegetal particle type. This corresponds to the thermal decomposition of the cellulose [Alvarez, 2004]. A significant gap can be noticed between the two measurements of the hemp shiv which does not allow concluding about a precise value. The result of a PCFC test on barley straw published by Palumbo et al. is also plotted [Palumbo, 2015]. It can be observed an important difference between the two straws: the straw studied in Palumbo et al. presents a lower HRR. The results could be influenced by the chemical composition of the particle type or the moisture content.

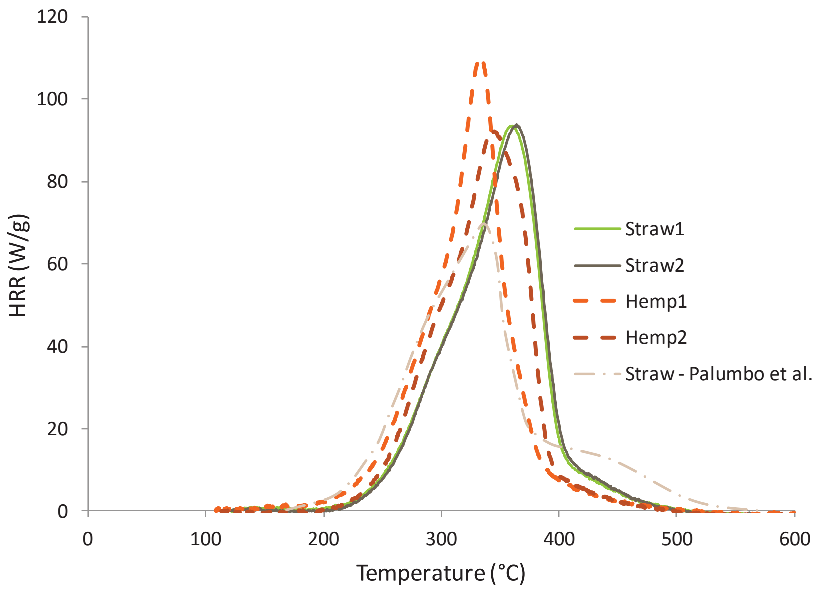

Figure 4-15. Heat Release Rate as a function of temperature for the plant aggregates

Other parameters can be deduced from this test. They are recapitulated in Table 4-5.

- The total Heat Release (HR), corresponding to the integral of the Heat Release Rate
- The temperature of start combustion (T_0), which is considered from a HRR of 10 $W.g^{-1}$
- The temperature corresponding to a maximal HRR (T_{max})
- The Peak of Heat Release Rate (PHRR)
- The mass loss fraction during the combustion (M_{loss})

Results are very similar between straw and hemp, with a total heat release very close to each other. However, the temperature of the degradation of the particle is slightly lower for hemp than straw and the peak of HRR is slightly higher for hemp but this is due to the scattered results of the hemp particles. The mass loss fractions of the two particle types are very similar.

Table 4-5. PCFC results for the plant aggregates

Raw material	HR (MJ/kg)	T_0 (°C)	T_{max} (°C)	PHRR (W/g)	M_{loss} (-)
Straw	8.3	255	362	93.8	0.83
Hemp	8.2	244	339	101.3	0.81

3.2. Ignition time and extinguishability

During the test, no flame was observed, even on samples containing plant aggregates, which is highly positive. The whole protocol described in the standard was thus not adapted to this type of material. However some observations could be made. The smoldering of straw and hemp shiv on the surface of the specimen occurred quickly at the beginning of the test, emitting smoke. The higher was the plant aggregate content, the higher was the smoke release. After removing the sample from the heat source, its surface was black (Figure 4-16), but after cooling it became slightly red, as fired bricks are. The cooling of bio-sourced samples occurred faster than earth alone samples.

Figure 4-16. S6 sample after ignition time test

3.3. Thermal conductivity

Vegetal matter is decomposed by high temperatures and the microstructure of the material is modified. Thermal conductivity is impacted by such transformation. After a fire, it is thus interesting to know the evolution of the material properties. From 400°C and higher, the cohesion of S6 samples is declined, when the straw is burnt. All the other formulations were kept cohesive.

Thermal conductivity for each formulation is displayed in function of the temperature experienced from 25°C to 800°C in Figure 4-17 and the evolution of the sample mass in function of the temperature is shown in Figure 4-18.

Thermal conductivity is decreased when the experienced temperature is increased. For the first stage, it can be explained by the evaporation of the water adsorbed by the material which had not sufficient time to re-adsorb much water. Between 200°C and 400°C, the decrease in thermal conductivity seems to be due to the decomposition of the vegetal matter which occurs around 350°C, generating an increase in porosity and thus a lower density. However, this decrease is not much important in comparison with the earth alone sample. This can be explained by the kinetics of the degradation process, which might be longer than the time of the stage temperature. Between stages 400°C and 600°C, the sample mass is quite stable, however thermal conductivity is still decreasing. It should be due to the deshydroxylation of the clay which is the loss of the constituting water OH and the transformation in metakaolin. An important decrease in thermal conductivity takes place between 600°C and 800°C, which can be linked to the decarbonation of the material.

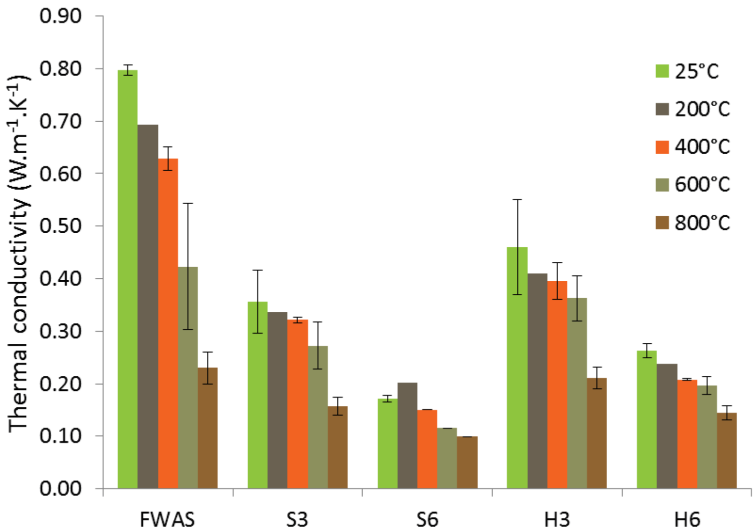

Figure 4-17. Thermal conductivity according to the experienced temperature

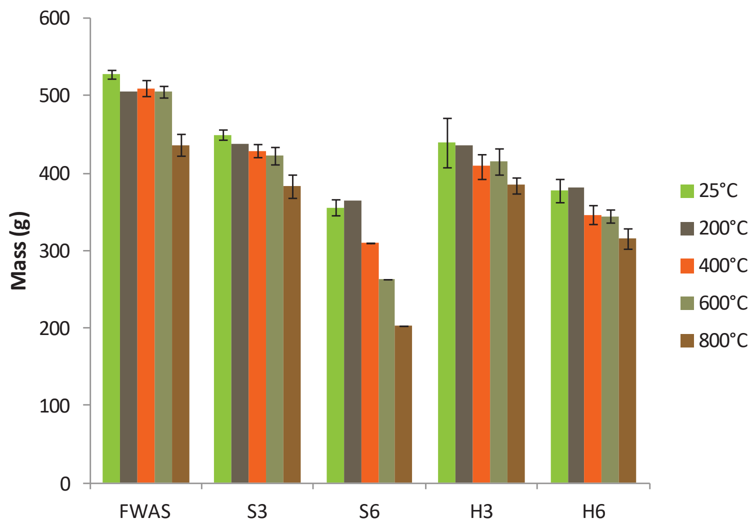

Figure 4-18. Sample mass according to the temperature experienced

3.4. Compressive strength

After a fire, the resistance of a building should be maintained. It is thus important to verify if the physical-chemical changes of the material lead to a high decrease in strength.

The results are presented in Figure 4-19. Between 25°C and 200°C, no real difference in strength is observed, standard deviations being quite important. From 400°C and higher, the effect of the temperature on compressive strength is different whether it is earth alone or earth with plant aggregates. For earth alone samples (FWAS), the compressive strength is increased at 400°C and 600°C (6.8 MPa and 14.6 MPa respectively) but is slightly decreased at 800°C (10.8 MPa). However, standard deviations are important. The increase of the resistance could be due to the transformation of kaolinite into metakaolinite between 460°C and 600°C, 550°C according to [Cultrone, 2001].

For samples containing vegetal matter, no significant difference is observed between the samples subjected to room temperature and samples which experienced a temperature of 200°C. Standard deviations are also quite important. At 400°C, the resistance decreased significantly due to the degradation of the vegetal matter around 350°C as shown by the PCFC results. This drop in strength increases with the amount of vegetal particles. Between 400°C and 800°C, the compressive strength progressively increases keeping however a strength value lower than the initial one. This increase may be linked to the firing of the earth, engendered by the loss of the constituting water and its transformation into metakaolin.

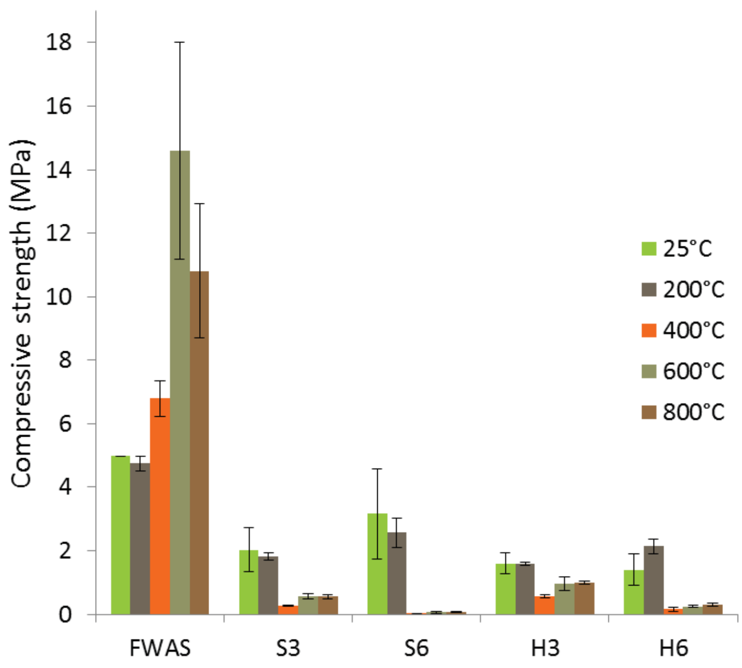

Figure 4-19. Compressive strength according to the experienced temperature

3.5. Temperature of the unexposed face test

Fire resistance can be characterized with an insulation criteria such as the one determined thanks to the test presented hereafter. A material is considered to protect against fire when a fire-like increase of the temperature induces a temperature increase of the unexposed face lower than 180°C measured with either thermocouple.

Temperatures of each face of the samples in function of the time are presented in Figure 4-20. The temperature programmed for the earth alone sample is slightly different from the others, the temperature step of 800°C lasting only 30 minutes instead of 2 hours for the other formulations.

It can be observed that the higher is the vegetal aggregate content, the better the materials seems to insulate from high temperatures. This result is in good agreement with the thermal conductivities previously obtained (Figure 4-17). There is a delay and a decrease of the temperature rise with the addition of 6% of plant aggregates.

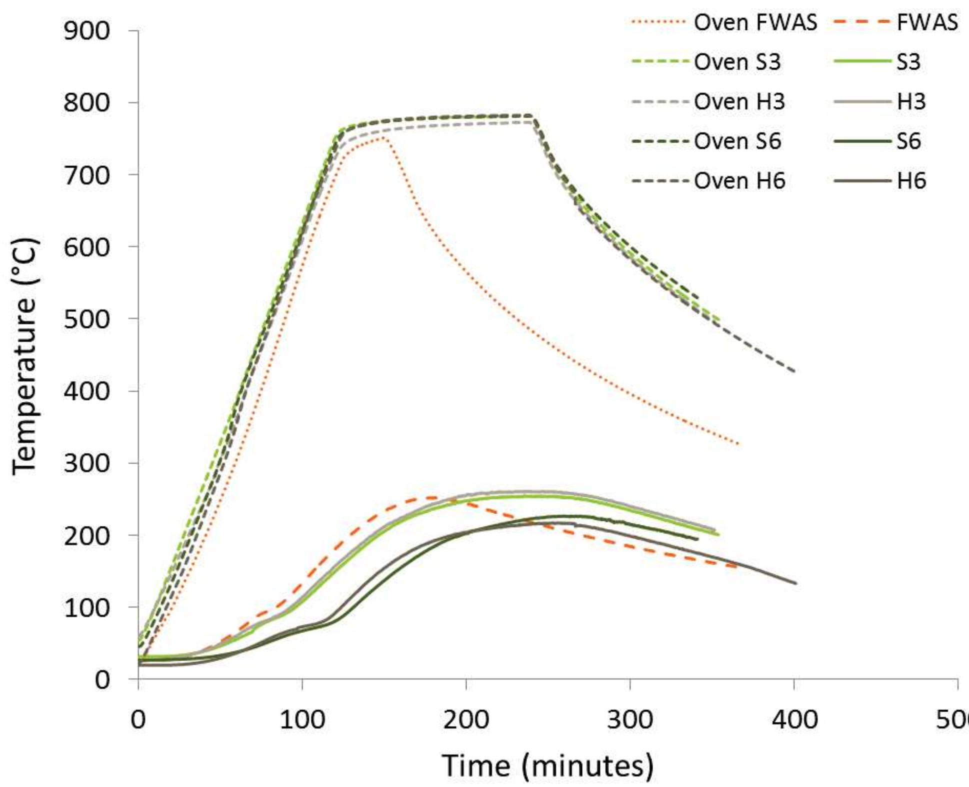

Figure 4-20. Exposed and unexposed face temperatures in function of time

The temperatures of the unexposed face measured in the worst case scenario are shown in Table 4-6. The increase in temperature ΔT is calculated thanks to the initial temperature T_0 and the maximal temperature reached T_{max}. The time in minutes that a sample could maintain an increase in temperature lower than 180°C is also displayed (Time ΔT < 180°C).

Table 4-6. Unexposed face temperatures

Ref.	T_0 (°C)	T_{max} (°C)	ΔT (°C)	Time (min) ΔT< 180°C
FWAS	26.4	252.3	225.9	132
S3	32.0	255	223.0	154
H3	25.8	261.2	235.4	145
S6	27.1	226.8	199.7	206
H6	20.3	217.3	197.0	191

For the different sample formulations, the rise in temperature is always higher than the required value of 180°C (between 197°C for H6 samples to 235°C for H3 samples). However, this required value is relative to a standard heating curve presented in the ISO 834-1 [ISO, 2014] which prescribe a temperature of 739°C at 15 minutes and 1049°C at 120 minutes. The heat curves of the current test are then less aggressive than the standardized ones. The conclusions about the increase in temperature drawn must then be taken carefully. The samples containing 6% of plant aggregates are the best thermal insulators, with temperature rises of 197°C and 200°C, which is quite close to the standard EN 1363-1 [CEN, 1999a]. The samples with plant aggregates maintained the unexposed face temperature lower than 180°C for longer time: higher than 3 hours for S6 and H6 samples against around 2h30 for S3 and H3 samples and 2h10 for a FWAS sample.

However, this increase in temperature is influenced by the material thickness, which is around 5 cm in this case of study. Whether it is a material used as a partition wall or a load-bearing wall, the thickness would be higher, meaning that the temperature rise would be lower. Other tests are necessary to evaluate that rise in the case of a thicker material.

4. Conclusion and outlook

The effect of straw and hemp shiv additions on the fire reaction and resistance of an earth matrix has been analyzed.

These results show the tendency of the bio-based material to not ignite itself. Although the PCFC test has shown a peak of heat release rate around 350°C for both plant aggregates, corresponding to the degradation of the cellulose, ignition-extinction test has proved that the composite material is still non-flammable. The smoldering is present only on the plant particles, making the material more porous and so a better thermal insulator. However, compressive strength of earth containing plant aggregates is decreased when the material is submitted to high temperatures.

