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Dental composite properties evaluation: from experimental approaches to the prerequisite of a chewing bench

Hazem Abouelleil Sayed

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Dental composite properties evaluation: From experimental approaches to the prerequisite of a chewing bench

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1. Introduction

The oral health is an integral part of the human health, which is necessary to maintain a healthy oral function and dental structures. The ability to process food to be adequately digested and absorbed, the ability to speak and communicate, and more over the ability to acquire a presentable social appearance, are all essential in the modern day life [1].

Historically, teeth were replaced and restored using natural human or animal teeth, stones or sea shells, ancient Egyptians, for example, had a dental specialist for the king. But the modern day dentistry did not start except in the 17th century, and even though the first attempts to restore teeth had mostly humble results, by the 20th century much development took place [2].

The last 30 years have seen a big development in the restorative materials technology and techniques; part of this is due to the advanced development in computer, technology and digital imaging. Another part is the type of material itself, the dental composite resin enables the addition and the diversification of its components whether the polymer or the filler [3]. This can be considered equally true with the glass ionomer cement and CAD/CAM block materials [4,5]. Dental products companies have seen large development with the growth in the dental field market, thus pushing forward for innovations and new patents and discoveries, together with more demand from patients for more efficient and more esthetic restorations. Modern biomaterial research resulted in the development of a wide variety of restorative materials and the innovation of new compositions and techniques [6,7].

Several factors seem to add to the complex process of making the adequate choice of restoration. The increased patient demand for esthetic at the age of selfies,

social networks and excessive photographing, esthetics seem to gain more ground as one of the principle factors that drive the choice of restorative material, sometimes on the cost of efficiency and survival rate. On the other hand, the dentist plays a role in guiding the patient to the correct choice of material in order not to sacrifice function for looks [8].

Another rising feature is the minimal intervention dentistry for managing dental caries and the remineralization materials and methods. The conservation of the dental tissue and pulp vitality is the scope of new research as the future of dental restorative dentistry [9].

The rising patient awareness of efficient oral hygiene, dental tissue conservation as well as good esthetics brought about more awareness and concerns about the biological effects of dental materials whether real or imagined. The media seem to play a major role in that issue, dentists are being routinely interrogated by their patients about mercury in dental amalgam and Bisphenol A in dental composite. While most of the concern is ungrounded, other issues like endocrine disrupting compound effects should be examined in depth before the development of new dental restorative materials [10].

Another important decision to be taken by the dental practitioner is the choice between direct and indirect restorations, each of which has its benefits and disadvantages. Direct restorations are inserted at the clinic within the same session, no laboratory procedures are needed, and no extra costs are required. Indirect restorations, on the other hand, are fabricated outside the oral cavity after taking an impression of the cavity (manual or digital) and are then inserted. This allows for better shaping of teeth contours and contact points, and also allows for the use of

extremely hard and tough materials in the fabrication process. Consequently, they are mainly indicated in large defects or replacement of large compromised existing restorations.

No ideal restorative material has been developed so far whether direct or indirect. The number of basic properties that should be fulfilled by dental restorative materials makes the task more challenging. The American dental association (ADA) required a list of basic criteria for restorative dental materials. These included that it should not be poisonous or harmful to the body, nor be harmful or irritating to the tissues of the oral cavity. It must resemble the natural dentition and help protect the tooth and tissues of the oral cavity. It must be easily formed and placed in the mouth and withstand the biting and chewing force in the posterior area of the mouth. Finally all of these criteria should not degrade by time.

Because of the above mentioned arguments and because of the aggressive oral environment, and in order to predict the behavior and the clinical performance of these materials, multiple thorough and complex tests are carried out to characterize their physical, mechanical, biological as well as their esthetic properties. Dental restorations have to function without failure or degradation while tolerating the humid oral environment and the microbial oral flora and being subjected to the masticatory and physical stresses caused by the fluctuating temperature and pH, the same holds true for the interfacial cement or adhesive layer that keeps the restoration in place.

Ferracane questions the ability to predict clinical success of resin based restorations only through laboratory testing, he claims that only clinical trials are able to provide the needed information. As a researcher in the field of chemistry and laboratory

testing, he exhibits the limits of such *in vitro* evaluation regarding the high number of parameters involved in clinical behavior of dental restorations [11].

However, *in vitro* characterization of physical properties and modulation of aging of restoration remain a prerequisite to develop new materials regarding costs and security before *in vivo* evaluation. Moreover, the present work claims that laboratory testing is essential in many ways, and can actually predict to a large extent the material performance. When properly designed and conducted, laboratory testing can provide valuable information about the actual causes of failure, and how different correlations between various properties can modify the restoration behavior.

The objective, through this work, was to develop transversal competences for a better understanding of *in vitro* evaluation as a way to predict clinical behavior.

Many parameters involved in the physical and mechanical properties of dental materials (dental composite as an example) have been compiled in figure 1, together with different tests used to predict its performance and longevity in the oral environment.

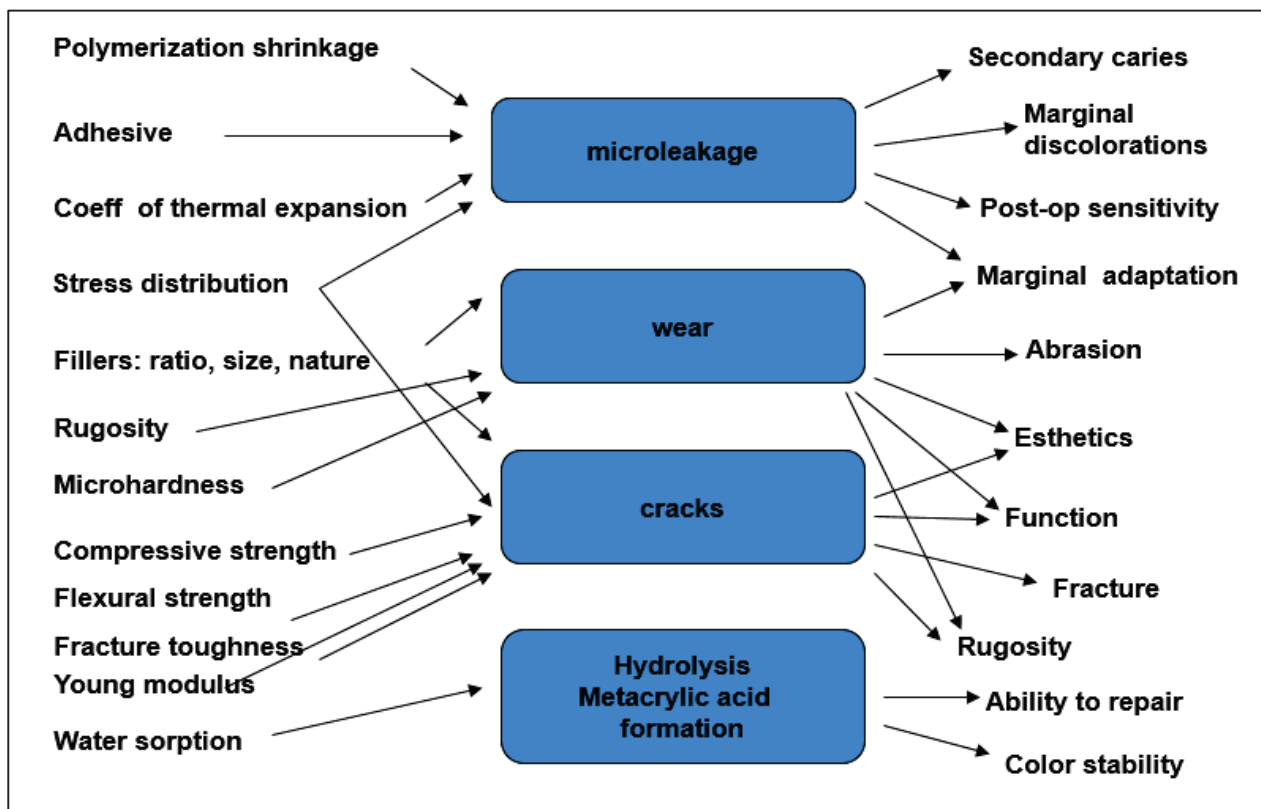
As can be shown, that sophisticated interaction between the different variants adds to the complexity of defining the role played by each of these properties.

We decided to investigate most of the parameters that impact the behavior of the restoration once in function.

The relevance of this work resides in the transversal approach it adopts, through simultaneously studying the restorative materials characteristics, to improve the

knowledge of their behavior and to create specific investigative tools (numerical, analogical) that we seek to develop in our laboratory.

Figure 1: Physical and mechanical properties of composite and their clinical implications (courtesy Pr Colon)



Different factors present on the left hand side of figure 1 were examined through different studies presented in this work. We selected the mechanical properties that correlate effectively with clinical outcomes the *flexural strength*, *flexural modulus*, *microhardness* and *fracture toughness*. These tests were performed on CAD/CAM block materials (indirect restorations) with different material compositions. They were also conducted on bulk composites and fiber composite (direct restorations) being

polymerized in bulk. They present the advantage of ease of insertion, and comprise different types of monomer composition and *filler* volumes including the fiber reinforced composite. *Filler ratio, size and nature* were then analyzed for their impact on the outcome of different materials.

The role of adhesive layer on the integrity of the restoration was the subject of several studies discussed in this work. Thus, while the restorative material may have mechanical properties sufficient to resist masticatory stresses, differences in the *coefficient in thermal expansion*, and in modulus of the *adhesive* layer would lead to marginal leak, secondary caries and eventual failure of the restoration.

With the continuous development of new dental restorative products, many important properties are overlooked. We can logically presume that using a limited number of tests cannot be sufficient to adequately predict the longevity of the restoration and that studies that characterize only one aspect of the restorative material would ultimately draw inaccurate conclusions. Hence, a more global method for evaluating dental materials should be adopted. The final aim, furthermore, is to launch, through an ANSM project (Agence Nationale de la Sécurité du Médicament), a new device that would integrate all the critical parameters needed for testing new materials.

This thesis is built through different scientific papers, each of them comprise part of our knowledge in the field of dental restorations. The overview of all these different topics is the basis of the manuscript leading to the final target of medialization of the clinical behavior of dental restorations.

2. Dental restorative materials

Types and composition of restorative dental materials

The restoration of dental tissue is a complex problem. The shape, function and appearance of the dental tissue to be replaced have to be restored in an efficient and simple way. The loss of dental tissue could be a result of caries process, attrition, abrasion, erosion or trauma, and regardless of the causative agent. It is mostly the amount, the location and the type of tissue to be replaced that dictates the technique of restoration and the material used, and sometimes the simple observation.

The complex anatomy of the dental structures and the amount of physical and mechanical stresses to which they are submitted, calls for a material that could withstand such a challenge.

Another equally important requirement is the handling of the material. Direct restorative materials have to go through a process of trituration, in which the material is inserted in a plastic phase into a tooth cavity (shaped to conform to the material used), then the materials becomes solid and begin to harden and acquire its final properties. Each of these steps is operator sensitive and requires time and effort. The other solution is the indirect restoration in which fabrication of the restoration outside the oral cavity (indirect technique) in the lab or using the CAD/CAM technology. The restoration is then cemented in place using the appropriate cement or adhesive. The insertion of the restoration again requires much time and effort to assure the entry in position and to avoid the affection of the weak interface between the material and the dental tissue.

The choice of the material of restoration and the technique of insertion depends on a multitude of factors, beginning with biologic and esthetic as well as economic factors. Moreover, the patient oral hygiene may dictate the kind of restoration to be used.

2.1. Restorative resin composite

Resins in dentistry, comprise a large variety of materials and functionalities, whether restorative, prosthetic or else. Composite, as a definition, is a physical mixture of materials. Dental composites typically involve a dispersed phase of filler particles distributed within a continuous phase (matrix phase). Historically, restorative composite resin began with the first successful innovation by Bowen in 1962 and the introduction of bis-GMA as monomer, able to form a resistant cross linked matrix, as well as the introduction of organic silane as a coupling agent to bind the filler particles to the resin matrix [2].

Restorative resin composite is basically composed of three distinct phases: the polymer resin matrix, the inorganic filler (mostly) and the filler resin interface. The polymer resin matrix is composed initially of a liquid monomer that converts to a highly cross linked polymer upon initiation of polymerization, whether chemically or using light. The filler particles enhance the mechanical and physical properties and reduce the fraction of resin in the final restoration. Finally, the filler resin interface couples these two components together [12].

The most used monomers are the di-functional long chained BIS-GMA and UDMA making them extremely viscous. Therefore, they are diluted with another di-functional monomer, TEGDMA, of much lower viscosity, in order to practically be able to add larger portion of filler particles and to manipulate the material. The

corresponding monomer composition can be shown in Figure 2. Much development has taken place in monomer types and compositions, for example, several bisphenol A free composites are now available in the market.

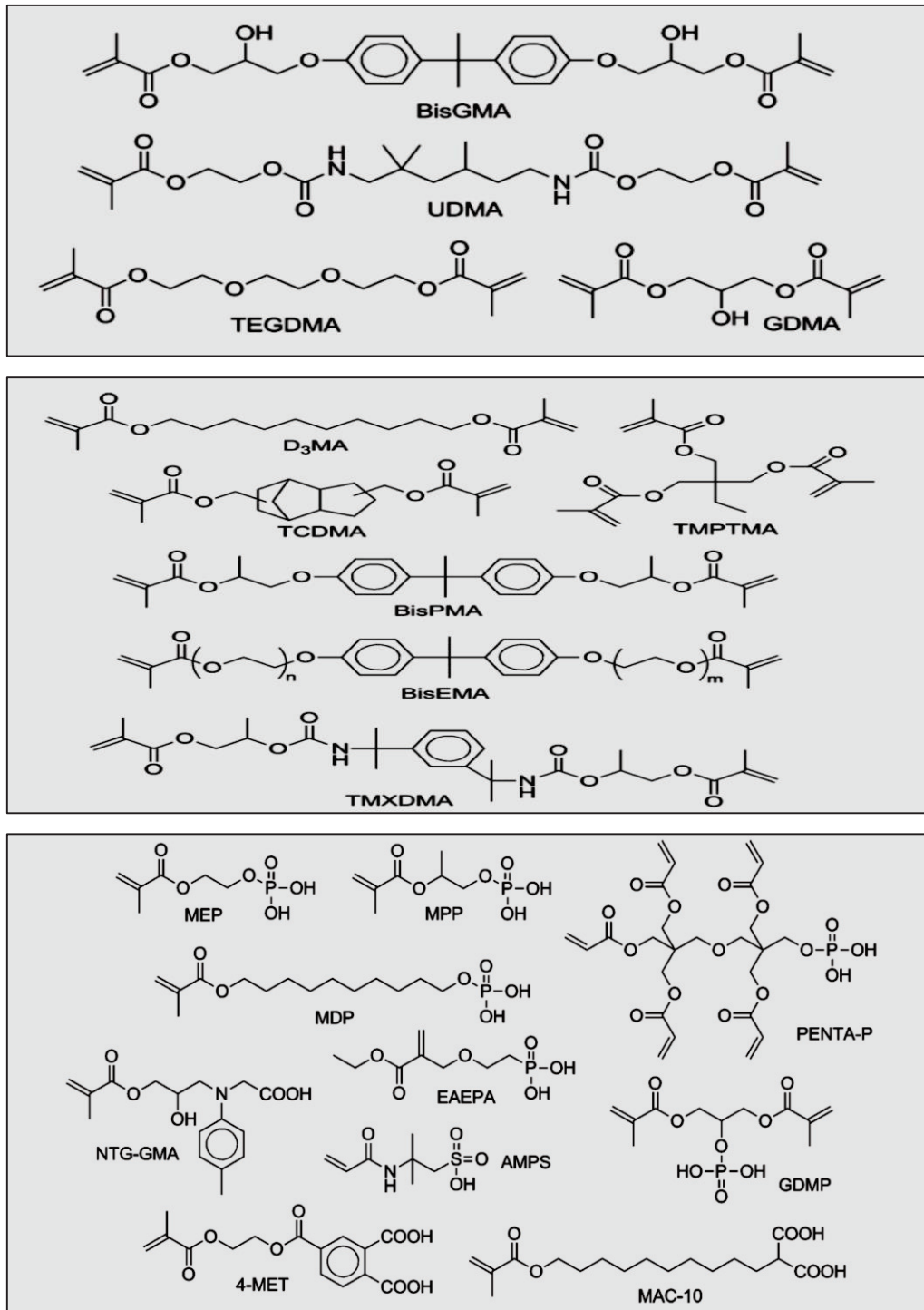


Figure 2: The composition of several monomer types used in dental composite resin restorations; (Moszner et al. 2012) [13].

The filler particle mainly used in dental composite is silica, but the filler composition is often modified with other ions to produce desirable changes in properties. Lithium and aluminum ions make the glass easier to crush to generate small particles. Barium, zinc, boron, zirconium, and yttrium ions have been used to produce radio opacity in the filler particles. On the other hand, excessive modification (by replacement of the silicon in the structure) can reduce the efficacy of silane coupling agents [2].

Composites, generally, are classified with respect to the components, amounts, and properties of their filler or matrix phases or by their handling properties. The most common classification method is based on filler content (weight or volume percent), filler particle size, and method of filler addition.

Composites also could be defined on the basis of the matrix composition. The corresponding size and distribution of filler particles can be shown in Figure 3.

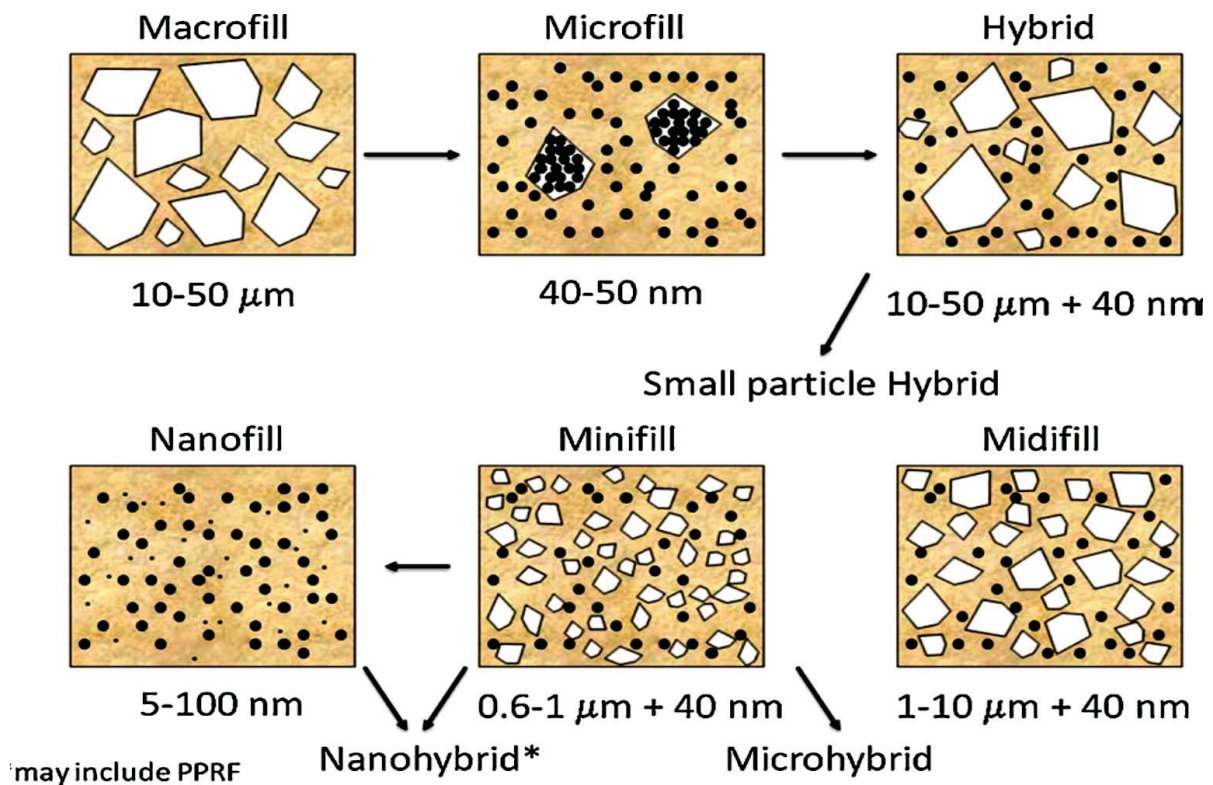


Figure 3: Differences in filler size and distribution in dental composite resin (Ferracane 2011) [3]

Almost all important properties of composites are improved by using higher filler levels. The only practical problem is that, as the filler level is increased, the fluidity decreases. The degree of filler addition is represented in terms of the weight percent or volume percent of filler. As the overall filler content increases, the physical, chemical, and mechanical properties generally improve. Relevant properties of different types of dental composite can be shown in Table 1.

Polymerization of acrylic resin monomers used in dentistry takes place in stages named activation, initiation, propagation, and termination. Activation involves the production of free radicals. Initiation is the step in which free radicals react with monomer units to create the initial end of a polymer chain. Propagation is the addition of monomer to the growing chain. Termination is the conclusion of the process as a result of steric hindrance, lack of monomer, or other problems.

Composites originally were designed for restoration of Class III, IV, and V tooth preparations, but now are used in modified forms for most other restorative dental uses. Based on their intended application, they can be used in all Classes (I-VI) of restorations, cements, bases, cores, veneers, or repair materials.

	Macrofill	Midifill	Microfill	Hybrid	Flowable	Packable	Mini-Nano-Hybrid
Material	Adaptic	Concise	Heliomolar	Herculite XRV	Ælitedflo	Surefil	Filtek Supreme
Manufacturer	J&J	3M	Kulzer	Kerr	Bisco	Dentsply	3M-ESPE
Filler level (weight %)	78	81	70	79	60	77	—
Filler level (volume %)	64	68	48	66	42	65	—
Depth of cure (mm)	—	—	—	6.1	5.6	5.5	—
Flexural modulus (GPa)	—	—	5.8	10.2	5.4	10.3	7.2
3-pt. flexure strength (MPa)	100	111	85	135	—	100	150
Compressive strength (MPa)	236	262	210	285	203	256	225
Diametral tensile strength (MPa)	—	—	36	45	34	34	35
Fracture toughness (MPa • m ^{1/2})	[Poor]	[Poor]	0.8	1.2	1.1	1.2	1.3
Diamond pyramid hardness (kg/mm ²)	—	—	70	68	—	96	85
In vitro wear (µm/100K cycles)	—	—	12	9	28	2	7

Table 1: Relative properties of different composite resin restorations, the values are reported from a variety of sources including manufacturer's product bulletins (Sturdevant's Art and Science of Operative Dentistry) [14].

Developments in resin composite restorations

Currently, the high demand for these restorations and their structural composition allow for significant room for advancements, particularly with respect to their mechanical properties: polymerization shrinkage, thermal expansion coefficient, fracture resistance, marginal leakage, and toxicity [15,16].

These shortcomings reduce the lifetime of composite resin restorations and represent the driving force for improvement in dental composites. Clinical evaluations and laboratory-based studies continue to highlight the need for improved dental restorative composites [17]. Each component of the resin composite represents an opportunity for improvement and is the target of recent research.

In fact, one of the main advantages of restorative composite resin is its limitless ability for development and modification. Some examples of the potential advances, that could develop restorative resin composite, include:

❖ Development of the initiator system

The use of benzoyl germanium initiators in place of the Camphorquinone / Amine. Systems show better shelf stability and depth of cure [18].

❖ Development of the polymerization reaction

- Soft start curing was studied in depth, and though it was originally hypothesized that it allowed for stress relaxation, it was found that there was subsequent reduction in the degree of conversion and mechanical properties [19].
- Polymerization-induced Phase Separation was considered to decrease the volumetric shrinkage taking place during the polymerization reaction [20].
- Hybrid polymerization reactions forming interpenetrating polymer network (IPN) where the material is formed from two distinct polymerizations with

significant bonds between the two. These materials exhibit low toxicity and reduced shrinkage stress [21].

- Ring-opening Polymerization reaction, that depend on opening of a cyclic structure to facilitate intermonomer bonding and crosslinking, lead to less volumetric shrinkage. The most pronounced example is the silorane composite [22].
- Bulk filling composites are claimed to be more efficiently polymerized, and are then to be inserted in thicker increments in posterior cavities.

❖ **Development in the Polymer component**

- Bile acids and Fluorinated derivatives of BisGMA into BisGMA / TEGDMA resins were found to reduce water sorption and volumetric shrinkage. But their high viscosity led to limited commercial incorporation of these monomers [23].
- Ultra rapid Monomethacrylates like Monovinyl (meth) acrylate monomers, showed rapid polymerization rate and resulted in adequate mechanical properties and higher degree of conversion compared to conventional composite systems [24].
- Acidic monomers enabled the adhesive layer to be eliminated. On the other hand, they increased the hydrophilicity of these materials limiting their use.

❖ **Development in the filler system and addition of components**

- Eliminating the silane-mediated interface between filler and matrix by direct mechanical interlocking by using mesoporous silica fillers or single-walled carbon nanotubes (SWCNT) as a secondary filler [25].

- Calcium phosphate nanoparticles with silicon nitride whiskers or other composites to promote remineralization [26].
- Fiber reinforced composites (FRCs): Composite made of a polymer matrix that is reinforced by fine thin fibers

More than 50% of the glass fibers used for reinforcement is E-glass. They are a mixture of amorphous phases and silicon oxide, calcium oxide, barium oxide, aluminum oxide and some oxides of alkali metals. Orientation of fiber plays an important role in increasing the strength. The reinforcing effect of the fiber fillers is based, not only on stress transfer from polymer matrix to fibers, but also on the behavior of individual fibers as crack stoppers. Xu et al. showed that increasing the glass fiber length generally increased the ultimate strength and fracture resistance. These properties have clinical significance and would affect the longevity of restoration [27]. Their conclusion was that the Quantity of glass fibers should be defined by volume percentage and not in weight percentage.

Coating of fiber with polymer matrix avoid voids between the matrix and the fiber that would lead to decreasing the load bearing capacity and water sorption. Other important factors are the adhesion of fiber to the polymer matrix and the distribution of fibers. Mechanical properties such as strength, stiffness, toughness and fatigue resistance as well as the linear coefficient of thermal expansion (LCTE) depend upon the geometry of the reinforcement and the fiber orientation (Krenchel's factor) [28].

Short fiber reinforced composite (everX Posterior™) has been introduced as a dental restorative composite resin. Studies showed improvements in the load bearing capacity short glass fiber composite

resin, has also exhibited control of the polymerization shrinkage stress by fiber orientation. Thus, marginal microleakage was reduced compared with conventional particulate filler restorative composite resins. The specific mechanical and physical strength and the specific modulus of these fiber reinforced composite materials may be markedly superior to those of existing resin-based composites and metallic materials.

A study was conducted by our group to test and compare fiber composite to other bulk fill composites. Several mechanical properties, correlated to the composite longevity in the oral cavity, were chosen. The results obtained showed that the mechanical properties of the fiber composite were among the best, making it suitable to be used in posterior stress bearing regions in the oral cavity.

Original Research Article: Comparison of mechanical properties of a new fiber reinforced composite and bulk filling composites

Comparison of mechanical properties of a new fiber reinforced composite and bulk filling composites

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Objectives: The aim of this study was to evaluate the mechanical and physical properties of a newly developed fiber reinforced dental composite. **Materials and Methods:** Fiber reinforced composite EverX Posterior (EXP, GC EUROPE), and other commercially available bulk fill composites, including Filtek Bulk Fill (FB, 3M ESPE), SonicFill (SF, Kerr Corp.), SureFil (SDR, Dentsply), Venus Bulk Fill (VB, HeraeusKulzter), Tetric evoceram bulk fill (TECB, Ivoclar Vivadent), and Xtra Base (XB, Voco) were characterized. Composite samples light-cured with a LED device were evaluated in terms of flexural strength, flexural modulus (ISO 4049, $n = 6$), fracture toughness ($n = 6$), and Vickers hardness (0, 2, and 4 mm in depth at 24 hr, $n = 5$). The EXP samples and the fracture surface were observed under a scanning electron microscopy. Data were statistically analyzed using one-way ANOVA and unpaired t -test. **Results:** EXP, FB, and VB had significantly higher fracture toughness value compared to all the other bulk composite types. SF, EXP, and XB were not statistically different, and had significantly higher flexural strength values compared to other tested composite materials. EXP had the highest flexural modulus, VB had the lowest values. Vickers hardness values revealed SF, EXP, TECB, and XB were not statistically different, and had significantly higher values compared to other tested composite materials. SEM observations show well dispersed fibers working as a reinforcing phase. **Conclusions:** The addition of fibers to methacrylate-based matrix results in composites with either comparable or superior mechanical properties compared to the other bulk fill materials tested. (*Restor Dent Endod* 2015;40(4):262-270)

Key words: Bulk composite; Fiber composite; Mechanical properties

Introduction

Dental composite resin recently became the material of choice for most patients and dental practitioners.¹ However, volumetric shrinkage and fracture are still considered as major concerns with dental composites.^{2,3} In order to overcome these weaknesses, attempts have been made toward increasing both their physical and mechanical properties.⁴ This necessitates the comprehensive appraisal of each of its components such as the resin matrix, the filler or the filler-resin interface, and their role in affecting the material properties. Different studies have investigated this in order to improve composite properties, either by varying the particle size, percentage, or by development of the polymer matrix chemistry.^{4,5}

Evolution in both filler and polymer technology in dental composite resins led to a wide selection of materials that provide the adequate properties required for each clinical situation.⁴ Yet, the use of dental composites in high stress bearing areas remains to be a challenge for the dental practitioner, since bulk fracture is still considered one of the primary reasons for failure.^{2,6} Bulk fill composites were introduced in an effort to improve the performance of composite resin restorations, which was inserted in 4 mm increments mainly in the posterior areas and considered to have higher physical and mechanical properties to endure the higher masticatory stresses. Moreover, the reduced treatment time decrease the risk of air entrapment or moisture contamination.⁷ They are also claimed to reduce cuspal deflection and promote light transmittance.^{7,8} Currently, various studies reveal the difficulty in comparing between the available materials due to variation in composition and viscosity.⁹⁻¹¹

Bulk filling composites usually have higher filler volume percentage, and sometimes a modified initiator system to ensure better curing in depth, as compared to conventional composites. While no long term clinical studies are available regarding their intraoral performance, Ilie *et al.* found bulk filling composites to have lower mechanical properties, except for flexural strength as compared to nanohybrid and microhybrid resin based composites.⁹ However, other studies found them equally successful compared to conventional composites.^{7,12} Many bulk fill composite resins have been investigated regarding different parameters like degree of conversion, polymerization stress or microleakage. Such studies have shown that bulk fill composite resins have similar properties as conventional dental composite resins.¹²⁻¹⁶

Finan *et al.* studied the degree of conversion, biaxial flexural strength and Vickers hardness of two flowable bulk composites (SDR and XB), and despite the differences between the two materials, found that the properties justify their use in 4 mm increments.¹⁷ The variation in material composition and viscosity, whether flowable or non flowable bulk composites, leads to differences in physical and mechanical properties among the bulk fill composites available in the market.⁷ Fiber reinforcement of conventional dental composites were also introduced with the aim of enhancing their physical and mechanical properties, and increasing their resistance to fracture. The enhancement of the material properties was due to the stress transfer from the matrix to the fibers depending on the fibers length and diameter. Garoushi *et al.* studied their effect, and found a significant improvement in the materials physical properties.¹⁸

It was deemed important to investigate the role of fibers added to composite compared to other commonly used bulk fill composites, and to examine the extent to which fiber reinforcement would enhance the mechanical properties of

the materials. Multiple laboratory investigations have been used to evaluate dental composite resins; standardized tests present the advantage of being easily reproducible in laboratories, and allowing values obtained by different institutes to be compared. Moreover, they provide preliminary information about the material suitability in the oral environment and the extent to which they conform to the indications prescribed by the manufacturer.¹⁹ Heintze *et al.* found that flexural strength and flexural modulus tests can be used as a good indicator for the material durability under stress, and correlate well with the clinical longevity.¹⁹ Fracture toughness test was considered by Ilie *et al.* as another important method that investigates the material's ability to endure stress without fracture and monitor the crack propagation inside the material before failure.¹¹ On the other hand, Vickers hardness assay, one of the most used mechanical experiments examines the material surface hardness, and scanning electron microscope observations reveal important information about the samples used and the mode of failure of the material.⁹ Standard ISO flexural strength and modulus tests consider only 2 mm thickness samples. However bulk fill composites are indicated to be used clinically in 4 mm thick increments, and accordingly investigating the material at this thickness seems more appropriate.

The aim of this study was to investigate the mechanical properties of a fiber reinforced composite compared to other commonly used bulk fill composites, and to consider its performance under laboratory settings. The null hypothesis was that there is no significant difference in mechanical properties (flexural strength, flexural modulus, fracture toughness, and Vickers hardness) among the fiber reinforced composite and other bulk fill composites.

Materials and Methods

Bulk fill dental composites used in the study were X-tra base (XB, Voco GmbH, Cuxhaven, Germany), Venus bulk fill (VB, HeraeusKultzer, Hanau, Germany), Filtek bulk fill (FB, 3M ESPE, St. Paul, MN, USA), Surefil SDR (SDR, Dentsply, Milford, DE, USA), Tetric evoceram bulk fill (TECB, Ivoclar Vivadent AG, Schaan, Liechtenstein), SonicFill (SF, Kerr Corp., Orange, CA, USA), and a fiber reinforced bulk fill dental composite resin, EverX Posterior (EXP, GC EUROPE NV, Leuven, Belgium). The compositions of bulk fill materials used, their shade as well as their lot numbers are listed in Table 1.

For the fracture toughness test, flexural strength and modulus tests, the number of samples for each of the materials used was 6. The tested samples were polymerized using GC G-light unit (GC EUROPE NV) from both sides for 40 seconds. A modified flexural strength test was performed using bulk fill samples with 4 mm² cross sectional areas polymerized only from the top side as done

Table 1. Materials, manufacturers, chemical composition of the matrix, fillers and filler contents

Material	Code name	Manufacturer	Resin matrix	Inorganic filler	Lot
X-tra base	XB	Voco, Cuxhaven, Germany	Bis-EMA, MMA	75 wt%, 58 vol% silica	1305261
Venus Bulk Fill	VB	HerausKultzer, Hanau, Germany	UDMA, EBADMA	65 wt%, 38 vol% Barium silicate glass and silica	100325
Filtek Bulk Fill	FB	3M ESPE, St. Paul, MN, USA	Bis-GMA, Bis-EMA, UDMA	64 wt%, 42 vol% Zirconia	N414680
Surefil SDR	SDR	Dentsply, Milford, DE, USA	TEGDMA, EBADMA	68 wt%, 44 vol%, Barium borosilicate glass	1202174
Tetric EvoCeram Bulk Fill	TECB	Ivoclar Vivadent AG, Schaan, Liechtenstein	Bis-GMA, UDMA, Bis-EMA	80 wt%, 61 vol% Barium glass filler	S01118
SonicFill	SF	Kerr Corp., Orange, CA, USA	Bis-GMA, Bis-EMA, TEGDMA	83 vol% Filler	4252491
EverX Posterior	EXP	GC EUROPE N.V., Leuven, Belgium	Bis-GMA, PMMA, TEGDMA	74.2 wt%, 53.6 vol% Short E-glass fiber filler, barium glass	1212261

Bis-EMA, ethoxylatedbisphenol A dimethacrylate; MMA, methylmethacrylate; UDMA, urethane dimethacrylate; EBADMA, ethoxylatedbisphenol A dimethacrylate; Bis-GMA, bisphenylglycidyl dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; PMMA, polymethyl methacrylate.

in the clinical situation. The wavelength of the light was between 380 and 520 nm with maximal intensity at 470 nm and light intensity was 1,150 mW/cm². The specimens from each group were stored in water at 37°C for 48 hours before testing.

Fracture toughness

To measure the fracture toughness (K_{IC}), rectangular glass molds that were lined with polyester strips (Striproll, Kerrhawe SA, Bioggio, Switzerland) were used to prepare single-edge-notched specimens. The cured samples (3 mm × 6 mm × 25 mm) were removed without using force. A sharp central notch of specific length (a) was produced by inserting a razor blade into an accurately fabricated slot at mid-height in the mold. The slot extended down half the height to give $a/W = 0.5$. The crack plane was perpendicular to the specimen length. The length of the crack was checked using a stereomicroscope.

Fracture toughness K_{IC} was calculated from the following formula:

$$K_{IC} = \left[\frac{3PL}{2BW^3} \right] Y$$

Where P is the peak load at fracture, L is the length, B is the width, W is the height, a is the average notch depth, and Y is the calibration functions for given geometry

$$Y = 1.93[a/W]^{1/2} - 3.07[a/W]^{3/2} + 14.53[a/W]^{5/2} - 25.11[a/W]^{7/2} + 25.80[a/W]^{9/2}$$

Flexural strength and flexural modulus

According to the ISO 4049, samples for a three point bending test were prepared in Teflon molds between two glass slabs, resulting in bar shaped specimens (2 mm × 2 mm × 25 mm). The test was conducted under a cross-head speed of 0.5 mm/min, with a span length of 20 mm and an indenter diameter of 2 mm. All specimens were loaded in a Universal Mechanical testing machine (Servo hydraulic - Adamel Lhomargy DY-34, MTS, Roissy-en-Brie, France). Flexural strength and modulus tests, were repeated on larger samples ($n = 6$, 4 mm × 4 mm × 25 mm), that were cured only from the top, using the same light and stored in water at 37°C for 48 hours before testing.

Flexural strength (O_f) and flexural modulus (E_f) were calculated from the following formulas:

$$O_f = \frac{3FmI}{2bh^2}$$

$$E_f = \frac{SI^3}{4bh^3}$$

Where Fm is the applied load (N) at the highest point of load-deflection curve, I is the span length (20 mm), b

is the width of test specimens and h is the thickness of test specimens. S is the stiffness ($S = F/d, N/m$) and d is the deflection corresponding to load F at a point in the straight-line portion of the trace.

Vickers hardness test

The Vickers hardness test was performed with Leitz microhardness device (Leitz, Wetzlar, Germany), under a force of 200 g for 30 seconds. Ten samples for each material were prepared using a 5 mm diameter Teflon mold, with either 2 mm ($n = 5$) or 4 mm thickness ($n = 5$), placed between 2 glass plates. The materials were polymerized only on one side for 40 seconds. The excesses were removed by polishing the 2 surfaces using abrasive paper discs of decreasing coarseness from 2,400 to 4,000 grits (Struers SAS, Champigny sur Marne, France) at 3,000 rpm under water irrigation. The top surface (polymerized) and the bottom surface (non-polymerized) were marked to be identified. Each sample was tested 5 times on each side, at 24 hours after immersion in distilled water at 37°C. The Vickers hardness was calculated using the formula:

$$HV = 1854.4 \frac{P}{d^2}$$

Where P (g) is the load applied, and d is the average of the 2 diagonals of the surface of the diamond indentation.

Scanning electron microscopy

Scanning Electron microscope (S800-1, Hitachi Europe Ltd., Whitebrook, Berkshire, UK) observations were conducted under x80, x100, and x250 magnification to

examine the fracture mode, and to measure the fiber’s diameter and length. Samples were dried, sputter-coated with metal, and observed. The type of fracture was determined for each specimen.

Statistical analysis

The statistical analysis of the current data was performed using the application of one-way analysis of variance (ANOVA). The results were compared between each test and between each material type using unpaired t -test. The results are reported as mean \pm SD. Statistical significance was accepted at $p < 0.05$.

Results

Fracture toughness and Vickers hardness of tested composite materials are presented in Figures 1 and 2. Flexural strength and flexural modulus are presented in Table 2. The fiber reinforced composite EXP had significantly higher fracture toughness value ($3.1 \text{ MPa}\cdot\text{m}^{1/2}$), compared to other bulk composites except for FB ($2.52 \text{ MPa}\cdot\text{m}^{1/2}$) and VB ($2.26 \text{ MPa}\cdot\text{m}^{1/2}$) where no significant difference was found with EXP. In the normalized flexural strength test (2 mm x 2 mm x 25 mm), SF (157.6 MPa), EXP (153.6 MPa), XB (150.4 MPa) and FB (140.0 MPa) were not statistically different, and these have significantly higher flexural strength values compared to other tested composite materials, except for FB which was similar to SDR. EXP had significantly higher flexural modulus (14.6 GPa), while SF (12.47 GPa), TECB (10.87 GPa), and XB (10.65 GPa) were not significantly different and came in second position. On the other hand, VB (5.02 GPa) had the lowest statistically significant value.

Table 2. Flexural strength (σ , MPa) and Flexural Modulus (E_{flexural} , GPa) for the 2 mm and 4 mm sample groups

	Flexural strength (σ , MPa)		Flexural Modulus (E_{flexural} , GPa)	
	2 mm	4 mm	2 mm	4 mm
SonicFill	157 \pm 16 ^K	147 \pm 20 ^k	12.4 \pm 1.6 ^A	6.5 \pm 0.5 ^{a*}
EverX Posterior	153 \pm 9 ^K	140 \pm 14 ^{kl}	14.6 \pm 1.6 ^A	6.9 \pm 0.5 ^{a*}
X-tra base	150 \pm 8 ^K	124 \pm 13 ^{lm}	10.7 \pm 1.3 ^A	5.7 \pm 0.4 ^{b*}
Filtek bulk fill	139 \pm 9 ^{KL}	139 \pm 9 ^{kl}	6.3 \pm 0.4 ^B	4.0 \pm 0.1 ^{c*}
Surefil SDR	129 \pm 13 ^{LM}	121 \pm 19 ^{lm}	7.2 \pm 1.2 ^B	3.9 \pm 0.4 ^{c*}
Tetric evoceram bulk fill	118 \pm 11 ^L	112 \pm 26 ^L	10.8 \pm 0.6 ^A	5.6 \pm 0.2 ^{b*}
Venus bulk fill	116 \pm 5 ^L	121 \pm 10 ^L	5.0 \pm 0.4 ^B	2.8 \pm 0.2 ^{d*}

Uppercase letters identify statistically homogenous groups for 2 mm thickness samples. Lower case letters identify statistically homogenous groups for 4 mm thickness samples. Asterisks identify statistical difference between 2 mm and 4 mm thickness samples of the same material ($p < 0.05$).

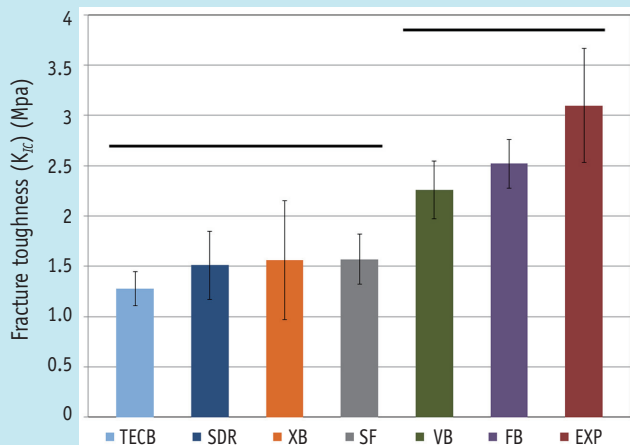


Figure 1. Bar graph illustrating fracture toughness (K_{IC}). Straight line indicates that there was no statistically significant difference between the groups. TECB, Tetric evoceram bulk fill; SDR, SureFil SDR; XB, Xtra Base; SF, SonicFill; VB, Venus bulk fill; FB, Filtek bulk fill; EXP, EverX Posterior.

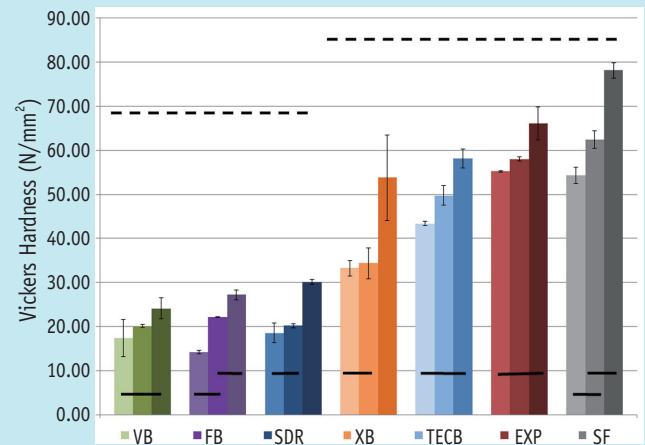


Figure 2. Bar graph illustrating Vickers hardness (N/mm^2) at different curing depths of 4 mm, 2 mm and at the surface. Dotted line (-----) indicates that there was no statistically significant difference between the materials. Straight line (—) indicates that there was no statistically significant difference within the same material at different curing depths. TECB, Tetric evoceram bulk fill; SDR, SureFil SDR; XB, Xtra Base; SF, SonicFill; VB, Venus bulk fill; FB, Filtek bulk fill; EXP, EverX Posterior.

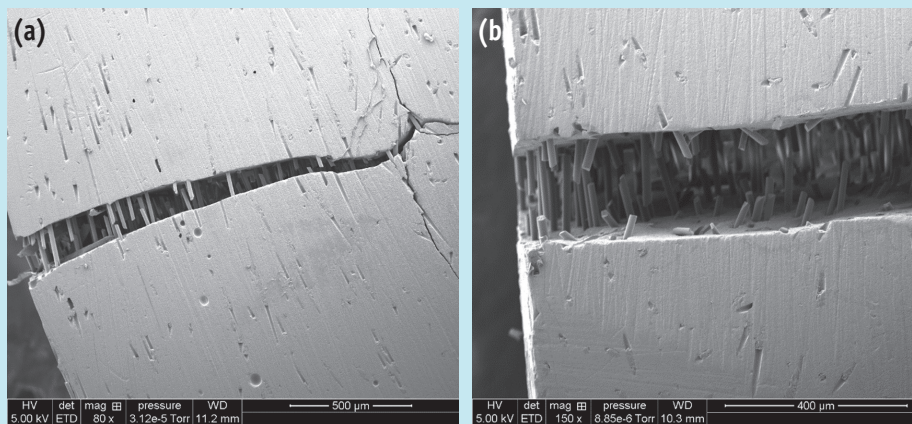


Figure 3. Scanning electron photomicrograph of fracture toughness sample (a) after failure; (b) the fiber orientation across the failure line are shown at higher magnification.

Considering the modified test (4 mm × 4 mm × 25 mm), there was no significant difference from those obtained in the original test, except for XB and they had the same order of the strength values from SF (147.67 MPa), EXP (140.04 MPa), and FB (139.62 MPa). On the other hand, the flexural modulus values decreased significantly in comparison with the normalized test, attaining almost half the original value, while remaining in the same order, with the highest flexural modulus for EXP (6.89 GPa), together with SF (6.55 GPa), followed by XB (5.7 GPa) and FB (4.01

GPa), which were less affected in comparison with original test.

Vickers hardness values revealed that SF had the highest value followed by EXP. The decrease in hardness between the surface and 2 mm and 4 mm depths were not significant for EXP, TECB, and VB, while other bulk composites revealed a significant difference between the curing depths.

SEM analysis revealed that the fibers stop the crack propagation along the fracture line, as shown in Figure 3.

Discussion

According to the results obtained in the current study, the null hypothesis was rejected, that is, fiber insertion into composite leads to significant increase in physical and mechanical properties, such as flexural strength, flexural modulus, fracture toughness, and Vickers hardness.

In this study, flexural strength and modulus were investigated. These tests are considered to be good indicators of the material resistance to fracture in normal masticatory conditions, taking in account the great variability in the results obtained between studies.^{19,20} The results obtained are in accordance with previous studies conducted on bulk composites. SF, EXP, XB, and FB had significantly higher flexural strength values, compared to VB and TECB which had the lowest.^{9,18,21} Moreover, as shown in previous works, the filler volume percentage is closely related to the flexural strength and flexural modulus values.^{9,18,21} This can be shown for SF with the highest filler volume percentage (83%) ranking the highest, TECB (61%) and XB (58%) follow next, while VB with the lesser filler volume percentage (38%) ranking the lowest. Interestingly, EXP (53.6%) performed relatively better in these two tests compared to its filler volume percentage, showing the role of the fibers in increasing the material stiffness and resistance to bending force during testing and probably during function.

In this work, the modified flexural strength and modulus tests were done on 4 mm increments cured only from the top side in an effort to mimic the clinical situation. This would eventually mean less matrix polymerization and, accordingly, a larger role of the filler type and percentage in the material behavior. The results obtained show that the flexural strength values remained significantly unchanged. In comparison to the original test, significant decrease in the flexural modulus values of the composites tested indicated a marked decrease in rigidity. This is probably due to an increase in thickness of the increments and decrease in the overall matrix polymerization. A probable explanation would be that, as a result of less matrix polymerization and the consequent lack of rigidity, the modified test samples were able to withstand flexure even at greater load relative to greater sample thickness (hence unchanged flexural strength) but with more deformation before final failure (hence lower modulus of flexure). These results, when confirmed with further studies, would throw more insight on an important aspect regarding the amount of deformation and the distortion of the material due to the decreased stiffness, most notably at the interface region. This would also provide some explanation for the discrepancies found between results obtained in the laboratories and those from clinical studies in which bulk materials are inserted in larger and thicker increments and cured only from one side.¹⁹

Results obtained acknowledge the role of fibers in increasing the material's resistance to fracture, and coincide with those of previous studies.^{18,22} The single edge notched beam method used in this study is one of the most commonly used fracture toughness test methods, which are used to predict resistance to fracture. The method is widely used in dental material research and is usually conducted by means of a 3 point bending apparatus, and the sharp crack created could be easily measured. This method is also very sensitive to the notch width and depth, thus making comparison difficult between different studies.^{11,18} In the present work, no correlation was found between the fracture toughness value and the filler volume percentage or the filler particle size.

The enhancement of the material properties was explained to be due to the stress transfer from the matrix to the fibers and also due to the action of the fibers in stopping crack propagation through the material.²³ It was found that the mere insertion of fibers is not enough to enhance the composite properties, that is, the fibers length and diameter play a critical role in this mechanism. Peterson found that fibers incorporated into a material, greatly enhances its mechanical properties, on the condition that the fibers have a length that exceeds a certain minimum length. This is known as the critical fiber length, which could be calculated using the following formula:²⁴

$$l_c = \frac{f^d}{2c}$$

Where the critical length (l_c) equals the ultimate tensile strength of the fiber (f) multiplied by the fiber diameter (d), and divided by twice the shear strength of the matrix interface (c)

The physical explanation of the strengthening and stiffening mechanism is that since the matrix has a much lower modulus than the fiber, the matrix strains more. The critical fiber length is therefore the minimum length at which the center of the fiber reaches its ultimate tensile strength when the matrix reaches its maximum shear strength. Accordingly, composite with fibers below critical length fail to show enhanced properties.¹⁸ In the present study, we were able to measure the fiber length and diameter using stereomicroscopy and SEM, and we found that EXP had a fiber diameter of 16 μm and a wide range of fiber length, with the average length lying between 1 and 2 mm similar to the values found in previous studies, thus exceeding the fiber length required.¹⁸ The fiber length and orientation can be shown in Figure 4.

One interesting observation found from the fracture toughness and the flexural strength test samples alike was that all fiber reinforced composite EXP samples remained attached, even after failure of the sample and formation of crack line, unlike the samples from other bulk fill brands

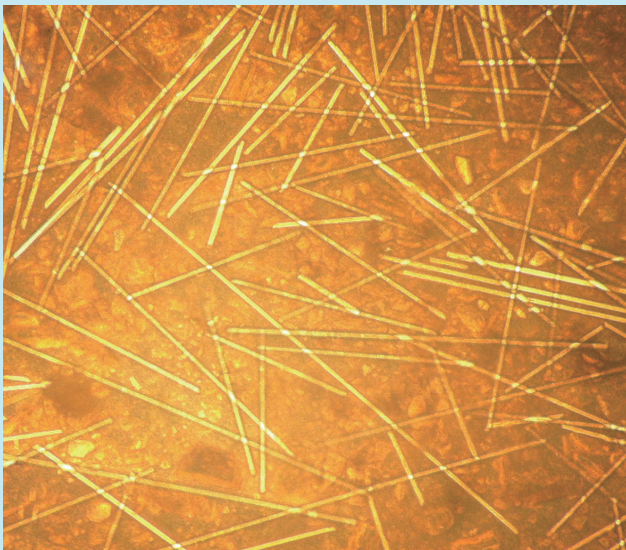


Figure 4. Microscopic image of EverX Posterior showing fiber length extending to the length of one millimeter and up to two millimeters.

which separated in two pieces the time the failure load was reached, as can be seen in Figure 5. Scanning electron microscope observations performed on fractured samples show the fibers traversing the crack line and between the fractured parts, as can be seen in Figure 3. Further investigation of this property is important clinically, since not only it shows the material resistance to fracture, but also its resistance to displacement at the more vulnerable interface, thus preventing cavitation and food impaction. Moreover, this property would render the material with better potential for repair.

The Vickers microhardness test samples show that SF, EXP, TECB, and XB have the highest values compared to SDR, FB, and VB. It is worth noting that though this method was criticized as an unreliable indicator of the curing quality, and that it overestimates the depth of cure. Flury *et al.* have shown that Vickers microhardness could be considered as an accurate tool for estimating the polymerization depth for bulk composite resins.^{25,26} Moreover, only SF and EXP had bottom surface hardness values that exceeded the 50 VHN considered ideal.²⁷ However, EXP is the only composite with Vickers hardness value at 4 mm depth that exceeded the 80% ratio compared to the top surface hardness as required in literature.²⁶⁻²⁹ The results thus obtained provide evidence that EXP could be used in 4 mm increments for tooth cavity fillings.

The present results were obtained in optimized laboratory settings, however, clinical conditions are not similar and the aspects like insertion and handling could have a potential effect on the mechanical properties of the materials and their performance *in vivo*. Another important

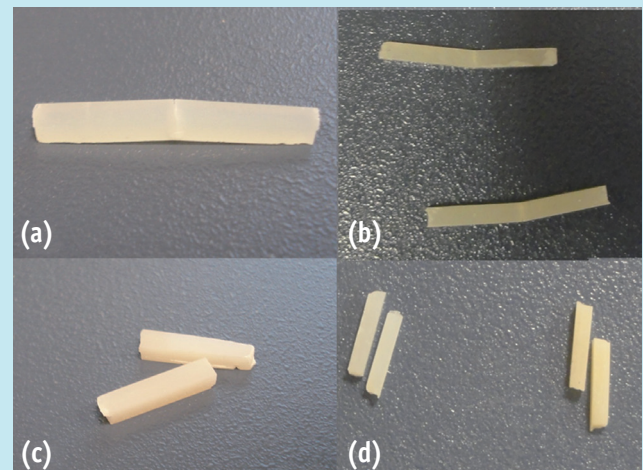


Figure 5. Samples of (a) fracture toughness and (b) flexural strength tests for EverX Posterior remained connected after failure, compared to other bulk composite samples after (c) fracture toughness and (d) flexural strength, which were completely separated into two fragments.

factor that should be taken into consideration as one of the limitations of the current study is the fiber alignment inside the composite in relation to the acting force, which is not necessarily consistent with the laboratory simulations performed during *in vitro* testing. Some of the important aspects considering the materials polymerization contraction and contraction stress were not included in the study. Further investigations should be conducted to test other material properties. According to the results obtained in this work, the fiber reinforced composite tested may be used as a restorative material in stress bearing areas. In order to acknowledge the results obtained with the present study, this should be followed by long term clinical studies to assure the materials performance under normal clinical conditions.

Conclusions

In the current study, fiber reinforced composite EXP had either comparable or superior resistance to fracture, flexural strength and modulus, as well as high microhardness values, compared to other bulk fill composite resins.

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Conflict of Interest: No potential conflict of interest relevant to this article was reported.

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In the light of the above study, and according to our results and those of various similar studies, it can be concluded that the tests performed on these composites reveal the variation in their mechanical properties. On the other hand, the presence of the mastication bench device would allow the mechanical testing of these composites under more relevant conditions and under forces that closely resembles these experienced in the human oral cavity.

The dental composites studied would be inserted and polymerized inside the mastication bench using the same clinical protocol utilized during a real clinical session. This would disclose more about the actual performance of the material, as could be observed in the example of the 2 and 4 mm thickness flexural test samples.

Moreover an important feature as fiber alignment in reinforced composite would be closely monitored with relevant ease.

1.1. Ceramics and CAD/CAM restorations

The word ceramics is used to designate materials having both metallic and nonmetallic ions in their compositional formula.

Dental ceramics are nonmetallic, inorganic structures, primarily containing compounds of oxygen with one or more metallic or semi metallic elements (aluminum, boron, calcium, cerium, lithium, magnesium, phosphorus, potassium, silicon, sodium, titanium and zirconium). Many dental ceramics contain a crystal phase and a silicate glass matrix phase [2].

They consist of silicate glasses, porcelains glass ceramics or highly crystalline solids. The properties of ceramics are customized for dental applications by precisely controlling the types and amounts of the components used in their production.

Ceramics do not react readily with most liquids gases or alkalis and weak acids, and they remain stable over long time periods. Dental ceramics are characterized by excellent strength, high hardness, biocompatibility, chemical inertness and esthetic potential.

Conversely, they are generally brittle and may fracture without warning when flexed extensively or when quickly heated and cooled, and they cause intensive wear of the opposing dentition.

The two reasons that seemed to limit the use of ceramics: their brittleness and the great effort and time required for processing. Recent advances in ceramic processing methods have led to much improvement in their mechanical properties and expanded the scope for their use in dentistry.

Classification

While several classifications exist for the dental ceramic materials as shown in figure 4, only the classifications based on microstructural composition and processing method will be discussed in this chapter.

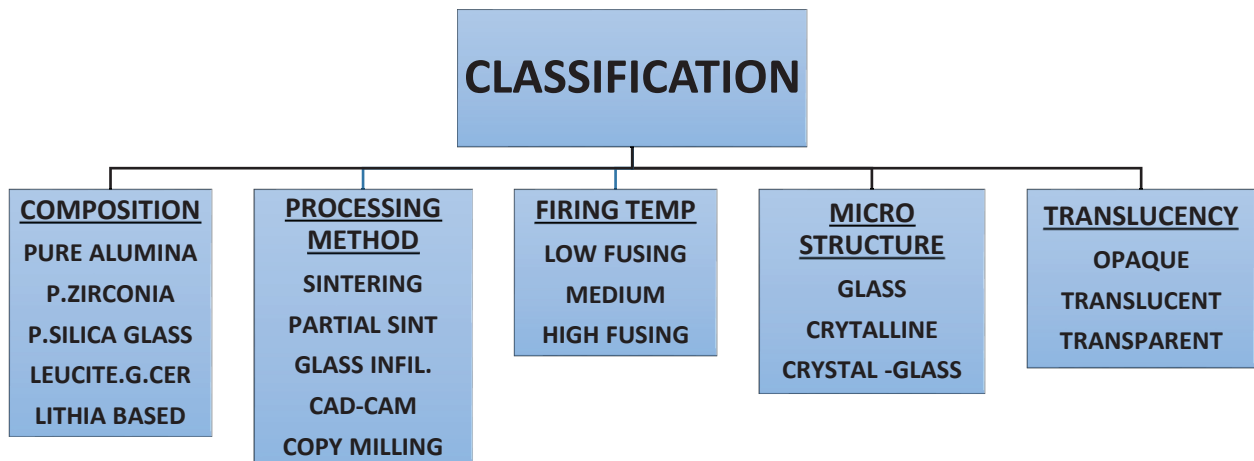


Figure 1: Different classifications of dental ceramics (Philips dental materials) [2].

Microstructural classification

Ceramics can be differentiated according to their composition of glass to crystalline ratio, into four compositional categories:

- Glass-based systems
- Glass-based systems (mainly silica) with fillers, usually crystalline (typically leucite or, more recently, lithium disilicate),
- Crystalline- based systems with glass fillers (mainly alumina)
- Polycrystalline solids (alumina and zirconia).

1.1.1. Glass-based systems (mainly silica)

Glass-based systems are made from materials that contain mainly silicon dioxide and various amounts of alumina. Glasses are 3D networks of irregular interatomic spacing pattern, resulting in an amorphous structure dissimilar from that of crystalline. [29]. Glass based systems are mainly used for veneering ceramics onto metal crowns or bridges. One example is Feldspars which are aluminosilicates containing various amounts of potassium and sodium, these are modified in various ways to create the glass used in dentistry. Synthetic forms of aluminasilicate glasses are also manufactured for dental ceramics.

During its heat treatment, *Ceraming* or conversion from a glass to a partially crystalline glass occurs. This controlled crystallization with the nucleation and growth of internal crystals, result in a multiphase solid containing a residual glass phase with a finely dispersed crystalline phase [30,31].

1.1.1.1. Glass-based systems with fillers

They differ from the pure glass category in that crystalline fillers or, particles of a higher melting glass, have either been added or grown in the glassy matrix. This provides improved mechanical properties and controls optical effects such as opalescence, colour and opacity. [32].

- Feldspathic porcelains are Low-to-moderate leucite-containing feldspathic glass. (Even though other categories have a feldspathic-like glass).
- High-leucite-containing glass (approximately 50%) is also based on an aluminosilicate glass. Leucite is a potassium aluminum silicate mineral with

high coefficient of thermal expansion. These materials have been developed in both powder/liquid, machinable and pressable forms [32].

- Lithium-disilicate glass ceramic are aluminosilicate glass that had lithium oxide added to it (IPS e.max[®]). They are supplied in a pre-crystallized form (blue state), which contains metasilicate and lithium disilicate nuclei. The block can be easily milled at this state, after which, the restoration is recrystallized in a chair-side ceramic oven at 850°C in vacuum for 20–25 min. The new crystalline phase (lithium disilicate $\text{Li}_2\text{Si}_2\text{O}_5$) makes up about 70% of the volume of the glass ceramic. This transition results in increased strength, thermal expansion and contraction. Lithium disilicate consists of randomly oriented small interlocking plate-like crystals, causing cracks to deflect, branch or blunt, thus providing a substantial increase in its flexural strength [33,34].

1.1.1.2. Crystalline-based systems with glass fillers

This structure has 85% crystal content. This system was developed as an alternative to conventional metal ceramics (Glass-infiltrated, partially sintered alumina In-Ceram[™] (Vita)). A ceramic core is formed onto a refractory die from fine slurry of alumina powder by a process known as “slip casting” [2,35].

Slip-casting, involves the condensation of an aqueous porcelain slip on a refractory die. Molten glass is infiltrated at high temperatures into the fired porous core by capillary action.

The obtained material is less porous, having higher strength and toughness and with fewer defects from processing than conventional feldspathic porcelains.

This glass-infiltrated core is later veneered with a feldspathic ceramic having excellent translucency and esthetic qualities. The Vita In-Ceram slip-casting system makes use of three different materials to gain a good compromise between strength and esthetics.

a. In-Ceram® spinell

Spinell (MgAl_2O_4) is a natural mineral of dental significance because of its extremely high melting point (2135°C) combined with its high strength.

b. In-Ceram® alumina

This structure is enriched by up to 60% aluminum oxide crystals by weight with a grain size of 10–30 μm to increase stability. Thus, In-Ceram alumina has strength of around 500 MPa.

On the other hand, intense refraction of light occurs at the aluminum oxide crystals in the feldspar, resulting in the opaque effect of such materials [36]. Accordingly, these poorly translucent materials are only suitable for crown frames fabrication with subsequent veneering.

c. In-Ceram® zirconia

It is composed of a mixture of zirconium oxide and aluminum oxide (33% zirconium, 66% Alumina), resulting in a marked increase in the flexural strength in the core framework. Zirconium oxide is particularly important in protecting the structure against crack propagation. This material has a very high strength of around 700 MPa and very poor translucency.

1.1.1.3. Polycrystalline solids

This dense, air-free, glass-free, polycrystalline structure is formed by directly sintering pure alumina and pure zirconia crystals together into a monophasic material without any intervening matrix [37,38].

Polycrystalline ceramics are formed from powders that can be packed only to 70% of their theoretical density, therefore they shrink in volume by around 30% during firing. Because of their great strength, these materials could only be fabricated using CAD/CAM systems.

a. Pure Alumina

High alumina ceramics generally contain a minimum of 95% pure alumina. The high purity alumina has enough strength that enables it to replace metal crown copings. The grain boundaries shift, during sintering, result in the formation of a closely interlocking crystalline structure of considerable strength (up to 800 MPa). High-alumina ceramics is clearly distinguished from aluminous porcelains by its constituents, the sintering process and the migration of atoms.

High alumina ceramics has a chalky white appearance making it necessary to be veneered by a feldspathic porcelain veneer. On the other hand, sintering shrinkage is no longer a problem.

The dispersion of ceramic crystals within the glassy matrix can increase both the overall strength and elastic modulus, as long as both have similar thermal expansion properties [39].

b. Pure Zirconia

The importance of Pure Zirconia in dental practice is because of its relatively high fracture toughness (9–10 MPa/m^{1/2}) and its flexural strength (900–1200 MPa), which is about twice that of alumina.

Most of the current zirconia ceramic materials used for dental prostheses are based on tetragonal zirconia particles that are fully stabilized with Yttria.

A phenomenon, known as low temperature degradation (LTD), is responsible, for the aging process, in progressive spontaneous transformation of the metastable tetragonal phase into the monoclinic phase, in the presence of water, at relatively low temperatures.

Stabilization of the cubic polymorph of zirconia, over wider range of temperatures, is accomplished by substitution of some of the ions in the crystal lattice with slightly larger ions. Hence, there are Yttria partially stabilized tetragonal zirconia polycrystals, Magnesium partially stabilized zirconia and Ceria stabilized zirconia/alumina nanocomposite (Ce-TZP/A).

Recently interest in the use of zirconia has increased due to its superior biocompatibility and biomechanical properties, but it has been concluded that conventional adhesive techniques do not yield high enough bond strength to substrates [38].

1.1.2. CAD/CAM

CAD/CAM is considered as a good substitute for both the dentists and laboratories in order to avoid time-consuming, technique sensitive and unpredictable traditional ceramic fabrication methods.

CAD/CAM help reduce the fabrication time of high strength ceramics. Moreover, industrially fabricated blocks are more homogenous with minimal flaws and compare favorably with other restorative options. The CAD/CAM concept can be shown in figure 5.

Some high strength polycrystalline ceramics such as stabilized zirconium dioxide could not have been practically processed by traditional laboratory methods. These materials have made possible the use of all ceramic crowns and short span bridges in posterior load bearing regions [34].

- **CAD/CAM glass ceramics**

The first CAD/CAM produced glass-ceramic inlay was fabricated in 1985 using a ceramic block comprising fine grain feldspathic ceramic. Later development resulted in enhancing the mechanical properties [40].

- **CAD/CAM and mica-based ceramics**

The mica minerals are a group of sheet silicate (so-called phyllosilicate) minerals, consisting of varying complicated formulas. Its machinability was made possible by the presence of tetra silicic Fluormica, crystals which are highly interlocked within the glassy matrix. These materials are no longer in the market since its cumulative breakage at 2 years was found to be too high.



Figure 2: The CAD/CAM concept and the block material (Montazerian et al. 2016) [41].

- **CAD/CAM with leucite-reinforced ceramics**

These were characterized by good marginal gap, internal fit and fracture load. A mid-term evaluation, of a 5-year clinical split-mouth investigation of all-ceramic partial coverage on molars, reported a survival rate of 97% after 3 years.

These materials were developed for chair-side single unit restorations and have a flexural strength of about 160 MPa. Clinically, it is recommended for single tooth restorations and is available in high translucency, low translucency and polychromatic blocks. The milled restoration can in the next step be stained and glazed.

- **CAD/CAM lithium disilicate and monosilicate reinforced ceramics**

Laboratory studies have shown that lithium disilicate (e.max® CAD) crowns are resistant to fatigue in cyclic loading. Another variant is the lithium silicate ceramic enriched with zirconia (Celtra Duo®, Suprinity®). This material has been recommended for use in fabricating inlays, onlays, veneers, anterior and posterior crowns and implant supported crowns. Short term clinical trials on single crowns showed survival rates up to 100%.

- **CAD/CAM and glass infiltrated alumina and zirconia ceramics**

The Vita InCeram™ fabricated for CAD/CAM machining are more homogenous as compared to those fabricated for slip-casting technique. CAD/CAM InCeram Spinell™ is the most translucent material of the group and is recommended especially for anterior crowns. CAD/CAM InCeram Alumina™ has been recommended for single anterior and posterior crowns. It was reported a survival rate of 92% after 5 years for premolar and molar crowns.

CAD/CAM InCeram Zirconia™ is an example of glass infiltrated zirconia (ZrO₂) toughened alumina, and has the highest strength of this group of materials. Its flexural strength was found to be favorable for bridge frameworks. The zirconia opacity, however, has limited its use to the posterior region as substructures for crowns or bridges with one pontic. In vitro studies showed that posterior bridges made of CAD/CAM InCeram Zirconia™ was similar in accuracy to ceramo-metal bridges, and produce a better fit than the slip cast InCeram Zirconia™.

The dense crystal lattice reduces crack propagation resulting in excellent mechanical properties, but the extreme hardness and strength of these blocks gave their milling the name “hard machining” and meant that well-fitting prosthesis could not be practically fabricated without CAD/CAM systems.

Polycrystalline ceramic is relatively opaque and a veneering ceramic layer is indicated for the required aesthetic results.

With the addition of stabilizing oxides such as ceria (CeO₂), magnesia (MgO) or yttria (Y₂O₃), a multi-phase material known as partially stabilized zirconia (PSZ) is formed at room temperature with cubic crystals as the major phase and monoclinic and tetragonal crystals as the minor phases. It is also possible to form a mono-phasic material consisting of tetragonal crystals only and the material is then called tetragonal zirconia polycrystal (TZP).

- **Polymer-infiltrated-ceramic-network**

Polymer-infiltrated-ceramic-network (PICN) material was developed to achieve a material with enhanced mechanical characteristics, compared to conventional restorative materials like ceramics and composites [42]. Polymer-infiltrated ceramic

network material contains 86% (by weight) porous feldspathic ceramic matrix infiltrated with a copolymer (urethane dimethacrylate and triethylene glycol dimethacrylate). The long-term aim is to imitate the mechanical behavior of a natural tooth [43].

The estimated improvements of the PICN CAD/CAM materials are reduced brittleness, rigidity and hardness together with improved flexibility, fracture toughness and better machinability compared to conventional ceramics. Moreover, they would induce less wear toward opposing teeth such as occurs with composites along with enamel like material attrition.

- **Resin-composite nano ceramics CAD/CAM blocks**

These material are not ceramics, but were introduced in an attempt to significantly improve the properties of resin composites for CAD/CAM applications.