Tests were realized during a short period, the experiments realized shall be repeated to reduce the spread of the results. It also should be interesting to extend the tests to a larger scale, at least to the brick size.

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Article G

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Assessment of fungal growth on construction materials based on experiments achieved with earth-based materials containing plant aggregates

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Abstract

As people spend more than 80% of their time indoors, the impact of buildings on the health of their occupants is a major issue. For example, diseases can be caused by mold, already present in a large number of dwellings. When developing a material, the sensitivity of the material to microbial growth should be assessed to avoid such problems. Fungal growth depends on environmental factors such as relative humidity and temperature, and also the nature of the substrate in the present case, the material. However, few studies have focused on this topic and no international standardized method exists to investigate fungal growth on building materials. The objective of this paper is thus to adapt a method from those already proposed in the literature in order to evaluate mold proliferation in a material laboratory. Fungal growth on earth material containing plant aggregates (namely straw and hemp shiv) was investigated under different climate conditions. The samples were inoculated with a strain of *Aspergillus brasiliensis* and were incubated for 12 weeks at 76, 84 and 93% of relative humidity and at temperatures of 30°C and 20°C. The influence of the straw on the microbial resistance was demonstrated: earth with straw was more sensitive to fungal growth than earth alone. Fungal growth took place after 4 weeks of exposure at 93% of relative humidity and 30°C. For the earth with no plant aggregates, the mycelium was observed for the first time after 8 weeks of incubation under the same conditions. Concerning the methodology, some improvements are suggested and image analysis appears to provide a promising lead for quantifying mold growth, although further work is required.

1. Introduction

Objectives in the development of construction materials are often multiple, for example, to obtain satisfactory mechanical and thermal properties simultaneously. The objective is not necessarily twofold, and other objectives can be defined, such as fire safety and occupants' health, visual aspects or durability. Among the potential objectives, mold risk has been a focus of attention recently. There are two possible reasons for this. First, buildings are being built to be increasingly airtight, which limits unwanted air infiltrations but also leads to increased level of indoor relative humidity, thus increasing mold risk. Second, numerous research projects and recent constructions are considering bio-based materials, most of them containing plant aggregates. These are claimed to be healthy and increase the indoor comfort of the occupants [1] but it is commonly believed that the use of plants would lead to an increased risk of mold growth. However, scientific proofs are rather scarce and, for this reason, more and more attention is being paid to mold growth on building materials [2-8], with applications of bio-based material becoming a topical issue. Some studies deal with wood-based or paper-based materials and also inorganic materials such as cement or gypsum plaster board [2],[8]. Fungal growth has also been studied in situ on straw bales with lime-based render for building envelopes [9]. Hoang et al. [3] have shown that bio-based materials are more sensitive to fungal growth because of the presence of nutrients and their high hygric capacity.

Additionally, it can be observed that the study of mold growth is not well established, at least in the field of construction materials. Therefore, no international standard methods exist, and current techniques are mostly adapted from practices in biology laboratories. Although such access to relevant devices has its importance, there are some significant differences in terms of purposes. When developing new materials, the objective is to compare a significant number of different samples quickly, before selecting the most relevant, which is characterized in more in detail in a second phase. As the situation stands now, mold growth is considered as a risk that should be avoided. To put it simply, the point of view in material development is that there are two possibilities for mold risk: the risk is acceptable or not. In biological studies, the point of view is different: the biological activity is continuous and influenced by multiple factors, one of them being the medium (the material). Also, many different species of mold are considered in biological studies, and some of them may interact. Finally, favorable conditions for growth differ from species to species. Therefore, it is hard to assess whether the risk of mold growth is acceptable for a given material.

In fact, this separation between material development and biological studies is not very strict and several studies have already been achieved on this topic. Indoor microbial growth on building materials was recently reviewed by Verdier et al. [10] who compared several methods for sampling and analyzing the proliferation of micro-organisms, and described the most common microbial communities and the building parameters. For the analysis of the results, a widespread method is the use of isopleths. An isopleth is a curve in a plane defined by temperature and relative humidity, which identifies the critical conditions for mold growth for a given material. This work was initiated more than a decade ago [11]. Also, the hazard classes of fungi (A or B/C) have been specified. Class A are highly pathogenic, not allowed in dwellings, and class B are allergenic or pathogenic in case of long exposure. Class C are not harmful to health, generating only economic damage. Other predictive models of proliferation are proposed in [2] and [4], still based on fungal growth tests on building materials. Isaksson et al. [5] studied pine sapwood under real climatic conditions, from which a predictive model was developed. However it was valid for pine only. More general models exist, such as the transient biohygrothermal model [11] and some are included in transient hygrothermal tools, such as ESP-r or WUFI. A comprehensive review and comparison of existing predictive models used at building scale are reviewed by Vereecken and Roels [12]. They conclude that every model relies on strong simplifying assumptions, which can lead to different results or analysis depending on the model used. However, this illustrates the interest in mold growth studies of the scientific community specializing in the field of building physics.

There is a clear need to move forward on this topic, one of the most obvious reasons being the absence of a suitable standardized protocol. This is one of the issues identified in the framework of the Bioterra project, a national collaborative project involving members from both materials and biological laboratories. This project is focused on earth-based materials, as they are assumed to favor mold growth and as limited research has been published on this topic up to now. Mold formation was observed on earth panels containing hemp shiv ten days after manufacturing in [13]. It was also observed after removal of the formwork of earth-straw walls in [14], particularly inside the building, where ventilation was less effective. The main objective of the project is to characterize the physical properties of earthen bio-based products [15],[16] as well as the proliferation of micro-organisms on these materials for various environmental conditions. The last target of the project is to propose solutions to limit the proliferation of these pathogenic micro-organisms. A first study on the micro-organism sampling method on raw materials and manufactured building materials is presented in [17].

In the present paper, fungal growth is studied on earth as the mineral matrix with the addition of two types of plant aggregates: barley straw and hemp shiv. The objectives are twofold. First, a critical discussion of the methodology used to assess mold growth is proposed, by considering the objectives of material characterization. To be more specific, the methodology mainly relies on microscopic observations on samples inoculated with a single strain. Samples were first sterilized by being exposed to high temperature, and then incubated at different constant hygrothermal conditions. The results will be analyzed to determine which samples present the highest risk for mold growth. The second objective is to give a more comprehensive understanding on the phenomenon of proliferation under such experimental conditions.

2. Short literature review on mold fungi in buildings

Indoor molds are microorganisms, most of which are members of the fungus family. The biodiversity of fungi is estimated at around 1.5 million species. Fungi are part of the eukaryotic organism group, meaning that a cell contains a nucleus. Fungi are known to be the main decomposers in ecological systems. Growth of fungi begins with the development from spores to hyphae, which are filamentous structures. The hyphae can be several centimeters long but only a few microns in diameter and so invisible to the naked eye. Several hyphae grow into an interconnected network called a mycelium. Their reproduction is asexual and occurs thanks to conidia, which are spores, borne by stalks called conidiophores.

Various species identified on indoor building materials have been listed by Verdier et al. [10]. The most frequently isolated were *Cladosporium*, *Penicillium*, *Aspergillus* and *Stachybotrys* genera. A correlation between the species and the type of material was investigated. *Penicillum* sp. were the most frequently found in all kinds of building materials. *Aspergillus* sp. were found on ceramic materials, paints and glues. *Stachybotrys* sp. were observed specifically on gypsum materials. Common species of these genera were *Cladosporium sphaerospermum*, *Penicillum chrysogenum*, *Aspergillus niger*, *Aspergillus versicolor* or *Stachybotrys chartarum*. Due to their allergenic or toxic effects, some of them were classified as potentially pathogenic species by the French High Council for Public Health [18]. It is estimated that 20% to 40% of the housing in Northern Europe and North America is impacted by indoor molds [18]. These molds produce Microbial Volatile Organic Compounds (MVOC), which are responsible for their unpleasant odor, and also, more importantly, spores and mycotoxins, responsible for various health issues [19]. The set of such health problems is known as the Sick Building Syndrome (SBS) or Building Related Illness (BRI). As people spend more than 80% of their time inside buildings [20], the impact is quite large. These pathogenic mold fungi have various health consequences. Sedlbauer lists three main diseases due to fungi [11]. Mycoses, usually caused by *Aspergillus* sp., *Cladosporium* sp., *Penicillium* sp. or other, can be dangerous particularly if the person is immunodeficient. Mycotoxicoses are intoxications due to toxic-active substances produced by fungi. Finally allergies can take different forms such as rhinitis, asthma, conjunctivitis, atopic eczema, etc. They are due to an excessive reaction of the immune system. Fungal growth can also have an impact on building materials, although this is more limited. Bio-fouling, expressed by discoloration, or bio-corrosion are of only minor importance [11],[19]. However, it should be noted that mycelium growth damages the material concerned by consuming the substrate, its source of nutrients [21].

Mold growth is highly dependent on various environmental factors, some of which have been identified as strongly influencing mold growth in most cases:

1. Water availability. In steady state conditions, fungal growth begins at around 80% of relative humidity (or water activity of 0.8) according to Fog Nielsen [19]. A minimal relative humidity of 71% was proposed by Ayesrt [22] for some of the species investigated, but most of them proliferated from 78%. However, significant amounts of mycotoxins, which are metabolites that are toxic for humans for example, are only

produced from 95% relative humidity [19]. The affinity of micro-organisms for water depends on the type of inoculum. Some of them are xerophilic, meaning that they grow in a dry environment, whereas others are hydrophilic. Thus, at a given relative humidity, the growth of micro-organisms depends on the species.

2. Substrate (or medium, or material). The proliferation depends on the quantity of nutrient available and the porosity of the material, which influences the moisture content at the surface. Fungal species have affinities for certain pH levels and salt contents [11]. Roughness of the surface material can facilitate fungal growth by concentrating nutrients and moisture in cracks or on asperities [23].
3. Temperature. The optimal temperature for many fungal species is between 20°C and 30°C [24],[25]. However, some microbial growth has been recorded between 0 and 50°C depending on the species [11],[22].
4. Time of exposure. The longer the material is exposed to humidity, the higher is the risk of microbial growth [19]. In the case of a material in a dwelling, the environmental conditions of relative humidity are variable. A short period of time under favorable conditions can lead to microbial growth, although there is a delay. This phenomenon can be explained by considering the Time-of-wetness (TOW) for example, which represents the hours of high humidity per time unit. This has been mentioned in a few studies [12],[26]. Finally, it has been reported that mold can survive for some time under unfavorable conditions.

Moreover, some oxygen is also required but the supply is assumed to be sufficient in a building or in most laboratory tests. Biotic influences may occur during the growth when several species are present together [11],[25]. Finally, it should be noted that mold fungi do not need light to grow [11].

Even under optimal conditions, microbial growth tests generally last several weeks or months, which is quite a long time. One of the consequences is that detailed data about germination time are rare [11] as such a delay is hardly acceptable during material development, where a shorter time scale is preferred, at least for the earliest stages of development.

3. Methodology

As underlined in the introduction, there is no consensus on the methodology that should be applied to study mold growth on building materials, although research has already been done on this topic. In this chapter, the main techniques found in the literature are presented. Some laboratory tests are based on standards intended for plastics (ISO 846 [27]) or insulation materials and their facings (ASTM C1338 [28]), for example. Recently, Johansson et al. [29] summarized these standards and proposed an innovative method for building materials. This constitutes the main basis for the present work but some adjustments will be proposed in the third part of this section.

3.1. Literature review on the experimental procedure for mold growth

3.1.1. Sterilization

Sterilization has to be achieved just before starting the study of mold growth in order to remove the fungi already present in the material. A simple method consists of exposing the samples to high temperature for a given time. It is mentioned in [17] that, even if not all the bacteria were removed with a heat treatment at 100°C, almost all molds were eliminated. In [3],[8], materials were sterilized with gamma rays, which is much more effective. However, this is costly and the device is rather unusual in material development laboratories. Some authors have chosen not to sterilize their samples.

3.1.2. Fungal selection and inoculation

The fungal species chosen for the study was *Aspergillus brasiliensis* (formerly *Aspergillus niger*), as in several other references [3],[30]. This was expected to make the phenomena occurring during the test easier to understand, and allow comparison with these earlier studies. A previous study [17] has shown that the main cultivable fungi growing on raw earth belong to the *Aspergillus* and *Penicillium* genera. Moreover, *Aspergillus niger* is often observed on building materials and has been implicated in health issues [31],[32]. This is a filamentous fungus, which has been observed all over the world in various environments (forests, dunes, indoors…). It can be pathogenic for humans. The optimal water activity (a_w) for its growth is around 0.95 [22],[33]. According to different authors, the optimal temperature is around 30°C: Ayesrt [22] observed the greatest growth between 30°C and 35°C, it occurred between 27°C and 37°C according to Passamani et al. [33] and the proliferation was greater at 28°C than at 20°C in the study by Lasram et al. [34].

Regarding inoculation, some authors suggested that natural inoculation would be more representative of real conditions [3],[35], while artificial inoculation was preferred by others. The latter technique consists of inoculating the specimens with an inoculum preparation, which accelerates the test and improves repeatability [29]. Moreover, it is easier to quantify the fungal growth when the initial state (spore quantity) is known.

In Hoang et al. [3], a single strain (*A. niger*) was inoculated, but various fungal species were used in [2],[5],[8],[30],[36]. The inoculation could be performed by means of a spray [2], micropipette [3] or dry cotton swab [8] in order not to modify the water activity.

3.1.3. Incubation

Most of the time, samples were put in a single climatic chamber, which regulated the hygrothermal conditions, or in closed chamber at controlled temperature where saturated salt solutions were used to maintain constant relative humidity. The incubation time was found to differ significantly among the studies, ranging from 42 days [35] to 30 weeks [8]. However, the temperature and relative humidity conditions were similar. Often, the

temperature was set close to 30°C and the relative humidity was kept high (above 90% [3],[35]), which are taken to be optimal conditions for fungal growth, leading to fast tests.
Some authors have investigated cyclic conditions to better represent indoor conditions in dwellings. In Latif et al. [6], for example, samples were placed at 90% RH for 2 days, then at 55% for 4 days. The alternation of these two relative humidity lasted 16 days. In the present study, several steady environmental conditions were investigated for 12 weeks, as recommended in [29].

3.1.4. Observation techniques and result analysis

The proliferation of fungi may be assessed by measuring the CO_2 production due to their aerobic respiration [9] or quantified by measuring the mass loss of the sample (consumption of nutrients by the fungi) [35]. However, the latter method presents problems of material loss during handling, nutrient intake by the fungi, which will also decrease the mass, and hyphae growth, leading to an increase of the mass. In the present study, observations ware made with a microscope once a week to follow the possible microbial growth. In fact, the most widespread technique used to follow fungal growth consists of regularly repeated observations with a microscope [2],[8],[35]. In [2], the observations were made under a laminar flow to avoid room contamination but, again, in a materials laboratory, such devices are not common.

The observations are often analyzed through a classification of different stages of proliferation. One example can be found in [2], where the rating scale ranges from 0 to 4:

- 0 means no mold growth;
- 1 is for the growth start, with one or few hyphae and no conidiophores;
- 2 represents sparse growth but with some conidiophores;
- 3 stands for patchy or heavy growth with many well-developed conidiophores;
- 4 means growth over practically the entire surface.

This technique does not depend on the fungus or its inoculation, so it can be applied in many different cases. Moreover, only a microscope is required, which is a widely available affordable device. A sample is considered to fail the test if the rate is greater than or equal to 2 on the above scale. It is the sporulation phase that is the most harmful to human health because spores can be released and cause allergies or other problems [37]. When several samples are considered, a choice can be made between two methods: considering either a median rate equal to two or the first sample to fail. Note that a similar analysis method named the “mold index” was proposed in [38], but 7 rates (from 0 to 6) were distinguished. Up to 2, the growth is visible only with a microscope, whereas it is visible with naked eye from 3 to 6 with a covered surface ranging from 30% to 100%. The main drawback is that the classification is subjective. This was acknowledged in [2], but it was also demonstrated that four different investigators obtained the same result, leading to the conclusion that the method was relevant. Other rating scales are proposed, which take account of the surface covered by fungal growth. However, this analysis may be more relevant if spray was used for inoculation [39] rather than a pipette.