These Resin-composites were polymerized under high pressure and high temperature (HP/HT) resulting - in higher degree of conversion and polymerization. As a result, these blocks exhibited dramatic improvement in flexural strength, hardness, density and Weibull modulus (a dimensionless parameter of used to describe variability in measured material strength of brittle materials), as compared to those of their photo-polymerized counterparts and, even better, than that of some glass-ceramic materials.

In addition to being easily repaired intra orally for minor defects induced by function, the CAD/CAM burs used to fabricate resin composite crowns or restorations are less likely to be dulled up over longer periods. Other studies even revealed that resin-composite materials may be less susceptible to chipping during the milling procedure.

MATRIX		FILLER	PROCESS	TRADE NAME
Esthetic ceramics - Veneering on ceramic Aluminosilicate glass (feldspatic or synthetic)		Colorants-opacifiers 4-5%	Powder	Vita VM7®
			Pressed	None
Zirconia ceramics veneering on ceramic Aluminosilicate glass (feldspatic or synthetic)		Moderate leucite, chemical modifiers (5-10%)	Powder	Vita VM9® IPS e.max Ceram®
			Pressed	IPS e.max ZirPress®
Metal ceramic systems (veneering on metal alloys) Aluminosilicate glass (feldspatic or synthetic)		Leucite 17-25 %	Powder	Vita VM® IPS in line®
			Pressed	IPS in line POM® Vita PM9®
Esthetic ceramics High glass content Aluminosilicate glass (feldspatic or synthetic)		High melting glasses nephline, albite Appr 40%	Cerec 3	Mark II®
			Cerec 3	Procad®
		Leucite 40-50%	Pressed	Empress esthetic® OPC®
			Powder	Optec® Cerinate® Mirage®
Structural ceramics		Lithium disilicate 70%	Inlab	Emax CAD®
Low glass content	Special silicate glasses (high lithium or lanthum)		Pressed	Emax Press®
			Inlab or Dental lab	In-ceram alumina® In-ceram spinell® In-ceram zirconia®
No glass content	Polycrystalline alumina	Mg3%	Inlab	Vita AL-Cubes®
			Cad/cam	Procera®
	Polycrystalline zirconia	Y 3-5%	Inlab	Vita YZ-Cubes® Emax ZirCAD®
			Cad/cam	Lava® Cercon® Procera®

Table 1: The different dental ceramic compositions and their trade names.

In order to study the mechanical properties of different CAD/CAM block materials, a study was performed on several commercially available block types. The mechanical properties of the block material were evaluated, as well as their internal fitness after being machined.

Original Research Article: Mechanical properties and internal fit of four CAD CAM block materials

RESEARCH AND EDUCATION

Mechanical properties and internal fit of 4 CAD-CAM block materials

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ABSTRACT

Statement of problem. Recent polymer-based computer-assisted design and computer-assisted manufacturing (CAD-CAM) materials have been commercialized for inlay restorations, a polymer-infiltrated ceramic-network (PICN) and composite resin nanoceramics. Little independent evidence regarding their mechanical properties exists. Internal adaptation is an important factor for the clinical success and longevity of a restoration, and data concerning this parameter for inlays made with these blocks are scarce.

Purpose. The purpose of this in vitro study was to evaluate and compare the mechanical properties (flexural strength, flexural modulus, Vickers hardness, fracture toughness) and the internal adaptation of these recent polymer-based blocks with a lithium disilicate glass-ceramic block.

Material and methods. The materials tested in this study were a PICN material (Vita Enamic), 2 composite resin nanoceramics (Lava Ultimate; 3M ESPE and Cerasmart; GCDental Products), and a lithium disilicate glass-ceramic (IPS e.max CAD). Mechanical properties were evaluated according to ISO norm DIS 6872:2013. Bar-shaped specimens (18×3×3 mm) were prepared and submitted to a 3-point bend test using a universal testing machine at a cross-head speed of 0.5 mm/min. In addition, identical cavities were prepared in 60 human mandibular extracted molars (n=15) and optically scanned to receive mesioocclusodistal inlays milled with the 4 materials tested in a CEREC Inlab milling machine. The replica technique and a stereomicroscope (×20) were used to measure the internal fit of the inlays at 9 preselected locations. All data were statistically analyzed using 1-way ANOVA and the post hoc Tukey multiple comparison or Games-Howell test ($\alpha=.05$).

Results. The mean flexural strength of the tested blocks ranged from 148.7 ±9.5 MPa (Vita Enamic) to 216.5 ±28.3 MPa (Cerasmart). The mean flexural modulus ranged from 23.3 ±6.4 GPa (Vita Enamic) to 52.8 ±10.5 GPa (IPS e.max CAD). The mean Vickers hardness ranged from 0.66 ±0.02 GPa (Cerasmart) to 5.98 ±0.69 GPa (IPS e.max CAD). The mean fracture toughness ranged from 1.2 ±0.17 MPa.m^{1/2} (Cerasmart) to 1.8 ±0.29 MPa.m^{1/2} (IPS e.max CAD). The values for internal discrepancy ranged from 119 ±55 μm to 234 ±51 μm. The mean internal discrepancy was significantly higher for Lava Ultimate ($P<.05$) than IPS e.max CAD and Cerasmart but not for Vita Enamic. The factor "material" was statistically significant in relation to the mechanical properties evaluated in this study ($P<.05$). The Pearson correlation was negative between the flexural strength results and the internal discrepancy of the materials tested ($R^2=0.941$; $P<.05$).

Conclusions. The mechanical properties of the CAD-CAM block materials tested were within the acceptable range for fabrication of single restorations according to the ISO standard for ceramics (ISO 6872:2008). IPS e.max CAD and Cerasmart were observed to have superior flexural strength and better internal fit. (J Prosthet Dent 2017;■:■-■)

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Clinical Implications

Novel polymer-based computer-aided design and computer-aided manufacturing restorative materials with a high flexural strength may provide restorations with improved internal adaptation.

Computer-aided design and computer-aided manufacturing (CAD-CAM) in dentistry have experienced rapid expansion over the last 10 years. Software and milling devices have greatly improved, especially with the recent introduction of a new range of digitalization tools/scanners.¹ Moreover, CAD-CAM technology has enabled the use of polycrystalline ceramics and the development of new materials containing polymeric matrix.^{2,3} CAD-CAM generated restorations for posterior teeth fabricated from a variety of materials showed acceptable clinical outcomes.⁴ The survival rate of CEREC-generated restorations was reported to be 97% for 5 years and 90% for 10 years.⁵

Currently, a wide range of block materials with different compositions and physical properties is available. Dense ceramics are characterized by high hardness and wear resistance values; yet, they cannot withstand elastic deformation because their Young moduli are much higher than that of dental tissues.² Recently, a polymer-infiltrated ceramic network (PICN; Vita Enamic; Vita Zahnfabrik) material and composite resin nanoceramics blocks (Cerasmart; GC Europe Lava Ultimate; 3M ESPE) have been introduced as alternatives to dense ceramics.² Vita Enamic is composed of a porous ceramic network (86%), which is then infiltrated with a polymer by capillary action.⁶ Composite resin nanoceramics blocks consist of a polymeric matrix reinforced by ceramic fillers, either nanofillers (Lava Ultimate; 3M ESPE) or nanohybrid fillers (Cerasmart; GC Europe). Industrial fabrication of these blocks under high temperature and high pressure has led to a higher volume fraction filler and higher conversion rates (85%) than with indirect composite resin fabricated in dental laboratories, thus significantly improving their mechanical properties.^{2,7-9}

These recently introduced materials are characterized by having a greater modulus of resilience than dental ceramics, and although they are less resistant to wear, they cause less wear to the opposing dentition.¹⁰ Moreover, manufacturers claim these materials are less susceptible to fracture and chipping (better machinability) because their Young moduli are close to that of dentin.^{2,10-13} Additionally, they are easier to repair and polish than glass-ceramics.^{2,14-18} Several studies have claimed that a lower modulus of elasticity may result in better machinability and more accurate adaptation, thus

establishing correlation between mechanical properties and internal fit.^{12,19,20}

With adequate bonding and sufficient mechanical properties, the clinical success of a restoration depends on good internal adaptation.²¹⁻²⁴ Poor internal adaptation reduces the material's mechanical behavior in terms of resistance to fracture.²²⁻²⁴

Although many studies have evaluated the marginal and internal adaptation of crowns, few studies have done so for inlays and onlays with machinable ceramic materials.^{22,25-29} Therefore, the purpose of this *in vitro* study was to determine and compare the mechanical properties and internal fit of 2 CAD-CAM composite resin nanoceramics (Cerasmart, Lava Ultimate) and a PICN material (Vita Enamic) with a machinable lithium disilicate glass-ceramic (IPS e.max CAD). Mechanical testing included flexural strength, flexural modulus, fracture toughness, and Vickers hardness, whereas for internal fit, the replica technique and stereomicroscopy of mesio-occlusodistal (MOD) inlays were used. A correlation was sought between mechanical properties and the internal fit of the tested materials.

MATERIAL AND METHODS

The 4 materials tested in this study and their compositions are listed in Table 1. The IPS e.max CAD specimens used in this study were not fritted. Flexural strength and modulus evaluation were done according to International Organization for Standardization (ISO) standard 6872.³⁰ Bar-shaped specimens (18×3×3 mm; n=16) were subjected to a 3-point bend test, using a universal testing machine at a cross-head speed of 0.5 mm/min. The flexural strength was calculated in megapascals (MPa) from the following formula³⁰:

$$\sigma_f = \frac{3PL}{2bh^2}, \quad (1)$$

where P is the breaking load (newtons), L is the test span (millimeters; center-to-center distance between support rollers), b is the specimen's width (millimeters), and h is the specimen's thickness (millimeters).

The flexural modulus (E_f) was calculated in gigapascals (GPa) from the following formula³⁰:

$$E_f = \frac{SI^3}{4bh^3}, \quad (2)$$

where I is the span length (20 mm), b is the specimen's width (millimeters), h is the specimen's thickness (millimeters), S is the stiffness (N/m), and $S=F/d$ (d is the deflection corresponding to load F at a point in the straight-line portion of the trace).

The test procedure for fracture toughness was carried out using the single edge V-notch beam method (simplified

Table 1. Type, manufacturer, composition, and lot number of tested CAD-CAM materials

Type	Brand	Manufacturer	Shade Size	Batch	Monomer	Filler	%Mass
Composite resin nanoceramic	Cerasmart	GC Europe	A2LT/14	14092	Bis-MEPP, UDMA, DMA	Silica (20 nm), barium glass (300 nm)	71
Composite resin nanoceramic	Lava Ultimate	3M ESPE	A2LT/14L	N721602	Bis-GMA, UDMA, Bis-EMA, TEGDMA	SiO ₂ (20 nm), ZrO ₂ (4–11 nm), aggregated ZrO ₂ /SiO ₂ cluster	80
Polymer infiltrated ceramic network (PICN)	Vita Enamic	Vita Zahnfabrik	EC42-M2T/EM-14	39440	UDMA, TEGDMA	Feldspar ceramic enriched with aluminum oxide	86
Glass ceramic	IPS e.max CAD	Ivoclar Vivadent AG	A2LT/C14	T38584	_	Crystalline lithium disilicate	

Bis-EMA, ethoxylated bisphenol-A dimethacrylate; Bis-GMA, bisphenol A diglycidylether methacrylate; Bis-MEPP, 2,2-bis(4-methacryloxyphenyl)propane; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

version) as described in ISO standard 6872,³⁰ revised third edition, for ceramic materials, and ISO standard 10477,³¹ for polymer-based crown and bridge materials. Bar-shaped specimens (18×3×3 mm) were used for each material type (n=10). The final V-notch depth was between 0.8 and 1.2 mm. Fracture toughness (K_{Ic}) was calculated for each specimen as follows: the span length was 15 mm, the S-to-W ratio was therefore 5, and the equation used was³⁰:

$$K_{Ic} = \frac{F}{b\sqrt{w}} \cdot \frac{S_1 - S_2}{w} \cdot \frac{3\sqrt{\alpha}}{2(1-\alpha)^{1.5}} Y \quad (3)$$

(note: $S_2 = 0$ in 3-point testing)

$$Y = 1.9109 - 5.1552\alpha + 12.6880\alpha^2 - 19.5736\alpha^3 + 15.9377\alpha^4 - 5.1454\alpha^5, \quad (4)$$

where F is the fracture load, b is the bending bar's width, w is the bending bar's height, S is the roller span, α is the a/w , a is the notch depth, and Y is the stress intensity shape factor.

The Vickers hardness tests were performed with the polished surface of the specimens already prepared for the other mechanical tests. The load used was 9.8 N for 20 seconds.

In the second part of the study, the internal fit of these 4 CAD-CAM blocks was evaluated. Sixty human mandibular extracted molars with no visible cracks or decay were selected (following informed verbal consent and in compliance with French legislation, the local ethical committee, and the World Medical Association Declaration of Helsinki 2008). Identical MOD cavities were prepared by a single operator (A.G.), using a high-speed handpiece with water spray cooling and an optical aid (Keeler loupes; magnification ×2.5). A new bur (6847 KRD and 8847 KR, Komet) was used for every 5 preparations. Cavities were optically scanned (CEREC AC Bluecam; Sirona Dental Systems), and virtual MOD inlays were created (CEREC Software 4.3; Cement space, 120 μm). Fifteen inlays of each group were milled using CEREC Inlab milling machine (MC XL model; Sirona Dental Systems). Internal fit was measured using the replica technique^{20,23,32-34} and stereomicroscopy (M165 C, ×20; Leica) at 9 preselected locations.

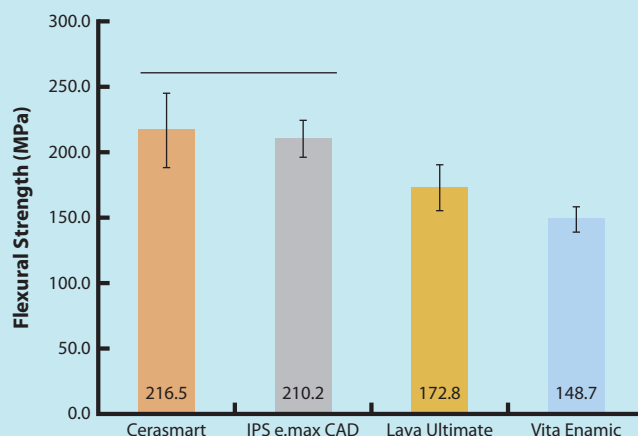


Figure 1. Mean flexural strength (MPa) ±SD. Straight horizontal line indicates statistically similar values ($P > .05$).

Statistical analysis was performed using 1 way ANOVA and Tukey multiple comparison or Games-Howell test whenever homogeneous variation of the data could not be assumed among the groups with statistical software (IBM SPSS Statistics v23; IBM Corp; $\alpha = .05$). Results for the mechanical properties and internal fit were analyzed accordingly. The data obtained for the internal fit were further separated according to the region involved. Each region was analyzed similarly to detect significant differences among the 4 materials. The Pearson correlation was performed between each of the mechanical properties tested and the mean internal discrepancy of each of the block materials.

RESULTS

Results for the flexural strength, flexural modulus, fracture toughness, and Vickers hardness are presented in Figures 1 to 4. Cerasmart (216.5 ±28 MPa) and IPS e.max CAD (210.2 ±14 MPa) have significantly higher flexural strength results than Lava Ultimate or Vita Enamic ($P < .05$). The highest flexural modulus values were for IPS e.max CAD (52.8 ±10.5 GPa), which was significantly higher than the other materials tested ($P < .05$). The lowest Vickers hardness values were for the 2 composite resin nanoceramics Cerasmart (0.66 ±0.04 GPa) and Lava Ultimate (0.95 ±0.08 GPa), whereas IPS e.max CAD (5.98 ±0.94 GPa) was significantly higher ($P < .05$). IPS e.max

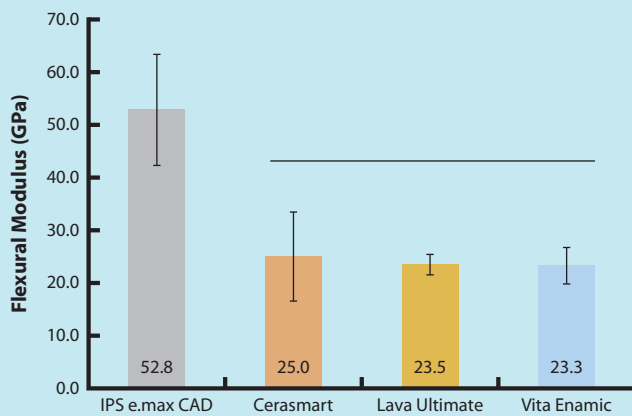


Figure 2. Mean flexural modulus (GPa) \pm SD. Straight horizontal line indicates statistically similar values ($P > .05$).

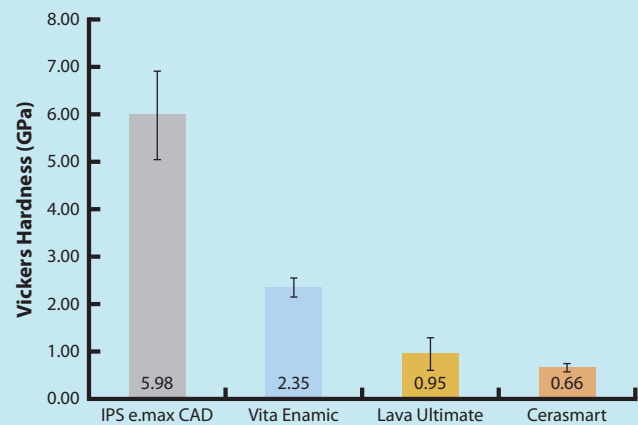


Figure 3. Mean Vickers hardness (GPa) \pm SD ($P < .05$).

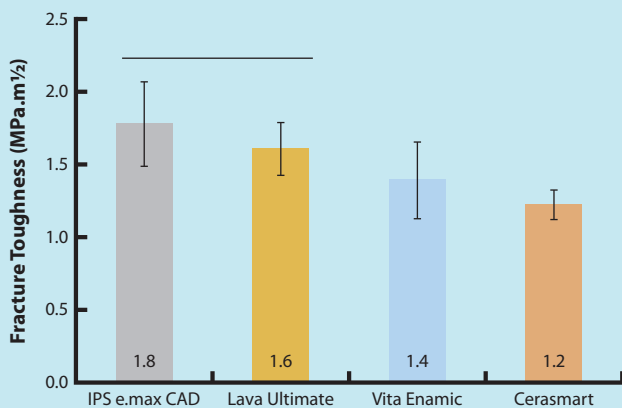


Figure 4. Mean fracture toughness (K_{IC}) \pm SD. Straight horizontal line indicates statistically similar values ($P > .05$).

CAD (1.8 MPa.m^{1/2}) and Lava Ultimate (1.6 MPa.m^{1/2}) had significantly higher mean fracture toughness values than Vita Enamic or Cerasmart ($P < .05$).

Results of the internal discrepancy for the 4 materials tested are shown in Table 2. The values ranged from 119 \pm 55 μ m to 234 \pm 51 μ m, and the mean internal discrepancy was significantly higher for Lava Ultimate than for IPS e.max CAD or Cerasmart ($P < .05$) but not for Vita Enamic ($P > .05$).

The Pearson correlation was negative between the flexural strength and the mean internal discrepancy for the materials tested ($R^2 = 0.941$; $P < .05$) (Fig. 5).

DISCUSSION

This study investigated the mechanical properties of a dense ceramic, a PICN material, and 2 composite resin nanoceramic blocks. Additionally, the internal adaptation of MOD inlays prepared from these materials was evaluated. Correlation between the mechanical properties and the machinability of each of these block materials was investigated.

Table 2. Mean \pm SD (μ m) internal discrepancy

	Cerasmart	IPS e.max CAD	Lava Ultimate	Vita Enamic
Pulpoaxial angles				
129 \pm 37 ^{ab}		122 \pm 35 ^a	161 \pm 64 ^b	119 \pm 55 ^{ab}
Axiocervical angle				
205 \pm 40		217 \pm 46	231 \pm 51	234 \pm 51
Axial walls				
129 \pm 41		126 \pm 42	158 \pm 79	129 \pm 49
Proximal seat				
127 \pm 39 ^a		125 \pm 35 ^a	179 \pm 45 ^b	157 \pm 38 ^b
Pulpal floor				
178 \pm 31 ^{ab}		178 \pm 16 ^a	220 \pm 48 ^b	208 \pm 30 ^b
Mean internal fit				
151 \pm 50 ^a		151 \pm 54 ^a	186 \pm 66 ^b	165 \pm 65 ^{ab}

Same superscript letters identify statistically similar groups ($P < .05$).

The IPS e.max CAD specimens were not fritted, because the main purpose was to evaluate the impact of mechanical properties on its machinability, which normally takes place before fritting. Thus, the results obtained were less than those that could be expected with the definitive fritted restoration.

The polymer-based blocks exhibited high flexural strength and low flexural modulus. The fact that Cerasmart showed higher flexural strength than Lava Ultimate could be attributed to its uniform nanoparticle filler composition compared with the large size range of fillers for Lava Ultimate.⁹ Two different composite resin nanoceramic materials do not necessarily behave similarly, and the structural composition seems to play an important role. The higher flexibility of composite resin nanoceramics is claimed to be due to the flexibility of the resin, which helps to reduce brittleness.⁸⁻¹⁰ The flexural modulus results obtained in this study are in accordance with those in previous studies comparing composite resin nanoceramic blocks and ceramic based blocks.^{8,9,27}

The Vickers hardness values obtained in this study suggest that the composite resin nanoceramic blocks will

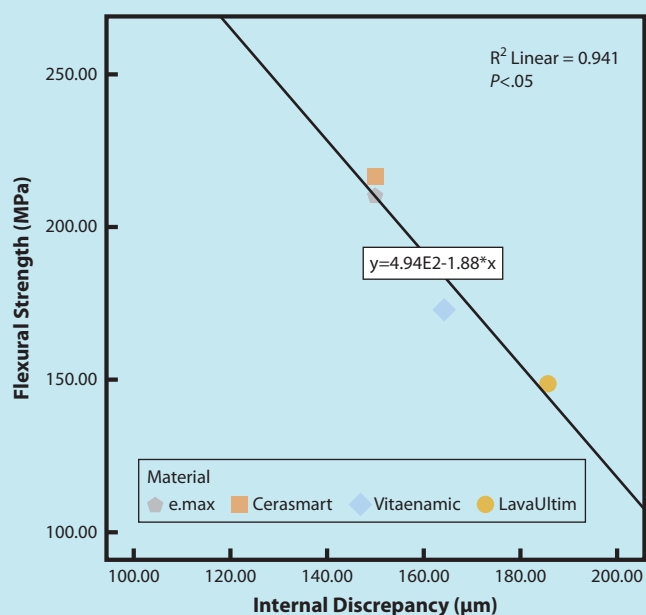


Figure 5. Pearson correlation for flexural strength and mean internal discrepancy.

have lower wear resistance than hybrid or ceramic blocks.^{2,9} The higher fracture toughness values of Cerasmart than of Lava Ultimate could be explained by the difference in type of fillers (zirconium-silicate), conferring more toughness to the material and rendering it more resistant to crack propagation.¹⁷

The flexural stress values were found to be negatively correlated with the mean internal discrepancy values, and higher flexural strength materials had lesser internal discrepancy values, hence better internal fit. Previous studies correlated the mechanical properties with machinability. Low hardness and modulus of elasticity have been shown to result in a greater amount of material being removed during grinding.¹¹ Conversely, other studies have reported that less brittle materials have lower edge chipping and better machinability.^{12,20,29} This finding did not apply in the current study, most probably because of the higher resistance to crack propagation of lithium disilicate ceramic and because its higher flexural resistance rendered it less prone to chipping. Moreover, because the flexural resistance of Cerasmart was higher than that of Lava Ultimate, it seemed to better withstand grinding without chipping, even though both are composite resin nanoceramic materials.

Internal fit is important for the longevity of the restoration, but threshold values have not yet been determined.²² In the present study, the replica technique was used to evaluate internal fit, and the reliability of this technique has been reported by several authors.^{22,25,32-34} Many studies have evaluated the internal adaptation of CAD-CAM crowns.^{32,33,35,36,37} However, studies concerning CAD-CAM inlays and onlays are scarce, and

materials tested in these studies were in most cases machinable ceramic materials and not polymer-based block materials.^{22,25,27-29}

A systematic review showed that for ceramic inlays and onlays, the internal fit ranged between 23 and 230 µm.²⁴ A study in which the method and configurations had many similarities to the present work showed closely similar values for IPS e.max CAD.²⁹

The present results show that larger discrepancies can be found internally on the occlusal seat and the proximal seats for Lava Ultimate and to a lesser extent with Vita Enamic. The fact that the other composite resin nanoceramic material failed to show the same tendency could be attributed to differences in the structural composition of the 2 materials.

The type of milling bur used and its particle size may affect restoration accuracy. Other variables such as the configuration of the virtual space in the software, the intrinsic properties of the CAD-CAM system, the choice of the rotary instrument in the milling machine and its speed may also influence the results.^{33,35,36} Future studies are recommended to evaluate and compare the replica technique for the internal fit of composite resin and glass-ceramic inlays and onlays with a virtual 3-dimensional analysis.^{26,37,38}

CONCLUSIONS

Within the limitations of this in vitro study, the following conclusions were drawn:

1. The mean flexural strength of Cerasmart and IPS e.max CAD was significantly higher than that of Lava Ultimate or Vita Enamic.
2. The mean flexural modulus and Vickers hardness of IPS e.max CAD were significantly higher than those of Cerasmart, Lava Ultimate or Vita Enamic.
3. The mean fracture toughness of IPS e.max CAD and Lava Ultimate was significantly higher than that of Vita Enamic or Cerasmart.
4. The mechanical properties seem to depend more on the structural composition of the material than on their chemical composition
5. A negative correlation was found between the flexural strength and the internal discrepancy of the tested materials.

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The previous article considered the difference in material composition between the various types of blocks tested, the obtained results revealed a valuable insight about the role of structural composition on the materials mechanical properties and its machinability.

The mastication bench, as a testing tool, would offer the advantage of evaluating different restorative materials with different composition under the exact same conditions and under the same relevant stresses. Unfortunately, this is practically impossible to obtain in clinical trials.

Moreover, the device would allow the monitoring of some important features such as the wear of the opposing dentition (mostly with ceramics), or the restoration margins and its adaptability.

A survey was done to compare our results with those of the current literature. The comparison charts can be shown in figures 6,7,8 and 9.

Our results were coherent with those of previous studies. Alternatively, the survey showed that some material types show more homogenous results between laboratories, in different studies, most probably due to the material structure and composition. This also varies according to the type of test used.

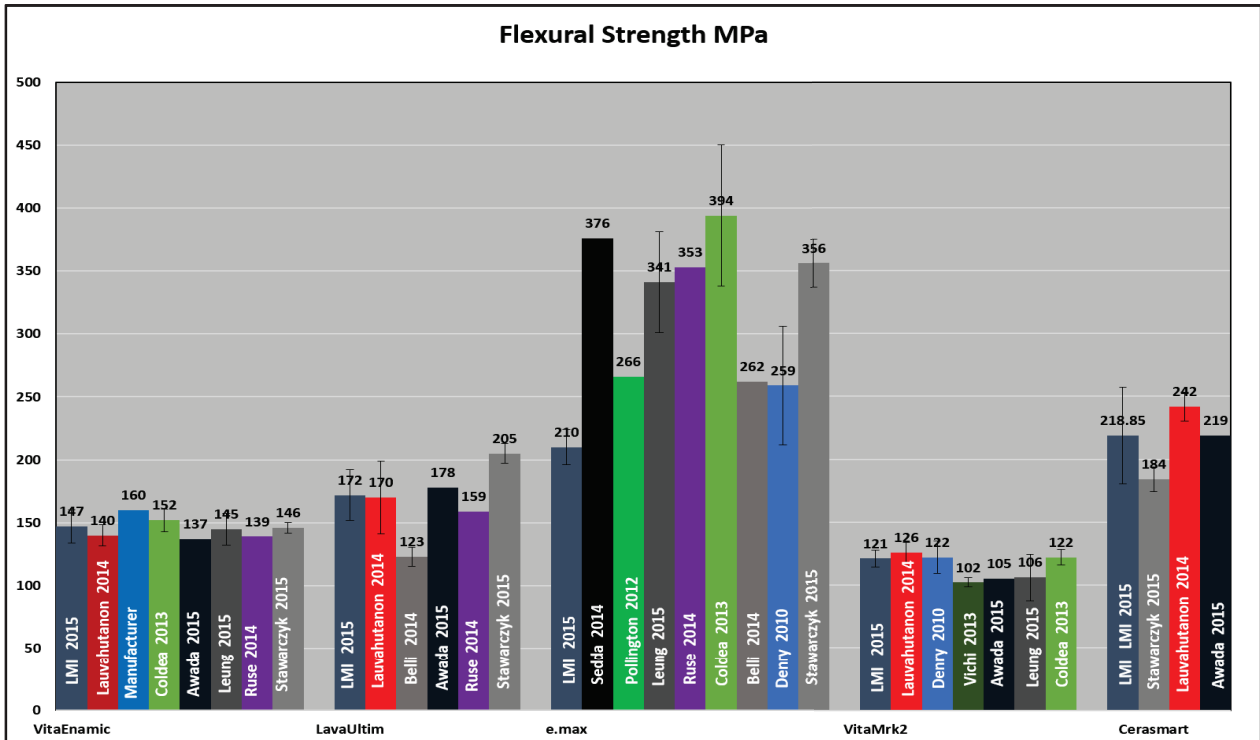


Figure 1: The flexural strength (MPa) results comparison chart [43-53]

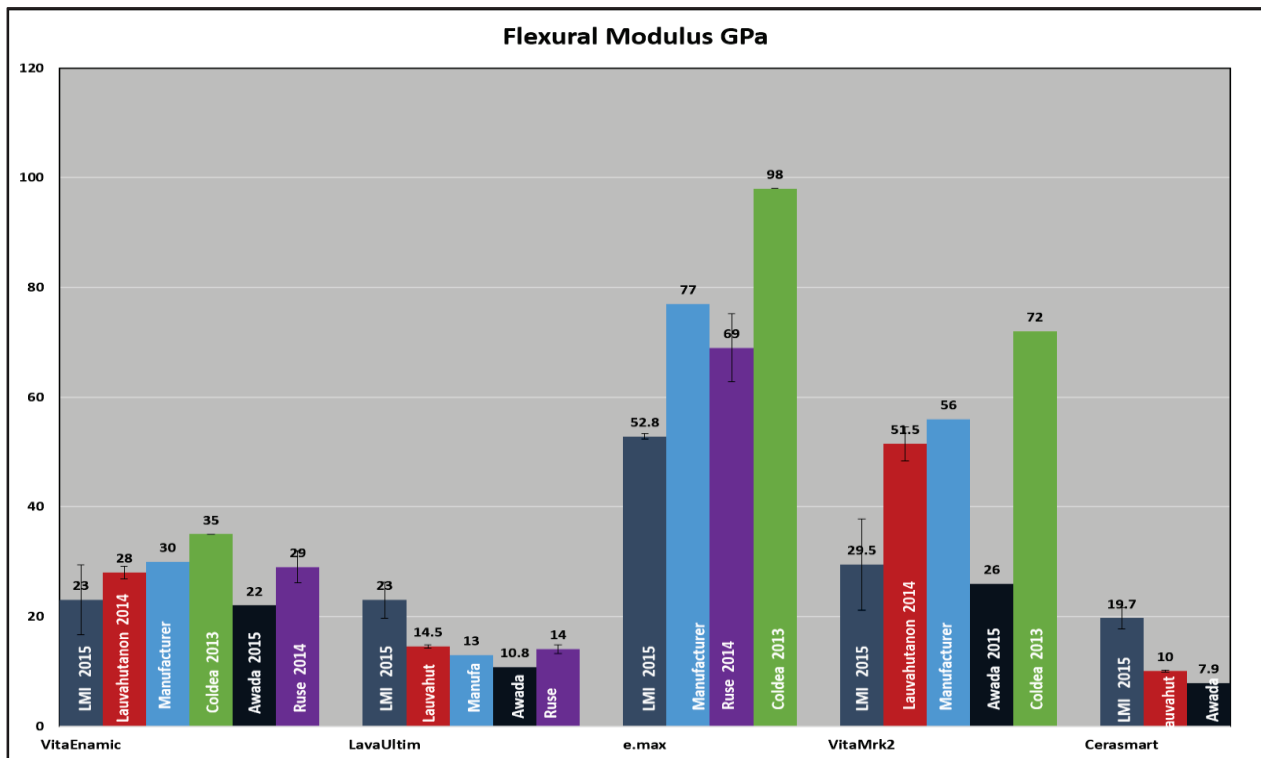


Figure 2: The flexural modulus (GPa) results comparison chart [43-45,47]

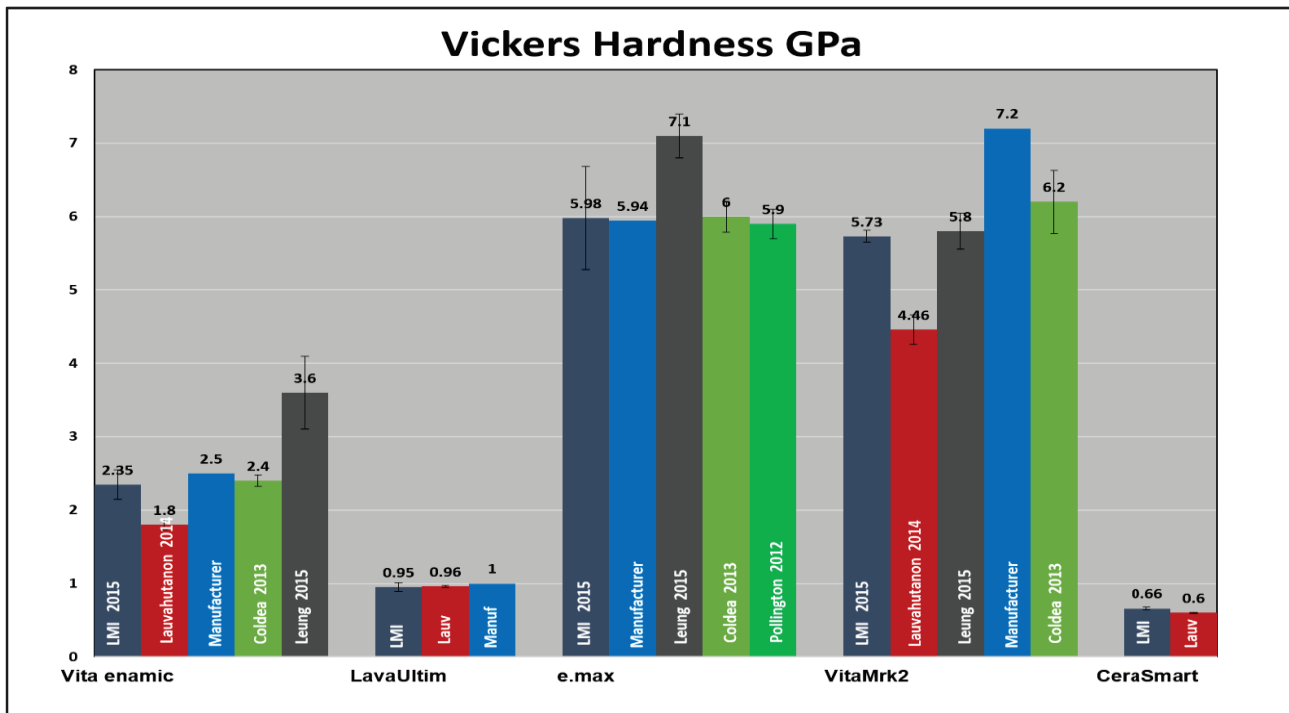


Figure 3: The Vickers hardness (GPa) results comparison chart [43,44,46,53]

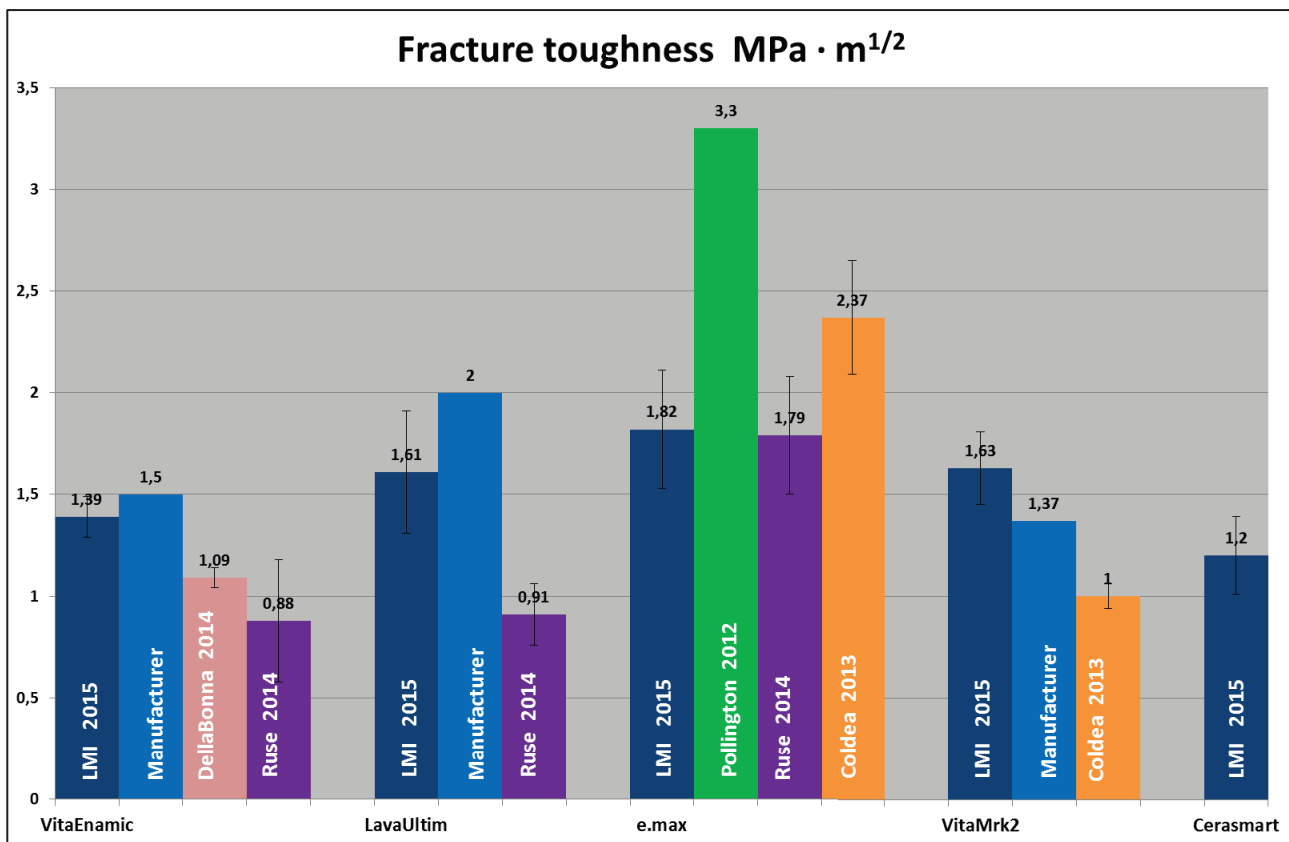


Figure 4: The fracture toughness (MPa/m^{1/2}) results comparison chart [43,47,53,54]

1.1. Metals and alloys

Metals represent one of the four major classes of materials in dentistry used for the reconstruction of damaged or missing oral tissues. All metals and alloys used as restorative materials in dentistry are crystalline solids. A wide variety of complex dental alloy compositions are used in dentistry, the most important are:

- Dental amalgams containing the major elements: mercury, silver, tin, and copper.
- Noble metal alloys in which the major elements are: combination of gold, palladium, silver.
- Non precious base metal alloys with a major element of: nickel, cobalt, iron, or titanium.