Still, this methodology remains subjective and qualitative, which stresses the need for other techniques allowing mold growth to be quantified. One simple alternative is to take pictures of the samples then to carry out image analysis to quantify the area covered by fungal growth. Hoang et al. [3] have used the software Imagej. Nielsen et al. [8] used stereo-microscopy, while Bekker et al. [39] developed a set-up called the "Fungal Observatory Climate controlled aUtomized Set-up" (FOCUS) which is based on the discoloration caused by the proliferation.

3.2. Raw materials and sample preparation

Raw earth is a material that is increasingly studied for its low environmental impact and its ability to buffer indoor moisture. Depending on the intended use, plant aggregates can be incorporated into the earth matrix to lighten the material and improve some properties of the composite, such as lowering its thermal conductivity [40].

In the present study, quarry fines from the aggregate washing processing (FWAS) were used as the earth. These fines are wastes generated by the washing of limestone aggregates produced for the concrete industry, among others. The sludge created is left in sedimentation basins until it is dry. It is then reduced to powder and used. These fines were composed of calcite (60%), kaolinite (11%), illite (11%), quartz (10%), dolomite (6%) and goethite (3%). FWAS had a pH of 7.8, which is an optimal value for the development of many microorganisms. The particles were extremely fine: 99% were smaller than 80 µm and the average particle size (D50) determined using the pipette analysis was 6.5 µm. Before being used, they were stored in plastic bags at room temperature. Two plant aggregate types were studied in this work: barley straw, the part of the cereal stem rejected during the harvest, and hemp shiv, the lignin-rich part of the hemp stem.

Three formulations were studied: earth alone (FWAS), earth with 3% of barley straw (S3) and earth with 3% of hemp shiv (H3) but only for the test at 20°C in order to limit the number of samples used. The raw materials were mixed by hand before adding water to reach the optimum Proctor content (corresponding to the highest density) and mixing mechanically until a homogeneous mixture was obtained. The specimens were manufactured by double static compression in cylindrical molds 5 cm in diameter and 5 cm high. Five specimens were made at once in the mold (Figure 4-21). They were separated by with four PVC discs 5 cm in diameter. Specimens were first dried for 24h at 40°C, then the temperature was increased by 0.1°C/min to 100°C and kept at 100°C until the weight became constant (weight variation less than 0.1% between two weighing 24 hours apart). This was done to accelerate the drying of the samples (in opposition to natural drying). Note that this temperature was set to be similar to the one used in brickyards before firing.

Figure 4-21. S3 specimen manufacturing

3.3. Methodology adjustments in the present study

3.3.1. Sterilization and inoculation

In the present study, sterilization was achieved by placing the specimens in an oven at 100°C for 24h. This value was the same as during the drying phase of the material. It was not higher in order not to modify the material, and avoid combustion of the plant matter, which starts at 250°C [41]. However, this temperature was assumed to be high enough to eliminate most of the molds, as mentioned above.

Here, artificial inoculation with a pipette was preferred so that the spore quantity delivered on each specimen was known exactly. The fungal suspension of *Aspergillus brasiliensis* (ATCC 16404 / CBS 733.88*)* was concentrated at 3.10^5 conidia.mL^{-1} and 5 µl was pipetted onto 5 different spots of the surface. Therefore, each surface received 25 µL, which represents about 7500 conidia, i.e. more than 800 conidia.cm^{-2}. Specimens were allow to cool for 30 minutes after the sterilization before being inoculated. Five samples were inoculated while two additional samples, serving as controls, were not.

3.3.2. Incubation

In a preliminary study, it was shown that, when the samples were placed in the same climatic chamber, the fungal growth occurred at the same time in the inoculated samples and the control samples (non-inoculated). To avoid this, the use of separated boxes was proposed by Thomson and Walker [9] to eliminate any cross-contamination. Saturated saline solution should be placed inside to maintain a constant relative humidity level. A similar protocol was followed here using different salts to obtain 3 different RH conditions:

- 93% of relative humidity with a solution of potassium nitrate (KNO_3);
- 84% with potassium chloride (KCl);

- 76% with sodium chloride (NaCl).

The individual incubation set-up was composed of a plastic box in which saturated saline solution was placed. Above this, the specimen was put on a holder supported by rigid foam wedges to ensure good stability (Figure 4-22). The box was sealed with Parafilm® in order to increase the air-tightness of the assembly. Prior to testing, all the plastic boxes were cleaned with alcohol to avoid any other source of contamination.

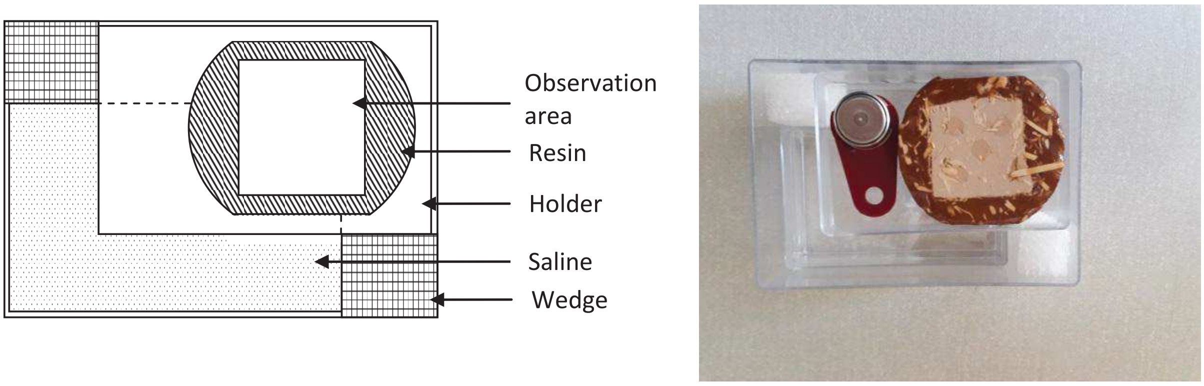


Figure 4-22. Scheme (left) and picture (right) of the individual incubation set-up

The materials were tested under two different temperatures (20 and 30°C), leading to a total of six different hygrothermal conditions, as summarized in Table 4-7. Assuming that the least favorable conditions would lead to no mold growth, fewer samples were prepared and used for the tests at low temperature and relative humidity (20°C together with 76% or 84% RH). For the tests at 30°C, the incubation set-ups were placed in a climatic chamber. For the tests at 20°C, they were placed in a room where the temperature was maintained at 20°C. The conditions were monitored by hygrothermal sensors placed in the specimen holder, within the plastic box. As no surface condensation was detected in the boxes, it was concluded that this protocol avoided liquid water droplets falling onto the samples, which would have biased the experiment.

Table 4-7. Conditions of incubation of the materials and quantification of the mixtures tested

Incubation	**Temperature**	**30°C**			**20°C**		
	RH	**75%**	**84%**	**93%**	**75%**	**84%**	**93%**
	Time (weeks)	12			12		
Materials	FWAS (37 samples)	5^i, 2^n	5^i, 2^n	5^i, 2^n, 2^u	2^i	5^i	5^i, 2^n
	S3 (37 samples)	5^i, 2^n	5^i, 2^n	5^i, 2^n, 2^u	2^i	5^i	5^i, 2^n
	H3 (14 samples)	-	-	-	2^i	5^i	5^i, 2^n
^{i}Inoculated, ^{n} Non-inoculated, ^{u} Uncoated and inoculated							

For microscopic observation, the plastic box was opened and the sample was removed with its holder without being touched. The only possible contamination during the observations would have come from the air of the room, but exposure lasted for only few minutes, once a week.

3.3.3. Microscopic observation

Preliminary tests showed the difficulty of achieving automatized image acquisition on raw samples. First, the sample surfaces were not perfectly flat. The two faces of the specimens were therefore polished to limit the surface roughness, prior to sterilization (note that the sides were also polished to allow the specimens to fit into the holder). The final thickness was around 0.8 cm, but the thickness was not expected to have a strong influence because mold growth mainly develops at the surface. Second, proliferation was faster on the edges of the specimens, i.e. where the fungus had not been inoculated. A similar observation was made by Hoang et al. [3] and may have two causes:

– Sterilization was not effective / homogeneous;
– The edges were much rougher than the sides, which favored local water accumulation and mold growth.

To solve this problem, Van den Bulcke et al. [4] proposed coating the sample edges with polyurethane. In the present study, the specimens were coated with resin (Synolite® 0288), except over a square surface of 3x3 cm², which was the investigated surface. In order to check the influence of the resin on the microbial growth, 2 samples of FWAS and 2 of S3 were left uncoated.

To complete microscopy observations, automatized image acquisition was also performed under a binocular microscope in order to assess the fungal growth by image analysis. The motorized stage allowed 165 snapshots to be taken of the sample over an area of 9 cm². The global image was then reconstituted by the computer software Ellix from Microvision Instruments. The objective was to determine the contaminated area of the surface by comparing the images at the end of the test with those from the beginning. However, the automatized acquisition lasted around 20 minutes for each sample, which made it hardly suitable for on-going monitoring of mold growth for all the samples. Consequently, this technique was applied only at the beginning and end of the whole period of incubation.

3.3.4. Additional analysis technique

Finally, this study was completed with a third analysis technique to identify the fungal species. This test, referred to below as the identification test, was carried out in a microbiology laboratory (Laboratoire de Génie Chimique de Toulouse). This non-destructive (for the material) analysis was carried out on the five inoculated specimens of mixture S3 following a protocol described by Simons et al. 2015 [17]. First, the surface of a specimen was sampled by applying a 9 cm^2 adhesive dressing (Hydrofilm - Hartmann®) to it. The dressing was then removed, put into a tube with 10 mL of a recuperation medium (sterile Phosphate Buffer Saline (PBS) + 1% of sterile detergent (Tween80)) and vortexed for 2 minutes. The suspension obtained was serially diluted and each dilution was deposited on a nutrient medium of Potato Dextrose Agar (PDA) with 0.05 $mg.mL^{-1}$ of Chloramphenicol (Cm) in triplicate technique. The specimens were finally incubated for 4 to 5 days at 22°C and the fungi enumerated. Afterwards, the Colony Forming Units (CFU) were counted and the fungal CFU concentration in the initial suspension was calculated. The identification of the fungal isolates was assessed by observing some aspects of the mycelia such as their shape or color. The hyphae and conidiophores were also observed by optical microscopy (magnification 400X) with a safranin-stain.

This technique is complementary to microscopic observations but it should be noted that sampling by the adhesive method is not free from bias [17]. First, although the whole surface area of the specimen was sampled, the inside of the material was not. Second, there is no guarantee that all the molds were sampled by the adhesive film or that all adhered molds were released during the vortexing. Nevertheless, this technique indicates the proportions of the various types of mold. Here, this technique was used on S3 samples only, as mold growth was not significant elsewhere.

4. Results and analysis

First, it is worth recalling that all the tests were limited to 12 weeks. No growth was reported at any time during the test for the lowest values of relative humidity (76% and 84% RH), for all temperatures and materials. At 20°C, no growth was observed on FWAS or H3 specimens. For the S3 specimens, mold growth was detected for only two samples at 93%. One of them was classified 1 from the second week to the end of the test, showing only one hypha. On the other one, fungal growth appeared 12 weeks after inoculation, directly at rate 2. So it was not significant and it can be concluded that mold growth occurred for one hygrothermal condition only (30°C, 93%RH). In addition, mold growth was found on inoculated samples only. Moreover, the results obtained with uncoated samples did not differ significantly from those obtained with coated samples, indicating that the resin did not influence the microbial growth on the specimens. Finally, the results differed according to the material. Overall, this increases confidence in the methodology.

4.1. Observation techniques

The proliferation started with a growth of tiny filaments a few micrometers diameter (see Figure 4-23) which extended over several millimeters above the material surface. The observation thus had to be made at high magnification and then the whole area had to be scanned to detect the hyphae. The magnification chosen, with a 10X objective, allowed a surface area of 0.93 mm² to be observed. At such high magnification, the specimen did not appear to be at all homogeneous, even for raw earth with no plant aggregate. Consequently, it was hard to detect the hyphae with this manual scanning method and they could even be confused with plant aggregate. This method was thus long and tedious, yet it seemed a good way to estimate the start of growth (rated 1 in the scale proposed by [2]).

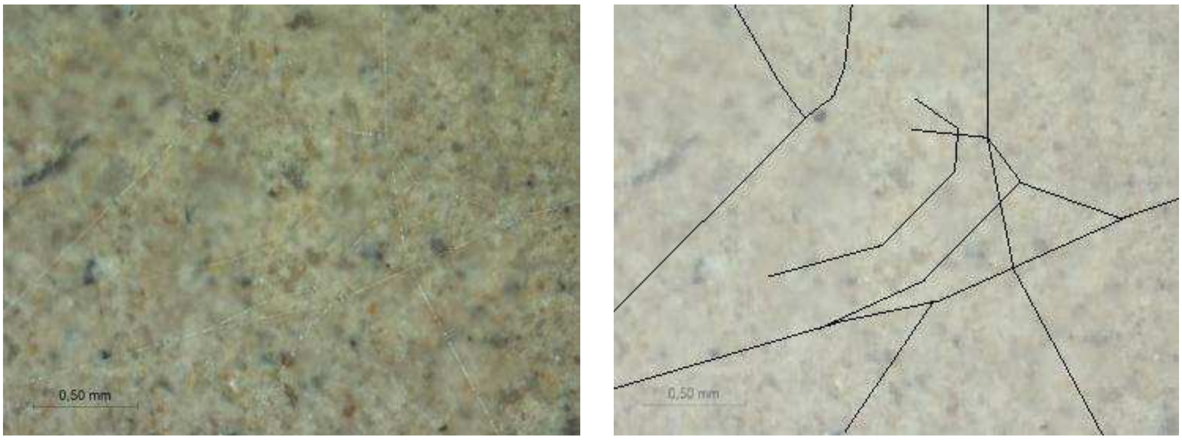


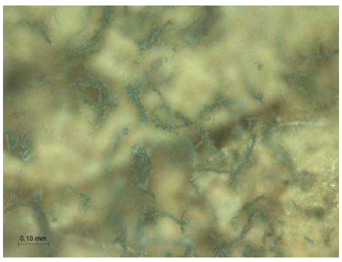
Figure 4-23. Hyphae of a FWAS sample at 93% RH and 30°C after 12 weeks: microscopic photo (left) and manual identification of hyphae routes (right)

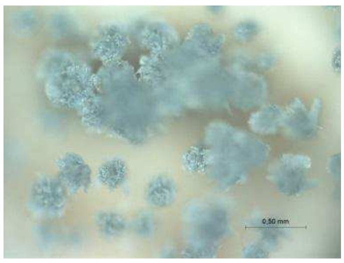
However, when the fungal growth was well established, it was easier to observe the molds. Marked fungal diversity was observed, with the varied appearances shown in Figure 4-24. On the first picture (I2 at 3 weeks), no conidia could be observed whereas black conidia could be observed at 5 weeks or on I1 at 4 weeks. Other molds observed on I3 or I4 presented other shapes, spherical or elongated, and colors: green or blue-green. These molds might not have been introduced only by the inoculation. The first occurrence of growth was not observed exclusively on inoculation sites; in the case of S3 specimens, the fungal proliferation often began on wisps of straw.


I2 - 3 weeks


I2 - 5 weeks


I1 - 4 weeks


I3 - 6 weeks


I4 - 4 weeks

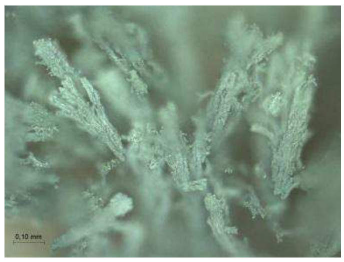

I4 - 5 weeks

Figure 4-24. Various fungal species observed with the microscope on S3 samples (30°C and 93% RH)

4.2. Image analysis

Figure 4-25 presents a picture obtained at the beginning of the test (one week after inoculation) and another of the same sample 5 weeks later. Here, we should recall that these images were obtained by juxtaposing several snapshots taken by an objective of magnification 4X. The overall resolution was about 2300x2300 pixels.