Moreover, Titanium, which is classified in four different grades, may also technically be considered as an alloy.

2. Mechanical and Physical properties of dental materials

Physical properties of a material are based on the laws of mechanics, acoustics, optics, thermodynamics, electricity, magnetism, radiation, atomic structure and nuclear phenomenon. Mechanical properties are a subset of physical properties based on the laws of mechanics [2].

Some of these physical and mechanical properties play a major role in the life expectancy of the restoration or prostheses in function. In this work, we will be focusing on some of these properties being the most critical in dental practice.

2.1. Thermal properties

Thermal conductivity, diffusivity and the coefficient of thermal expansion are physical properties based on the laws of thermodynamics. Dental materials used should be thermally compatible with dental tissues, the absence of this compatibility would not only lead to patient discomfort but also to mechanical stresses which would induce microcracks, and the loss of the restoration or prostheses over time [55].

Several traditional methods are used for characterizing the thermal properties of dental tissue, these include: thermal couples, infrared cameras, flash laser method, differential scanning calorimeter and traditional calorimeter cup.

Lin et al. examined the state of the art of methodologies, used in analyzing the heat transfer process, across dental materials and dental tissue, whether experimental settings or mathematical modeling, taking in consideration the temperature fluctuation in the oral

cavity (-5 – 76.3 °C). They found that, *in vitro* methods mostly failed to implement all the events occurring during the temperature variation process (blood flow inside the pulp cavity for example). On the other hand, they criticized mathematical modelling as being over simplified and unrealistic not considering the variation in structure of enamel and dentin, the thermophysical properties of these tissues do not only vary between individuals and different sites, but also according to their microstructure (volume fraction of dentinal tubules for example) [56].

2.1.1. Thermal conductivity

According to Fourier's law for heat conduction, thermal conductivity (k or λ) is the property of a material to conduct heat. It is measured as the quantity of heat in calories per second passing through 1cm of a material with cross section 1cm^2 and having a temperature difference of 1 k (1°C), measured under steady state conditions in which the temperature gradient does not change. It is, thus, quite logical that materials with higher thermal conductivity transmit more thermal energy.

A classic example is metallic restorations that transmit thermal energy to pulp tissue, and, thus, could not be inserted in moderate or deep cavities without base material that would act as an insulator.

Composite and ceramic restorations, on the other hand, are thermally nonconductive, but the difference in thermal properties with the dental tissues may submit the adhesive interface to increased stress leading to eventual failure of the restoration.

2.1.2. Thermal diffusivity

Thermal diffusivity is a measure of the speed, at which, a temperature change will spread through an object when one surface is heated. It is calculated by dividing the thermal conductivity of a material by the product of its density and heat capacity.

The rapid change in temperature, which occurs during the ingestion of foods and drinks, imply that materials or tissues that are able to absorb most of the thermal energy, without transmitting it to the pulp or the surrounding tissues, would provide more biological protection. Thus, thermal diffusivity of restorative materials seem to be more important than thermal conductivity. Various methods were used to measure the thermal diffusivity of dental filling materials.

	Thermal conductivity k W /mK	Thermal diffusivity α m ² /s	Density g cm ⁻³	Specific heat cal/ g K	Coefficient of thermal expansion 10 ⁻⁶ /°C	young's modulus GPa
Enamel	0.93	4.69 · 10⁻⁷	2.97	0.18	11.4	84.1
Resin composite (inorganic filler)	1.1	6.15 · 10⁻⁷	2.4	0.19	33	16.6
Resin adhesive (no inorganic filler)	1.4	1.9 · 10⁻⁷	1.1	0.27	62	1.1

Table 3: Thermal and physical properties of dental tissues, restoration and adhesive interface. [55,57-59]

2.1.3. Coefficient of thermal expansion

The coefficient of thermal expansion is defined as the change in length per unit of the original length of the material when its temperature is raised 1°C (1K). It describes how the size of an object changes with a change in temperature. Several methods are

used to designate the coefficient of thermal expansion but for solids linear coefficient of thermal expansion is the one mostly used.

Differences in the coefficient of thermal expansion play an important role for veneering material as in case of crown and bridge prostheses. Changes in coefficient can lead to building up of huge stresses that would lead to the separation of the veneer material and failure of the restoration.

Another important aspect is the difference in thermal properties, especially the coefficient of thermal expansion, between the restorative material and dental tissues at the interface. This would lead to stress build up and gap formation between the restoration and the dental tissues, leading to eventual failure of the restoration.

Various studies examined thermal effect on pulpal tissue, or bone tissue (during implant placement), they all stressed the importance of monitoring heat energy generation and production during drilling. It is also important that the material thermal properties could be as close as possible to those of dental tissues to avoid the stress build up during ingestion of and cold food and drinks in the oral cavity [2].

Another important aspect is the rate at which thermal changes take place and the effect of these on the material tooth interface.

2.2. Mechanical properties

A mechanical property is defined as the physical science dealing with forces that act on the bodies and the resultant motion, deformation, or stress that those bodies

experience. All mechanical properties are measures of the material resistance to deformation, crack growth, or fracture under an applied force and the induced stress.

The stress strain curve of each material reflects its ability to resist stress. The strength of a material is the ability to resist induced stress without fracture or permanent deformation (plastic strain, see Figure 10). The stressing rate is also of importance on the long run for predicting the resistance of the material to load.

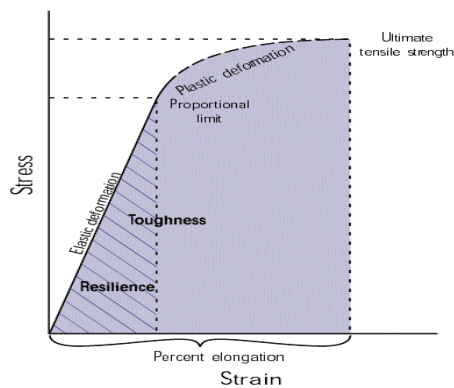


Figure 10: The stress strain curve showing the elastic limit (Philips dental materials) [2].

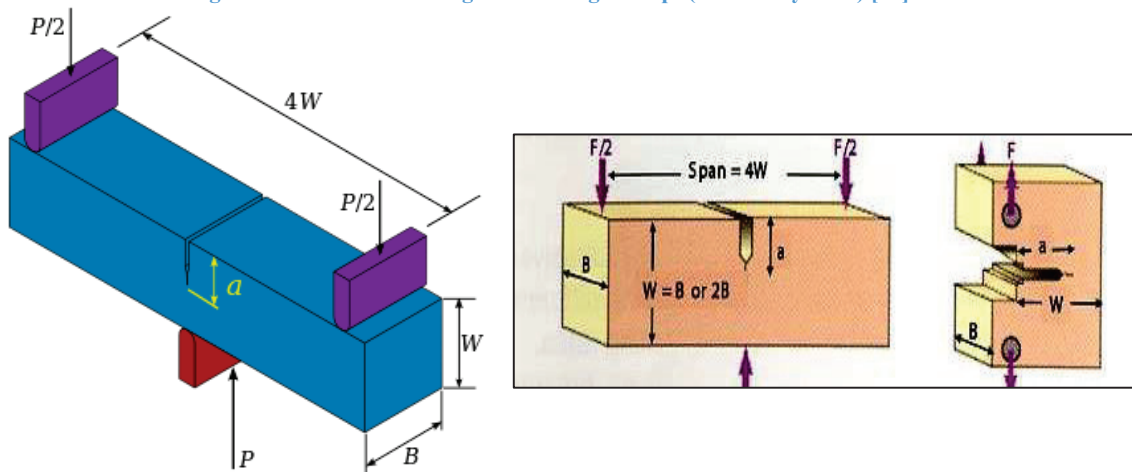
2.2.1. Fracture strength

Fracture strength is the ability of the material to endure stress without fracture and to resist the crack propagation before failure.

The principles of linear elastic fracture mechanics (LEFM) were developed in the 50s. The size of the cracks or defects during processing, production and handling of a material determines its strength, but its fracture toughness is generally independent of the size of the initiating crack. It should be taken in consideration that fracture mechanics are most important, in the understanding of failure mechanism of dental materials, and in the development of new ones [60].

Fracture mechanics are also important in testing and analyzing interfacial fractures and dental adhesives. According to Soderholm, the difficulty encountered lies in the complex stress conditions present in transitional regions and the visco elastic properties of the adhesive joint [61].

Figure 11: The fracture toughness testing concept (Mecholsky 1995) [62].



Various methods and test techniques are used for measuring the fracture toughness. These methods include double torsion, indentation crack length/fracture, indentation strength, Chevron notch bend specimen, double cantilever beam, single-edge notched beam (SENB), single-edge pre-cracked beam, fractography approach, or compression pre-cracking. The most common method used for dental materials is the single-edge notch test and the short rod Chevron notch test on cylindrical, rectangular, and prismatic specimens [63].

2.2.2. Flexural strength (Modulus of rupture)

It is defined as the ability of a material to resist deformation under load. For restorative materials in occlusion bearing areas.

The ISO standard requires a flexural strength of at least 80 MPa for restorative materials. For this test, bar-shaped specimens are made, stored in water for 24 h and at 37 °C, and loaded in 3-point bending test until failure in a universal testing machine. This test is a simultaneous collective measurement of tensile, compressive and shear stresses, the principal stress on the upper surface is compressive whereas those on the lower surface are tensile. The flexural strength in the 3-point bending test is calculated with the following formula:

$$\sigma_f = \frac{3F_m l}{2bh^2}$$

Flexural strength of a material has a great predictive value and correlates directly with the *in vivo* clinical success [64]. Some factors have an important impact on the flexural strength of dental composites. The chemical composition as well as the filler type and percentage reflects heavily on the flexural strength and modulus of the material. Moreover, long water storage or extended thermocycling are known to affect the flexural strength of dental composites [65].

2.2.3. Flexural modulus (Elastic modulus)

Flexural modulus represents the relative stiffness or rigidity of the material, which is measured by the slope of the elastic region of the stress strain graph.

Because the elastic modulus of a material is constant, it is unaffected by the amount of elastic or plastic stress induced in the material. It is independent of the ductility of a material since it is measured in the linear region of the stress strain plot. Thus, elastic modulus is not a measure of its plasticity or strength. Materials with a high elastic modulus can have either high or low strength values. Its importance in the dental domain lie in its ability to show the amount of deformation the material can show under stress.

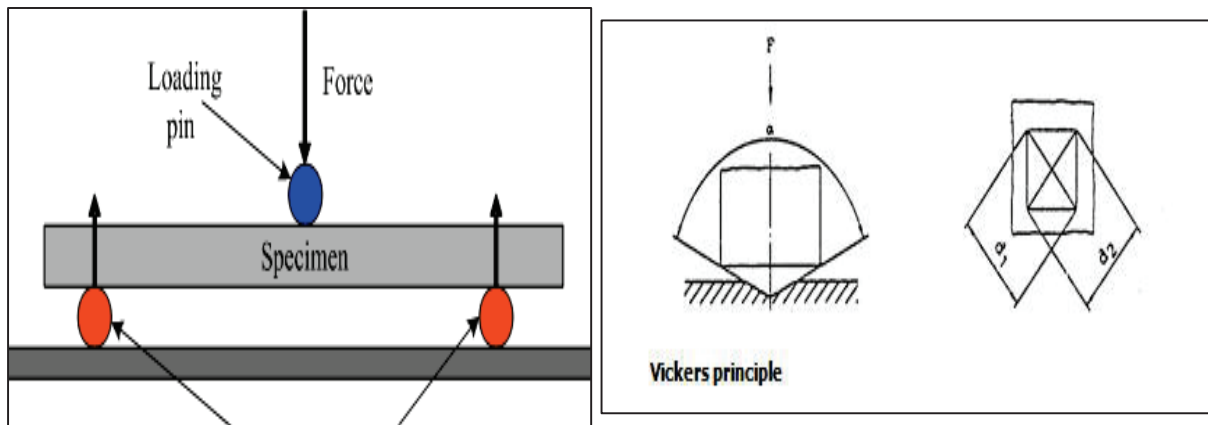


Figure 12. The flexural strength and modulus test, and Vickers hardness test (Sato et al. 2005) [66].

2.2.4. Hardness

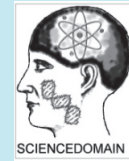
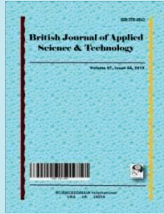
Hardness is mostly defined as resistance to indentation. It results from interaction of numerous properties among which compressive strength, proportional limit and ductility. It can also be used to measure the degree of cure of resin composite.

There are several types of surface hardness tests. Most are based on the ability of the surface of a material to resist penetration by a diamond point or steel ball under a specified load. The tests most frequently used in determining the hardness of dental

materials are known by the names Barcol, Brinell, Rockwell, Shore, Vickers and Knoop. The Knoop and Vickers are classified as microhardness tests, and they are ones mostly used in the dental field especially with dental composites.

A study was done to examine the impact of thermal changes and the differences in thermal properties of the interface components on the integrity of the adhesive region, the results obtained in the presented study highlighted the extreme importance of such effects.

Original Research Article: Thermal shock effect on enamel – resin composite Interface.



Thermal Effect Shock on the Enamel-composite Restoration Interface

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Authors' contributions

This work was carried out in collaboration between all authors. Authors CJ and HA designed the experiment, wrote the protocol and wrote the first draft of the manuscript. Authors HA and BG managed the analyses of the study and literature searches. All authors read and approved the final manuscript.

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ABSTRACT

Aim: To study the effect of thermal shock on the enamel – composite restoration interface as compared to standard thermal cycling protocol.

Methodology: Box shaped cavities were prepared in thirty mandibular third molars, the cavities were restored using two step etch and rinse adhesive: Adper™ Scotchbond™ 1 XT (3M™ ESPE™, St. Paul, USA), and nano-hybride resin composite Filtek™ Z250 (3M™ ESPE™, St. Paul, USA). Specimens were divided in 3 groups. The first group was thermal cycled for 600 cycles, the second group was submitted to 600 thermal shock cycles using Oral B waterjet device, and the third group was a control group. Teeth specimens were evaluated for dye leakage using 2% Basic Fuchsin dye for 24 hours, all bonded teeth were subsequently sectioned perpendicularly into

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0.9 ± 0.1 mm² sticks that were loaded on universal testing machine to obtain the ultimate tensile strength. Values were analyzed with one way ANOVA post hoc Tukey HSD (SPSS version 23) with 95% confidence interval.

Results: Both thermal shock and thermal cycling groups had significantly higher dye leakage values along the interface as compared to the control group. The microtensile bond strength values were significantly lower for the thermal shock group as compared to the control group, no significant difference was found between the thermal cycling and the control group.

Conclusion: Thermal shock was shown to induce more stress on the interface, which may lead to cracks and gap formation overtime.

Keywords: Thermal shock; thermal cycling; dental composite; restoration interface.

1. INTRODUCTION

In modern day practice, resin composites are the first choice for most dental practitioners, not only because of increased patient demand for more esthetic restorations, but also due to advances in material composition and polymerization techniques, leading to better mechanical and physical properties and increase in the life expectancy of the restoration [1,2].

Dental composite restorations are composed of inorganic filler dispersed in a resin based oligomer matrix; a coupling agent such as silane is used to bond these two components. Long term aging significantly reduces the mechanical properties, proposedly because of degradation of the polymer network and leaching of the unreacted polymers [3]. According to long term studies, secondary caries is considered to be the main cause of resin restoration replacement. The tooth material interface seem to be the most vulnerable area where the effect of moisture, polymerization contraction together with mechanical stresses and the stresses caused by fluctuating temperature and pH; lead to eventual failure of the adhesive interface and gap formation [3,4,5]. Another important point, is that even though the thermal diffusivity and thermal conductivity of composite restorations are close to that of the human teeth, the adhesive resin layer is markedly different due to its polymer composition and absence of filler content [6,7].

No single method exists for evaluating the effect of aging on the mechanical properties of resin composites and the tooth material interface. Aging in water is the conventional method used to simulate intraoral aging; basic standardized tests demands a minimum of 24 hours of water immersion before testing [8]. It was reported that water immersion can lead to significant reduction in the mechanical properties of resin composites (30- 55%). The varying results obtained with

different materials are attributed to difference in polymer matrix composition or type of filler. It is believed that water sorption and swelling of the network lead to reduction in friction between the polymer networks and softening of the material [3,9,10].

On the other hand, the oral environment encompasses a wide range of temperatures (-5 to 76.3°C). The differences between the physicothermal properties of the tooth components and the restorative material used, lead to the development of thermal stresses with the maximum stress on the bonding interface. These together with other masticatory stresses can easily induce the failure of the bonding interface, and hence failure of the restoration [11].

The exchange of hot and cold food and drink usually results in an abrupt and sudden change in the oral temperature, meanwhile the temperature of the dental tissues, restorative materials and the bonded interface between them occurs at a finite rate according to their heat transmission properties [12,13].

Several methods exist for the characterization of the thermal properties of the tooth components, yet significant differences have been obtained with the reported results, this could be due to the heterogeneity of the samples, or more over to the heterogeneity of the dental structure itself. The heat transfer mechanism inside a restored tooth is very difficult to measure, mostly because of the complex tooth geometry, and the varying thermophysical properties of the different constituents. The thermal performance of restored teeth was reported to differ significantly from intact ones, due to differences in thermal properties of tooth components and the restorative material [14].

Thermal cycling is considered as the most effective method for simulating the aging process

in the oral cavity. Conversely several numerical methods such as the finite element method (FEM) and the finite difference method (FDM) models were developed to analyze the temperature transfer across enamel, dentin and various restorative materials, but significant discrepancy still exists between experimental measuring and mathematical modelling [9,14].

It was found that regardless of the protocol, significant decrease in bond strength occurs after thermal cycling [13,15]. Accelerating the rate of thermal change across the tooth surface, would presumably lead to more stress build up. The difference in heat transfer rate and the thermal coefficient of each of the constituents of the tooth material interface would play the main role in stress build up, while the resistance to bond failure would depend mainly on the mechanical properties of the interface components, most notably the elastic modulus [14].

In this work an experimental setting was done that would convey sudden thermal change to the tooth surface, as compared to conventional thermal cycling, and compared to a control group. The null hypothesis would be that no difference exists in the integrity of the interface between the two groups as compared to the control group.

2. MATERIALS AND METHODS

2.1 Specimen Preparation

Following removal from patients (ages 17-27) (following informed verbal consent and in compliance with French legislation, the local ethical committee and the World Medical Association Declaration of Helsinki 2008), thirty caries-free freshly extracted third molars, were kept in a 0.5%-chloramine solution at 4°C temperature for five days then in distilled water, until further processing (ISO/TS 11405 norm). After cleaning and removal of superficial debris from the surface using a scalpel blade, occlusal surface was flattened and standardized box shaped cavities (4 × 3 × 3 mm) were prepared using diamond and tungsten carbide burs in a high-speed handpiece under copious water spray. The depth of cavities was standardized by marking the burs at 3 mm length prior to use, and the measure was controlled using a periodontal probe. Burs were replaced after ten cavities and no bevels were added at any margin of the preparation. Cavity floors were inspected for

absence of pulp exposure. The teeth were kept wet until the adhesive treatment procedure started. The adhesive used were two step etch and rinse: Adper™ Scotchbond™ 1 XT (3M™ ESPE™, St. Paul, USA), and the resin composite used was the nano-hybride Filtek™ Z250 (3M™ ESPE™, St. Paul, USA). Materials were manipulated according to the instructions by the manufacturer: enamel and dentin were etched and then rinsed with water for 10 seconds. Excess water was blotted using a mini-sponge. Immediately after blotting, 2 consecutive coats of adhesive were applied to etched enamel and dentin for 15 seconds with gentle agitation, gentle air blast was applied for five seconds to evaporate solvents. The adhesive was then light cured for 10 seconds. The composite was inserted incrementally in 3 layers cured for 20 seconds each. Restored teeth were inserted in cold cure resin, except for the crown portion to enable handling for the dye leakage, and micro tensile bond strength measurement. Specimens were then divided in 3 groups (10 each) and stored in distilled water for 24 hours at 37°C.

The first group was thermal cycled for 600 cycles (30 seconds dwelling time and 3 seconds interval) (5°C -55°C), The second group was submitted to 600 thermal shock cycles (10 seconds hot and 5 seconds cold with no interval) (5°C - 55°C) using Oral B waterjet device regulated electronically with a special electronic board to obtain the required duration and number of cycles (Fig. 1). The third group was a control group that was kept in distilled water inside an incubator at 37°C.

2.2 Dye Leakage

Teeth specimens were covered with two layers of nail polish except for the composite restorations and 1mm around the cavity margins, they were then dipped in a 2% Basic Fuchsin dye for 24 hours, the dye film on teeth surface was then polished off with a polishing disc, and each tooth was then sectioned 2 vertical sections through the center of the restoration using diamond-disc-operating saw at slow speed and under constant irrigation (Isomed, Buehler Ltd, Lake Bluff, IL, USA).

The sectioned teeth were then assessed using a stereomicroscope (Olympus CKX41, Olympus-Europe, Hamburg, Germany) and image analysis software program to measure the length of dye penetration along the interface.

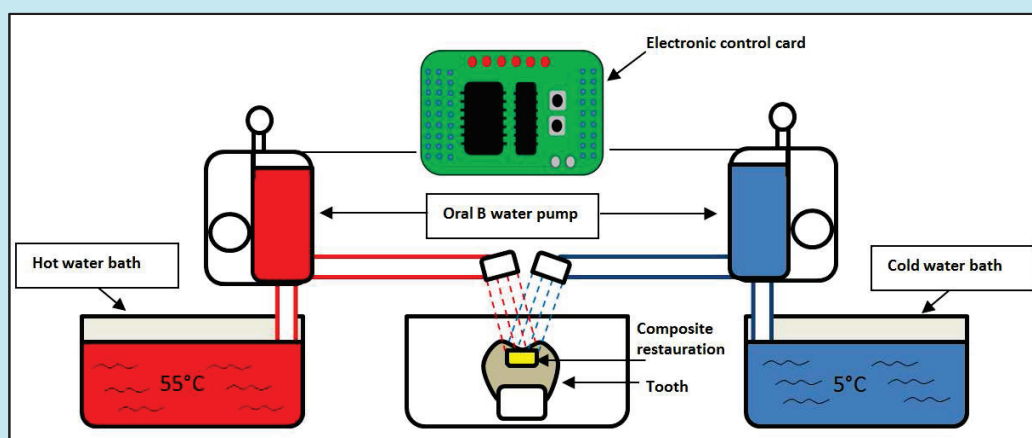


Fig. 1. The experimental setup for the thermal shock experiment

Dye penetration at the restoration tooth interface was scored for the enamel margins

Score 0: No leakage visible at the tooth restoration interface.

Score 1: Penetration of dye along the cavity wall but less than one-half of the length.

Score 2: Penetration of dye along the cavity wall but short of the axial wall.

Score 3: Penetration of dye to and along the axial wall.

The worst score from the all sections of each specimen was recorded.

The microleakage data were analyzed using Kruskal-Wallis statistical tests at a significance level of 5%.

2.3 Microtensile Bond Strength Testing (μ TBS)

All bonded teeth were subsequently sectioned perpendicularly and through to the bonded interface into $0.9 \pm 0.1 \text{ mm}^2$ sticks using diamond disk wafering blades 15HC (Buehler, Dusseldorf, Germany) at slow speed and under constant irrigation (IsoMet® Low Speed Saw, Buehler, Lake Bluff, IL, USA). Two stick samples were retrieved from each tooth. The bonded surface area was calculated before each test by measuring the width with digital caliper.

Each specimen was attached to an aluminum device constituted of two symmetric parts, having a central notch (2 mm of depth and width) in order to allow auto alignment. Device surfaces

were cleaned with alcohol. Tensile load was applied using a universal testing machine (DY34, Adamel Lhomargy SARL, Roissy-en-Brie, France), at a crosshead speed of .5 mm/min, to obtain the ultimate tensile strength, using a load cell of 1 KN.

Bond strengths of sticks from the same tooth were averaged and the mean taken as one statistical unit. Sticks that failed prematurely were included in the data and given the value of 2 MPa.

The obtained values were analyzed with one way ANOVA post hoc Tukey HSD (SPSS version 23) with 95% Confidence Interval ($P=0.05$).

3. RESULTS

The results obtained for the dye penetration test are shown in Table 1.

The control group was significantly different from the thermal shock and the thermal cycling groups, while both groups were not statistically different.

Results for the bond strength are shown in Table 2.

Fracture mode was determined at $\times 50$ magnifications with a stereoscopic microscope (Wild Heerbrugg TYP 376788, Wild Heerbrugg, Switzerland) and recorded as cohesive failure and adhesive failure; results are shown in Table 3, the samples that failed prematurely were considered among the adhesive failure group. Fig. 2 shows examples of the adhesive and cohesive failure samples.

Table 1. Dye penetration percentage for each group

Margins for the study groups	0	1	2	3	Mean
Thermal shock	-	5	3	2	1.7
Thermal cycling	-	5	4	1	1.6
Control	4	6	-	-	0.6

A total of 9 samples failed prematurely for the thermal shock group, 3 for the thermal cycling group, while no premature failure was found for the control group.

Table 2. Microtensile bond test results (MPa), similar letters denote statistically homogenous values

	Shock	Cycling	Control
Mean	6,04 ^A	9,11 ^{AB}	10,38 ^B
Std, deviation	(5,03)	(4,44)	(4,98)

Table 3. Number of adhesive and cohesive failures in each of the three groups

	Adhesive	Cohesive
Thermal shock	16 (80%)	4 (20%)
Thermal cycling	14 (70%)	6 (30%)
Control	5 (25%)	15 (75%)

4. DISCUSSION

Thermal cycling has long been used as the standard method for aging to predict the clinical reliability of various restoration types. Though the entire of the oral environment seem too complicated to be reproduced, it has long been perceived that thermal and mechanical stresses play an important role in the deterioration of the physical and mechanical properties of the restorations. Mostly resin composite restorations and the adhesive interface are the most vulnerable to the oral environment, in comparison with metallic or ceramic restorations [9,16].

In this study amplifying the effect of thermal transition between hot and cold was used to investigate the effect of thermal change on the enamel resin restoration interface. In this work the temperatures used as reference in literature between 5 and 55°C were used. Some studies have been reported using more elevated temperature whether in actual experimental setup or in simulation computer models, the justification for such exaggerated values was found by the authors to be of little scientific evidence [13,14].

During the act of eating and drinking hot and cold food or drinks, the temperature transfer to the tooth surface occurs abruptly. The thermal conductivity and more significantly the thermal diffusivity of the material control the thermal energy transfer inside the material contained by the fluctuating temperatures inside the oral cavity [10,17].

In heat transfer analysis, thermal diffusivity α is divided by density and specific heat capacity.

$$\alpha = k/(\rho c_p)$$

- k is thermal conductivity (W/(m·K))
- ρ is density (kg/m³)
- c_p is specific heat capacity (J/(cal/ g K))

According to Fourier's law of heat conduction the heat flux per unit area q (W/m²) is given in terms of the temperature T by

$$q = -k \Delta T$$

The surface heat transfer coefficient is responsible for the rate at which the temperatures exchange between the tooth surface and the hot or cold food or drink. Its value depends on the nature of the conductive and convective heat transfer processes in the layer of liquid adjacent to the surface of the tooth [11,12]. Understandably this reveals the importance of the existing difference in thermal properties between the three components; dental enamel, restorative filled resin composite and unfilled resin adhesive [18,19].

The differences in physical properties and composition between the components of the interface (Enamel, adhesive resin and composite resin restoration) accordingly would mean that the thermal energy flow inside of each of these components occur at a different rate. Moreover the corresponding amount of thermal energy needed to change the temperature or to affect thermal contraction or expansion is also different [12].

According to Table 4, the thermal diffusivity of enamel is more than double that of the filler free

resin adhesive layer [18,20], meaning that the temperature would travel two times faster into the enamel to the depth X, when at the same time the adjacent resin adhesive and resin composite restorative haven't reached the same temperature. The difference in temperature and coefficient of thermal expansion; would lead to increased stresses between the two (enamel and filler free resin adhesive layer). These thermally induced elastic stresses would lead to the appearance of micro cracks over time.

The magnitude of the stress resulting from a temperature change from T_0 to T_f could be calculated using the equation:

$$\sigma = E_{al}(T_0 - T_f) = E_{al}\Delta T$$

Where E is the modulus of elasticity and α_l is the linear coefficient of thermal expansion.

The increase in the rate of change with the thermal shock protocol would also mean less time for the resin to gain or lose thermal energy at depth due to its inferior thermal diffusivity values, accordingly subjecting the interface to

more internal stresses [21]. The thermal shock resistance parameter TSR takes in account the material elasticity able to absorb such stresses [22], and is given by:

$$TSR \cong \frac{\sigma k}{E_{al}}$$

The enamel adhesive interface is composed of resin tags mechanically interlocked inside the enamel; constraining its expansion/contraction with thermal changes. Furthermore unfilled resin (adhesive) has relatively higher thermal expansion coefficient compared to that of enamel and even that of the filled composite resin [19].

It was suggested by many authors that that temperature fluctuations during meals are frequent and variable and that alterations in oral temperature occur rapidly while the return to baseline temperature occurs more slowly [10,13]. More over the effect of thermal shock has been examined in a number of studies perhaps to reveal its overlooked impact on the adhesive interface, as shown by the previous equations [10,12,22].

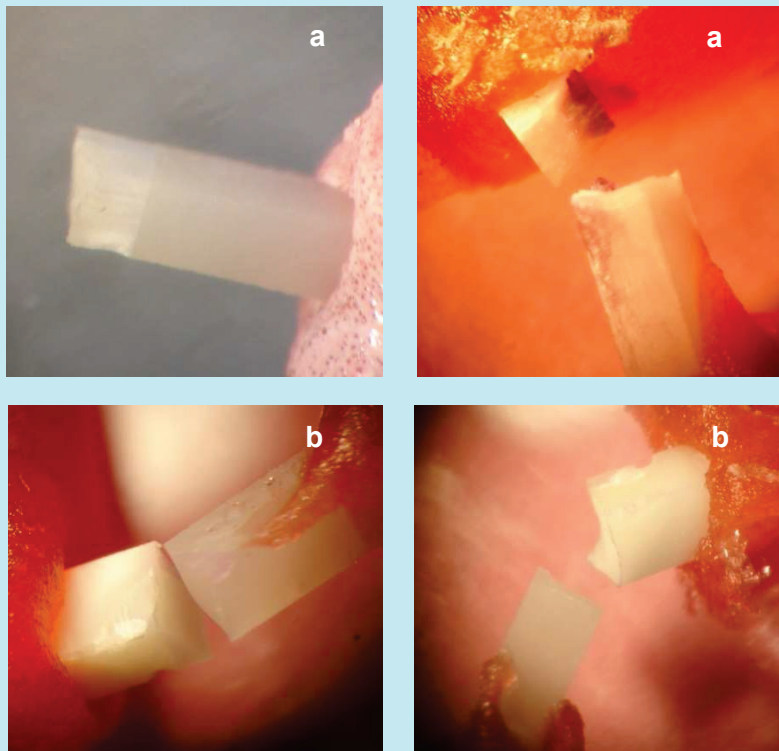


Fig. 2. Different types of failures; adhesive (a) or cohesive (b) as revealed under stereo microscopy

Table 4. Thermal and physical properties of dental tissues and the resin restoration adhesive interface

	Thermal conductivity k W/mK	Thermal diffusivity α m ² /s	Density g cm ⁻³	Specific heat cal/g K	Coefficient of thermal expansion 10 ⁻⁶ /°C	Young's modulus GPa
Enamel	0.93	$4.69 \cdot 10^{-7}$	2.97	0.18	16.9	84.1
Resin composite (inorganic filler) Filtek™ Z250	1.1	$6.15 \cdot 10^{-7}$	2.4	0.19	33	16.6
Resin adhesive (no inorganic filler) Adper™ Scotchbond™	1.4	$1.9 \cdot 10^{-7}$	1.1	0.27	62	1.1

Numerical simulations through mathematical modeling have tried to reproduce the complicated oral environment with its complex dental geometry, material properties and *in vivo* biological functions. Yet in spite of the significant amount of research done; discrepancy between the results obtained with these models and experimental measurements show that some of the factors were not considered during the development of these models. The magnitude of the actual stress build up across the interface is frequently underestimated [14,23].

In the present work it was shown that thermal stresses on the enamel adhesive interface had a significant effect on the integrity of the marginal seal of the restoration, after taking in consideration the specific composition and thermal properties of the resin composite restoration and the resin adhesive used [24,25]. The effect was intentionally exaggerated through using a cavity configuration with an elevated C factor. It has been shown with previous studies that the cavity configuration can increase the amount of stress on the marginal adhesive interface [26].

In this work the results obtained for the dye penetration test, show clear tendency to gap formation and dye penetration for the thermal shock samples, and indicating that abrupt changes in temperature could have a more deteriorating effect on the interface, seemingly because of the increased stresses generated due wise [27].

The dye leakage method was criticized by several authors, for its inability to quantify the marginal leakage phenomenon. On the other hand the method was considered by other study

groups as an overall evaluation of the interfacial integrity after aging [27].

The results obtained for the micro tensile bonding test showed significant difference in microtensile values between the control group and the thermal shock group, but not with the thermal cycling group, thus demonstrating effect of thermal shock on creating stresses and weakening the bonded adhesive interface. The microtensile bond strength is a widely accepted method for evaluating the bond strength across the interface [28]. In his review Heintze found that microtensile bond testing is more accurate in comparison to other methods used to evaluate the interface strength and the stresses that affect it [27].

An important point to be taken in consideration is the premature failure of the samples; the scientific community pointed the importance to integrate those into the results, while the absolute value was not a matter of agreement [29,30]. The value used in this study to represent premature failure have been 2 MPa, which represents half the minimal bond strength value obtained during testing and in order not to use markedly low values. It should be taken in consideration that 9 out of 20 samples failed prematurely for the thermal shock group, while only 3 for the thermal cycling group, and none for the control group samples. On the other hand the type of fracture whether cohesive or adhesive (Fig. 2) and the number of premature failures as represented in Table 2; show clearly the effect of thermal stresses on the adhesive interface.

The samples studied under scanning electron microscopy as shown in Fig. 3, provided proof that the initiation of failure was mainly between the enamel and adhesive resin part of the interface.

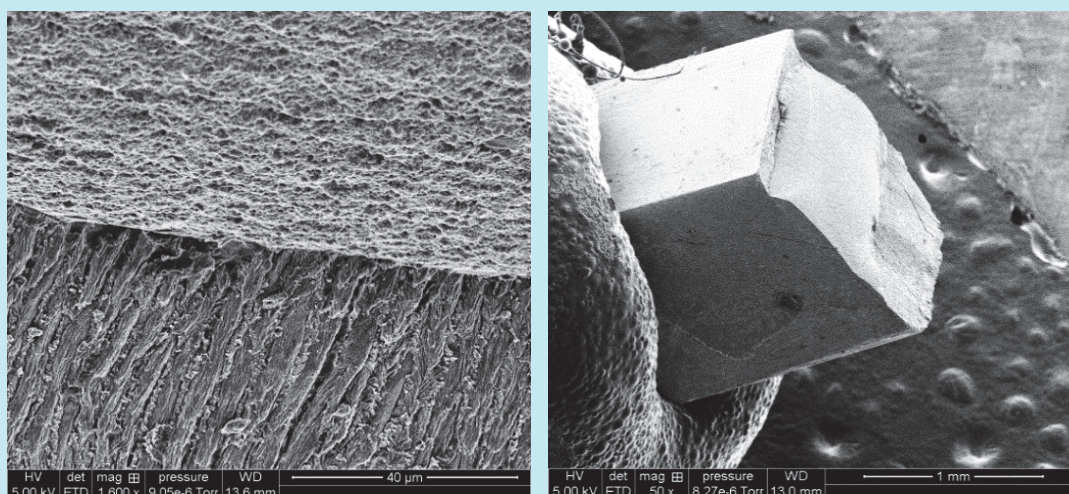


Fig. 3. Scanning electron microscopy images showing initiation of failure between enamel and the adhesive layer

The samples were subjected to 600 thermal cycles corresponding to one month of function in the oral cavity, and which is considered by the norm ISO to be appropriate for simulating the aging of biomaterials *in vivo* [8,13]. The experimental setup has not taken into account the exact time interval between the thermal shock and the thermal cycling (5 and 10 seconds for the thermal shock with no interval between the hot and cold, and 30 /30 seconds for thermal cycling with 3 seconds for the samples to pass between the hot and cold water baths). The slow transition and adaptation that is compensated in the thermal cycling method was meant to be eliminated in thermal shock setup, moreover the continuous waterjet projected on the tooth surface would allow closer contact and more efficient temperature transfer [31].

Adhesion to dentin is more vulnerable to stress and failure. In the present study enamel was chosen as a substrate for testing the interface, mainly because in the clinical situation, enamel anatomically covers the dentin, and the enamel resin interface is the part more exposed in the oral cavity and more subject to temperature changes, physical and mechanical stresses, while most of these are much attenuated by the time they reach dentin [10].

The major shortcomings of the present study are the limited number of samples, together with the limited number of thermal cycles used during the test. In this study only one type of composite resin and adhesive were used, a large variety of adhesives and composite resins should be

used to cover the varied compositions and thermophysical properties of existing materials. Future work should evaluate the temperatures, time durations and experimental setup used in the current study.

5. CONCLUSION

Within the limitations of this work, it was shown that thermal shock induces more stress on the enamel-composite restoration interface, which may lead to cracks and gap formation, possibly leading to eventual failure of the restoration overtime.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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The presented study investigated the impact of abrupt changes in temperature on the longevity of restorations in the oral cavity. The use of the mastication bench device would be very valuable in the evaluation of the impact of similar physical stresses. Moreover, it would allow the integration of prolonged cycles and the systematic assesment of such effects. It would also permit not only evaluation of these stresses on the interface region but equally on the dental tissues and the restorative materials.

The effect of these thermal and physical stresses on the mechanical properties would be evaluated under controled testing procedures with the use of the mastication bench.

4. Dental Materials evaluation

4.1. Types of tests used to evaluate dental materials

Requirements for a Laboratory Test

Laboratory tests are useful for testing new operative techniques and materials before they are used in clinical practice. The methods employed, however, should meet the following requirements (FDA 1978):

- The results must be reproducible, meaning that the same results should be obtained when the test is repeated under the same conditions and with the same materials.
- The parameters which influence the test results must be known.
- The variability of the measured values must be low and within an acceptable range, the ratio of the standard deviation to the mean (The coefficient of variation) should be under 20%, and its value determines the number of specimens per group.
- The devices used for the test or for measuring the test parameters and post-testing conditions of the specimens must be suitable and qualified for the given purpose. The device may have to be calibrated before performing the test or measurement. Proof and documentation must be established.

The advantage of ISO Standards is that the defined test methods are accurately described and can be performed in laboratories with relative ease. The specifications for

material properties are the greatest common denominator between the representatives of industry, authorities, and universities, who work together in the standardization committees [67].

4.2. ISO Tests

The dental committee of the International Standards Organization (ISO/TC 106) has already developed over 160 dental standards. ISO dental standards play a major role in controlling the quality and safety of products used by dental healthcare professionals in dental treatments.

The ISO/TC106 committee has 46 member countries (25 actively participating and 21 observing). In addition to the full technical committee, there are seven sub-committees and some 44 working groups, which have input and participation from close to 300 international dental experts from the member countries. The ISO committee also collaborates with other organizations like the World Dental Federation (FDI) and the World Health Organization (WHO).

Besides its main function in attaining performance standards and requirements, it also plays an important role in the classification and coding systems of various dental instruments and equipment.

The establishment of these standardized regulations involves extensive procedures and arguments. Finally, the publication of an ISO Standard requires vote, in support of the document, by at least 75% of the member bodies (countries). In three years

a technical specification is reviewed to decide whether it will be confirmed for a further three years, withdrawn or become an international standard.

The importance of these international standards can be revealed by the fact that 90% of the population worldwide suffers, at some time in their lives, from dental disease that require clinical treatment and that a high proportion of the population use oral hygiene products [64,68-69].

The dental committee of the International Standards Organization establishes several requirements, among these:

- Handling properties of the material.
- The esthetics of the material.
- The longevity of the placed filling. (Flexural strength, fracture strength, surface hardness, water sorption, solubility, polymerization shrinkage and shrinkage force).

Other regulatory bodies define different sets of standards, for example, the mark CE (European Community) means that the product corresponds to the basic requirements and standards of the CE guidelines. Starting of June 1998, all products brought onto the market in the EU or the European Economic Area and Switzerland must bear the CE mark. On the other hand, this set of standards is no guarantee for quality.

Another set of standards are the American Dental Association (ADA) Guidelines for acceptance. ADA Guidelines demand two clinical tests of the product over 18 months in addition to the laboratory test before awarding the product the “Seal of Acceptance”.

This is the reason why most dental companies reject the program, as clinical studies considerably delay the marketing of a product.

A great advantage of standardized testing is that the values obtained in different institutes can be compared to each other. Furthermore, these in vitro tests provide physical values that are highly useful for assessing the clinical suitability of the materials.

The disadvantage of the standards, however, is that some of them lie below the ideal range, because the limits are based on a consensus between the manufacturers and the testing institutes.

On the other hand, critics of the laboratory tests according to ISO claim it to be very far from clinical reality, as the test set-ups don't use natural teeth, but mostly manufacture standardized material specimens.

In a review by Heintze et al, the ISO testing for dental composite was examined, according to the standard ISO 4049 used in Dentistry (ISO 2009a). This standard describes both testing methods and the minimal requirements for polymer-based filling restorations and luting materials.

In his work, he investigated to what extent these tests have implications over the clinical performance of dental composites. Among these, are tests that evaluate wear, polishability, optical properties, handling properties and expansion after water sorption.

Other important properties are the Modulus of elasticity and Microhardness, which depends largely on the filler particles (size, weight percent, volume percent) and chemical

composition. Moreover studies have shown that the harder the material the greater its wear resistance [64].

Another important feature for dental composites is the polymerization shrinkage, which is tested by three standardized methods: the bonded disk method, the Archimedes test and the photo-elastic method. The shrinkage of current composites range from 1.5 to 3 vol% and does not seem to have the critical influence on marginal staining and secondary caries that has been shown in the laboratory.

The depth of cure test determines the degree of conversion obtained and attained by the composite layer according to its thickness. In the test, composite is placed in the hole (6 X 4 mm) of a stainless steel mold and polymerized. The unpolymerized portion is removed with a plastic spatula, and the remaining composite thickness is measured and divided by two. Composites usually present a depth of cure of at least 2 mm. The ISO standard specifies a minimum of 1.5 mm (ISO 2009a).

Another method of determining depth of cure is to measure the Vickers hardness of the top and bottom of different thickness specimens. In this method, a pyramid-shaped diamond instrument, with a defined speed and a load of 10 N, is pushed into the material and the diameter of the diamond impression in the material is recorded.

For a material to be qualified as completely cured, the surface hardness of the bottom must be at least 80% of that of the top. Studies have shown that this value correlates well with half the depth of cure test as specified in the ISO standard [70].

The ISO standard stipulates a handling time of at least one minute before the composite starts to polymerize in ambient light. The clinical relevance of this test lies in

the information, about how long the dentist can handle the material before the ambient light cures it. In this test, the composite is illuminated for 60 seconds, under predetermined conditions, with a UV filter, and then compressed between two glass plates to a thin film, to detect any in homogeneities due to premature polymerization.

The ISO testing for Radiopacity is set by comparing the composite material to an aluminum standard. A standardized specimen and the aluminum standard are x-rayed together, the distance between the x-ray tube and the film is 300–400 mm. The optical density of the tested material is then compared to the aluminum standard, and must be greater than or equal to that of the standard. This test shows that although the protocol is clinically relevant, the minimum value is set too low.

Another important test, with established clinical relevance, is the standardized test for water sorption. High water sorption negatively influences the swelling, discoloration, and transparency of the material [71].

During water sorption, first the bond between the matrix and the filler is weakened, and finally the accumulated water decreases the material strength [72]. The consequence of this material alteration is that other measured parameters like the flexural strength and abrasion resistance also undergo detrimental changes [73].

The flexural or bending strength is a measure of the fracture resistance of a restorative material in occlusion bearing areas.

The ISO standard demands a flexural strength of at least 80 MPa. For this test, bar-shaped specimens are made, stored in water for 24 h and at 37 °C, and then loaded until failure in a universal testing machine (crosshead speed 0.75 mm/min [+/-0.25]). The

flexural strength was found to possess a direct clinical correlation and great predictive value for the success of restorative materials in practice.

4.3. Tooth-material interface

The tooth material interface or the adhesive interface in case of dental composites and ceramics, represent a unique challenge to the testing and evaluation process.

The fact that the interface is composed of two or three materials with different physical and mechanical properties, the way, in which the stress is distributed among the interface components, and the analysis of failure of the adhesive interface, all represent different aspects of the problem. Several methods are used to test the interface:

4.3.1. Direct analyses of bond strength

4.3.1.1. Shear strength measurement

The main problem with the Shear strength measurement is that the operator seems to have a considerable impact on the test result. The distribution of stress along the interface is not uniform adding to the complexity of the testing method.

4.3.1.2. Macro- and microtensile tests

While Macro tensile bond strength test were conventionally used in the past, Microtensile bond strength methods has largely replaced them.

Microtensile bond strength method present several advantages over the conventional Macro tensile test. Fewer teeth are needed, differences in dentin adhesion according to region can be evaluated. However, the method is labor-intensive and technique sensitive.

Conversely, high in vitro values do not necessarily indicate good clinical performance, but generally the macro- and microtensile test results correlate better with retention loss of cervical restorations than do shear bond tests.

4.3.1.3. Push-out test

The push-out test is mostly used for radicular root canal material testing. One advantage of the push-out test is that no pre-test failures occur at the bonded surface of the specimen, and that the coefficient of variation of the test results, seems more acceptable.

4.3.2. Indirect analyses of bond strength

4.3.2.1. Dye penetration (Microleakage)

It was found that all types of restorations exhibit some sort of microleakage, and that dentin margins exhibit more dye penetration than enamel margins [74].

Silver nitrate staining is the preferred method for viewing the marginal bond defects, it is claimed that this method could be used to assess the quality of the bond. Silver ions are able to infiltrate the microscopic gaps, at the interface between dental tissue and

restorative material or adhesive material, where they precipitate and form inclusions of metallic silver which can then be visualized through X-ray [75].

On the other hand, the dye leakage method was criticized for several reasons. It is unable to quantify the strength of the bond, and it has irreproducible results. In fact, results obtained from different testing institutes could not be compared [76].

Moreover, no correlation could be found between dye leakage and the occurrence of marginal gaps [64,74,77].

4.3.2.2. Micro scanning tomography (microCT)

The main conventional microleakage tests have several disadvantages. Irreversible destruction and loss of information of the samples, limited number of locations could be evaluated, since the leakage could be only assessed in two dimensions.

Other techniques, such as spectro-photometric dye-recovery methods, serial grinding and imaging methods and the 'whole wall technique' are equally destructive and share the same limitations.

In addition to being a non-destructive technique, moreover, Micro-CT can reach a potential resolution in the submicron range. Furthermore, coupled with silver nitrate infiltration can enable better assessment of the interface and the presence of marginal gaps [74].

Micro-CT, on the other hand, is a non-destructive technique which can, moreover, reach a potential resolution in the submicron range. Coupled with silver nitrate infiltration can enable better assessment of the interface and the presence of marginal gaps [74].

	Reproducibility	Qualitative / Quantitative	Operator dependent	Destructive	Number of samples	Clinical relevance	cost
<i>(microCT)</i>	Yes	Quantitative / semi Quantitative	No	No	Little	Slight	Elevated
<i>Dye-penetration (Micro leakage)</i>	Slight	Semi Quantitative	Yes +++	Yes	Many	No	Low
<i>Microscopy</i>	Slight	Qualitative + semi Quantitative	Yes +++	No	Many	No	Low
<i>Shear strength</i>	Slight	Quantitative	Yes (sample prep)	Yes	Many (per tooth)	Slight	Intermediate
<i>Macro / micro tensile tests</i>	Slight	Quantitative	Yes (sample prep)	Yes	Many (per tooth)	Slight	Intermediate
<i>Push-out test</i>	Slight	Quantitative	Yes (sample prep)	Yes	Many (per tooth)	No	Intermediate

Table 4. Different tests for tooth restoration interface evaluation.

The evaluation of the adhesive interface using a new method to avoid the faulty stress distribution across the interface, and to explore the difference between the dentinal substrate effects on the adhesive properties, is the aim of this study.

Original Research Article: Evaluation of interfacial type of fracture using two adhesive systems



Evaluation of Interfacial Type of Fracture Using Two Adhesive Systems

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Abstract

The aim of this study was to concentrate all the loading forces of a bond strength test on the dentin-adhesive interfaces either bonded with an etch-and-rinse or a one-step self-etch adhesive system; the results were compared to that of a glass ionomer cement. Superficial and deep dentin discs were prepared from freshly extracted teeth and bonded to a one with two-step etch-and-rinse Adper™ Scotchbond™ 1XT (3M ESPE, Seefeld, Germany), and one-step self-etch Adper™ Easy Bond (3M ESPE, Seefeld, Germany), and a control group with glass ionomer cement GC Fuji IX (GC Corp, Leuven, Belgium). The 4 by 4 mm bonded areas were subjected to shear stress and targeted only at the thickened adhesive layer. Bond strength and crack length measurements were obtained. Interface morphology and fracture mechanisms were observed using scanning electron microscopy. Deep and superficial dentin bond strength measurements were statistically significant difference showing higher values for the deep dentin than for the superficial dentin. Crack length measurements for Adper™ Easy Bond showed a higher value than with Adper™ Scotchbond, GC Fuji IX. Scanning electron microscopy demonstrated cohesive failures inside the adhesive layer for both adhesive types. The test setup, with the thickened adhesive layer, made the results more dependent on the mechanical properties (stiffness) rather than on the adhesive properties of the adhesive material itself and reflects its ability to resist the fracture load.

Keywords: Dentin adhesive; Adhesive testing; Interfacial

Introduction

Adhesive restorative dentistry relies on adhesion to tooth structure and depends on the bonding substrate. While bonding to enamel is relatively reliable, adhesion to dentin is much less predictable due to the tubular structure of dentin [1], dentinal fluid transduction and the presence of smear layer [2,3]. In adhesive dentistry, resin monomers replace minerals removed from the intertubular and peritubular dentin after the collagen matrix is expanded after an initial priming of the tissues. Following this interaction by either etch-and-rinse or self-etch components, adhesive resin monomers replace the removed minerals and become micro-mechanically interlocked in the created porosities [1,2].

In order to predict their mechanical properties, dental adhesive systems are submitted to a variety of laboratory tests of their sealing ability by measuring the mechanical bond strength [4].

This is obtained from the load needed to break the bond between dentin and composite; when related to the cross sectional area of the interface, this is referred to as the nominal bond strength [2,3]. The failure can be induced through tensile or shear loads; the tests can be achieved either as macro tests (with relatively large bonded areas with bonding surfaces around 7mm²), or micro tests with smaller bonded areas (with bonding surface around 1 mm²) [2,5-8]. They can be used for the screening of new adhesives in the study of their mechanical properties [7].

Conventional macro-shear tests or tensile tests are easy to perform; micro-shear or micro-tensile tests are more demanding and require a technique-sensitive specimen preparation. A high discrepancy can be found regarding the results of the mechanical bond tests for the same adhesive owing to the laboratories and the tests performed [8]. It is believed that this is explained by the non-uniform interfacial stress distribution [9,10] and the numerous interactions of experimental factors, such as the substrate or methodological factors. Moreover, a wide variation exists in the physical construction of the test with such variables as the geometry of the sample and the physical parameters of the test (knife shape, sample holder geometry, cross head speed) [6-10]. Therefore, the stress build-up and force distribution plays a major part

in this variation, and much work has been done to sort out the problem [9,11,12]. Finite element and fractographic analysis were used to predict stress distribution across the interface [2]; it was concluded that once dentin cohesive failure takes place, the calculated failure strength no longer represents the interfacial stress [9]. Soft matter physicists [13] calculated that the fracture toughness and the corresponding energy needed for rupture, depends on the type of mechanical test chosen to measure it; therefore the fracture energy strongly depends on the visco-elastic properties of the adhesive, and the velocity of the fracture [9-13].

Several studies have described the relationship between dentin depth and mechanical bond test results. The structural variation of dentin - i.e. depth, number of tubules and the thickness of peritubular layer - affects the obtained bonding values which differ according to the superficial or deep dentin [14,15].

Following these observations an experimental set up was designed to avoid the stress dissipation effects on the mixed physical assembly that comprises the composite and the adhesive. This resulted in a modified type of shear bond testing aimed at the 500 μm adhesive layer, without putting stress on the composite part. The adhesive layer was thickened in order to concentrate the load in the adhesive. We investigated the bond strength of dentin-adhesive interfaces either with an etch-and-rinse or a one-step self-etch adhesive system; we used a glass ionomer cement, which has an ionic exchange mechanism of chemical adhesion, as a control. Therefore, the aim of the study was to test the two following hypotheses, when the load is directly applied

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to the adhesive layer: - self-etch and etch-and-rinse adhesives exhibit similar values of adhesion; - superficial and deep dentins do not differ in regards to the adhesion values.

Materials and Methods

Specimen preparation

Following removal from patients with informed consent, caries-free freshly extracted third molars, were kept in a 0.5%-chloramine solution at 4°C temperature for five days then in distilled water, until further processing (ISO/TS 11405 norm). After cleaning and removal of superficial debris from the surface, occlusal enamel was removed. Two parallel sections to the occlusal surface were cut to obtain 2 mm-thickness dentin slices. A diamond-disc-operating saw was used at slow speed and under constant irrigation (Isomed, Buehler Ltd, Lake Bluff, IL, USA). The superficial dentin slices were cut below the dentin-enamel junction; the deep dentin sections were made 2 mm below the first ones, above the roof of the pulp chamber. The obtained dentin slices were then prepared according to the following two-steps procedure: first, the surfaces were mechanically polished using an initial sequence of 120-400-800 grit waterproof abrasive papers of silicon carbide. (Escil, Chassieu, France) Secondly, an additional and final polishing was performed with 6 mm, 3 mm and 1 mm diamond abrasive papers in order to maximize the removal of the smear layer (Struers A/S, Ballerup, Denmark). At the end of the polishing process, all the specimens were ultrasonically cleaned for 8 min in water, rinsed thoroughly in ultra-pure water, and finally kept in deionized water at 4°C temperature.

Each group consisted of 15 samples prepared one with two-step etch-and-rinse Adper™ Scotchbond™ 1XT (3M ESPE, Seefeld, Germany), and one-step self-etch Adper™ Easy Bond (3M ESPE, Seefeld, Germany), and a control group with glass ionmer cement GC Fuji IX (GC Corp, Leuven, Belgium). For each adhesive two dentin substrates were considered (superficial and deep).

Adhesion area and Adhesive layer thickness control

In order to minimize the adhesion area variation in bond strength measurements, the adhesive thickness was geometrically constrained; a polysiloxane rubber-base mold with a constant area of (4 X 4 mm) surface was applied under mechanical pressure over each sample of the polished dentin surface.

A constant volume (9 µl) was delivered within the mold; this resulted in a 500 µm thickness of the adhesive layer on each sample.

Bond strength measurements and crosshead speed

Bond strength measurements were made using a Universal Mechanical testing machine MTS™ (Servo hydraulic - Adamel Lhomargy DY-34, France), where a blade was forced perpendicularly to the adhesive interface (Figure 1). Data were recorded with the MTS™ software attached to the machine.

We used a 15° angle beveled sharp blade with a 15 µm cutting edge, so that the load of the blade would target only the thick adhesive layer. A digital stereomicroscope was used to obtain the blade bevel and cutting edge measurements; all experiments were conducted with a low cross head speed at 0.5mm/min.

Crack length measurements

The crack length measurement was calculated as the distance travelled by the blade into the substrate - before separation of the

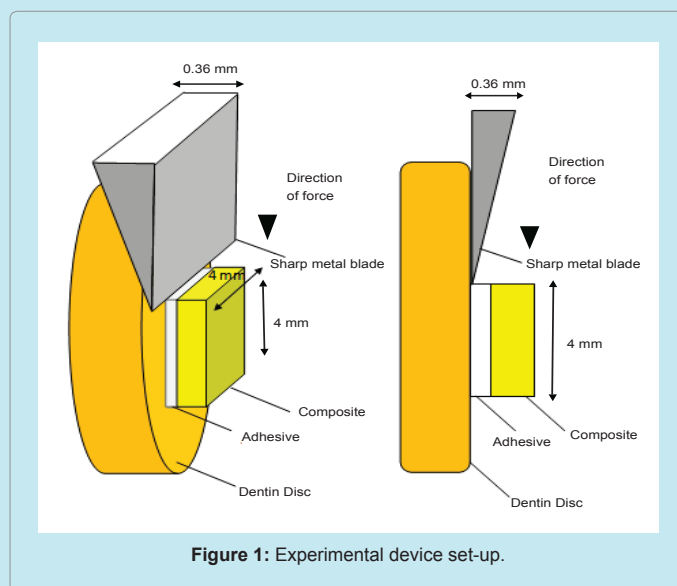


Figure 1: Experimental device set-up.

adhesive from the dentin surface. This was obtained with the data from the Universal Mechanical testing machine MTS™ when testing the interfacial bond strength.

Scanning electron microscopy

In order to locate the position of the crack within the specimen, scanning Electron microscope observations (S800-1, Hitachi Europe Ltd. Whitebrook, Berkshire SL6 8YA Parc, United Kingdom) were conducted on randomly selected specimens from each group. Samples were dried, sputter coated with metal and observed. The level of separation was determined for each substrate under magnification. The failure mode was assessed as: adhesive (along the interface without composite or dentin involvement), cohesive (totally in dentin or in composite) or mixed (at the interface involving dentin or/and composite).

Statistical analysis

Statistical significance of the results was calculated by means of the SPSS™ Software version 17.0.2, and the bond strength results and crack length measurements data were analyzed by ANOVA. Multiple comparisons were done using the Fisher's test (0.001 < p < 0.05).

Results

The bond strength values were recorded according to the type of dentin - superficial or deep- for each adhesive. The crack length measurements were obtained from the mechanical testing machine records and plotted to the applied load at the rupture point. No pretest failure occurred among the samples, none of the adhesive discs did prematurely detach in any of the groups.

Bond strength measurements

The forces needed for debonding were calculated in MPa. The bond strength measurements vary according to the dentin substrate and to the adhesive type. Higher values were noted with the self-etch than with the etch-and-rinse adhesive which appear to exhibit different behaviors.

Bond strength values according to the superficial or deep dentin: Bond strength values according to the dentin substrate depth are presented in figure 2. Deep and superficial dentin bond strength

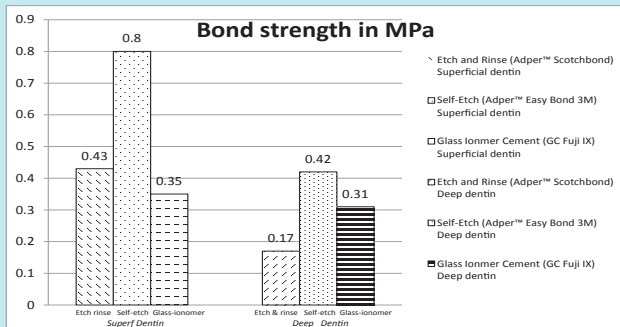


Figure 2: Bond strength values according to type of adhesive used and according to type of dentin substrate.

measurements were respectively at 0.43 ± 0.27 MPa and 0.17 ± 0.12 MPa for the etch-and-rinse adhesive (Adper™ Scotchbond), with a mean difference at 0.265 MPa ($p=0.0033$). They were at 0.8 ± 0.35 MPa and 0.42 ± 0.31 MPa with a mean difference at 0.377 MPa ($p<0.0001$) for the self-etch adhesive (Adper™ Easy Bond 3M). There was a statistically significant difference showing higher values for the superficial than for the deep dentin. With the glass ionomer cement (GC Fuji IX) the values were at 0.35 ± 0.18 MPa and 0.31 ± 0.14 MPa with a mean difference of 0.032 MPa ($p=0.7228$); no statistically significant difference was noted between the dentin substrate depths. Statistical relevance of these results is presented in table 1.

Bond strength values according to the type of adhesive: Bond strength values according to the type of adhesive are presented in figure 2. Considering both dentin substrates superficial and deep dentin, a significant difference was found between etch-and-rinse and self-etch adhesives: for superficial dentin there was a mean difference of 0.363 ($p<0.0001$); for the deep dentin there was a mean difference of 0.251 ($p=0.0060$) with higher values for the self-etch adhesive. Also, a significant difference was found between the bond strength values of self-etch (Adper™ Easy Bond 3M) and the glass ionomer cement (GC Fuji IX) with the superficial dentin with a mean difference of 0.451 ($p=0.0001$), while for the deep dentin there was no significant difference for the bond strength values with a mean difference of 0.106 ($p=0.2387$). Similarly there was a significant difference between bond strength values of the etch-and-rinse (Adper™ Scotchbond) and the glass-Ionomer Cement (GC Fuji IX) for the superficial dentin (mean difference 0.088, $p=0.3262$). Statistical relevance of these results is presented in table 1.

The crack length measurements in mm

Crack length measurements according to the dentin substrate: The crack length measurements before separation are presented in figure 3. For the self-etch (Adper™ Easy Bond 3M) there was no significant difference between superficial dentin and deep dentin; the crack length measurements were respectively at 0.69 ± 0.2 mm and 0.51 ± 0.2 mm). The statistical relevance of these results is presented in table 2.

Crack length measurements according to the adhesive type used: Higher values are noted for self-etch than for etch-and-rinse adhesive. There was a significant difference between self-etch (Adper™ Easy Bond 3M) and etch-and-rinse (Adper™ Scotchbond) on both superficial dentin (mean difference 0.39 - $p<0.0001$) and deep dentine (mean difference 0.232 - $p=0.0029$). In addition a significant difference was calculated between self-etch (Adper™ Easy Bond 3M) and glass ionomer cement (GC Fuji IX) with a mean difference of 0.36 ($p<0.0001$) for

superficial dentin and weaker significance for deep dentine with a mean difference of 0.14 ($p=0.0567$).

Correlation between load in MPa and the distance travelled by the blade before separation: No correlation was found in figure 4 drawn between the load in MPa and the distance travelled by the blade before separation for the different adhesive types according to the dentin substrates. The values for deep dentin are organized in a linear fashion; one can possibly conclude that the substrate is homogeneous and that the mechanical behavior is essentially related to the material.

material	Etch-and-rinse (Adper™ Scotchbond)	Self-Etch (Adper™ Easy Bond 3M)	Fisher	P value
Superficial dentin	0.43 (0.27)	0.8 (0.35)	b	< 0.0001
Deep dentin	0.17 (0.12)	0.42 (0.31)	b	0.006
ANOVA	b	b		
P value	0.003	< 0.0001		

^aMeans are not significantly different (Fisher's test)

^bif the p-value <0.05, the MPa for debonding is significantly different

Table 1: Fisher's test results for Bond strength values of the different adhesive types.

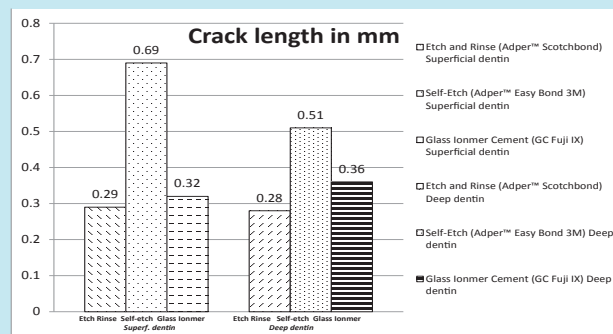


Figure 3: Distance in mm according to type of adhesive used and according to type of dentin substrate.

material	Etch-and-rinse (Adper™ Scotchbond)	Self-Etch (Adper™ Easy Bond 3M)	Fisher	P value
Superficial dentin	0,29 (0,1)	0,69 (0,2)	b	< 0.0001
Deep dentin	0,28 (0,1)	0,51 (0,2)	b	0.0003
ANOVA	b	b		
P value	0.83	0.0059		

^aMeans are not significantly different (Fisher's test)

^bif the p-value <0.05, the MPa for debonding is significantly different

Table 2: Fisher's test results for crack length measurements of the different adhesive types.

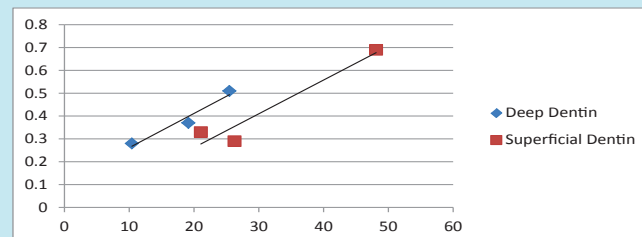


Figure 4: Relationship between load in MPa and distance in mm for the three adhesive types and the two dentin substrates.

The values for superficial dentin are more scattered; thus this substrate appears more heterogeneous resulting in larger value dispersion, possibly depending on the material. Moreover, the plotted lines are parallel for both dentin substrates and suggest a linear relation between the load applied and the crack propagation into the material.

Scanning electron microscopy results: Several samples were inspected by scanning electron microscopy to detect the level of separation between the adhesive complex and the dentin disc. The crack was restricted within the adhesive layer, as shown in figures 5 and 6, resulting in an adhesive failure.

Discussion

On behalf of the reported results in the present study, both null hypotheses were rejected. Self-etch and etch-and-rinse adhesives did not perform similarly in the experimental physical set-up described here. Higher values were found for self-etch (Adper™ Easy Bond 3M) than for etch-and-rinse adhesive (Adper™ Scotchbond). This was noted for both superficial and deep dentin; in addition the level of significance was increased for superficial dentin which demonstrated higher bond strength values.

In the present experiment, the adhesive bonding area and thickness measures were maintained constant by using a polysiloxane rubber base mold that was applied under pressure, to facilitate the interpretation of the nominal strength values [16,17]. Stress concentration at the adhesive interface is a major factor that complicates the interpretation of the nominal bond strength results. The nature of the test set-up further reflects the ambiguity of the results [9,18]. The present experimental

set-up tried to address the common problem of stress distribution found with bond strength testing methods. Though the complex stress distribution seems unavoidable, we attempted to enlarge the adhesive layer thickness so that only the layer would be submitted to the main load. Electron microscopic observations images obtained confirmed that the targeted area was solely the adhesive layer, resulting in a failure within the adhesive layer.