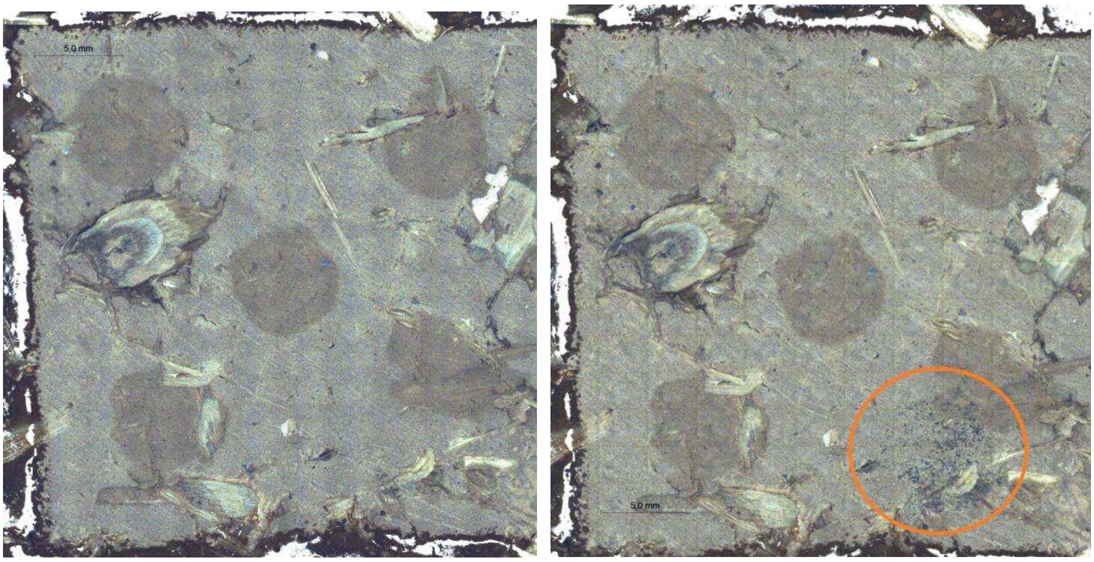


Figure 4-25. Pictures obtained by the automatized image acquisition device with an S3 sample (I3) after one week (left) and six weeks (right) of incubation at 30°C and 93%RH.

Basically, image processing relies on visual changes (either in color or brightness). By modifying the basic image settings (brightness, contrast and gamma correction), mold growth could be identified at six weeks (orange circled zone in Figure 4-25). This is in line with other studies, e.g. [42], where a good correlation between the image analysis and the visual study was obtained thanks to the discoloration caused by the fungi. However, this was not systematically the case, and some growth could be identified by microscopic observation while it remained undetected by image analysis. This is in line with the conclusions presented in [43], where it is stated that some molds were blue-green but most were colorless or had a similar color to the background. Apart from the color of the mold, it can be observed that the straw inclusions and the inoculation sites are standing out from the background, which makes the detection of mold growth more difficult.

Image analysis was performed punctually when mold growth was detected with the naked eye. The methodology can be roughly broken down as follows:

- Segmentation: the objective was to reduce the number of grey levels needed to distinguish mold growth from the background. Here, the number and size of these segments was determined by using a Particle Swarm Optimization (PSO) algorithm [44]. Consequently, the number of grey levels was reduced from 256 to only 3;
- Binarizing: this step distinguished two zones, so that areas with mold growth could be pointed out;
- Filtering: very small areas may have resulted from the methodology rather than from mold growth. Consequently, they were removed from the final image;
- Ratio calculation: the pixels corresponding to mold growth were summed so that the percentage of mold growth coverage could be computed.

This technique was successfully applied to a 7x7 mm² area extracted from Figure 4-25. The result of mold growth detection is presented in Figure 4-26, the resolution of which is about 512x512 pixels. The relative area covered with mold growth was computed as 22% in this case. This exemplifies the potential of image analysis applied to mold growth.

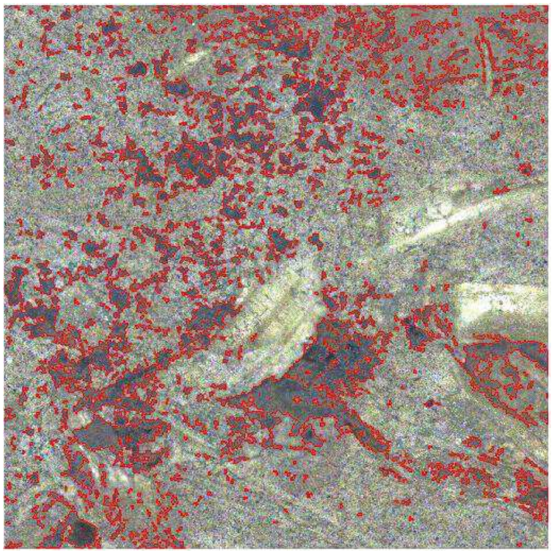


Figure 4-26. Identification of mold growth (red contoured areas) by image analysis

However, this technique could not be applied to the whole surface of the sample, because of the poor quality of the reconstructed image. As illustrated in Figure 4-27, the final image is an assembly of several snapshots, for which the boundaries are visible in some cases. Furthermore, the growth developed above the sample, i.e. in the third dimension. This caused small variations in the focal position, which is also visible on the final image, making it unsuitable for image analysis.

Figure 4-27. Highlight of the poor quality of the reconstructed image

Hence, the conclusions on the application of image analysis to the study of mold growth are mixed. On the one hand, it is proved that this technique is a good candidate for quantifying mold growth coverage. On the other hand, the present methodology is not robust enough to be applied systematically. As improving image analysis falls beyond the scope of this work, the study of mold growth will rely on microscopic observation alone in the remainder of the paper.

4.3. Rating of the mold growth

The fungal growth is described as a function of time and was analyzed using two criteria:

1. The rating attributed each week (to each sample and the median value) for S3 specimens (Figure 4-28) and FWAS specimens (Figure 4-29) ;
2. The Kaplan-Meier survivor curves proposed by Singer and Willet [45] and presented by Johansson et al. [2]. A threshold is defined and corresponds to a sample obtaining a rating of 2 here. The survival rate is defined as the ratio of samples that passed the test at a given time (i.e. with a rating lower than 2). This second criterion is somewhat more severe as the evaluation is binary and the scatter on the results has a more limited impact. Consequently, the times to observe results obtained with this criterion should be systematically shorter than the times obtained with the median value criterion. Still, this criterion is useful for a quick comparison among different tests.

Only the five inoculated specimens of each formulation were used to plot Figure 4-28, Figure 4-29 and Figure 4-30.

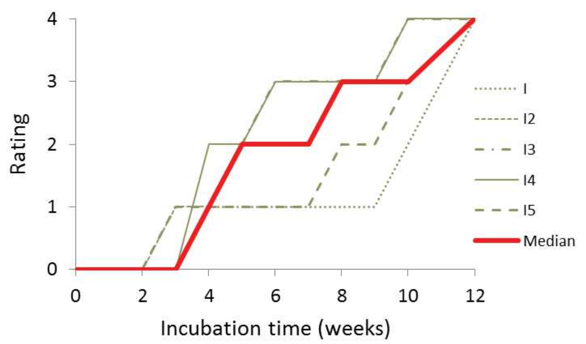


Figure 4-28. Rating for inoculated samples of S3 (93% RH, 30°C)

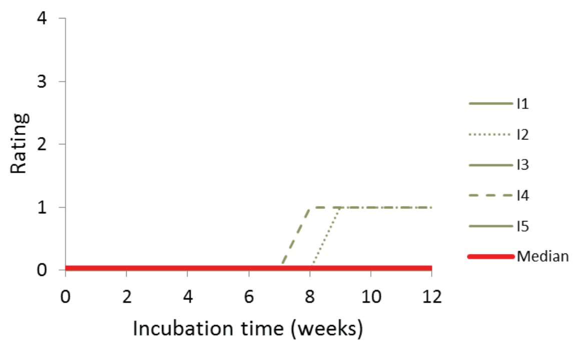


Figure 4-29. Rating for inoculated samples of FWAS (93% RH, 30°C)

The first observation of hyphae (rated 1) took place at 3 weeks after inoculation for an S3 sample at 93% RH and 30°C while its median rating reached 2 at 5 weeks of incubation (Figure 4-28). The graph shows a large variation of rating between the five specimens. For example, after 9 weeks, one sample was rated 1, one other was rated 2 and the other three were rated 3. Even with a same strict protocol, the results were scattered.

Concerning FWAS specimens, the first observation of hyphae took place at 8 weeks after inoculation in the same conditions (Figure 4-29). Median rating for FWAS specimens was 0 until 12 weeks. As can be seen on Figure 4-29, only 2 samples out of 5 showed a start of growth within 12 weeks and it reached only the rating of 1.

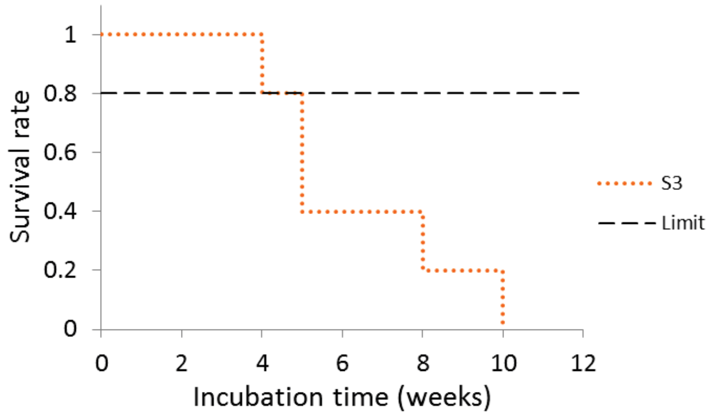


Figure 4-30. Survival rates of S3 specimens according to incubation time (93% RH, 30°C)

The second method is shown by Figure 4-30 and presents the survival rates of S3 specimens according to incubation time. The survival rate was set to 0.8, which in our case, means that the sample was assumed to fail the test as soon as a spot of mold growth was detected on one of the samples. For S3 specimens, the threshold was reached after 4 weeks, whereas it was 5 weeks with the first method (median value in Figure 4-28). Note that all S3 specimens failed the test at 10 weeks.

The longer the incubation time was, the more resistant to microbial growth the material was considered to be. As expected, straw seemed to be more favorable to fungal growth than earth or hemp shiv. Straw constitutes a carbon source that is useful for proliferation. This result was already observed by Hoang et al. [3] with the case of sunflower panels and plywood boards, which were very favorable to microbial growth. The growth may also have been facilitated by the inclusion of plant particles, which made the material more heterogeneous. It has thus been reported that rough surfaces or surfaces with cracks may concentrate moisture and nutrients more easily and then provide favorable conditions for fungal attachment and growth [23],[46]. The fact that fungal growth was less on hemp shiv samples than on straw samples can be explained by their chemical composition. It was shown in [47] that lignin was resistant to microbial attacks. According to a previous paper [48], a lignin content of 17.2% was determined for hemp shiv while it was only 5.5 for barley straw. This good resistance of hemp to fungal growth was already observed in [49]. It was shown in that paper that hemp had no risk of developing any mold at all below 95% of RH, whatever the temperature, while fungal growth was likely to occur from 75% RH.

Irregular observations were continued during an additional time for the samples at 30°C. The main result was that the two non-inoculated samples of S3 and one of FWAS also presented fungal growth at 93% of RH. These samples reached a rating of 4 between 14 and 17 weeks for S3 specimens and between 17 and 21 weeks for the FWAS sample. Moreover, the rating of the two inoculated samples of FWAS presenting fungal growth was increased to two between 15 and 16 weeks. Inoculation seems only to speed up the proliferation.

4.4. Identification of the growing species

After 12 weeks of incubation, all five S3 samples showed fungal growth with conidiophores, reaching the rating of 4. Thus the number of Colony-Forming Units per mL of suspension was counted (expressed in log10(CFU/mL)) and the growing species were identified on those samples (Figure 4-31).

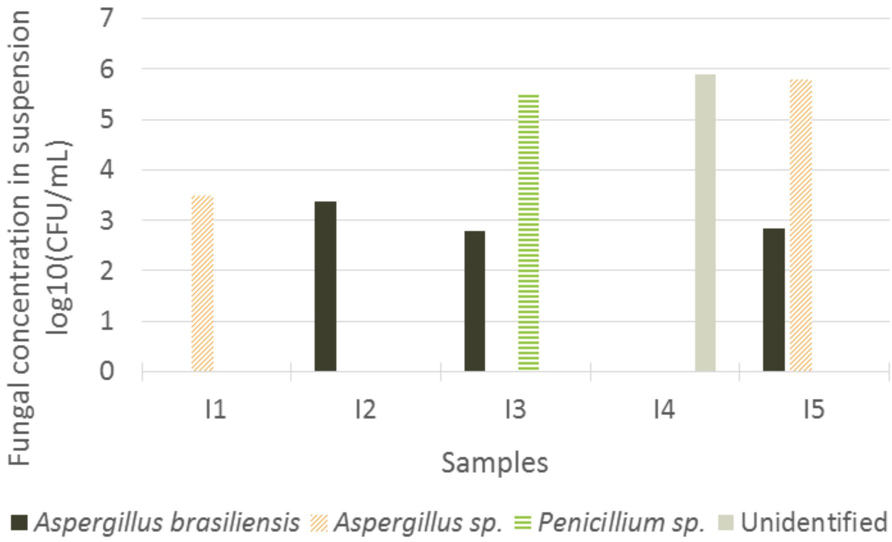


Figure 4-31. Colony-forming units sampled per mL of the suspension of inoculated S3 samples

This diversity of molds was already noted with the microscopic observation (Figure 4-24). The first striking result is that *Aspergillus brasiliensis*, which was the inoculated strain, was not the predominant species, except on one of the 5 samples (I2), and was not even found in two samples (I1 and I4). However, it was found in three specimens, albeit in an amount well below the other fungal species grown. *Aspergillus* sp. was identified in two specimens (I1 and I5). This was the same species, but different from *A. brasiliensis*. *Penicillium* sp. was identified as the majority species on one sample (I3), and the major isolate of the last sample (I4) could not be identified, but belonged to a different genus that was not *Aspergillus* or *Penicillium*.

This last result was unexpected: despite the care taken during the experiments, the methodology did not allow the growth of the inoculated mold to be observed. There was little chance of cross-contamination between the samples because the fungal species found were different for the five samples. So two possibilities can be considered:

1. The method used for sterilization was not effective. According to [50] quoted in [11]), most spores and mycelia are destroyed by heating for a short time at 80°C. However, some spores of *Aspergillus* sp. and *Penicillium* sp. can survive this hostile

temperature. The diversity of the micro-organisms observed might thus come from the raw material itself, particularly from the straw, as has been shown in a previous study [17]. Besides, the earliest tests showed that heat sterilization was not as effective in the core of the sample;

2. There was external contamination with the room air, containing airborne molds. This would have happened during observation, when the top of the individual incubation set-up was removed.

If the observed contamination was due to a lack of sterilization or deposited from the air during the observation, a similar random contamination would appear in both inoculated and non-inoculated samples. However, no growth was observed on control samples (non-inoculated) during the first 12 weeks, whereas inoculated samples had several mycelia on their surface. A parameter other than the sterilization or air contamination seems to facilitate unwanted mold growth, and the one remaining possibility is inoculation. Again, two assumptions can be considered:

1. The inoculated strain could facilitate the growth of other strains already present in the material or deposited afterwards. This biotic influence has been shown in some studies [11],[25].
2. The growth could also be explained by the addition of water through inoculation. Liquid water might help to activate or accelerate the fungal growth of species not removed by sterilization or deposited afterwards [29]. Another test was carried out to validate this assumption by using distilled water, as performed in [3], instead of the *A. brasiliensis* inoculum. Fungal growth was observable on the two samples over the same time frame as the samples inoculated with *A. brasiliensis*. Hyphae were observed from the second week for one sample whereas hyphae and green spores were seen after 6 weeks of incubation for the other one. Thus, the addition of liquid water through inoculation seems to cause the observed fungal growth on the material, more than the addition of fungal strains.

Even though the results obtained here were not those expected, the identification technique stands as complementary to microscopic observation. To the author’s knowledge, it has not been applied to building material up to now, but it represents an interesting prospect and should be considered for further works.

5. Discussion

The whole biological process of the experiment was not fully understood and controlled. The identification test led to unexpected results, questioning the methodology presented here. Nevertheless, this study also led to valuable results and promising perspectives can be foreseen. As the point of view is twofold, the discussion is broken down in two parts.

From the biological point of view, the identification of unwanted mold fungi questions the efficiency and the relevancy of the inoculation, and to a lesser extent, of sterilization. Also, scanning the whole surface with a microscope was not a successful way to quantify the microbial growth, even though the procedure was automatized. However, the first results presented here are encouraging. The problem of the contrast between the mold and the material is well known. The use of fluorescent techniques or genetically modified strains could lead to significant improvements. Another alternative would be to use a high definition camera combined with an adequate lens to obtain a relevant magnification. The strong advantage compared to the present set-up would be that the whole surface could be pictured at one time. Finally, it was found that the use of the identification technique, although not free from bias, was relevant and complementary to microscopic observation. Therefore, the systematic use of this technique at the end of the mold growth test is suggested.

From the point of view of material development, this test is time consuming and requires a significant number of samples. The assessment of the emergence of the growth, i.e. the definition of the rate increases from 0 to 1, was found to be cumbersome. However, as molds are harmful only from rate 2, this stands as the most interesting rate for the choice of a material. Time could thus be saved by looking only for molds at rate 2, where the presence of spores eases the detection. It is acknowledged that this analysis is quite limited considering the complexity of mold growth. However it highlights differences, as illustrated in Figure 4-30. All the samples with straw failed the test after 10 weeks, while none with earth only failed. This result is significant and useful in the process of developing new construction materials. Finally, some practical elements were found to be influential and should be considered for future studies:

- Tests should be achieved in individual boxes for each specimen to avoid cross-contamination;
- Polishing the surfaces and coating the edges and sides of the samples is recommended to limit undesirable proliferation, because of their greater roughness;
- Five inoculated samples plus two non-inoculated samples seems to be a minimum for a mold growth study.

6. Conclusion and outlook

In this paper, a methodology for studying mold growth has been proposed and applied to earthen bio-based materials. Various climatic conditions were tested on earth specimens or earth with straw specimens. The influence of the straw on the microbial resistance was demonstrated: earth with straw is indeed more sensitive to fungal growth than earth alone. Fungal growth took place after 4 weeks of exposure at 93% of relative humidity and 30°C. For earth with no plant aggregates, the mycelium was observed for the first time after 8 weeks of incubation under the same conditions. Finally, no proliferation at all was observed at 20°C in either case. As the bio-resource used in a composite was found to influence the fungal growth, it would be interesting to evaluate the fungal resistance of a composite material containing rice husk, generally recognized as rot-proof [51] or cork particles, which are hydrophobic. Preliminary tests in our laboratory have given promising results. Rice husk composites seem to have a better resistance to molds than barley straw composites.

The methodology was also discussed: some improvements were suggested and image analysis appears to be promising for quantifying mold growth. Finally, it is acknowledged that more tests would be necessary to determine the isopleth curves. These could eventually be implemented within transient hygrothermal models, in order to compare the risk of mold growth for several materials under dynamic conditions representative of realistic indoor situations.

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Concluding remarks

Several durability properties of earthen materials containing plant aggregates have been assessed in this chapter.

Some durability properties of five material formulations are tested in article E. The adaptation of standard procedures to bio-based earth materials is not always relevant and considerable work is needed to standardize representative tests. Work is, in fact, currently in progress to revise the French standard for compressed earth blocks. The results still allow good comparisons between the materials tested. Good compatibility of straw, hemp shiv and rice husk with earth has been found but cork granules have been shown to have poor cohesion with the earth matrix. Plant aggregates can lead to an improvement or a degradation of the durability properties, so a compromise needs to be reached. Rice husks seem quite promising, with excellent resistance to impact and good resistance to dry abrasion, erosion and water absorption. One of the greatest weaknesses of earth materials is their sensitivity to liquid water. One common way to "improve" the water resistance of earth is to stabilize it, often with Portland cement. However, as has been clearly demonstrated by (Van Damme and Houben, 2017), this is not relevant, either technically or environmentally. It is advised to adapt the architectural practice to the material with the basic rule of "good boots and a good hat". From an experiment in situ exposed to natural weather, unstabilized rammed earth has been estimated to last for 60 years without maintenance and aesthetic problems (Bui et al., 2009). To develop this type of material, some testing procedures closer to natural conditions will be needed (Van Damme and Houben, 2017). If necessary, other stabilizers can be investigated as long as they are environmentally friendly, efficient and inexpensive. One approach would be to use natural polymers, such as cactus pulp (Mattone, 2005) or alginate (Galán-Marín et al., 2010). The use properties of the material studied in the present work, such as mechanical or hygrothermal performances, should also be investigated to conclude on its suitability as building material.

Article F describes how earth materials containing 0, 3 or 6% of straw or hemp shiv behave with respect to fire. First, the fire reaction is assessed. Although lignocellulosic resources burn from 250°C, the material with up to 6% of plant aggregates is still not flammable. Concerning fire resistance, the materials were tested after a high temperature exposure (up to 800°C). The influence of plant aggregates on thermal insulation is positive in comparison to earth alone. The higher the plant aggregate content is, the better is the thermal insulation. However, their influence is negative in terms of compressive strength. The exposure to high temperature burns the plant aggregates, leading to an increase in the material porosity. This transformation of the material explains the decrease in strength and the increase in thermal insulation. However, an increase in temperature improves the compressive strength of the earth, by transforming the kaolinite into metakaolin. This study was performed over a short period of time (1 month) and few samples were thus tested, leading to large discrepancies in the results. More tests should therefore be done. Moreover, it has not been possible to compare the results with standards and to classify the material (fire-resistance class), because the tests described in the standards are intended for real-size material. The materials investigated at small scale in the present study should also be tested at bigger scale (at least brick or wall scale).

Fungal growth on bio-based earth materials is investigated in article G. The methodology proposed still requires some improvements. Not all the phenomena (notably biological ones) are understood yet and the observation from rate 1 is very time consuming. However, interesting results have been obtained concerning fungal growth, rating and kinetics. The earth material containing barley straw seems to facilitate the onset of mold. Fungal growth is observed on this material from the second week of incubation at 30°C and 93% of relative humidity whereas its first appearance on samples of earth alone is 7 weeks after inoculation. Moreover, after the 12 weeks of the test at high relative humidity and 30°C, all the samples containing straw had reached rate 4, whereas only two samples of earth alone had shown any evidence of mold development – which did not go beyond rate 1. Concerning tests performed at the same high relative humidity (93%) but a lower temperature of 20°C, only one sample containing straw reached rate 2 and one reached rate 1 at the end of the test. In addition, no fungal growth was observed on earth alone or earth with hemp shiv samples. For lower relative humidity (84% and 76%), no mold at all was observed whatever the material or the temperature. Such conditions (30°C, 93%RH) might be occasional, as a result of an accident, or water damage for example. These conditions are nevertheless highly unlikely in a dwelling during such a long period. The bio-resistance of specimens of earth alone seems to be very good and the material can still be considered as healthy. Finally, it would be interesting to investigate an earth material with rice husks, known as rot proof and which showed quite good results in the first part of the chapter. Preliminary studies seem to show good mold resistance of rice husk. It would also be interesting to compare these earthen composite materials with conventional materials, such as gypsum boards for example, regarding fungal growth.

In conclusion, it can be noted that the addition of plant aggregate seems to decrease the durability properties of the material, specifically water resistance, fire behavior and mold resistance. However, the durability is still comparable with that of other building materials. More investigations to determine the properties with other plant aggregate contents (6% at least) or an optimization of the manufacturing process would be needed to complete the present study. Other durability properties might be interesting to study, such as freezing-thawing resistance, drying-wetting resistance and resistance against insects or small animals.

General conclusions and perspectives

Conclusions

This work is a contribution to the development of more resource-efficient materials and techniques, for sustainable and healthy buildings. Earth-based bricks containing plant aggregates were investigated, at laboratory scale in this thesis. Although the earth built heritage is large worldwide, earth material is much less studied than other masonry types such as concrete blocks or fired bricks. The few existing standards about earth are quite empirical and a small number of tests are proposed. They often deal with earth used alone or stabilized with hydraulic binders, but barely take any plant addition into account. Moreover, the number of studies focusing on this type of material is still small. The raw materials are quite non-conventional in the field of Civil Engineering materials, and many of the tests therefore had to be adapted.

It was possible to expand the experimental campaign thanks to various project collaborators. The extrusion of the specimens was performed with the technical center (CTMNC) of Limoges. Several campaigns were carried out there. The durability tests achieved in Lisbon, as well as the fire behavior study conducted in Barcelona, were not initially planned, but greatly improved the knowledge of the material. Finally, the microbial proliferation investigation was investigated in close collaboration with another laboratory of the University of Toulouse (the Laboratory of Chemical Engineering (LGC)).

The state-of-the-art review has shed light on the small number of studies that exist on bio-based earth materials, even although a renewed interest in these materials was observed. These studies included quite a large number of papers dealing with mechanical properties or even thermal properties. Nevertheless, hygric and durability properties have been very little investigated on this type of materials. Furthermore, plant aggregates or fibers (used in an earth matrix) are themselves barely multi-characterized. The review showed that a wide variety of soils achieved good performance levels. It is thus more relevant to focus on the building product performances than the intrinsic characteristics of the clay: granulometry or mineralogical composition. The influence of plant aggregate incorporation in an earth matrix was investigated in this bibliographic review. This type of addition always decreases the dry density, the thermal conductivity and the shrinkage, and improves the ductility of the composites. However, concerning compressive and flexural strength, no general tendency could be observed as many parameters: earth nature, type of bioresource, formulations, testing protocols, manufacturing process, etc., differed among the various studies, making comparisons among materials complicated.

The raw materials used in this thesis work were one earth and several plant aggregates. The earth was supplied by a project partner, Carrières du Boulonnais. It came from the limestone aggregate washing process, was very fine (average particle size of 6.5 µm) and it was mainly composed of calcite, containing only 11% of illite and 11% of kaolinite. The three main plant aggregates chosen were straw, which is quite common in several earth techniques such as wattle and daub and cob; hemp shiv, already quite well studied thanks to the various hemp concrete studies; and, finally, corn cob, which is quite readily available in the region and unusual. These plant aggregates showed low bulk densities and good thermal properties. The preliminary campaign of extrusion, conducted in Limoges, gave promising results with regard to the extrusion of earth with a fairly large amount of plant matter. It was possible to extrude earth with a straw content of up to 6% by weight, even though the results were far better at 3%. The formulations of the composite materials thus involved a weight content of 3 and 6% of plant aggregates, even when the samples were compressed.

The second chapter, dealing with the three agro-resources, investigated their availability in France. Overall, the availability corresponds to the quantity produced less the quantity required for human consumption and for fertilizing the soil. Barley straw is a resource having good availability, with more than 4 million tons available each year. Corn cob and hemp shiv also present interesting potential with 240000 and 17000 tons of by-products available each year. The plant aggregates were also characterized. Their bulk density, highly dependent on their microstructures (tubular pores), is around 60, 150 and 500 $kg.m^{-3}$ for the straw, hemp shiv and corn cob respectively. Their thermal conductivity, linked to their density, is also quite small, as low as 0.044 $W.m^{-1}.K^{-1}$ for straw. The geometrical description of the particles was based on image analysis: straw and hemp shiv showed an elongated shape while corn cob was more spherical. Their sorption-desorption capacity also seemed very interesting.

Once the objectives had been defined, the state-of-the-art realized and the raw materials characterized, the thesis work was divided in two major parts: the use properties and the durability properties of the composites.

The use properties were assessed on earth composites made with 0, 3 and 6% of barley straw, hemp shiv or corn cob. First, the mechanical properties were investigated. Compressive strength was tested on compressed specimens while flexural strength was tested on extruded specimens. The importance of the testing protocol was discussed. For example, the difference between the compressive test with friction and the one with reduced friction could reach 59% in the case of CC3 specimens. Without a standardized protocol, no comparison can be made among the values from different studies. A significant decrease of compressive strength was observed with the addition of plant aggregates, and the compressive strength relative to a strain of 1.5% fell below the limit value of 1.3 MPa (except for the corn cob addition) required in the New-Zealand standard (NZS 4298, 1998) for earth bricks without plant aggregates. An increase of the wall thickness would thus be necessary to maintain the load bearing capacity of the elements. No significant influence of surfactant additions was observed on the flexural behavior.

Then, the hygrothermal properties of the same materials were assessed. The effect of the addition of plant aggregates was more obvious on thermal conductivity. The higher the plant aggregate content was, the lower was the thermal conductivity. It is highly dependent on the density, or more specifically on the volume content of the plant matter. The values of water vapor permeability obtained show that all the formulations were very permeable to moisture transport, although a small decrease was observed for a plant aggregate content of 6%. This high permeability result thus seems to be mainly due to the earth characteristic. Concerning the sorption-desorption isotherms, the storage capacity of the material is slightly increased by the plant addition. A comparison of the two methods used, saturated salt solutions and DVS, showed higher equilibrium curves in the case of measurement by the DVS. However, the curves presented the same shape and tendency. Finally, a theoretical value of the moisture buffering of the different formulations was calculated. The moisture buffering was excellent for all the materials, particularly for the samples containing 6% of corn cob.

Thus, the thermal insulation performance was improved by the plant matter addition, up to a decrease of thermal conductivity by 75% for the mixture S6. In the case of an outer wall, this decrease would allow the thickness of the additional insulator to be reduced. However, it would not be enough to avoid using an insulator while maintaining the thermal performance levels required by the current thermal regulations. Moreover, the plant matter incorporation, with the resulting lightening of the material, would decrease its thermal capacity and limit its thermal inertia, which is particularly positive for the summer comfort of the building. In terms of moisture buffering, an addition of 3 or 6% of plant aggregates did not significantly improve the hygric behavior of the earth alone, which already showed an excellent buffering capacity. Furthermore, the incorporation of low plant content induced a significant drop in mechanical strength, which would require an increase in the wall thickness to maintain a sufficient load bearing capacity. Given the results achieved, it appears that, to be thermally interesting, the incorporation of plant matter in an earth matrix should involve large mass proportions (dry density of the composite lower than 500 $kg.m^{-3}$ (Labat et al., 2016; Vinceslas et al., 2017). The consequence would be that the material would be neither extrudable nor load bearing. Conversely, thanks to their moisture buffering capacity and thermal inertia, the use of extruded earth bricks without plant matter would be interesting in buildings for load bearing interior walls, or outer walls if they were externally insulated.

Some durability properties were then evaluated. The main weakness of earth materials is their vulnerability to water. Some tests to evaluate the resistance of five material formulations to abrasion, erosion or impact were carried out in Lisbon. The adaptation of standard procedures to bio-based earth materials was not always relevant, but the results allowed good comparisons to be made between the materials tested. Good compatibility of straw, hemp shiv and rice husk with the earth was found. With respect to cork granules, poor cohesion with the earth matrix was revealed. Plant aggregates can lead to an improvement or a degradation of the durability properties, thus, a compromise needs to be found. Rice husks seemed quite promising, with their excellent resistance to impact and good resistance to dry abrasion, erosion or water absorption. However, the use properties of this material, such as mechanical or hygrothermal performance, should also be investigated to conclude on its use as a building material.