The obtained values are comparable to previously reported fracture toughness values [19], by concentrating the load; the set-up tries to propagate a crack in the adhesive joint by using a sharp blade without exerting pressure on other components of the adhesive interface.

The measurements somewhat go against the common knowledge that etch-and-rinse adhesive systems perform better than other classes of adhesives for bond testing [3]. Yet an important difference must be pointed out regarding the method used in the present study, the mechanical properties of the adhesive material itself was the measured element as targeted by the sharp blade tip which endured most of the concentrated force. This is in contrast to the blunt knife used in the shear test method that spreads the load on all components of the adhesive bond complex [10]. We concluded that the material stiffness and its capability to withstand stress before failing were the main factors influencing the results. The comparable results in both the bond strength measurements and the crack measurement length for each material equally support the above conclusion. The force displacement curves obtained for the self-etch showed that distance travelled by the blade into the material before failure significantly exceeded that with the other two materials thus showing a higher flexibility and resilience; the adhesive layer would act as an elastic buffer. The elastic bonding concept, as described by Van Meerbeek [20], could serve as an explanation for the higher bond strength obtained with this material in our study.

The two types of adhesives used in this study appear similar in regards to the monomer composition presented in table 3, with Bis-GMA and HEMA as the main components, also containing ethanol and water and the presence of silica fillers to increase material strength, while the main difference lies in the presence of acidic monomers in the self-etch adhesive. On the other hand the higher values obtained in this study with the Self-Etch adhesive Adper™ Easy Bond adhesive would be related to the presence of a higher percentage of the more bulky and stiffer monomer Bis-GMA; in addition the dentin surface preparation minimized the presence of the smear layer thus decreasing the bonding strength for this type of adhesive [20].

As expected the glass ionomer cement had low mechanical properties and performances in this strength test; as a self-adhesive material it has good clinical adhesive properties depending on a chemical bonding to tooth structure, but exhibits a weaker performance during *in vitro* test methods which concentrate stress loading [18,19].

In the present study we found a significant difference between the deep and superficial dentin bond strength measurements. Deep dentine has a lower mineral content, a greater number and area of dentinal tubules, a smaller area of inter-tubular dentine, and is inherently wetter *in vivo* than superficial dentine; moreover, the critical stress intensity factor in superficial dentin can double that of deep dentin [14,15]. Our data compare to previously reported results considering the dentin depths [14].

It is worth noting that the results obtained in this study were associated with quite large standard deviations, it was found that even with fracture toughness which is an intrinsic property, values

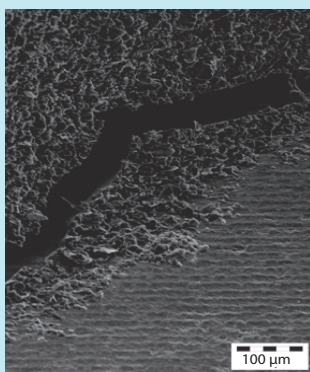


Figure 5: Fracture line (arrow) inside the adhesive layer of the self-etch adhesive.

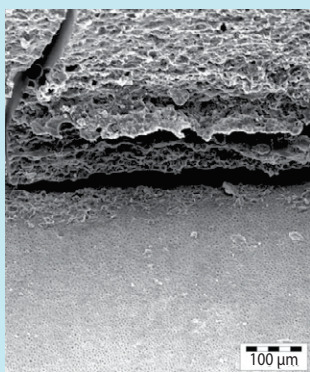


Figure 6: Fracture line inside the adhesive layer of the etch-and-rinse adhesive.

	Composition	Batch #	Manufacturer
Etch-and-rinse (Adper™ Scotchbond)	Bis-GMA 10-20% HEMA 5-15% Polyalkenoic acid copolymer 5-10% Water Ethanol Silica particles initiators	51010	3M ESPE, Seefeld, Ger- many
Self-Etch (Adper™ Easy Bond)	Bis-GMA 15-25% HEMA 15-25% functionalised polyalkenoic acid Water 10-15% Ethanol 10-15% Silica particles initiators methacrylated phosphoric esters, 1,6 hexanediol dimethacrylate, methacrylate 5-10%	437866	3M ESPE, Seefeld, Ger- many

Other names for Adper™ Scotchbond: Adper™ Single Bond Plus (USA)
Adper™ Single Bond 2 (Latin America, Asia, Australia)

Table 3: Composition of the two types of adhesives used.

are usually associated with large standard deviations (>25%) [16]. The elastic property differences across interfaces can lead to high interfacial stresses during interfacial fracture toughness. A wide scatter for individual results in dentin adhesion studies is reported [21] and the coefficients of variation range between 28 and 36% for shear tests, 21% and up to 40% for micro-shear tests [6]. Non-uniform stresses generated within the shear zone have a significant effect on the mode of failure. Finite element analysis showed that complex stress states occur at the interface and resulting in unstable crack propagation [8].

Our study design depended largely on the viscoelastic properties the materials used and their ability to withstand stress rather than the different modes of adhesion. Earlier studies emphasized the importance of material stiffness and its ability to withstand loads and shock during function [14,20,22]. It has been suggested that the dentin-composite joint should be reasonably flexible to minimize stress concentrations in the bond during function. A number of factors influence the relative stiffness of this interface zone including the elastic moduli of its individual components, the thickness of these component layers, or the degree of interaction between them. Preliminary studies found a significant positive correlation between relative interfacial stiffness and interfacial fracture toughness through varying the adhesive resin layer thickness and modulus [23]. Previous studies that tested the adhesive layer properties [24] concluded that a flexible adhesive layer could resist polymerization shrinking stress and emphasized the role of adhesive constituents on mechanical properties [25,26].

Our results stress the role of dentin substrate type, and suggest a clinical relevance regarding the type of dentin involved in the cavity design. *In vitro* adhesion to dentin studies should not consider dentin as a uniform substrate; variations in dentin depth can influence the results of bond strength measurements.

Future work should include a wider variety of adhesives whether self-etch or etch-and-rinse both filled and unfilled. By concentrating the load to the adhesive layer, the soft matter physicist approach will provide quantifiable data, to study the role of chemical constituents in the mechanical properties of adhesives.

Conflict of Interests

With the submission of this manuscript the authors would like to certify that all of the named authors were involved in the work leading

to the publication of the paper and our Institute's representative is fully aware of this submission. And all the named authors have read the paper before its submission; the authors have declared that no competing interests exist.

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The following study explored the effect of irradiation on dentin adhesive properties.

Original Research Article: Tensile Bond Strengths of Two Adhesives on Irradiated and Nonirradiated Human Dentin.

Research Article

Tensile Bond Strengths of Two Adhesives on Irradiated and Nonirradiated Human Dentin

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The aim of this study was to assess the effect of radiotherapy on bond efficiency of two different adhesive systems using tensile bond strength test. Twenty extracted teeth after radiotherapy and twenty nonirradiated extracted teeth were used. The irradiation was applied *in vivo* to a minimal dose of 50 Gy. The specimens of each group were randomly assigned to two subgroups to test two different adhesive systems. A three-step/etch-and-rinse adhesive system (Optibond FL) and a two-steps/self-etch adhesive system (Optibond XTR) were used. Composite buildups were performed with a nanohybrid composite (Herculite XTR). All specimens were submitted to thermocycling ageing (10000 cycles). The specimens were sectioned in 1 mm² sticks. Microtensile bond strength tests were measured. Nonparametric statistical analyses were performed due to nonnormality of data. Optibond XTR on irradiated and nonirradiated teeth did not show any significant differences. However, Optibond FL bond strength was more effective on nonirradiated teeth than on irradiated teeth. Within the limitations of an *in vitro* study, it can be concluded that radiotherapy had a significant detrimental effect on bond strength to human dentin. However, it seems that adhesive choice could be adapted to the substrata. According to the present study, the two-steps/self-etch (Optibond XTR) adhesive system tested could be more effective on irradiated dentin compared to three-steps/etch-and-rinse adhesive system (Optibond FL).

1. Introduction

“Radio-induced caries” are a well-known consequence of the radiotherapy of head and neck cancer malignant tumors. Hyposalivation which is induced by irradiation [1, 2], dietary changes [3], and oral flora modifications [4, 5] are considered as the most important etiological factors of these caries [6]. Radio-induced caries begin near the gum and surround the cervical zone of the tooth leading to coronaradicular fracture [7]. The loss of mechanical autocleaning of these surfaces as a result of decreased salivary flow probably explains this location.

While there is lack of data published on this topic, evidences suggest a conservative approach using adhesive restorations [8]. Haveman and Redding have shown that

conventional glass-ionomer cement (GIC) had poorer results than the resin-modified glass-ionomer cements (RMGICs) and composite fillings in patients treated by radiotherapy [9]. Moreover, according to several studies, it is not recommended to use the GIC as restorative material for patients suffering hyposalivation and having a daily fluoride application [10–12]. Composite resin restorations are an alternative for both esthetic and wear resistance.

The loss of adhesive restorations can be due to an alteration of dental tissues as a consequence of head and neck irradiation. A significant decrease of dentin microhardness has been observed after irradiation [13]. These observations were accompanied by reduction of the stability of the enamel/dentin junction [14]. The disturbance of

enamel/dentin junction could result in the formation of a gap (10 μm), loss of prismatic structure, and bacterial colonization associated with the obliteration of the dentinal tubules and odontoblastic process atrophy [15, 16]. These characteristics can be observed via scanning electron microscopy (SEM) [17, 18]. Furthermore, the radiogenic destruction of the dentin collagen could result in bonding failure [19].

As the loss of these restorations is time dependent, it was suggested as a reliable method to test the durability of the bond strength by accelerated ageing [20–27]. Thermocycling tests evaluate the stress of adhesive interface to water infiltration, mechanical and contraction/expansion tension by an alternative immersion in cold water (5°C) and hot water (55°C) [28]. This can result in cracks which propagate along the adhesive interface, a process known under the name of “percolation” [29]. This method of ageing is suitable for dental adhesive systems and recommended by the International Organization for Standardization (ISO, TR 11450) [30].

The purpose of this study was to evaluate the incidence of the radiotherapy on tensile strength of two adhesives on the human irradiated and nonirradiated dentin.

2. Material and Methods

2.1. Sample Preparation. Forty human extracted teeth (incisors, canines, premolars, and molars) were collected (gathered following informed consent). Twenty came from irradiated patients suffering from head and neck cancer. These teeth received a minimal dose of 50 Gy and were extracted because of periodontal disease. Twenty other teeth came from nonirradiated patients and were used as control group. All teeth were collected and stored in physiological solution for a period not exceeding two weeks; then, they were stored in distilled water at a temperature of 5°C. Class I cavities on molars and class V cavities on other teeth (4 × 4 × 2 mm) were prepared with a cylindrical medium-grit (100 mm) diamond bur (FG 068-040, Komet France SA, Paris, France) under constant water irrigation. The burs were changed for every 8 teeth.

2.2. Experimental Design and Bonding Procedures. Each group was randomly divided into 2 subgroups of 10 teeth. The subgroups were restored using a two-step/self-etch adhesive system (Optibond XTR, batch number 5092152, Kerr France, Créteil, France) or a three-step/etch-and-rinse adhesive system (Optibond FL, batch number 4995918, Kerr France, Créteil, France). The adhesive materials were used following manufacturer’s instructions (Table 1).

Restorations were made using a nanohybrid composite resin (Herculite XRV Ultra, Kerr France, Créteil, France) with 2 layers of 1 mm thickness. Photopolymerization of the resin-based materials was performed using a LED light curing unit (Elipar S10, 3M ESPE, Cergy-Pontoise, France) at 1450 mW/cm².

Subsequently, the resin-bonded samples of each group underwent artificial ageing using thermocycling machine (10000 cycles for 2 weeks) with baths at temperatures of 5°C and 55°C (Table 2) and 30-second dwelling time. The storage solution of thermocycling baths was changed weekly.

2.3. Sticks Preparation. Thermocycled teeth were included in resin to allow fixation during microtensile sample preparation. Four to six slices, 1 mm thick, were cut perpendicularly and through to the bonded interface using Diamond Disk Wafering Blades 15HC (Buelher, Düsseldorf, Germany) under constant irrigation (IsoMet Low Speed Saw, Buelher, Düsseldorf, Germany). The sticks were then individualized and measured (± 1 mm wide square section). The most peripheral sticks with residual enamel were excluded. A maximum of 4 sticks of the tooth central part were used trying to minimize the regional variability of dentin. The bonded surface area was calculated before each test by measuring the width with digital caliper.

2.4. Microtensile Bond Strength Testing (μTBS). Each specimen was attached following the methodology described by Perdigao et al. [31]. An aluminum device constituted of two symmetric parts, having a central notch (2 mm of depth and width) in order to allow autoalignment. Device surfaces were cleaned with alcohol. Tensile load was applied with a universal testing machine (DY34, Adamel Lhomargy SARL, Roissy-en-Brie, France), at a crosshead speed of 1 mm/min, to obtain the ultimate tensile strength, using a load cell of 1 KN.

2.5. Failure Mode Analysis. Fracture mode was determined at $\times 50$ magnification with a stereoscopic microscope (Wild Heerbrugg TYP 376788, Wild Heerbrugg, Switzerland) and recorded as cohesive failure and adhesive failure.

2.6. Statistical Analysis. The experimental design included (i) two fixed crossed factors: irradiation [yes(I)/no(NI)] and adhesive system (XTR/FL) leading to 4 subgroups and (ii) a random factor (tooth) nested in each subgroup: 10 teeth per subgroup with one to four replicates per tooth. The conditions for the application of statistical treatment were carefully verified. The effect of the tooth factor on the explained variable (bond strength of sticks: μTBS) was first assessed by a mixed linear model on the full dataset. In case of nonapplicability of this mixed model, we conducted a one-way nonparametric ANOVA per subgroup using Kruskal-Wallis test. Missing data were supposed to be missing at random and no data imputation was performed.

In case of no tooth effect on μTBS , normality of μTBS data was checked graphically and using the normality Shapiro test for each of the 4 subgroups. In case of nonrespect of normality in one subgroup, pairwise distributions comparisons were performed between subgroups. Four comparisons were *a priori* of interest: between the two control subgroups (NI:XTR versus NI:FL), between the two irradiated subgroups (I:XTR versus I:FL), and for each adhesive system: (NI:XTR versus I:XTR) and (NI:FL versus I:FL). Correction for multiple comparisons was performed to maintain the family-wise error rate at the significant level of 5%. For 4 pairwise comparisons, Bonferroni correction gave a significant level of 2-tailed single test equal to 0.05/4, that is, 0.0125. Data were reported as mean \pm SD per subgroup. Statistics were performed using the R language, version 3.1.2 available on the <https://cran.r-project.org/> website. Package nlme was used to perform mixed linear model.

TABLE 1: Adhesive systems reference and composition.

Product name (manufacturer)	Class of adhesive	Composition	Batch number
Optibond FL, Kerr France, Créteil, France	3-step/etch-and-rinse adhesive	<i>Gel etchant:</i> 37.5% H ₃ PO ₄ , water, and fumed silica <i>Primer:</i> (Ph = 1.8): HEMA, GPDM, MMEP, water, ethanol, photoinitiator (CQ), and BHT <i>Adhesive:</i> Bis-GMA, HEMA, GPDM, GDMA, photoinitiator (CQ), ODMAB, and fillers (fumed SiO ₂ , barium aluminoborosilicate, and Na ₂ SiF ₆)	4995918
Optibond XTR, Kerr France, Créteil, France	2-step/self-etch adhesive	<i>Primer:</i> (pH = 2.4 before application, reduction in 1.6 to the application in dental structure). Acetone, water, ethanol, HEMA, photoinitiator (CQ), and GPDM <i>Adhesive:</i> ethanol, HEMA, sodium hexafluorosilicate, MEHQ; nanosilica, barium; photoinitiator (CQ)	5092152

HEMA: 2-hydroxyethyl methacrylate.
 GPDM: glycerol dimethacrylate dihydrogen phosphate.
 MMEP: mono(2-methacryloyloxy)ethyl phthalate.
 CQ: camphorquinone.
 BHT: butylated hydroxytoluene.
 Bis-GMA: bisphenol A glycidyl methacrylate.
 GDMA: glycerol dimethacrylate.
 MEHQ: monomethyl ether of hydroquinone.

TABLE 2: Different constituents and brands of the thermocycling machine.

Bath of hot water	Brand, Fisherbrand (water bath heated, digital PID control UK plug 12L)
Bath of cold water	Fisher (Bioblock Scientific 18201)
Waterproof box for electric system	Schneider Electric, Telemecanique crouzet (ACM)
Timer for arm	Crouzet (Top 948, LCD MULTI-FUNCTION TIMER)

3. Results

Three teeth and one tooth out of 10 were missing in NI:XTR and I:FL subgroups, respectively. Linear mixed model was not appropriate because of the nonnormality of the normalized residuals ($p < 10^{-3}$). No effect of factor tooth was significant in each subgroup using Kruskal-Wallis test with p values ranging from 0.46 (I:XTR) to 0.84 (I:FL).

Due to the different number of samples by tooth, we obtained 15 observations for subgroup NI:XTR, 31 for NI:FL, 25 for I:XTR, and 27 for I:FL. The two subgroups relative to XTR exhibited nonnormal skewed distribution with p values $< 10^{-2}$.

Means and standard deviation of μ TBS are graphically presented in box plots in Figure 1.

On irradiated dentin, both adhesive systems (XTR and FL) did not show any significant difference with μ TBS in I:XTR subgroup equal to 12.2 ± 5.3 MPa (mean \pm SD) and in I:FL subgroup 11.3 ± 2.8 MPa ($p = 0.97 > 0.0125$). On nonirradiated dentin, they did not show any significant difference on bond strength with μ TBS in NI:XTR subgroup equal to 14.5 ± 4.8 MPa and in NI:FL subgroup 16.4 ± 6.2 MPa ($p = 0.42 > 0.0125$).

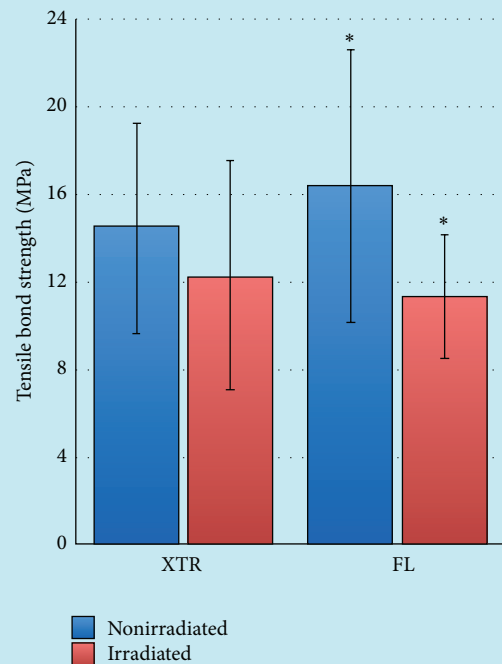


FIGURE 1: Mean of microtensile bond strength with standard deviation (MPa) according to the irradiation and the adhesive system (XTR/FL). *Significant difference between results ($p < 0.0125$).

Regarding FL groups, the value was significantly different between nonirradiated and irradiated dentin ($p = 0.0009 < 0.0125$). μ TBS was observed 1.5 times higher in nonirradiated subgroup in case of FL (33% decrease from nonirradiated to irradiated subgroups). On the other hand, no statistical differences were found for XTR adhesive system ($p = 0.040 > 0.0125$) with μ TBS observed 1.2 times higher in nonirradiated

TABLE 3: Adhesive and cohesive failure distribution.

	Adhesive fracture	Cohesive fracture
FL nonirradiated	48%	52%
FL irradiated	52%	48%
XTR nonirradiated	67%	33%
XTR irradiated	78%	22%

subgroup (16% of decrease from nonirradiated to irradiated subgroups).

The failure type for each group is summarized in Table 3. Adhesive failures at the composite resin/dentin interface were mainly observed for specimens treated with XTR. For FL adhesive system, there were as many adhesive fractures as cohesive failure.

4. Discussion

Head and neck cancers are one of the most common cancers [32]. Surgery and/or radiotherapy are the treatment of choice for such cancers [33]. Among the adverse effects like xerostomia or osteoradionecrosis, it has been demonstrated by several authors that radiation affects hard tissues [13, 34–37]. Regarding these consequences, some studies evaluated the bond strength on irradiated teeth. However, thermocycling for a sufficient time was not considered [38, 39]; furthermore, teeth were irradiated outside the oral cavity and after extraction [39, 40].

The procedure of the present study considers both the use of *in vivo* irradiated teeth and a sufficient thermocycling ageing protocol.

Teeth were stored in physiological saline solution immediately after extraction at the dental clinic and then in distilled water at 5°C. Even though Goodis et al. noticed that the physiological salt solution could have an action on dentin permeability and on traction resistance, unlike distilled water [41], Retief et al. have shown that saline solution does not influence the chemical and physical properties of human dentin [42].

Cavities were prepared using diamond bur under continuous water cooling to bring a higher traction resistance, compared with the abrasive 80-grits paper and to the diamond bur without irrigation [43]. As the experimental conditions should be standardized, dental composite resins were bonded on flat surface despite the overestimated bonding strength resulting in comparison to clinical conditions [44].

Regional differences in dentin anatomy and permeability have a significant influence on dentin bond strength [45, 46].

Photopolymerization time was applied according to the manufacturer recommendations and using the same light curing unit [47]. For all groups, the same resin composite and the same shade were used to avoid any influence of the composite material on bonding [48]. Several studies have shown the influence of the thermocycling ageing on adhesive systems strength [23, 26]. The standard (ISO TR 11450) recommends 500 cycles [30]. To simulate one-year ageing, as in the study of Gale and Darvell, a 10000-cycle experiment has been performed [28].

Several studies involving the two adhesive systems used in this work have been performed and have shown similar results [26, 49, 50]. Furthermore, according to De Munck et al. meta-analysis [51], Optibond FL, is the current reference in term of dentin bonding efficiency, on all the adhesives. These studies have been made on normal dentin. Nevertheless, the results obtained in this study showing μ TBS decrease (33%) in irradiated dentin for FL subgroups are consistent with literature. It is reported that the ionizing radiations may have an effect on the collagen fibers of dentinal tubules [19, 52]. Moreover, the changes described in the crystalline structure of dental hard tissues after irradiation seem to affect tensile strength [53–56].

With the XTR adhesive system, the weak decrease of μ TBS (16%) in irradiated dentin could be due to the chemical connections between the carboxylic or the phosphate groups of functional monomers and the phases of dissolved hydroxyapatite. These chemical connections would contribute to a better cohesion of the infiltrated resin after polymerization and, probably, in better resistance in the hydrolysis of this zone [57].

The results are in agreement with those of Naves et al. and S. Yadav and H. Yadav [40, 57]. Nevertheless, another similar study [58] did not find significant differences between irradiated and nonirradiated groups according to four adhesive systems, taking in consideration that no process of artificial ageing has been applied. In the present study, teeth were irradiated *in vivo* and, then, underwent adverse effects like hyposalivation.

5. Conclusion

The changes resulting from the irradiation on the hardness, the crystalline structure or the collagen matrix, seem to influence the adhesive agents bond strength to dentin. The dental substrate might have experienced radiation effects that could compromise bonding ability by impairing hybrid layer formation.

Under the limitations of this *in vitro* study, it appears that, regarding the type of adhesive system, radiotherapy may affect the microtensile bond strength of composite restorations on irradiated dentin. Therefore, it is advisable for a clinician to restore all cavities before radiotherapy and initiate caries prevention modalities in patients undergoing radiation therapy.

Further studies are needed to help the practitioner to adapt the choice of the adhesive system after radiotherapy of head and neck.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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The previous studies investigated the interface region, which plays a crucial role in the longevity of restorations. The results revealed the importance of the dental tissue substrate in obtaining a good adhesive interface, and the importance of evaluating the complex interface region in relevant way.

The mastication bench, as a device that simulates *in vivo* conditions, would allow the evaluation of different dental tissue substrates under similar conditions similar to that of the functioning oral cavity. It would also allow testing different adhesive system types under the same conditions.

A great advantage with such a device would be that it allows monitoring the interface region over extended cycles or time periods, using different methodologies, and thus obtain a more global understanding of the evolution of this region over time and under the impact of different types of stresses.

5. Oral simulators

5.1. Human oral cavity

Although the oral cavity environment seem impossible to replicate with all the temperature and pH fluctuation, bacterial contamination, and the various loads forced on the teeth. Still, In vitro studies can, to a certain extent, simulate the aging process in the oral cavity with a certain amount of success.

Chewing simulators are used in the dental domain to test dental materials, and to simulate the oral environment in an aim to evaluate the actual performance of restorative or prosthetic dental materials during function. Chewing simulators can test wear, loading of crowns and bridges to monitor the incidence of cracks and fractures, to test the integrity of tooth-material interface, and finally to evaluate the bio-adaptability of dental materials and material leakage from restorative materials [78].

Several reviews were made to compare the existing simulators to point out the advantages and disadvantages of each, and if they actually render the same results for the same material, and correlate adequately with the clinical outcomes [79,80].

5.1.1. Temperature

Since the early 50s, it has been noted that changing temperature of restored teeth in *in vitro* studies produces some sort of an exudate around the cavity margins. Thermal cycling has been, since, utilized as a tool to simulate the aging process in the oral cavity. Temperature fluctuation in the oral cavity has been reported to be between -5°C to 70°C [81].

The tooth material interface seems to be the region especially vulnerable to thermal changes. Differences in thermal properties would easily lead to the accumulation of stresses and opening of the margins. Though there is consensus regarding the effect of thermal cycling on dental materials, the results obtained between studies are not comparable, probably due to technical differences and study design [81]. More standardization of these methods is important before concrete conclusions could be drawn regarding the performance of a particular material as compared to others.

Device	Manufacturer	Number of test sites	Actuator	Force range	Force profile	Thermocycling	Integrated force sensor
ACTA	ACTA three body wear machine Willytec	12	Spring weight	0-50 N	linear	No	No
CoCoM	Zurich University	6	Solenoid	-	sawtooth	Yes	No
Alabama	Alabama University	4	Spring	84	-	Yes	No
MTS	MTS Minneapolis	1	Hydraulic	4-50 N	Haversin waveform	-	Yes
OHSU	Proto-tech Portland	2-4	Solenoid	0-100 N	Sine wave	No	No
Willytec	Willytec	8	Weight	1-11 kg	Sine wave with impulse	Yes/No	No

Table 5. Comparison of different operating parameters of existing oral and wear simulators (Heintze 2006) [78].

5.1.2. pH

Hydrolytic degradation is the most significant aging process that dental materials undergo inside the oral cavity. Hydrolysis is a biomolecular reaction, which comprises water and the functional group possessing the labile bond; this depends on the type of chemical bond, pH, copolymer composition and water uptake. pH can affect degradation rate through catalysis, especially with hydrophilic monomers [82].

It was found that the dissolution of enamel due to acid attack leads to surface roughening of about 0.4 μm . In his work, Frasisconi showed that after being subjected to pH erosive challenge, glass ionomer cements showed the highest microhardness losses, followed by resin composites and amalgam, probably because of the dissolution of the siliceous hydrogel layer. A marked decrease in the surface hardness of resin composite was also noted. Moreover, the dental literature shows that acidic media results in higher wear of dental enamel when compared to restorative materials [83].

Chemical degradation starts by absorption of water that diffuses internally through the resin matrix, pores and filler interfaces, leading to softening and hydrolytic degradation. The corroded surface is then worn under load, exposing a fresh surface and the cycle is restarted. Clinically, this would lead to loss of contour, surface roughness and plaque retention by the restoration [84].

Ortengren et al. found that pH and time facilitated the elution from resin composite material, especially methacrylate, TEGDMA and hydroquinone monomethylether [84]. Other studies shown that acidic solutions increased the diffusion coefficient, water sorption and solubility for some composites [85,86]. Other group of studies showed that an alkaline pH can have more effect on composite restorations, and that even a barium

oxide glass filler can produce a pH around 9.3 affecting the water stability of the resin-to-filler bond at the silane interface [87].

5.1.3. Bacteria

Over 100 trillion microbial cells inhabit the human body, living in symbiosis with the host.

In the oral cavity, there are more than 700 bacterial species, including at least 11 bacterial phyla and 70 genera. Individuals that practice oral hygiene have, from one thousand to one hundred thousand bacteria living on each tooth surface, whereas those who do not regularly practice dental hygiene can have between 100 million and 1 billion bacteria on each tooth surface. Among these, many are in fact beneficial in preventing diseases [88], while some have been implicated in oral diseases such as caries and periodontitis, which are among the most common bacterial infections in humans.

It is mostly because of an imbalance between host-microbiota interactions, a dysbiotic community or a deregulated immune response that results in a disease or harmful situation [89].

Several bacterial species have been isolated from biofilms associated with caries lesions. Dental caries are usually associated with increased proportion of streptococci such as *Streptococcus mutans* (*S. mutans*), *Streptococcus sobrinus*, *Streptococcus mitis* and *Streptococcus oralis*, also lactobacilli and bifidobacteria.

S. mutans is considered the main etiological agent in caries development. This is related to its high adhesion capability, and acidogenicity, both on teeth and oral restorative materials.

It has been established that resin-based composites are particularly susceptible to the development of cariogenic biofilms [90].

Among the factors that influence the bacterial biofilm formation over the surface of resin-based composite restorations are: surface roughness, surface free energy, surface chemistry, surface topography and surface chemistry and polishing treatment.

5.1.4. Mandibular movements

Mandibular movements comprise a complex series of interrelated six-dimensional rotational and translational activities. Although both temporomandibular joints cannot function entirely independently of each other, they also rarely function with identical simultaneous movements.

Types of Movements

Two types of movements occur in the temporomandibular joint: rotational and translational [79].

Rotational Movement: Rotation is the process of turning around an axis. Rotational movement of the mandible can occur in all three reference planes: horizontal, frontal and sagittal.

Translational Movement: Occurs when the mandible moves forward, as in protrusion. The teeth, condyles, and rami all move in the same direction and to the same degree.

Human chewing cycles are usually reproduced with two servo-hydraulic actuators which are easy to make and to program. Recent simulators based on hexapod design,

equipped with six degrees of freedom, can faithfully reproduce mandibular kinematics and simulate all chewing movements after programming [91,92,93].

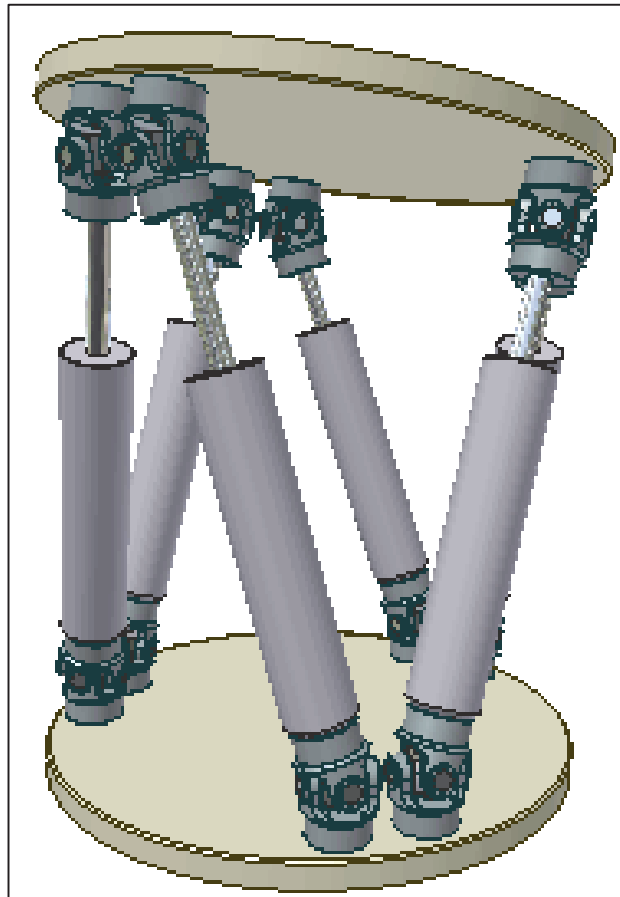


Figure 13: hexapod design used for 6 degrees freedom simulation [92].

5.2. Biocompatibility and Material release in the oral cavity

Biocompatibility of a material is defined as its ability to elicit an appropriate biological response in a given application in the body. The material should not be harmful to the pulp and soft tissues, should not contain toxic substances or produce allergic responses, and should not be carcinogenic [2,94,95].

Unfortunately, determining the biocompatibility of dental material is an extremely complex task. In fact, no test can be done to ensure that there will be no adverse effects on all the subjects who use this substance.

The biocompatibility of a material does not depend, only on its chemical or physical nature, nor only on the duration of exposure and the type of tissue exposed, but on the substances eluted from it as well.

Biocompatibility tests are classified on three levels:

- Group I: Primary tests (or in vitro tests)

The material or its extract is put in contact directly or indirectly with some biological system outside the organism. They are relatively fast and inexpensive, moreover, they are easily standardized. The main problem is their weak relevance to clinical outcome.

- Group II: Secondary tests (animal tests)

These involve the placement of the test material in an organism. A variety of test animals are available, depending on the relevant tissue involved. Though they show more clearly the biological response of an intact biological system to the

material in question, they are extremely costly, time consuming and difficult to control. Finally animal testing is essential before clinical use.

- Group III : Usage tests (clinically in animals and humans)

When performed in humans, it is called clinical trial. Animal choice in these tests is much limited, because the animal used should be similar anatomically and physiologically to humans. Conversely most clinical trials have limitations; most often only subjective symptoms are retrieved with no microscopic or histologic evaluation of the results. Moreover usage tests are extremely costly, difficult to control and usually involve legal and confidentiality issues.