The fire behavior of the composites with straw or hemp shiv was assessed in Barcelona. The results show the tendency of the bio-based material not to ignite. Although the PCFC test showed a peak of heat release rate around 350°C for both plant aggregates, corresponding to the degradation of the cellulose, an ignition-extinction test proved that the composite material was still non-flammable. Smoldering was only detected on the plant particles, the earth being fired. Considering these first results, the addition of small quantities of plant material into an earth matrix does not seem prejudicial to its incombustibility. Nevertheless, additional tests are needed to conclude on the fire behavior of the composites, especially at element or wall scale.

Finally, the fungal growth resistance of the bio-based earth materials was investigated. Based on a brief literature review, a methodology to assess the microbial proliferation on this type of materials was proposed in this last part. Although the protocol still needs some improvements (difficult to quantify, time consuming...), some interesting results were obtained. The fungal growth was assessed according to five rating classes, from 0 to 4. From rate 2, which corresponds to the emergence of conidia, the sample was considered to have failed the test. The earth material containing barley straw seems to facilitate the mold onset. Fungal growth was observed on this material from the second week of incubation at 30°C and 93% relative humidity, whereas its first appearance on earth-alone samples came 7 weeks after inoculation. Moreover, after the 12 weeks of the test at high relative humidity and 30°C, all the samples containing straw had reached rate 4, whereas only two samples of earth alone had reached rate 1. No fungal growth was observed at lower relative humidity. The appearance of mold on samples containing straw at 20°C and 93% of relative humidity was much slower and less noticeable than at 30°C. These particular climate conditions of 30°C and 93% of relative humidity are not likely to occur in a dwelling, except after accidents, which would thus be temporary. Thus, earth-based materials with plant aggregates seem to be resistant to fungal growth, even at high relative humidity, if they are not exposed to liquid water.

To conclude, the three main plant aggregates tested in this research work allow certain properties, such as thermal conductivity or resistance to impact, to be improved. Their behavior is quite similar, but barley straw seems to be the best compromise: it presents the greatest availability, the lowest bulk density and thermal conductivity and the best ductility. Rice husk also seems interesting because, although it is less available, no attempts to give it added value have been made yet. However, earth material without any plant matter has the highest mechanical strength, and durability to fire and to fungal growth. It is, moreover, a very good moisture buffer thanks to its high permeability to water vapor. The use of a defined building material should be adapted according to its qualities and drawbacks. For example, the sensitivity of earth to liquid water needs to be taken into account when designing the construction so that it can be protected.

Perspectives

Various aspects of material characterization were investigated in this thesis work. However, many points, presented below, could be developed or investigated in the future.

First, some additional investigations concerning the raw materials would be useful. The influence of plant aggregates was assessed when they were added to only one type of earth, but it might be interesting to validate the results obtained with a different type of earth (mineralogical composition, granulometry, etc.). Other very available resources could be tested. Instead of the corn cob used here, corresponding to the hard part of the cob, which has quite a high density, corn pith could be studied. Several studies have begun research into this lignocellulosic resource, particularly for insulation materials, and have observed promising results (Palumbo et al., 2016). Rice husk gave promising results concerning durability properties and a preliminary study on fungal growth was carried out on this type of composite, which seems to present very high resistance. It might thus be interesting to evaluate the use properties as well as the microbial proliferation resistance, with this kind of plant aggregate. Earth, with plant aggregates or not, is very sensitive to water. It could be interesting to test the same mixtures stabilized with natural polymer, for example beetroot and tomato residues (Achenza and Fenu, 2007), cactus pulp (Mattone, 2005) or alginate (Galán-Marín et al., 2010).

With regard to mechanical properties, additional work is still necessary. Various works have shown the diversity of mechanical tests existing for earth bricks. Research on harmonization and standardization of mechanical testing procedures for earth bricks, containing plant aggregates or not, should be done. In this work, although the treatment with surfactants did not improve the mechanical strength of the composites, it did cause a slight decrease in the strain at rupture. More investigation is thus required to optimize its effect on strength, in particular concerning the treatment process. Compressed specimens were used to study the influence of plant aggregates and surfactant additives during compression tests whereas extruded specimens were tested in flexion. In further work, it would be interesting to determine whether or not the adhesion between earth and fiber differs according to the process used: compression or extrusion. This study was performed on samples at laboratory scale. Nevertheless, it seems that the interaction between the mortar and the bricks is very important with regard to the mechanical strength (Bui, 2008). Further research is thus required on masonry assemblies made of bio-based earth.

Further work concerning hygrothermal properties should be performed. For example, the experimental measurement of the Moisture Buffer Value would be interesting to verify one of the material's main strengths. The value measured should also be compared with the theoretical MBV calculated here. Moreover, a test complementary to the MBV would be the measurement of the heat storage capacity and restitution of heat. It would allow the thermal inertia of the material to be quantified, which is of major interest for bioclimatic construction. Finally, it would be interesting to estimate the theoretical sorption capacity by calculation using the values corresponding to the raw materials. This would avoid the problem of representativeness due to the small size of the sample and would save time by reducing the number of tests. There is, however, the problem of the influence of the aggregate on the structure of the earth matrix at the interface between matrix and plant aggregate. This phenomenon has already been quite widely studied in the concrete field, but not yet in the case of unfired earth. Another interesting development would be the study of hygrothermal properties at wall scale, and monitoring an earth-based wall in an occupied room would give some useful information about how the moisture and thermal aspects are regulated.

Durability properties still need a considerable amount of research, particularly to develop suitable tests. Concerning fire behavior, the study could be completed by actual size tests, in order to compare the results with the standards. Moreover, for a better understanding of the transformations occurring in the material after exposure to high temperature, the new mineralogical composition could be assessed by XRD. Further work is necessary concerning fungal growth resistance. The study of the proliferation between 84 and 93% of relative humidity would allow the critical relative humidity to be determined, which would help to fix the isopleths of these materials. Moreover, the contact of the material with liquid water facilitated fungal growth. Thus, an investigation of the fungal resistance in case of accidental capillary rise of liquid water is required. Another durability test that, to the best of our knowledge, has never been performed on earth material containing plant aggregates would be freeze-thaw tests. This aspect might be important in the case of an application of the material for an outer wall.

Finally, in order to quantify their impact on the environment, it would be interesting to conduct a life cycle assessment of the materials developed.

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Appendix

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Development of bio-based earth products for healthy and sustainable buildings: characterization of microbiological, mechanical and hygrothermal properties

A. Simons, A. Laborel-Préneron, A. Bertron, J.E. Aubert, C. Magniont, C. Roux and C. Roques

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Development of bio-based earth products for healthy and sustainable buildings: characterization of microbiological, mechanical and hygrothermal properties

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Abstract – The impacts of buildings on the environment and on the health of the inhabitants are priority issues nowadays. For many environmental, social and economic reasons, the demand for building products made of materials such as earth and bio-based materials is increasing. Under certain conditions, mold growth can be observed on the surface of such materials, which raises many questions about their use in buildings. In the framework of the "BIOTERRA" ANR project, the aim of the study was to develop and characterize an earth based material incorporating plant fibers from both abiotic and biotic points of view. Compressive strength, thermal conductivity and water vapor permeability of this material were determined. Microorganism sampling methods intended for raw materials and cylindrical specimens were optimized, and the microflora profile of these materials was then obtained. The results showed that the straw addition led to a decrease of compressive strength and an increase of thermal insulation. However, it did not influence water vapor permeability coefficient. Raw materials and manufactured specimens contained mainly *Bacillus* sp., *Aspergillus* sp. and *Penicillium* sp. Other compositions of this bio-based material will be characterized. Sampling methods developing here can also be used to identify the microflora of existing earthen buildings.

Key words: Bio-based building material / sick building syndrome / mechanical properties / hygrothermal properties / mold

Résumé – Développement de produits de construction biosourcés à base de terre crue pour des bâtiments sains et durables : caractérisation des propriétés microbiologiques mécaniques et hygrothermiques. Les impacts de la construction sur l'environnement et la santé des occupants sont devenus aujourd'hui des enjeux prioritaires. Pour de nombreuses raisons environnementales, sociales et économiques, les produits de construction à base de matériaux tels que la terre crue et des matériaux biosourcés connaissent un essor important. Sous certaines conditions, le développement de moisissures peut être observé, soulevant de nombreuses questions quant à leur utilisation. Incluse dans le projet ANR « BIOTERRA », cette étude a eu pour objectif de développer et caractériser, à la fois sur le plan abiotique que biotique, un matériau à base de terre crue avec ajout de fibres végétales. Sa résistance à la compression, sa conductivité thermique ainsi que sa perméabilité à la vapeur ont été déterminées. Des méthodes de prélèvements des microorganismes dans les matières premières ou les produits fabriqués ont été optimisées. Un profil de la microflore de ces matériaux a ainsi été obtenu. Les résultats abiotiques ont montré que l'ajout de paille faisait diminuer la résistance en compression du matériau composite mais permettait une diminution de la conductivité thermique. Cet ajout n'a toutefois pas influencé le facteur de résistance à la

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perméabilité à la vapeur d'eau. Les essais biotiques quant à eux ont révélé que les différents matériaux contenaient principalement des microorganismes appartenant aux genres *Bacillus* sp., *Aspergillus* sp. et *Penicillium* sp. D'autres compositions de ce matériau biosourcé seront caractérisées. Les méthodes de prélèvement développées ici pourront également être utilisées pour l'identification de microflores de bâtiments en terre crue existants.

Mots clés : Matériaux de construction biosourcés / syndrome du bâtiment malsain / propriétés mécaniques / propriétés hygrothermiques / moisissures

1 Introduction

Recent years have seen renewed interest in low-environmental impact housing in industrialized countries, and the impacts of building materials on the health of their inhabitants and on the environment have become priority issues. Some old building materials, such as earth, are being examined from this point of view. Scientific research on earth construction has been expanding significantly for about thirty years. Nevertheless, there are very few publications focusing specifically on unfired earth, although this material is widely used around the world. Nowadays, more than two-thirds of the world's population still live in unfired earth houses [1]. This building technique was used in France for centuries and a large heritage of unfired earth building methods (mud-bricks, cob, etc.) exists in different regions. Earth building has several advantages, such as improving comfort in the house, providing good thermal inertia [2] and offering natural regulation of the humidity of indoor air [3]. Earth can also be transformed into bio-based materials with the addition of aggregates or fibers of plant matter (straw, flax, hemp, etc.), in order to enhance the thermal insulation and lighten the material. In addition, these materials are low cost and have very low environmental impact (local and renewable raw materials with low embodied energy that contribute to carbon storage).

However, microbial proliferation can sometimes be observed on these materials [4], as in other common building materials. Under certain conditions, such as high and uncontrolled humidity (minimal water activity between 60% and 90%) and a temperature between 10 °C and 35 °C [5], molds may grow and form visible mycelia on building walls [6–10]. When molds are visible to the naked eye, development of the mycelium is already very advanced, which can imply health risks. Molds and bacteria may then cause poor indoor air quality, which is one of the most important issues in building. The pollution of indoor air is linked to Sick Building Syndrome (SBS), and may cause health problems for inhabitants [11]. The main microorganisms involved in SBS are molds. Fungal development can cause production of allergens, mycotoxins or volatile organic compounds (VOC), and also fungal infections and diseases [12–17]. Genera involved in health problems are mainly *Aspergillus, Cladosporium, Penicillium, Stachybotrys, Ulocladium* and *Chaetomium* [10, 18, 19]. Bacterial involvement in these problems is less common or less well known and there are few studies discussing the problem. The main bacteria identified on wet area inside buildings are Gram positive bacteria [20], such as *Streptomyces,* and also mycobacteria [21–23]. Adverse effects observed are similar to those of fungi, and include mycobacteria parietal compound in the ambient air, or the production of toxins by *Streptomyces*, which may cause inflammatory reaction [24, 25]. Molds on building materials may be initially present in raw materials, or brought in during the fabrication process or by the outdoor air. Potential origins of microbial contaminations are many and varied, and a large diversity of microorganisms may be encountered.

The ANR collaborative project "BIOTERRA" aims to identify, characterize and provide solutions to microbial growth on earthen bio-based products (bricks and plasters) used in the construction and renovation of healthy, sustainable buildings. The final ambition is notably to identify how the properties of earthen bio-based products – especially the hygroscopic properties, condition the possible growth of microorganisms on these materials, in relation to the environmental conditions. This project will also aim to develop and validate methodologies for the sampling and identification of microbial strains and the study of their growth on building products. With this in mind, a preliminary study was carried out in order to develop and characterize an earth based material incorporating plant aggregates from both the abiotic and biotic point of view.

In the present paper, mechanical and physical characteristics of manufactured specimens were measured, such as compressive strength, thermal conductivity and water vapor permeability. Microorganism sampling methods intended for raw materials and building products were set up and optimized. Finally, microbial isolates were characterized, and a first microbial profile of these materials was obtained.

2 Material and methods

2.1 Material

Quarry Fines from Washing Aggregate Sludge (FWAS) were used for this investigation. These fines were composed of calcite (63%), dolomite (3%), kaolinite (11%), illite (9%), quartz (10%) and goethite (3%). FWAS had a pH of 7.8, which is an optimal value for the development of many microorganisms. FWAS were extremely fine: 99% of the particles were below 80 μm and the average particle size (D50) determined using the pipette analysis was equal to 6.5 μm. Before being used, they were stored in plastic bags at room temperature.

Barley straw, in pieces 10 to 30 mm long, was also tested in different proportions in the earth matrix. The straw was also stored in plastic bags at room temperature.

2.2 Manufacturing

Three different mixtures were prepared for the various tests: (i) specimens made with FWAS only and specimens containing (ii) 3% and (iii) 6% of straw by weight content, marked S3 and S6 respectively. No binder (cement or lime) was added to the mixtures. The water content of the mixtures, determined by the Proctor test, was around 14% for FWAS, 19% for S3 and 21% for S6. To manufacture the specimens, earth and straw fractions were poured into a blender and mixed by hand. Then, water was added and the materials were mixed mechanically in the blender until a homogeneous mix was obtained (3 min). The raw materials were mixed the day before molding.

Cylindrical specimens 5 cm in diameter and 5 cm high (Φ5H5) (Fig. 1), intended for compressive strength tests, vapor permeability measurements and biotic tests, were manufactured by double static compression at the Proctor density. Specimens ($150 \times 150 \times 50$ mm^3, Fig. 1) for thermal conductivity measurements were rectangular prisms, manufactured in the same way.

The specimens were first dried at 40 °C for 24 h, then the temperature was increased by 0.1 °C/min to 100 °C and kept at 100 °C until the weight became constant (weight variation less than 0.1% between two weightings 24 h apart). This rise in temperature, inspired from the industrial process, was kept slow to keep shrinking homogeneous and to avoid mechanical stresses. The specimens were then stored in a room regulated at 20 °C and 50% relative humidity (RH) and were tested as soon as they were in equilibrium with the environment (about one week later). It is important to note that the specimens used for the two parts of this paper (mechanical and hygrothermal properties and microbiological study) were prepared using the same procedures. The dry densities of the FWAS, S3 and S6 specimens were 1.99 g.cm^{-3}, 1.52 g.cm^{-3} and 1.20 g.cm^{-3} respectively.

2.3 Mechanical and hygrothermal characterization methods

2.3.1 Compressive strength

The compressive strength tests on the Φ5H5 specimens were performed using a 100 kN capacity hydraulic press. The values of compressive load and vertical displacement were registered during each test. The vertical displacement was measured with a transducer that was in contact to the lower steel plate. The load was applied at a constant deflection rate of 3 mm/min. This speed was chosen as an intermediate value between the 1.2 mm/min specified in the French standard NF XP 13-90 [26] (intended for compressed earth blocks) and the 5 mm/min used in Reference [27] (intended for hempcrete). Three specimens of each mixture were tested in two different tests: one test with the specimen in direct contact with the steel plates (generating friction) and the other including a system avoiding friction. In the latter case, a 2-mm-thick piece of Teflon and a thin neoprene piece – with a drop of oil between the layers – were put between the earth sample and the steel (neoprene in contact with the specimen, and Teflon in contact with the steel). Teflon was used because of its low friction coefficient and neoprene because of its high mechanical resistance. Specimens were tested as soon as they were in equilibrium at 20 °C and 50% relative humidity. The Young's modulus of each specimen was then calculated from the linear part of the stress-strain curve (elastic deformation).