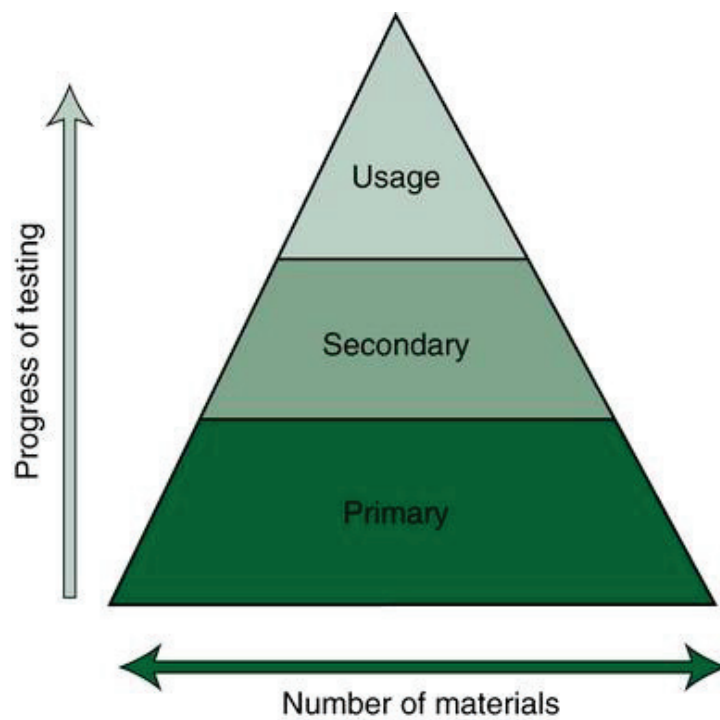


Figure 14 : Biocompatibility testing pyramid (Wataha 2001) [96]

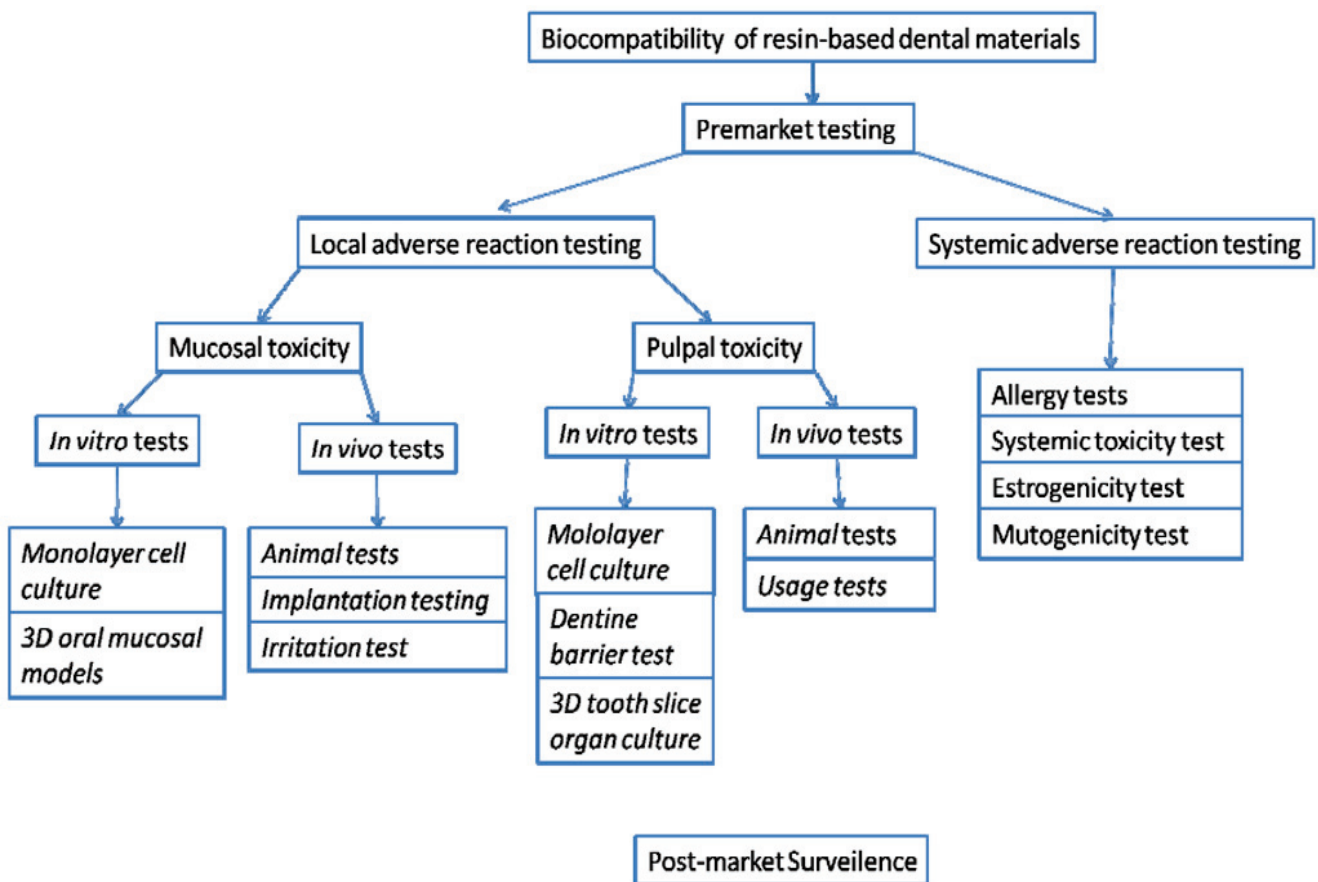


Figure 15. Biocompatibility testing (Wataha 2001) [96].

Even though standardization of biocompatibility tests is essential to ensure the safety of dental products, several ISO standards are involved with dental materials evaluation. Examples are ISO 10933 and ISO 7405 which include the dental barrier test. On the other hand, standardization is limited by the rapid advancement of new materials and technologies.

Moreover, the acceptability thresholds are difficult to attain among manufacturers, academics and the public [94,95,97,98].

5.2.1. Bisphenol A

Even though the level of Bisphenol A released from dental resins seem much lower than the maximum acceptable dose, lately, much public concern was stimulated about the use of Bisphenol A in dental treatment.

Moreover, a recent study revealed increased material elution with the use of bulk composite, mostly due to inadequate polymerization of the deep levels of the thicker restoration increments [99].

Bisphenol A is known to affect different organs and physiologic functions, such as reproduction and sex determinism, brain development, and behavior. It may also increase breast cancer risk and lead to obesity.

The actual mechanism of Bisphenol A toxicity is still the subject of research, even though specific target genes associated with specific disease states have been identified in differentiated cell types in epidemiologic surveys [100].

The most important aspect of this toxicity is being considered as an endocrine disrupting chemical (EDC), and would then have Nonmonotonic Dose Responses, meaning that a dose which is much lower than that currently accepted by regulatory authorities as the No Observed Adverse Effect Level (NOAEL) would result in actual toxicity.

5.2.2. Other material release

Other materials are also released from dental restorations. For example, Nickel, which is used in removable partial dentures and some orthodontic appliances, is known to be the most allergenic metal known.

Beryllium is also used in dental alloys and beryllium-containing particles that are inhaled and reach the alveoli of the lungs may cause a chronic inflammatory condition called berylliosis.

Another prominent example is the long lasting controversy about the toxicity of amalgam and its mercury content [2].

An investigation of the scientific literature on the subject of potential toxicity of Bisphenol A and other substances in dental restorative resins, BPA based monomers being the most commonly used in the composition of restorative composites is the aim of the following paper.

Dental composites and Bisphenol A - Potential toxicity and ways to reduce it - Systemic review.



Potential Toxicity of Bisphenol A and Other Related Substances in Dental Restorative Resins

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ABSTRACT

Background: Dental composites are now the restoration of choice for most practitioners, due to their ease of handling and superior esthetics. At the same time the actual risk of use of Bisphenol A (BPA) and BPA derivatives in dental composites, their degree of elution, and BPA exposure levels remain controversial.

Objective: The objective of this review is to consider these claims, and to examine the literature on different methods to counter this potential toxicity. A search of the literature was conducted for studies focusing on BPA toxicity in dentistry, up to 5 years (January 2009). The preferred reporting items for systematic reviews were used as guideline method.

Methods: Electronic databases: PubMed and ScienceDirect were used. In addition, studies were identified by searching of selected journals. The results were examined for relevance. Criteria for selection included were toxicity assessment, chemical composition, type of cure and duration, and materials affecting toxicity.

Conclusion: As a conclusion, the potential toxicity of BPA and BPA derivatives in dental composites still needs to be confirmed. The review focused on new methods for BPA toxicity assessment, the effect of additions like antioxidants and antimicrobials, together with new developments in dental composite chemical formulations, especially the monomer chemistry, and their effect in counteracting BPA toxicity *in vitro*. Overall, a more complete and comprehensive analysis is required, further studies are needed to fully acknowledge these conclusions *in vivo* and clinical situation.

Keywords: Bisphenol A, Toxicity, Restorative materials, Dental

INTRODUCTION

Dental composites are actually the restoration of choice for most practitioners, due to their ease of handling and superior esthetics, and although their physical



and mechanical properties have much improved, most of their components whether non-polymerized monomers, additives and fillers, have been shown to have definite toxicity, the most concerns in literature have been the release of Bisphenol A (BPA).^[1] Needless to say that BPA exists already in various products used on daily basis, most significantly food containers; thus BPA concentration in saliva was found to be at least 0.07 ng/ml even before placement of resinous restorations of any kind.^[2,3]

BPA itself is not used in dental composite resins, but it may be only found in dental resins as an impurity,^[4,5] the main reason is that moisture from saliva and salivary enzymes (esterase) inhibit its polymerization by causing hydrolysis of the second hydroxyl groups. On the other hand BPA derivatives are frequently used in dental resins; these derivatives are liquid monomers that polymerize into a solid after either chemical or light curing.^[4] Among the mostly used are BPA glycidyl dimethacrylate (bis-GMA), BPA dimethacrylate (bis-DMA) and BPA diglycidylether (BADGE) as well as BPA ethoxylate dimethacrylate (bis-EMA) and urethane-modified bis-GMA. The BPA derivative bis-DMA was found to hydrolyze into BPA; thus responsible for the Bisphenol A detected in extracts from certain composites, on the other hand, no studies have addressed the potential for other BPA derivatives used in dental materials to hydrolyze to BPA.^[1,4,6] Other potentially toxic methacrylate monomers including HEMA and TEGDMA are commonly found in the composition of resin based dental materials.^[7]

During the polymerization reactions, large chains are formed and crosslinking takes place, reducing the mobility of the monomers, and leading to entrapment of unreacted residual monomers. It was found that the degree of conversion varies depending on the resin-based material between 50 and 70%,^[8-11] the trapped uncured monomers can then leach out into the oral cavity. More leaching can take place due to further degradation of the resin composite, this usually takes place through mechanical swelling, water sorption and enzymatic degradation, as the degradation increase it results in more porosity and consequently more monomer leaching.^[8]

The potential toxicity of these leached monomers can be either locally on the pulp and surrounding tissues, or systemic on the body as a whole. On the cellular level it was found that restorative materials containing resin are cytotoxic, especially after mixing, in the dental field this may lead to pulp irritation and gingival inflammation and retraction. Moreover, according to some reports elution could continue for up to 1 year after the initial polymerization.^[12] Other studies discussed the probable synergistic effect of different monomers in increasing their toxicity toward pulp cells.^[13] Another effect of monomer release from resin restorations could be in the form of favoring bacterial proliferation, especially the microorganisms implicated in caries formation, as with MA and TEGDMA, therefore, contributing to the development of secondary caries.^[14]

The systemic toxicity presents a more complicated problem, multiple toxicological and epidemiologic studies have shown the noxious effects of BPA, for example epidemiological studies found that children with resin-based composites had worse psychosocial outcomes on some measures of neurodevelopment 5 years after placement,^[15] and that Prenatal exposure to BPA was associated with chromosomal defects, other group of studies claimed that it is implicated in local and systemic allergic-related reactions.^[16] The French National Agency for Food Safety and Occupational and Environmental Health (ANSES) concluded that according to available scientific literature, BPA has recognized impacts on animal health and suspected impact on human health and recommended reducing the exposition to BPA for humans by substituting it with other components. Protective measures should specifically target infants and young children as well as pregnant and nursing women.^[1,17]

BPA has been shown to contribute to cancer development and progression through interaction with estrogen receptors α and β , leading to changes in cell proliferation, apoptosis, or migration. BPA has also been shown to be involved in multiple oncogenic signaling pathways.^[18] BPA has also been shown to disrupt the thyroid hormone system at the gene expression level.^[19]



From another perspective, the question whether to consider BPA and other monomers used in dental materials as a toxic substance or as endocrine disrupting chemicals with Nonmonotonic Dose Responses^[20,21] would have a huge impact on the relevance to human health of the “low-dose effects”, which can sometimes be 1000 times lower than the dose levels currently accepted by regulatory authorities as the No Observed Adverse Effect Level (NOAEL).

Under the EU chemicals policy REACH (Registration, Evaluation, Authorization and Restriction of Chemicals), manufacturers and importers are required to register their substances with the European Chemicals Agency (ECHA). For a substance manufactured or imported in a quantity ≥ 1 ton/year manufacturers must compile a Technical Dossier on the physico-chemical, human health and environmental properties of that substance. For a substance manufactured or imported in a quantity ≥ 10 tons/year, like BPA, a Chemical Safety Assessment must be carried out and documented in a Chemical Safety Report (CSR).

The leading manufacturers of BPA formed the BPA REACH Consortium and developed the registration dossier for BPA, based on the EU BPA Risk Assessment Report 2003, and updated 2008.

The BPA dossier was submitted to ECHA in August 2010 under the REACH regulation “substances of very high concern” (SVHC) may be subject to “Authorization” in order to ensure that the risks from these substances are properly controlled.

In May 2010, The Polycarbonate/Bisphenol An industry group of Plastics Europe examined authorization under REACH published a report and concluded that BPA does not meet the criteria of an SVHC under REACH. Following the regulation, authorization is not required for “intermediates”; that is substances that are converted during chemical processing. BPA is predominantly used as an intermediate in the manufacture of polycarbonate plastic and epoxy resin.

In November 2013, ECHA announced to request further data on BPA in the area of skin contact and environmental exposure. The BPA REACH Consortium accepted the request and is currently working to provide the additional input within the timeline by the end of 2015. In March 2014, the Risk Assessment Committee supported an opinion proposing BPA as reprotoxic 1B. If adopted, enforcement will most likely not apply before early 2017. The classification will not affect compliance with food legislation: BPA can continue to be used in food contact applications for consumers.

Another part of the dilemma lies in the fact that the predetermined endpoints used in standard toxicological testing, have nothing to do with the modern technological methods that take in account endocrine disrupting compounds; that disrupt crucial cell-signaling pathways, and that cannot be detected by classical endpoint assays.^[20,22] The rodent studies reporting low-dose effects of BPA are claimed as irrelevant for the assessment of risks to human health by some groups because of different toxico kinetics between species.^[22] More accurate and sensitive methods are thus required to provide the necessary scientific data required by regulators to assess the safety features and the risk assessment of BPA.

With the evolution of new types of composite dental restorations, and the subsequent continued development of new resins and monomers, the main challenge that presents itself is to resolve the problems of shrinkage and secondary caries and at the same time preserve a good level of biocompatibility with dental tissues. In this work-several studies dealing with BPA toxicity in dentistry were reviewed; we focused on the best methods developed to reduce BPA toxicity, whether through new ways to assess toxicity, development of new chemical compositions, the effect of type of cure and time, and finally the effect of various additions on increasing or decreasing the level of toxicity. The objective of this work is to give an overall view of the current situation, and to examine potential

pathways that were tested to counter the probable harmful effects of PBA and other related substances in dental restorative resins.

DATA SOURCES

A search was conducted for articles dealing with BPA toxicity and other related substances in dentistry dating for 5 years (January 2009), our study included all remarkable articles published between 2009 and 2014, and consequently, the study has enough power to provide relevant results. The key words used were “Bisphenol” AND “toxicity” AND “dental composite”. The electronic databases searched were: PubMed and Science direct, in addition, more articles were included through manual search of selected journals.

STUDY SELECTION

The number of studies obtained was 211, duplicates were removed, and searches were limited to publications in English (187). At each stage, the search strategy was validated by the authors. The abstracts of these articles were examined and of those identified, articles that did not discuss the toxicity of dental composites, or provide background information (review articles) were excluded (Figure. 01). The reference lists of these articles were searched to identify any other articles relevant to the subject. Articles found were classified according to the main themes: Toxicity assessment. Chemical composition. Type of cure and time. and Materials affecting toxicity.

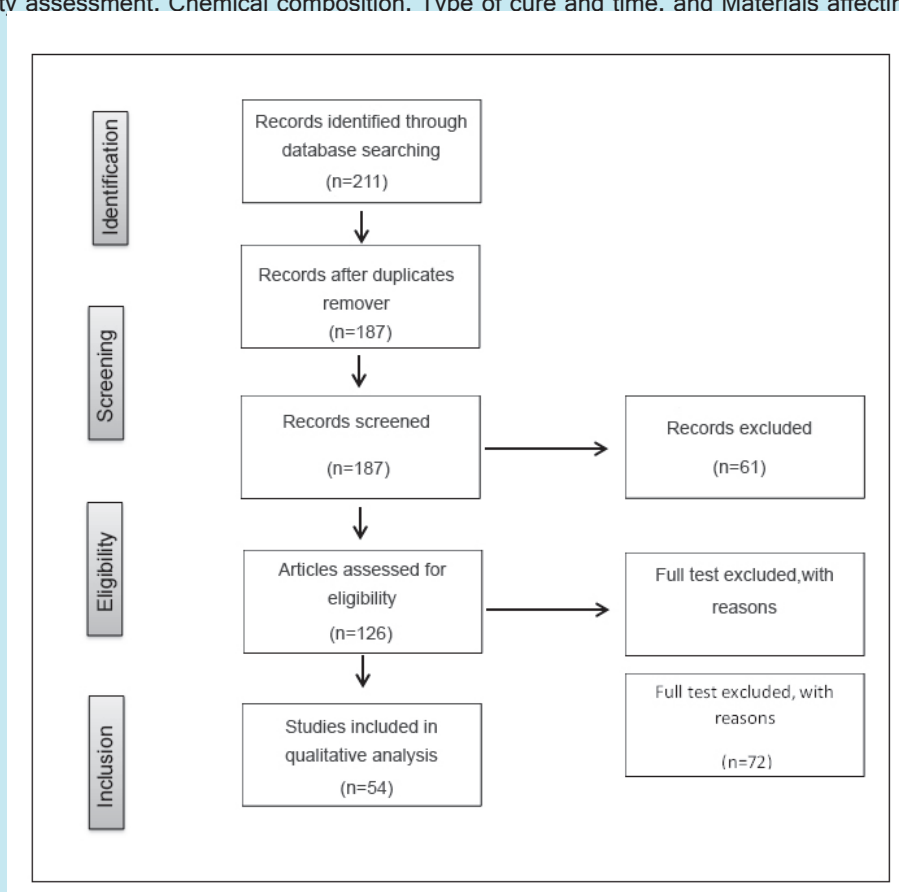


Figure. 01 *Flow of information through the different phases of the search strategy*



TOXICITY ASSESSMENT

Assessment of dental material toxicity can be either through measurement of toxic components elution from the restorative material, through direct measurement of its toxic potential of the cells/tissues, or its ability to reach the pulp, gingival tissues and/or the systemic circulation.

Several methods exist for components release quantification from dental resin-based materials. The gravimetric measurement of samples before and after extraction of components is the least expensive method.^[8] On the other hand to determine the individual release of separate compounds, sophisticated analytical methods should be used, (GC) gas chromatography is more suitable for analysis of low molecular weight compounds, whereas (HPLC) High performance liquid chromatography and liquid chromatography (LC) are more applicable for analysis of high molecular weight compounds. Mass spectrometry (MS) gives information on the molecular mass of the compound and allows the detection and quantification of compounds based on their ionization, and computation of the mass-to-charge ratio. It can be used as a sophisticated detector in conjunction with one of the above mentioned analyses and allows detection of degradation products.^[8] Infrared spectroscopy and Fourier transform infrared spectroscopy are nowadays regarded as outdated, mainly because the interpretation of the spectrogram is difficult and not molecule-specific. It is worth noting that a great diversity exists between studies using these methods, mostly due to lack of standardization of the test methodology and the test set-up.^[11,23,24]

The actual toxicity assessment *in vitro* on living cells is performed through cell culture. Franz and coworkers found that cell culture toxicity data are highly model dependent^[25] and that different ratios of specimen size to cell layer surface/volume of cell culture medium produced differences in cytotoxicity when the same material was tested. Their recommendations were that an internationally standardized protocol for specimen production is required to obtain comparable results between studies.^[26]

Other investigators tried a different approach through studying the mobile secondary free radicals release by dental composites stored in hydrophilic media,^[27] on the other hand Urcan et al.^[28] used the xCELLigence system, a real-time and continuous monitoring system that allows label-free assessment of cell proliferation, viability and cytotoxicity to investigate cytotoxicity of the most common monomers/comonomers in dental resin composites.^[28] others still analyzed the degradation of a model dental composite leading eventually to the release phenomenon.^[29,30]

For screening new dental monomers, Pérez-Garrido et al.^[31] used a quantitative structure-activity correlation (QSAR) model that could distinguish mutagenic from non-mutagenic species in new developed dental materials, and identify the molecular features that most contribute to the mutagenic effects of these chemicals, so that their presence should be avoided in the design of new monomers.

New techniques like focused ultrasonic solid-liquid extraction or electron paramagnetic resonance spectroscopy are evolved to monitor BPA traces or hydroxyl radical release.^[27,32] Asimakopoulos et al.^[33] in their review discussed the problems related to control of contamination during sampling and handling of specimens, as well as data analysis and interpretation during the process of BPA biomonitoring, stressing the importance of implementing strict rules during sampling and handling of specimens before drawing final conclusions.

Researchers are in a continuous request for new methods for the toxicity assessment; some authors focused on the method sensitivity, others focused more on rapid or easy methods. Recently, Willershausen and co-workers used the Alamar blue assay to evaluate the *in vitro* biocompatibility of a bioceramic root end material.^[34] More recently, Attik and collaborators described a sensitive method for a dental composite cytotoxicity evaluation using



time lapse confocal imaging.³⁵time lapse confocal imaging.^[35]

New approaches are being explored in order to obtain reliable information about material toxicity, especially with the advent of new composite resin materials, and the integration of monomers with new chemical formulas each day.

Chemical composition

BPA derivatives like BisGMA, BisEMA, EBPADMA and/or UDMA are mostly used for the development of dental composites, they allow for the development of restorative composites with better mechanical properties, rapid polymerization and low shrinkage. However, these monomers generally result in low methacrylate conversion, leaving significant amounts of unreacted monomers that would elute from the restoration over time. TEGDMA (not a BPA derivative) is mostly used in order to reduce viscosity, enabling larger percentage of filler to be incorporated into the resin matrix, for acquiring dental restorative composite with better mechanical properties.^[36]

Miao et al.^[37] found that in comparison to Bis-GMA/TEGDMA resin system, the composites containing EBPADMA exhibit greater compression strength, higher depth of cure, higher light transmission and lower volume shrinkage.³⁷ Moreover several studies found urethane-di, -tri, and -tetramethacrylates products had lower polymerization shrinkage and higher flexural strength properties compared to bis-GMA-based resins. Derivatives of urethane dimethacrylate are able to increase the molecular weight, reduce water sorption, and/or increase mechanical properties by incorporating aromatic or aliphatic groups.^[38] Yet on the other hand the urethane-based polymers absorb significantly more water than the aromatic-based materials, leading to passive and enzymatic hydrolytic degradation of the polymer matrix ^[39] which not only change mechanical properties of urethane- based polymers, but also initiate the release of unbound monomers and degradation by-products. UDMA may also induce a broad spectrum of cyto- and genotoxic effects in human cells.^[40,41]

In recent times, with the arouse of public concerns about bisphenol toxicity, monomer development efforts shifted more toward bisphenol-A alternatives: Eliades et al.^[42] studied the formulation of benzoic ring-free, high molecular weight molecules to replace Bis-GMA. The toxicological and inflammatory potential of newly developed materials as silorane-based composites, sometimes present contradictory results. Wellner and co-workers^[43] studied the release of 24 cytokines from human leukocytes and found that Filtek™ Silorane stimulates the leukocytes to a higher release of cytokines when compared to TetricEvo Flow, Indicating a higher sensitization potential for Silorane. At the same time Krifka et al.^[44] found silorane-based composite resin to have no direct cytotoxicity, and only a very slight increase in ROS production.

Another example is the use of bile acids as starting materials to form multimethacrylate monomers, they showed reduced volume shrinkage and promising mechanical properties; but, exhibited extremely high viscosities (higher than BisGMA).^[36] Polyhedral oligomeric silsesquioxane methacrylates (POSS-MA) were found to improve the mechanical properties even in small amounts. In another report dimethacrylates based on cycloaliphatic epoxides showed kinetics and mechanical properties comparable with those of BisGMA.^[45] Another alternative to BisGMA were Methacrylated beta-cyclodextrin derivatives found to exhibit flexural strength and volume shrinkage comparable with those of BisGMA/TEGDMA.^[46] Previous work has also demonstrated that Toluene 2,4-diisocyanate (TDI) modified resin matrices were found to trap toxic resin monomers in the complicated resin structure leading to lesser cytotoxicity,^[47] Poplawski et al. ^[48] examined the cytotoxic and genotoxic effects of glycidyl methacrylate (GMA), they warned against a potential long lasting exposure to this compound due to its broad spectrum of GMA genotoxicity, including DNA double-strand breaks.^[48] Zanchi ^[49] studied an experimental HEMA-free three-step adhesive system Bis-EMAs, (Bis-EMAs) he found that they have low ability to penetrate into wet demineralized



dentin mainly because of their hydrophobicity. He postulated that it would be less toxic, but recommended cytotoxicity tests and long-term evaluation before arriving at that conclusion.

Various studies classified monomers according to their degree of toxicity; D'Antò et al. found hydrophilic monomers such as HEMA less cytotoxic than the more hydrophobic monomers Bis-GMA, UDMA and TEGDMA,^[50] while other studies found BisG.MA as the most cytotoxic, followed by UDMA, HEMA and MMA,^[51] Lin in et al.^[52] found that while increasing the BisGMA content increased the mechanical properties, it reduced the conversion attained. And since its ultimate conversion is lower, it is therefore more likely to have a toxic effect, another group of studies found that BisGMA and UDMA were released from composite resins in much lower levels than toxic concentrations.^[53]

Moreover several factors control the degree of toxicity of resin materials used in dentistry; Anand et al.^[54] found that unreacted double bonds in dental composites influence biocompatibility rather than its degree of conversion, and that the magnitude of double bonds depends on the polymerization and chemical composition, this affect biocompatibility especially if they possess lipophilic properties as previously pointed out by Durner et al.^[8] who correlated the Lipophilicity of substances with their genotoxicity. Molecular weight also plays an important role concerning toxicity and pulp reactions that control monomer diffusion through dentin since the diffusion coefficient is inversely related to molecular weight.

The degree of elution of the released components depend on various factors, mainly the extent of the polymerization reaction (the 40 seconds usually used for the polymerization of resin composites seem insufficient to prevent a high release of monomers),^[55] another factor is the size of the released components, as smaller molecules are eluted at a faster rate, finally the chemistry of the solvent and the action of pH on the extraction of components from polymerized and unpolymerized composite. Moreover, Durner et al. ^[8] found that the degree of conversion of urethane dimethacrylate and bisphenol-Aglycidyl dimethacrylate varies from different manufacturers. In another report, it was found that residual monomers and not breakdown products, eluted only in small quantities during the first 90 days, but in high quantities thereafter.^[56]

According to Lin et al.^[57] filler type/content has little effect on cell viability, while the degree of conversion, hydrophobicity and roughness had marked effect. Other components of resin based dental materials may indirectly contribute to the increase of toxicity

As an example; in a study by Curtis et al.^[58] it was found that larger surface area to volume ratio of the fillers present in the nanofilled materials as compared to conventional composite increased water uptake and resultant degradation of the filler/matrix interface. Another factor is the degree of dentin permeability; Porto and collaborators found that total-etch adhesives remove the smear layer and smear plugs, and widen the entry to the dentinal tubules, allowing for the permeation of solutes and solvents, thus increasing the risk of toxicity. Self-etching adhesives on the other hand do not completely remove the smear layer, and are considered safer because the resinous monomers cannot penetrate very deeply into the dentinal tubules.^[59]

In perspective, there is limited commercial use of the new dimethacrylate derivatives, since the improvement in overall properties as compared to the conventional BisGMA/TEGDMA based resins seems only moderate for the present time (Table. 1). It can be shown from the table that most commercial dental composites depend on their composition on the conventional BisGMA/TEGDMA formulation. Moreover it must be noted that while BPA and BPA derivatives are being put under scrutiny, studies show that new BPA free materials claimed to be safer sometimes are equally toxic, a clear example is TEGDMA, Several studies state that TEGDMA; one of the main compounds leaching from polymerized resins is highly cytotoxic and moderately genotoxic.^[53,60-62] In conclusion while primary reports for new materials and additions seem mostly encouraging; sufficient *in vitro* and *in vivo* tests



Material	Manufacturer/batch	Resin matrix
Adamant® Cavifil	Vivadent/E 52179	BisGMA, UDMA, TEGMA
Arabesk Top	VOCO/94816	BisGMA, UDMA, TEGDMA
Ariston pHc	Vivadent/B21705	BisGMA, UDMA, TEGDMA
Beautifil	Shofu/050143	BisGMA, TEGDMA
Brillant	Coltene/IA350	BisGMA, BisEMA, TEGDMA
Charisma®	Heraeus Kulzer/10023	BisGMA, TEGDMA
Clearfil™ ST	Kuraray/00002A	BisGMA, TEGDMA
Esthet X®	BisGMA, BisEMA, TEGDMA	Dentsply/0112121
Filtek™ Silorane	3M ESPE/A-094	3,4-Epoxy cyclohexylethyl cyclo-polymethylsiloxane
Bis-3,4-epoxycyclohexylethyl-phenylmethylsilane	Clearfil Majesty	Bis-GMA, TEGDMA
Herculite® XRV	Kerr/909065	BisGMA, TEGDMA
Clearfil Majesty™	Kuraray	Bis-GMA, TEGDMA
Point 4™	Kerr/102A48	BisGMA, TEGDMA
Prodigy	BisGMA, TEGDMA, EBADM, UV-9	Kerr/812898
Synergy® Duo Shade	Coltene/IG079	BisGMA, BisEMA, TEGDMA
Tetric®	Ivoclar-Vivadent /C16884	BisGMA; UDMA, TEGDMA
Tetric® Ceram	Ivoclar-Vivadent D00037	BisGMA, UDMA, TEGDMA
Venus®	Heraeus Kulzer/010022	BisGMA, TEGDMA
Z100™	3M ESPE/2EG	BisGMA, TEGMA
Filtek™ Z250	3M ESPE/20011016	BisGMA, UDMA, BisEMA

Table. 1 *Materials, manufacturer and chemical composition of the matrix*

Note: *Composition as supplied by the manufacturers*

On the other hand, the light-curing polymerization process with its different parameters whether light spectrum, power density, and polymerization time has a direct influence on the degree of polymerization of the composite, eventually leading to different releasing rates of BPA or unreacted BPA based monomer.^[65] Santini et al.^[66] found that polywave LEDs significantly improved both the degree of conversion of materials which contain TPO initiator. Miletic et al.^[67] in their comparison of polywave and monowave LED light-curing unit (LCU), found polywave LED LCU more efficient in curing TPO-containing materials compared to CQ-amine only or the combined TPO and CQ-amine system.

Sigusch and collaborators^[65] found that the concentration of the released monomers UDMA and BisGMA in the composite varies with the light energy used, they also found that the size of the filler has an influence on the transmission of the curing light by the material, they recommended the use of high power density units to decrease the release of toxic substances; and that composites and light curing units should be harmonized with one another for achieving maximal biocompatibility. Durner et al.^[8] studied the various factors controlling the elution of substances from polymerized and non-polymerized dental restorative materials, they found that, the wavelength, wavelength-distribution and intensity of the light source, as well as the distance light source – dental material, the hardening time, the compo-



sition and the color of the dental material are all factors that control their degree of toxicity.

Materials affecting toxicity

In the various studies reviewed in this work, some materials were added to the dental restorative with the intention of decreasing its toxicity, while other materials were added only to improve the mechanical properties, decrease the polymerization shrinkage of dental restoratives, or for antibacterial effect, never the less these materials had an important effect on the toxicity of dental resins, mostly indirectly through their action on the polymerization reaction and the amount of monomers eluted from the dental restorative materials.

Morgan et al.^[68] studied the potential protective effect of cinnamon against BPA-induced oxidative stress, on the other hand multiple studies examined the effect of adding antioxidants to counter the cytotoxicity of monomers. It was shown that N-acetyl cysteine (NAC) was able to prevent cell damage induced by all materials tested, incorporation of NAC into a dental resin has been shown to restore the suppressed viability and function of dental pulp cells or oral fibroblasts on the resin substrate to a biologically relevant degree, and to protect against BPA-induced cognitive dysfunctions and oxidative stress in rats.^[50,69-76] It was found that NAC acts not only as a direct oxidant scavenger, but also improves the intracellular glutathione systems compromised by oxidative stress.^[72] Other studies, considered the action of chitosan in decreasing the risk associated with the use of UDMA.^[40]

Not all additions have effect on the polymerization reaction, and the toxicity potential of resin materials; for example the incorporation of 2% proanthocyanidin into dental adhesives,^[77] or silane treatment modifications to increase the bond strength between composite resins and leucite reinforced feldspathic ceramic.^[78] Also the addition of synthetic antimicrobial polymers with their prolonged antimicrobial activities and non-toxic and non-irritant properties; had no effect on the polymerization reaction, compared with ordinary low molecular weight antibacterial agents, which present some disadvantages, such as toxicity and short-term antimicrobial ability.^[79] Several reports investigated the antibacterial and mechanically strong Nano composites incorporating a quaternary ammonium dimethacrylate (QADM), nanoparticles of silver (NAg), and nanoparticles of amorphous calcium phosphate (NACP). They found that Ag has a low toxicity and good biocompatibility with human cells, and a long term antibacterial effect due to sustained silver ion release.^[80-82]

On the other hand, some additions had an effect on the curing reaction; silicone-based additives can accelerate or retard the reaction, depending on the presence or absence of DMA.^[83] While liquid Rubber (LR) though increased the mechanical properties lowered the crosslink density of 50/50 Bis-GMA/TEGDMA resin.^[84] Musanje et al.^[85] studied the optimal concentration of photo initiators, whether CQ or ethyl-4-dimethylaminobenzoate, needed for maximum polymerization, and sufficient depth of cure, that would consequently affect the amount of available free monomers to be leached. Polydorou^[86] studied the effect of bleaching on the elution of monomers from two modern composite materials, the bleaching agents tested reduced the amount of some of the monomers released from the two composite materials, and he concluded that the contact of bleaching agents with composite materials may not have any detrimental results for the human health.

To our knowledge, most of the materials added to the dental restoratives in an effort to reduce its toxic effects whether directly or indirectly haven't yet been used in commercial composites, and albeit the initial success for these materials to combat toxicity; more studies are yet to be performed *in vivo*, and long lasting effects have yet to be considered.

Conclusions

The present systemic review aims to provide an exhaustive summary of current literature related to the potential cytotoxicity of BPA and other materials in dental restorative resins. A controversy still exists about the actual risk



of use of BPA and BPA derivatives in dental materials, and the degree of elution from dental restorations, and whether toxicity studies conducted on animals reflect their actual toxicity on humans.

It must be noted that while BPA and BPA derivatives are being put under scrutiny, studies show that new BPA free materials claimed to be safer; sometimes are equally toxic.

Any of the factors that affect the degree of convergence and the depth of cure of the dental resin, have a direct effect on the degree of BPA toxicity and the degree of eluted toxic monomers. Several studies have shown that the simple act of gargling water for 30 seconds after application of the dental sealant or composite or washing the surface for 30 seconds with an air-water syringe while suctioning fluids and debris from the mouth, has been shown to decrease salivary BPA levels to nearly baseline, and pumice on a cotton ball or in a rotating rubber dental prophylaxis cup was highly effective in removal of residual monomer and eliminating absorption of bis-DMA, bis-GMA, and TEGDMA.^[1,87]

The development of dimethacrylate derivatives of BPA has been an active research area. Recent developments regarding public perceptions of bisphenol toxicity may have a strong influence on steering future monomer development efforts toward BPA alternatives.

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The mastication bench device would be able to increase our understanding of the elution of BPA and other noxious material inside the oral cavity.

The ability of the mastication bench to collect the circulating artificial saliva or other solution used inside the device in order to be analyzed over different time intervals, would reveal important information about the quality and quantity of different eluted materials.

Moreover, it would help reveal the effect of mechanical, physical and chemical stresses on the phenomenon of increased elution.

5.3. Chewing bench

The chewing bench is an oral simulator that is supposed to integrate several parameters to reproduce the oral cavity environment to the maximum possible.

5.3.1. Design

The chewing bench uses a hexapod design (figure 16) to reproduce the mandibular movements. Six electric motors (brushless technology) are used to replicate mandibular kinematics using a special program to control the movements.