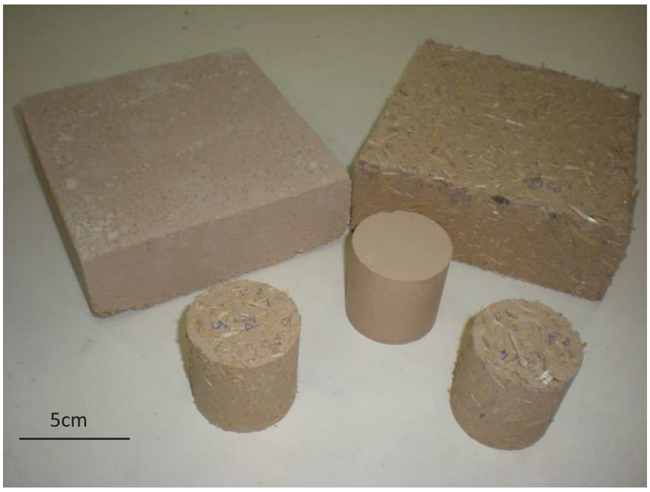

Fig. 1. Cylindrical and parallelepiped specimens.
Fig. 1. Éprouvettes cylindriques et parallélépipédiques.

2.3.2 Thermal conductivity

Thermal conductivity properties were assessed on three $150 \times 150 \times 50$ mm^3 rectangular prisms for each composition. The measurements were carried out with the EP500 guarded hot plate apparatus for earth alone and for earth with 6% of barley straw. Before testing, the specimens were dried at 100 °C and placed in a desiccator to cool. They were wrapped in a thin plastic film to avoid any humidity uptake during the measurement, which was performed at 25 °C with a difference of temperature of 10 K between the two plates. Steady state was assumed to be reached when the change in conductivity was lower than 1% in 60 min.

2.3.3 Water vapor permeability

The experiment used Φ5H5 specimens, which were kept at 20 °C and 50% relative humidity. This test was performed with a wet cup, as presented in the French Standard NF EN ISO 12572 [28]. The cup contained a saline solution of potassium chloride used to regulate the

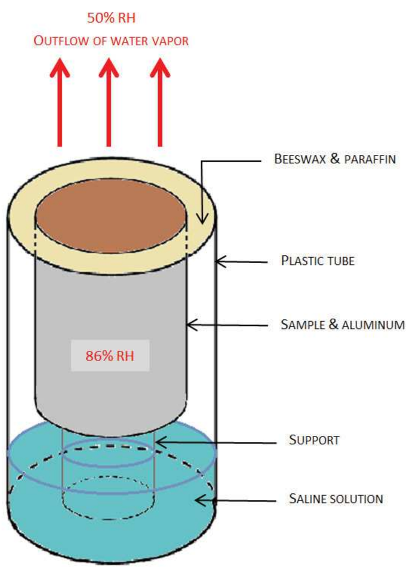

Fig. 2. Schematic diagram of the wet cup.
Fig. 2. Schéma de principe de la coupelle humide.

relative humidity at 86% (Fig. 2). This cup, with the specimen on its top, was placed in a chamber regulated at 23 °C and 50% relative humidity. The difference of humidity created an outgoing flow of water vapor, measured by regular weighing. The specimens were surrounded by an adhesive waterproof aluminum tape on the lateral face. They were then placed on a plastic support so as not to be in contact with the saline solution, as can be seen in Figure 2. The whole setup was finally sealed by a mix of 60% beeswax and 40% paraffin. The water vapor diffusion resistance factor (μ) was determined for the three compositions.

2.4 Microbial sampling and characterization methods

In order to sample the microorganisms contained in raw materials, the materials were suspended in aqueous sterile saline solutions and the influence of some key parameters (shaking time, addition of detergent, etc.) was evaluated. Sampling was more difficult on manufactured specimens because microorganisms were included in the matrix. A specific method, using adhesive sterile tape, had to be set up and optimized. All microbial assays were conducted under controlled conditions. Each assay was performed in triplicate in 2 independent tests.

2.4.1 Sampling and quantification on raw materials

Several techniques were used on the raw materials in order to optimize microorganism sampling methods. Each material (FWAS: 1 g; straw: 0.25 g) was mixed with 10 mL of sterile Phosphate Buffer Saline (PBS) at room temperature. Sterile detergent (Tween 80) was added to make the sampling of conidia easier. Different final detergent concentrations (1%, 5% and 10%) were tested. Suspensions were shacken at 300 rpm for 10 min. Shaking time was also extended to 30 min with a final detergent concentration of 5%. Three masses of FWAS in different volumes of buffer (1 g/10 mL, 5 g/20 mL and 25 g/100 mL) were also tested with 30 min shaking time. After homogenization by vortex, a range of dilutions of suspension were prepared in sterile distilled water. The suspensions and dilutions were deposited on various nutrient media, which were incubated at different temperatures: Tryptone Soy Agar (TSA) medium was incubated for 2 days at 32.5 °C to enumerate aerobic and aero-anaerobic bacteria; Potato Dextrose Agar (PDA) with 0.05 mg/mL of chloramphenicol (Cm) was incubated for 5 days at 22 °C to enumerate fungi. After incubation, the colonies formed were counted (CFU: Colony Forming units).

Another method to sample microorganisms on straw was tested, using a Smasher™ blender (AES Laboratories). Straw (2.5 g) was placed in a sterile bag with a membrane inside to separate solid particles from liquid after blending. Then, 100 mL of PBS with detergent was added at room temperature. Two final concentrations of detergent (1% and 5%) were tested. The bag contents were blended for 2 min. Blending time was extended to 5 min with a 5% final concentration of detergent. A range of dilutions was used and CFU were enumerated as described above.

2.4.2 Evaluation of release of microorganisms from adhesive dressing

An adhesive dressing was artificially contaminated in order to validate the release of microorganisms from it. Adhesive sterile dressings (Hydrofilm®) were cut into pieces (about 4 cm × 4 cm). One milliliter of a suspension of *A. brasiliensis* (*niger*) (ATCC 16404 / CBS 733.88) conidia (10^7 CFU/mL) was deposited on each piece of adhesive dressing. Then, the pieces were put face with deposited conidia up under a laminar flow in a Biosafety Cabinet (BSC). Water containing fungal conidia evaporated by the air of the laminar flow. When all the water had evaporated, the pieces were removed from the BSC. Adhesive dressings were put into a tube and 10 mL of PBS with a 5% final concentration of detergent was added. They were agitated by a vortex for 5 or 10 min. The suspension and a range of dilutions were deposited on nutrient medium (PDA with 0.05 mg/mL of cm) and were incubated at 22 °C for 5 days. After incubation, the colonies formed were counted.

2.4.3 Sampling and quantification on manufactured specimens

Sampling with pieces of adhesive dressing was carried out on Φ5H5 specimens made of only FWAS and S3 specimens at 2 different times of the manufacturing process: before the drying stage, directly after the compression step, and after the drying stage, when specimens

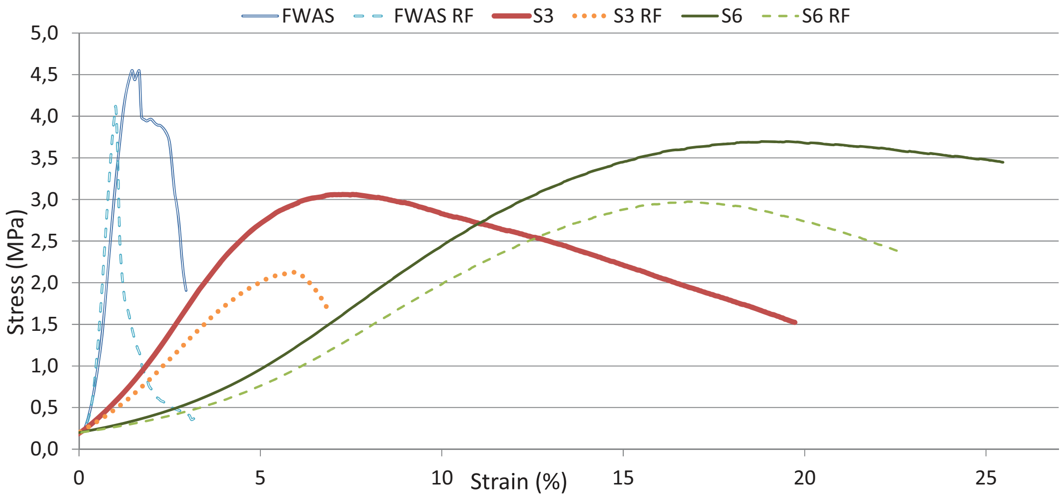

Fig. 3. Typical stress-strain curves of the different specimens with friction at the interface with the plates and reduced friction (RF).

Fig. 3. Courbes contrainte-déformation types de différentes éprouvettes avec frettage et avec frettage réduit.

were taken out the thermal chambers. Adhesive dressings were pressed on to manufactured specimens for 5 min. Then, the same protocol as described in Section 2.4.2 (with 5 min agitation and without evaporation step) was used to put the sampled microorganisms in suspension. A range of dilutions and a numeration were done as described at Section 2.4.1.

2.4.4 Characterization of microorganisms

Some aspects (color, size, relief) of the colonies and mycelia were first observed on the isolation medium. Bacterial isolates were Gram stained and mold isolates were stained with cotton blue. Then, the aspect of cells and hyphae was observed by optical microscopy (×400 to ×1000) so that bacilli/cocci Gram+/Gram- could be distinguished for the bacteria, and molds could be identified at the genus level.

2.4.5 Statistic tools

Averages and standard deviations were calculated for each condition. A Student test was performed to compare means. The tests were carried out on R software. A p-value below the threshold for statistical significance (0.05) is shown by an asterisk above the means concerned in Figures 5 to 8.

3 Mechanical and hygrothermal characteristics of the products

3.1 Compressive strength

Figure 3 presents typical stress-strain plots for the different compositions and protocols (with friction at the interface between the specimen and the press and with reduced friction). For each composition, the compressive strength measured in the tests with friction was greater than that in tests with reduced friction because of the confinement. The compressive strength of the specimen composed of earth alone was higher than that of S6 and S3, which is in accordance with density values of the various specimens. The average strengths for the test with friction were 4.1 MPa for the FWAS, 3.2 MPa for S3 and 3.8 MPa for S6. All the results were above the minimal value of 2 MPa imposed by the New Mexico standards [29] for adobe construction and by the German Standards DIN 18 945 [30]. The ultimate compressive strength of S6 specimens was higher than that of S3 specimens. This can be explained by a consolidation phenomenon due to the compressibility of the straw. Specimens not reinforced with straw showed an abrupt rupture after the maximum load whereas ductility improved with the increase of barley straw content. However, it is important to note that the ultimate strain was high for S3 and S6 specimens: between 5% and 8% for S3 and between 15% and 20% for S6, whereas it was only between 1% and 1.3% for FWAS. The addition of straw increased the ductility of the composite. In calculating building structures, such deformations of the material cannot be tolerated. Thus the maximal stress does not constitute a relevant indicator for compressive mechanical performance.

In order to compare the materials, it was chosen to limit the strain to 1.5% and to keep the corresponding compressive strength value, as described by [27] for hemp concrete. The maximal compressive strength was kept in cases when the failure occurred before 1.5% strain (for the specimens of FWAS alone). These values are presented in Figure 4 with the Young's modulus values for a test with friction. It may be noted that for a given deformation, compressive strength decreases with the density, which is in accordance with the decrease of elastic modulus with the addition of straw.

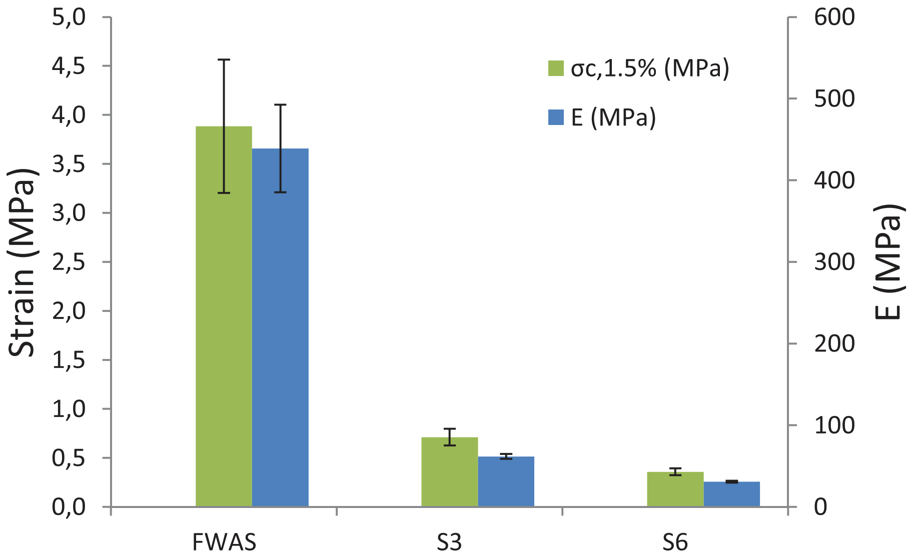

Fig. 4. Compressive strength at 1.5% strain (σc,1.5%) and Young's modulus with friction at the plate- specimen interface
Fig. 4. Résistance en compression pour 1,5% de déformation (σc, 1,5 %) et module d'Young (essai avec frettage).

Table 1. Water vapor diffusion resistance factor (μ).
Tableau 1. Coefficient de résistance à la diffusion de vapeur d'eau (μ).

	FAC			P3			P6		
μ	4.2	4.8	4.8	5.0	4.5	4.6	6.1	6.3	6.4
μ_{moy}	4.6 ± 0.3			4.7 ± 0.3			6.3 ± 0.2		

3.2 Thermal conductivity

Tests were performed on three specimens for the two compositions. The average values of thermal conductivity obtained were 0.57 ± 0.04 W.m^{-1}.K^{-1} for FWAS specimens and 0.14 ± 0.01 W.m^{-1}.K^{-1} for S6 specimens. The results show that the thermal conductivity decreased by about 75% with the addition of straw in comparison with the FWAS specimen. This decrease of thermal conductivity with an addition of plant aggregates has been widely reported in the literature [31,32]. It is linked with the decrease in density of the composite material. The improvement of thermal insulation demonstrates the interest of studying fibered earth bricks.

3.3 Vapor permeability

The assemblies were weighed daily over ten days. Using the measurements and the calculation presented in the NF EN ISO 12572 standard [28], the water vapor diffusion resistance factor (μ) was deduced for each composite specimen. The average values calculated from three specimens are presented in Table 1.

All the average factors were very close (5–6), but these results do not bring out the influence of the straw. These values were very low, which means that earth is a very permeable material. The results could be compared with reference values of other materials according to the 2012 French Thermal Regulations [33]. For instance, the concrete factor is around 80 and the calcareous stone factor is around 150. The permeability behavior of earth bricks is comparable to that of porous construction materials such as wood concrete or gypsum (lower than 10).

4 Sampling and characterization of microbial flora of raw materials and products

Preliminary tests showed that the microflora of the raw materials was mainly composed of bacterial spores and fungal conidia. Therefore only these two types of microorganisms were considered in the following tests.

4.1 Sampling on raw materials

Various parameters were tested, in order to enhance microorganisms sampling on FWAS (Fig. 5) or straw (Fig. 6). For FWAS, an increase of the shaking time (Fig. 5A) did not improve the recovery of bacterial and fungal spores. FWAS was easily suspended in the buffer by a simple vortex and FWAS particles in suspension could be directly deposited on a medium or diluted. Similarly, the detergent concentration (Fig. 5B) did not have any effect on the recovery of bacterial spores. However, the use of detergent at 5% final concentration doubled the recovery of fungal conidia. Finally, an increase in the FWAS / volume of buffer had no effect on the recovery of the bacterial and fungal spores (data not shown). Therefore, the conditions chosen were a shaking time of 30 min, a final detergent concentration of 5% and a solid/liquid ratio of 1 g/10 mL.

Regarding the straw, a 30-min shaking time (Fig. 6A) significantly improved (by about 1 log 10) the recovery of both bacterial spores and mold conidia. Straw

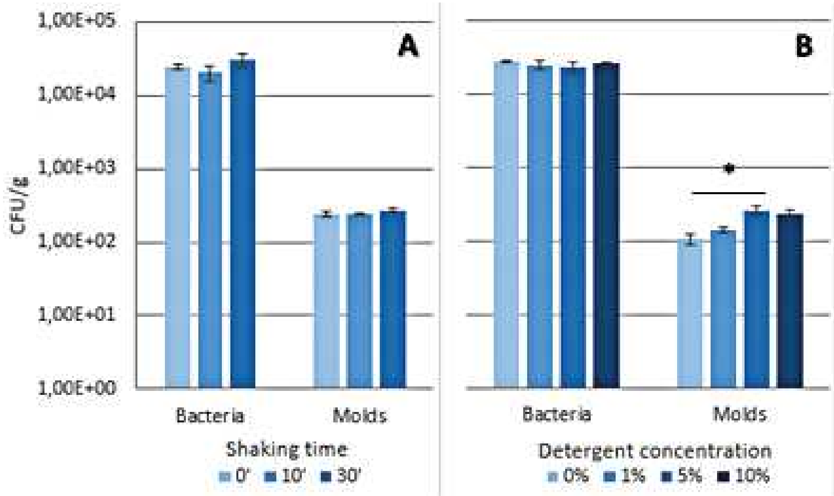

Fig. 5. Colony-forming units (average ± standard deviation; 2 independent assays in triplicate) sampled per gram of FWAS according to shaking time (A) or detergent concentration (B).

Fig. 5. Unités formant colonie (moyenne ± écart-type ; 2 essais indépendants en triplicat) prélevées par gramme de FWAS en fonction du temps d'agitation (A) et de la concentration en détergent (B).

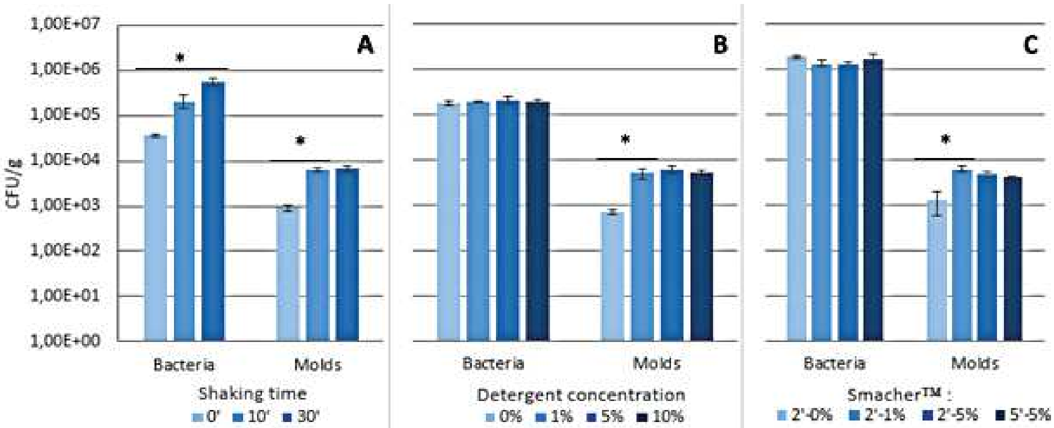

Fig. 6. Colony-forming units (average ± standard deviation; 2 independent assays in triplicate) sampled per gram of straw according to shaking time (A), detergent concentration (B) and Smasher^{TM} blending (C).

Fig. 6. Unités formant colonie (moyenne ± écart-type ; 2 essais indépendants en triplicat) prélevées par gramme de paille en fonction du temps d'agitation (A), de la concentration en détergent (B) et du mélange par Smasher^{TM} (C).

was rougher than FWAS and microorganisms could be blocked on it, so were not easily suspended. As observed previously, the use of detergent had no significant effect on the sampling of bacterial spores (Fig. 6B). However in the case of molds, an addition of detergent significantly enhanced the recovery of conidia by about 1 log10, even with a final concentration of detergent of 1%. The use of a surfactant enabled a better suspension of conidia, thanks to lipophilic interaction with conidia membrane and hydrophilic interaction with PBS [34].

Figure 6C presents the results of the sampling when a Smasher^{TM} was used. The detergent still did not have any significant effect on the recovery of bacterial spores but it increased the recovery of fungal conidia as observed with the previous method. In addition, a 5-min blending time (instead of 2 min) did not have any effect on the recovery of microorganisms. Compared to the shaking method, the blending method enhanced the recovery of bacterial spores by a factor 4 but no difference occurred for fungal sample. The use of the Smasher^{TM} for straw was therefore advantageous in comparison to a shaking step, with a better recovery of bacterial spores and a shorter processing time.

4.2 Evaluation of the release of microorganisms from the adhesive dressing

To determine the influence of the vortexing time on the release of microorganisms from the adhesive dressing, fixed conidia of *A. brasiliensis* fixed were vortexed for 5 or 10 min. The results are presented in Figure 7. When only homogenization by vortex was used, as few as 3.65×10^5 conidia were recovered even though 8.7×10^6 conidia were

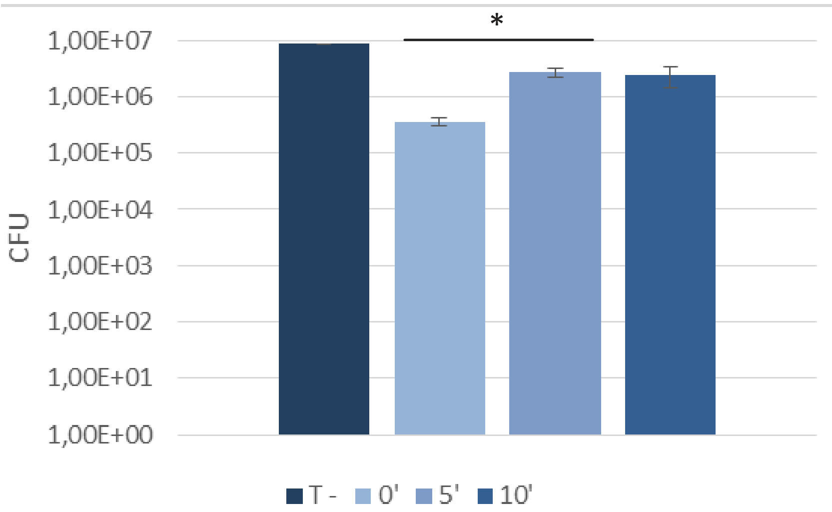

Fig. 7. Colony-forming units (average ± standard deviation; 2 independent assays in triplicate) of *A. brazilensis* recovered according to vortexing time.

Fig. 7. Unités formant colonie (moyenne ± écart-type ; 2 essais indépendants en triplicat) d' A. brazilensis recouvrées en fonction de la durée de vortex.

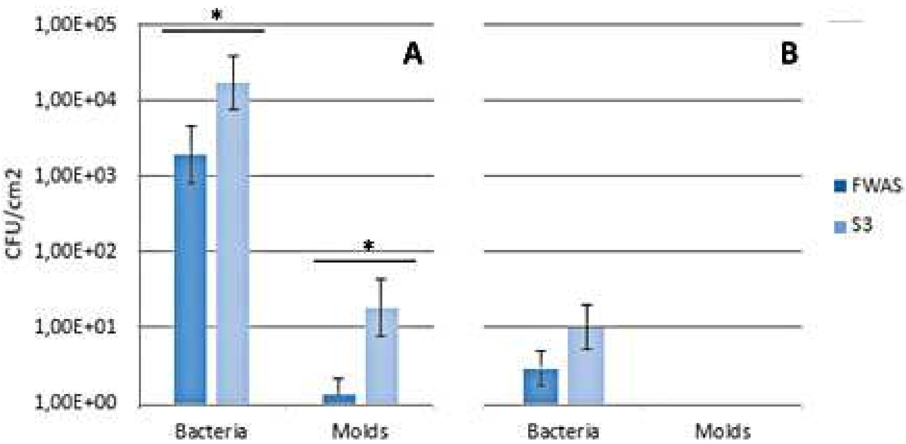

Fig. 8. Colony-forming units (average ± standard deviation; 2 independent assays in triplicate) of microorganisms recovered by adhesive dressing sampling on undried (A) and dried (B) specimens.

Fig. 8. Unités formant colonie (moyenne ± écart-type ; 2 essais indépendants en triplicat) de microorganismes recouvrées par prélèvement avec film adhésif sur des échantillons non séchés (A) et séchés (B).

deposited. With a longer vortexing time (5 or 10 min), recovery of conidia increased significantly by around 1 log10, and 2.8×10^6 conidia were recovered. No significant difference was observed between 5 or 10 min of vortexing, so a 5 min vortexing time seemed enough to release fixed conidia, although the recovery of conidia was about 30%.

4.3 Sampling on manufactured specimens

Figure 8 presents CFU enumerated after sampling of cylindrical specimens made of FWAS only or sampling on S3 specimens surfaces before or after the drying stage. Undried S3 specimens contained 1 log10 more bacteria and fungi than undried FWAS specimens. Although undried S3 specimens contained only 3% of straw, the addition of vegetable fibers led to detection of a significant quantity of microorganisms on the surface of the manufactured specimens. Molds could not be detected with FWAS specimens. The initial concentration of fungi in raw materials (2×10^2 CFU/g) was too low to be determined with this sampling method. Moreover, as presented above, the release of fixed conidia was about 30%, so all sampled microorganisms might not be observed.

Dried specimens contained 3 log10 less bacteria than undried specimens. Same difference of counted bacteria between FWAS and S3 specimens was observed after the drying stage. No enumeration was presented for fungi on dried specimens because too few isolates were obtained.

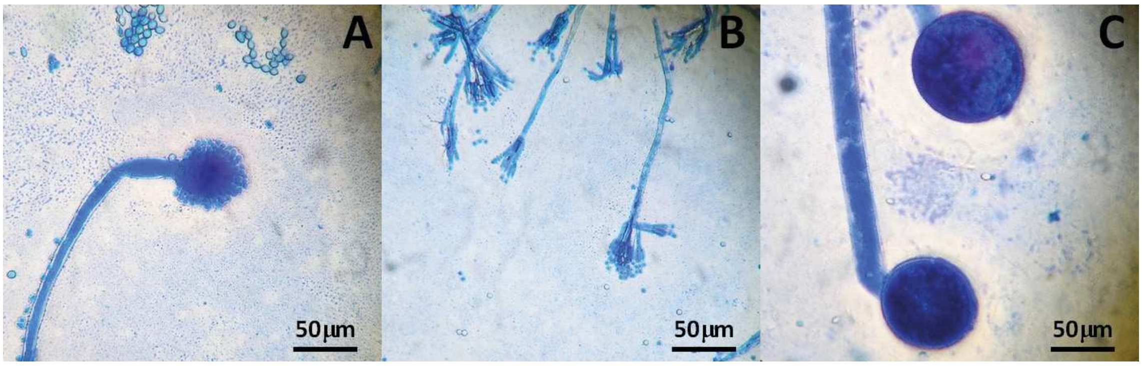

Fig. 9. Observations by optical microscopy of *Aspergillus* sp. (A), *Penicillium* sp. (B) and *Rhizopus* sp. (C) isolates after staining with cotton blue.

Fig. 9. Observation par microscopie optique d'isolats d'Aspergillus sp. (A), de Penicillium sp. (B) et de Rhizopus sp. (C) après coloration au bleu de coton.

4.4 Characterization of isolates

The isolates obtained during tests were characterized by macroscopic and microscopic phenotypes. On FWAS, most of the bacterial isolates (more than 85%) were Gram + sporulated bacilli able to grow in aerobic conditions and so considered as *Bacillus* sp. Fungal genera observed were mainly *Penicillium* (around 40%), *Aspergillus* (35%), *Cladosporium* (10%) and more rarely *Rhizopus* (2%) and *Ulocladium* (2%) (Fig. 9). As for FWAS, bacterial isolates from straw were mainly Gram + sporulated bacilli (more than 85%), and mold isolates mainly belonged to *Aspergillus* (around 95%) genera, with some *Penicillium* (2%) and *Rhizopus* (2%) isolated. The bacterial and fungal phenotypes observed on straw showed only half the diversity of those obtained on FWAS. Before packaging in plastic bags, FWAS were stored outdoors, which implied potential contamination by the ambient air or by rain. Moreover, FWAS could offer a more varied medium for the growth of microorganisms.

Finally, isolates obtained on cylindrical specimens were compared with those obtained on raw materials. Although few mycelia were sampled, they were the same as those observed on raw materials.

5 Conclusion

The effects of additions of 3% and 6% of barley straw on the compressive strength, water vapor permeability and thermal insulation of earth bricks were investigated. The compressive test results showed that the specimen containing only FWAS had the highest strength. Adding straw decreased the compressive strength, by 7% for the S6 specimens, but improved the ductility. The tests carried out with reduced friction generated lower resistances than the other test. The effect of barley straw was not significant for the water vapor permeability factor but, when the straw content was increased by 6% in mass, the thermal insulation was increased by 75%. These results confirm the interest of using light plant aggregates in earth bricks to improve the thermal insulation of these materials and thus to make significant savings in the energy used for heating buildings. The interest of adding such plant aggregates in the earth has been demonstrated and the compressive strength still seems to be compatible with the use as a building material. Moreover, high ductility is an advantage in case of a use as a filling material inside a wooden-frame for example, which can deform itself with climate conditions. The effect of the addition of other types of plant aggregates will be studied in further experiments. Moreover, it will be necessary to study the hygroscopic properties of bio-based earth products in greater depth because these properties will strongly influence the possible growth of microorganisms.

The optimization of techniques to sample microorganisms showed that a longer shaking or blending time increased the recovery of microorganisms on straw, and the use of a blender improved the sampling. An addition of detergent also appeared to be very important in the recovery of fungal conidia. As expected, most fungal isolates on raw materials belonged to the *Aspergillus* or *Pencillium* genera, molds which are common in the environment. Most of the bacterial isolates were *Bacillus* sp. but isolation of anaerobic bacteria will be carried out in further work to extend the types of microorganism explored. First sampling using adhesive dressing did not recover enough conidia to ensure the characterization of the microflora of manufactured specimens. Despite this limit of quantification, this technique is a non-destructive sampling method that enables direct sampling in houses or constructions. The purpose of using this method was to detect microorganisms at a contamination level where moulds were visible on the material. Sampling on specimens surface revealed that dried step removed almost all microorganisms. Considering our preliminary results, one of the most important tool will be to define if visible contamination of specimens surfaces occurs after proliferation of microorganisms deposited on surfaces by the environment or initially present in the interior of the specimens.

On the basis of these sampling methods, the next steps of this work will be the identification and characterization of microbial diversity and proliferation on earth constructions and bio-based earthen products. Specimens will be collected *in-situ*, on existing earth buildings. Then, microflora profiles will be obtained by using microbial isolations and genomic approaches (high-throughput DNA sequencing). The proliferation and adhesion of mycelia and biofilms on bio-based manufactured materials will also be studied to determine the environmental conditions favorable to their growth (temperature, relative humidity, etc.).

The first results concerning the hygroscopic properties of earthen bio-based product (vapor permeability) have to be completed in further experiments (especially regarding the sorption-desorption properties) but they confirm the high capacity of these materials to exchange quickly water vapor with the indoor air. The ultimate goal of this work will be to identify the links between the hygroscopic properties of earthen bio-based products, the environmental conditions (temperature and relative humidity) of indoor climate and the ability for microorganisms to proliferate at their surface. Finally, conditions of use of these types of materials in the construction and renovation of healthy, comfortable and sustainable buildings should be defined.

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