In the simulator dental arch, different types of teeth could be inserted including natural teeth, different restoration materials and prosthesis can be tested and compared at the same time.

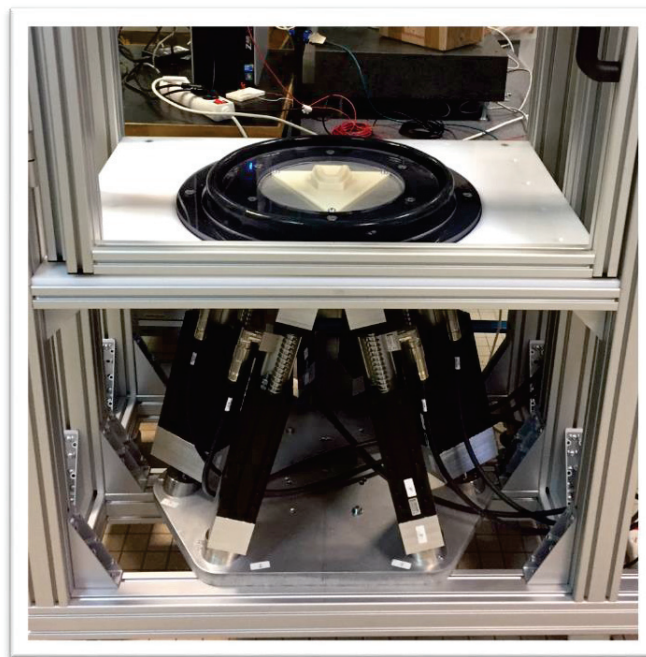


Figure 16 : design of the hexapod used in the Chewing Bench

5.3.2. Construction and Working Principle

The number of teeth in each arch will be 14. Simulation of the periodontal ligament apparatus will be used. Natural teeth or ceramic material could be used in the antagonist arch according to the load and wear patterns to be tested. The type of occlusion used will be balanced occlusion to maintain the same amount of force through various regions.

A water tight environment will be established using a silicon casing, while a fluid injection device will inject various types of artificial saliva with various temperatures and pH values.

Low-pressure sprays from four tubs containing artificial saliva and different solutions with varying pH values and at different temperatures ranging between -5 and +70°C; will be injected on the teeth. Electronic solenoids, and a water pump control the passage of liquids. Special sensors present inside the cavity of the chewing bench will monitor the exact temperature and pH of the injected solutions.

5.3.3. Configuration settings

The material chosen for the chewing bench is PolyetheretherKetone (PEEK), this material allows working with solutions with a pH ranging from 2 to 9. It corresponds to the pH range present in human food. In addition, this material stands temperatures in ranges between -5°C and +70°C, needed to produce thermal shocks of large amplitude to simulate the hottest as well as the coldest foods. The disadvantage is that PEEK is very rigid.

Cheeks made of silicone will be used for their flexibility, in order to easily reproduce the mastication trajectory. Different slurries with different compositions and foodstuffs could be used interchangeably with silicone, to test the different textures and consistencies of food material. These will mimic the action of different foods (thermal and chemical parameters) on dental restorative materials while using artificial saliva. The different parameters for the chewing bench are shown in the table 6.

Parameters Values

Oral cavity volume of	25cm ³
Maximal forces	500N
Trajectory	Mastication cycle
Solution	Normalized artificial saliva
Temperature of solutions	Between -5°C + 55°C (up to 70°C)
Ph of solutions	Between 2-9
Maximal number of cycles	5000

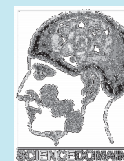
Table 6. Parameters of the chewing bench oral simulator

5.3.4. Loading force and direction

The three motors deliver a maximum force of approximately 700N, the velocity of each cycle descent speed would be 33 mm/s at 1.1 Hz as calculated. The time to simulate 20,000 cycles corresponds to one month of clinical service [101].

The following pilot study presents the idea of the chewing bench and its working principle, such as define in 2014-2015. We have to point out that the values presented were later modified to better represent the oral mandibular movements.

[Original Research Article: Development of a chewing simulator for testing dental materials: a pilot study.](#)



Development of a Chewing Simulator for Testing Dental Materials: A Pilot Study

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Authors' contributions

This work was carried out in collaboration between all authors. Authors CJ, HA and AS designed the device, wrote the protocol, and wrote the first draft of the manuscript and managed literature searches. Authors HA and BG managed the analyses of the study and literature searches. All authors read and approved the final manuscript.

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ABSTRACT

This paper presents the primary results of the development of a chewing bench prototype. The final aim of the device is to reproduce the human oral cavity environment in order to predict ageing of dental materials, it automatically imitates chewing cycles and reproduces the physical and chemical changes observed during meals. A dental articulator used for prosthodontics was chosen as an ideal structure for simulating human mandible kinematics; it has the advantage of being water tight compared to a hexapod device. Using Open Meca® software and three motors the extreme movements of the mandible were replicated. Four thermally controlled tubs were used to mimic physical and chemical changes observed during meal. The chewing bench provides a valuable tool for the evaluation of dental materials; its relevance is based on the simultaneous presence of all parameters that affect dental materials during function (mechanical, thermal and

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chemical). It is the first stage of work which will be validated a posteriori. This chewing bench would hopefully reduce the gap between in vitro performance and in vivo observation and serve as a benchmark for existing materials and as a device for testing new ones.

Keywords: Mastication; dental simulator; modeling; mandible movements.

1. INTRODUCTION

The past decade has witnessed rapid advancement in various fields of dental materials, in particular restorative ones. This advancement has led to the emergence of new products on the market and improvement in existing products [1].

Despite this, there is often a gap between in vitro performance of dental materials and clinical observations. This gap is related to the oral cavity environment; responsible for the ageing of dental materials [2,3,4,5].

Generally, failures in dentistry have multifactor origins, and wear and ageing of dental materials inside the oral cavity are related to different phenomena that vary among individuals and even in time for a given individual, these include; chewing forces, mandibular movements as well as physical and chemical changes during meals [2,4,6,7,8,9,10,11].

Recent research about the failure of dental materials sets more focus on enzymatic activity and slow degradation, and while secondary caries and fracture are considered the main causes for limiting the longevity of restorations, wear remains to be a significant mode of failure [12]. On the other hand, the ability to reproduce the complex oral environment even to a limited extent, continue to provide valuable information, about different materials ability to maintain their properties during function. Unfortunately other methods; whether animal studies or clinical testing, that could provide such information before hand, are expensive, time consuming and complicated. Furthermore, human mastication can't be reproduced by other species, since teeth shapes, mandibular kinematics and type of food are different [1,13].

In recent years, several wear simulators have tried to reproduce the oral environment for testing dental materials as closely as possible to in vivo conditions [11]. Starting with early trials as that of DeLong and Douglas [14] that reproduced human chewing cycles with two servo-hydraulic

actuators [1,13,15] and up to recent simulators based on hexapod design; and equipped with six degrees of freedom, and that could faithfully reproduce mandibular kinematics and simulate all chewing movements after programming [1,16].

One of the main drawbacks of such devices is that they allow for single parameter investigations [4], typically chewing forces; while other parameters are not considered despite their potential role in material aging [4,6]. Newer devices claim to enable chemical, thermal and mechanical testing at the same time; the most recent is the "Rub & Roll" device by Ruben et al., while for the present time; none of these devices include all parameters in a systematic manner [17,18].

Multiple reviews discussed the results obtained by different simulators available; they also provided a critic for the information obtained with such devices; in an in depth study of the various wear methods used, Heintze et al. found little correlation with clinical results, when comparing a large number of composite resin materials, yet he still concluded that these methods are important for categorizing various types of material used, mostly for new materials that are introduced in the market [19].

In light of the above observations, a simulator is being developed (referred to as a Chewing Bench) that allows for the evaluation of dental material. This device will be capable of simulating the oral cavity and most of its parameters (mechanical, chemical and thermal), predict ageing of dental restorative materials and to imitate human chewing cycles and to make the materials undergo physical and chemical changes observed during meals, as well as clinical or pathological conditions.

The aim of this study is to present a prototype which may be able to reproduce the oral environment and all parameters (trajectory, chewing force, chemical or thermal changes during meals) that contribute to wear in order to predict the ageing of dental materials (Fig. 1).

2. MATERIALS AND METHODS

2.1 Design

The chewing bench consists of an artificial jaw with three degrees of freedom during the chewing cycle, based on the semi-adaptable dental articulator (Fag, Quickmaster®, Fig. 2) designed for realizing dental prosthodontics. It produces movements which are reversed compared to humans because its maxilla is movable and its mandible is still [20]. The scanning of the dental articulator (Fag) movement was performed using Romer jib SIGMA 2022 equipped with a Laser G-SCAN camera. This method allowed observing the triangulation deformations made by the articulator. Point clouds were obtained which were processed using Rapid form (Rapid Form; 2004) and Catia (Catia, VR18) software.

Open Meca software (Open Meca, 2007) was used to reproduce extreme mandibular movements. The software allows us to produce a three-dimensional mechanical linkage (Fig. 3). Three programmable electric motors with brushless technology were used (Fig. 4); two of the motors move from side to side and the third motor rotates, imitating the mandibular translation and rotation movements that begin in the temporomandibular joint.

In order to simulate the teeth, existing database can be used to reproduce different teeth (canine, incisive, premolar and molar) which then can be inserted into corresponding holes on the dental arch in order to be involved in the masticatory process. Natural teeth can replace any tooth so reproduced. This method allows testing several teeth at the same time, comparing different restoration materials simultaneously, and changing the antagonist material used.

2.2 Construction and Working Principle

The teeth are placed in corresponding holes in a dental arch form, whether natural or artificial teeth issued from anatomical database, according to the test performed. The number of samples corresponds to the number of teeth in the arch; that is 14 for each of the two arches. Periodontal ligament simulation could be easily acquired through the use of the rubber sockets as those present in the Zurich wear simulator [15].

Materials to be tested will be inserted into the cavities prepared in the teeth, in case of restorative materials, or shaped into teeth form in case of ceramics. The antagonist arch can be changed according to need whether with natural teeth or ceramic material. The dynamic occlusion pattern selected is balanced occlusion to maintain force equilibrium over all the regions during chewing simulation. The design allows for a water tight environment, needed for the fluid injection device integrated in the mastication bench; responsible for varying the medium's pH and temperature.

A thermo-chemical device was developed especially for the chewing bench. Four tubs containing artificial saliva and different solutions with varying pH values and at different temperatures ranging between -5 and +70°C; low-pressure sprays of these solutions injected on the teeth with the possibility of making very fast changes of temperature (Fig. 5). The passage of liquids is controlled by electronic solenoids, and a water pump. The exact temperature and pH of the injected solutions will be monitored by special sensors present inside the cavity of the chewing bench.

2.3 Configuration Settings

2.3.1 Loading force and direction

The three motors allow reaching a maximum force of approximately 500N which corresponds to a majority of individuals for a full arch (as compared to a maximum of 700N) [21,22,23], and is greater than current in vitro tests which reach a force of 150N situated at the level of the first molar [13].

According to the trajectory and motors used, the velocity of each cycle descent speed would be 33 mm/s at 1.1 Hz as calculated [15], and the time to simulate 20,000 cycles correspond to one month of clinical service [24].

2.3.2 Parameters of the chewing bench

Other parameters and specifications of the chewing bench are shown in (Table 1).

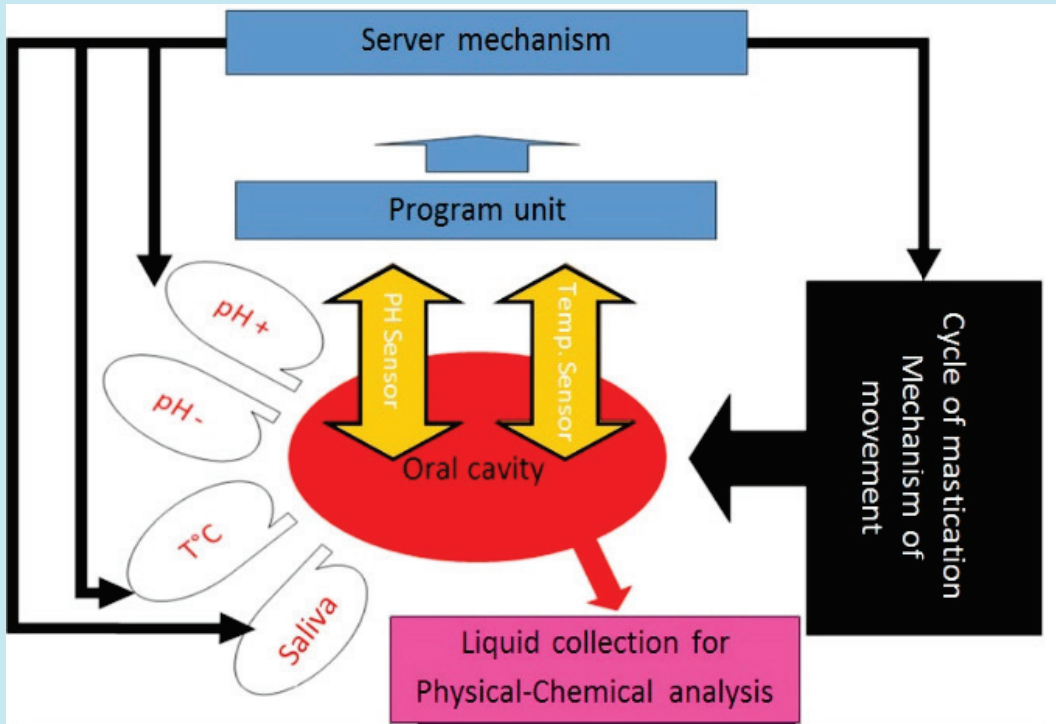


Fig. 1. Chewing bench principle

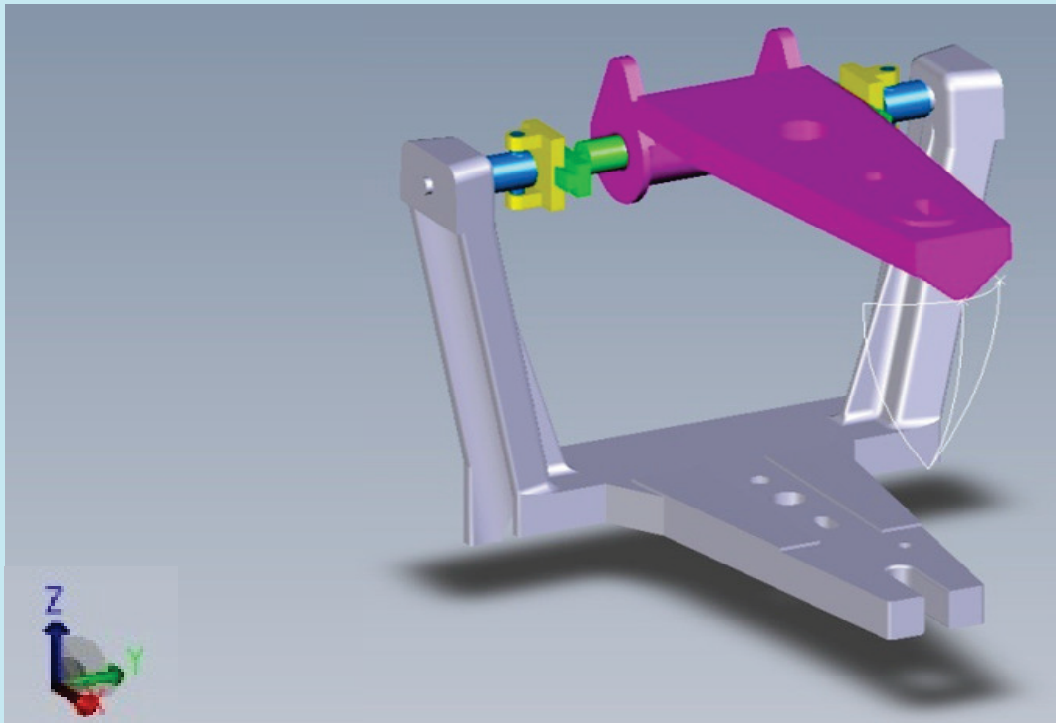


Fig. 2. Dental articulator (Fag Quickmaster®)

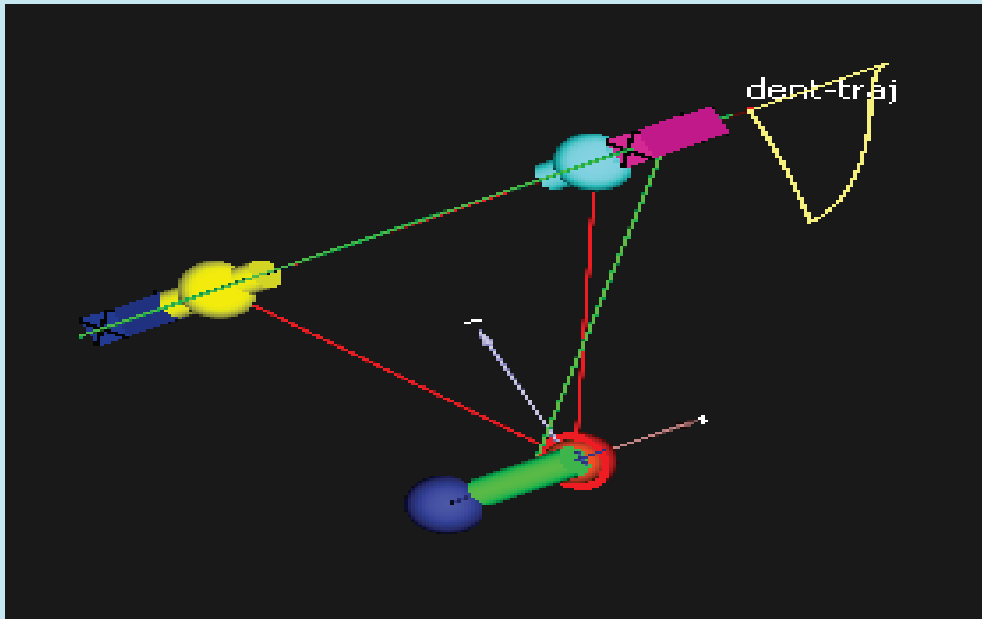


Fig. 3. Modeling mandible movement with open meca to simulate the movement trajectory in the X, Y and Z axes

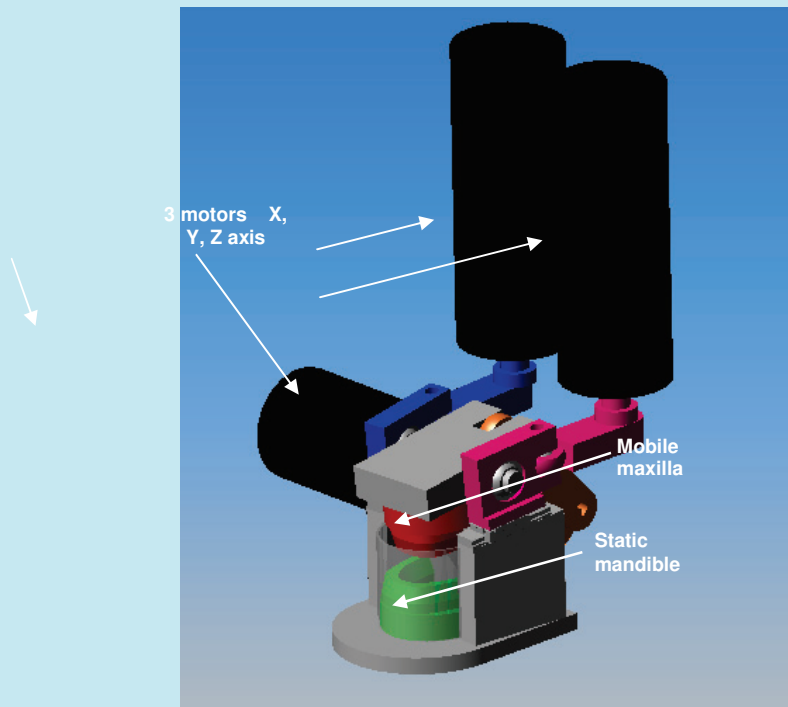


Fig. 4. Simulation of the oral cavity with the chewing bench and motors

Table 1. Parameters of chewing bench

Parameters	Values
Oral cavity volume of	25cm ³
Maximal forces	500N
Trajectory	Mastication cycle
Solution	Normalized artificial saliva
Temperature of solutions	Between -5°C + 70°C
Ph of solutions	Between 2-9
Maximal number of cycles	5000

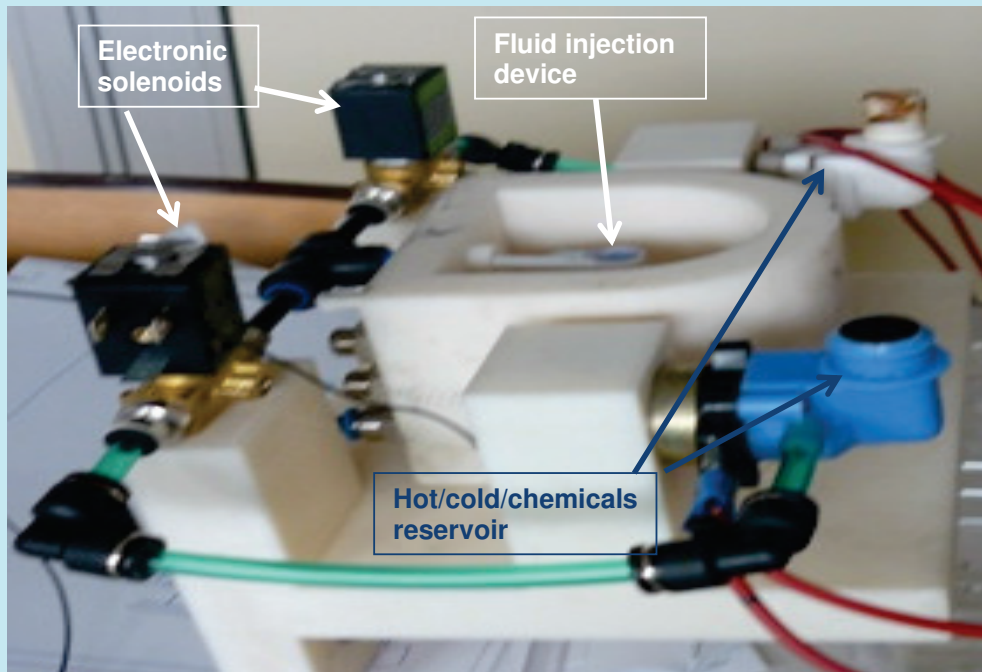


Fig. 5. Thermo-chemical device using pumps for spraying

The material chosen for the chewing bench was PolyetheretherKetone (PEEK), the properties of which are shown in Table 2; this material allows working with solutions with a pH ranging from 2 to 9. It corresponds to the pH range present in human food. In addition, this material stands temperatures in ranges between -5°C and +70°C, needed to produce thermal shocks of large amplitude to simulate the hottest as well as the coldest foods. The disadvantage is that PEEK is very rigid; cheeks made of silicone will be used for their flexibility, in order to easily reproduce the mastication trajectory.

Silicone rubber has been tested successfully; its texture is as close to food as possible, so it can be used for food simulating [13,25]. Different slurries with different compositions and foodstuffs could be used interchangeably with silicone, to test the

different textures and consistencies of food material; these will mimic the action of different foods (thermal and chemical parameters) on dental restorative materials while using artificial saliva.

Table 2. Properties of polyetheretherketone (PEEK) (Polyetheretherketone, www.goodfellow)

Parameters	Value
Young (MPa)	3,7-4
Hardness	M99
Poisson	0,4
Friction	0,18
Density (g.cm ⁻³)	1,26-1,32
Spécificheat	1340
Acid strength	Good
Base strength	Good

3. DISCUSSION

Mastication is an essential function in human development. It is complex and involves several parameters (chewing forces, temperature, pH, and saliva). The device presented in this paper is capable of reproducing an artificial oral environment for testing dental materials in conditions which are very close to in vivo conditions.

The dynamic fatigue and aging have been recognized to limit the longevity of restorative and prosthetic dental materials, and while wear is an important factor involved in the process, other factors seem to play an important role as well, especially at the tooth material interface [5]. The presented device not only tries to mimic the complex mandibular movement, to simulate mechanical wear and fatigue, but also integrates thermal and chemical effects, in systematic and programmable manner.

The proposed chewing bench has three degrees of freedom, it cannot thus accurately reproduce the complete kinematics of the human mandible [22] compared to the hexapod design [1,16]. This limitation is related to the semi-adaptable dental articulator (Fag) chosen as a starting model; however, this choice should not in our opinion significantly change the overall results obtained.

Further development of the chewing bench includes the ability to collect and analyze the eluted material after the chewing cycle. Whether during the chewing cycle, or after the passage of a number of cycles, using different testing solutions, the funnel shaped base of the bench will help collect it using a pump, while a special computer program and a charley robot can assign the collected solution corresponding to specific time of dispersion, thus allowing the examination of the effect of different solutions used at different scale of time, different methods can then be used to analyze the collected solution.

4. CONCLUSION

Having concluded the first stage, worked out the requirement and accepted the technical solutions, manufacturing settings and working tests of the chewing bench can now begin. Additionally, comparison of data from the chewing bench and those observed clinically may be necessary in order to validate the protocol.

Testing dental materials is very important and the identification of released products and their quantification in order to measure their toxicity is a vital human health issue. Therefore, this chewing bench would hopefully reduce the gap between in vitro performance and in vivo observation and serve as a benchmark for existing materials and as a device for testing new ones.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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6. Discussion

Each paper, part of this thesis, contents a scientific discussion and the confrontation to the literature.

We would like here, to focus about the interest of combining all these studies in a new modulation of *in vitro* evaluation throughout a simulator. It is also the opportunity to define the limits of such sophisticated device.

Throughout the studies included in this work, the main objective was to reach a more global knowledge of the way, dental materials are evaluated before being inserted into the oral cavity, and subjected to the various strains of the oral environment.

The dental restoration or prosthesis should maintain its form and function throughout prolonged periods, and logically should have a set of mechanical and physical properties that resist aging and deterioration.

The final objective was to bring about a general understanding of the process of dental materials characterization and assessment, and to shed more light on the hidden aspects of interaction between different properties in the aim of improving knowledge in material aging.

A great deal of emphasis was given to the choice of materials to be tested, and that it would represent the current trends in dental practice and the latest developments in material composition. Equal highlight was given to the choice of testing methodology and laboratory testing techniques and their correlation to the clinical outcome.

In our first study (Comparison of mechanical properties of a new fiber reinforced composite and bulk filling composites), we choose a bulk composite because this new material reduces time for filling placement, responds to market demand and limits the operator dependent parameter.

Hence, different types of bulk composites were tested and compared for their mechanical properties. The standard ISO test methods were used for evaluating the composites, and even though they were criticized for discrepancies between various studies, yet as stated earlier standardized testing allow for comparison between material performance (in the same study) and give an indication about their behavior in actual clinical settings.

In the above mentioned study, we tried to explore further the mechanical properties of these materials, knowing that they are indicated to be inserted in 4mm increments unlike conventional composites (2mm increments), so a modified flexural strength test was designed, including 4 mm thickness polymerized only from the top, in order to simulate the clinical condition of placement.

This non-standardized test, that was performed on the samples, revealed important information about the correlation between *in vitro* results and the clinical behavior in the oral cavity (the effect of one side curing on the mechanical properties versus top and bottom curing for *in vitro* testing) and the importance of adapting the standardized testing methods accordingly.

An adhesive system, necessary for resin composite restorations, is an additional parameter involved in the durability of the restoration. The adhesive system used should be tested and evaluated to ensure the restoration longevity.

In another study, (Evaluation of interfacial type of fracture using two adhesive systems), we choose to modify the conventional bond strength method, using shear force, to avoid the faulty stress distribution pattern that tend to distort the results obtained with such method.

Our results exposed new insight on the role of the viscoelastic behavior linked to the resinous adhesive in resisting the stress at the tooth restoration interfaces. Such insight reveals important aspects, usually poorly identified, but is essential in the long term behavior (the effect of the adhesive layer modulus and viscoelastic properties for example as revealed in our study). The continuous development of the adhesive materials composition, and the addition of fillers, enzymes or antibacterial agents, modifies the mechanical properties of these materials, and the viscoelastic behavior in our opinion is a fundamental property.

Mineralized dental tissues are biological tissues with many variations: porosity, level of mineralization, and water content. Any changes that would affect the chemical, physical and mechanical qualities of enamel and/or dentin would result in unpredictable deterioration of the tooth material-interface.

Our study (Tensile Bond Strengths of Two Adhesives on Irradiated and Nonirradiated Human Dentin) shows the decline in the adhesion between the irradiated dental tissue and the composite.

Needless to say those effects may be only revealed after the aging process. The action of thermal cycling may disclose more information about such effects, and the standardized testing underlines the importance of artificial aging on the material properties during *in vitro* testing.

The chemical composition is the main aspect to consider especially in the field of CAD CAM restorative materials.

Therefore, in our study (mechanical properties and internal fit of four CAD CAM block materials), different CAD CAM block materials were tested from different groups of material, whether ceramic based, resin composite based or a mixture of both. Each of these material groups possesses physical and mechanical properties, making it more suitable for a particular type of restoration.

Ceramic materials because of their optical properties, hardness, wear resistance and even brittleness, seem more appropriate to replace enamel anterior veneers for example). Hybrid or resinous based block materials having lower elastic modulus and wear resistance have improved milling properties and ability to repair.

Stresses to which the restorative and prosthetic materials are subjected to, varies to a great extent according to the age, sex, and dietary habits of each individual. Some of these stresses are more aggressive and tend to render the material or the interface more prone to failure.

In our study (Thermal shock effect on enamel – resin composite interface), we evaluated the thermal shock effect, which could take place routinely in the oral cavity, on the enamel resin interface. This type of provoked stress is rarely tested in the experimental *in vitro* settings, and explains why some materials seem to perform better in laboratory settings more than in the real oral environment.

As shown in our study, the thermal shock seemed to result in weakening the enamel – resin composite and make it more susceptible to failure, these types of stresses should accordingly be evaluated more in depth to avoid premature failure of resin composite restorations.

This approach was useful for evaluating the association between dental tissues, adhesives and restorative material properties in the behavior of the interface.

Stresses induced, and the aging process of the restoration may lead to the elution of degradation products and noxious constituents.

Dental materials inserted in the oral cavity, should not only show superior physical and mechanical properties to be able to endure the aggressive oral environment, but should as well induce no toxic, mutagenic or allergic reactions. The direct irritation or toxic effect

of dental materials on the pulpal or gingival tissue is the first step in the characterization of biological behavior. The systemic toxicity of such materials is much more difficult to evaluate, and have to be done through long term studies to detect the possible adverse effects these materials have on human body.

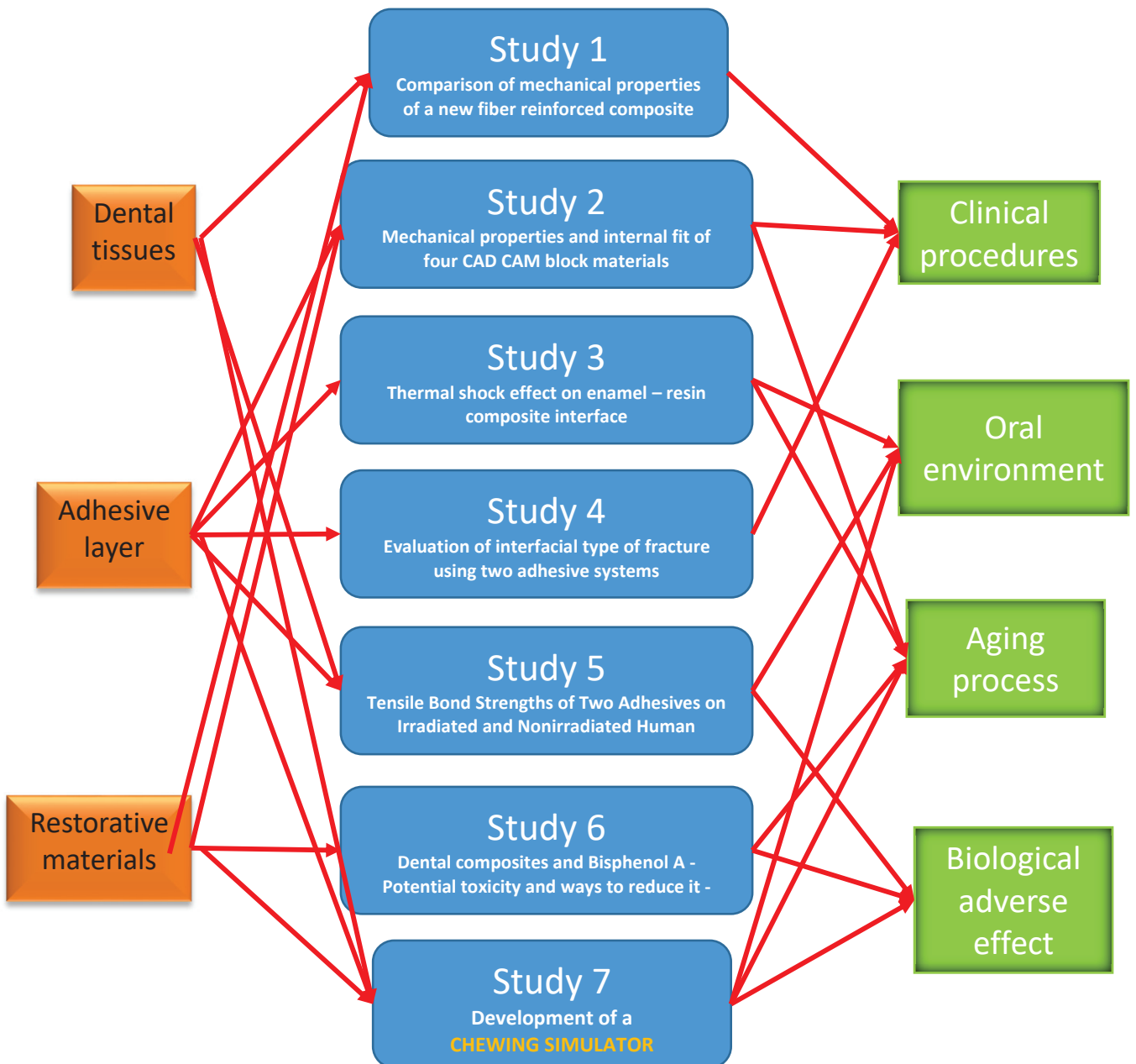


Figure 17 : An overview of the studies performed and their impact.

This holds especially true for Bisphenol A included in dental resins, as well as for substitute monomers that are used to replace bisphenol A.

An exhaustive systemic review was done on potential toxicity of Bisphenol A in dental composites and ways to reduce it, to explore the different studies that evaluated the long term effects of exposure to Bisphenol A.

The most alarming of these, is the possibility of an endocrine disrupting effect, or more precisely the NonMonotonic Dose Responses (NMDR) which would mean the doses needed to produce such effect are much smaller than the measured regulatory doses. The work done evaluated different potential harmful effects and also reflected about the possible measures to avoid the leakage of bisphenol A from dental restorations.

Our work clearly demonstrates that materials in laboratory settings seem to perform rather better than they do in the real oral environment, whatever the evaluated properties are.

Moreover, despite the existence of different ways to evaluate a new restorative material, manufacturers seem more interested in exhibiting the improved superiority of their products while researchers are more focused on the weak points.

Since new dental materials are developed each year, our work suggests an effective and standardized method to simulate *in vivo* behavior.

Accordingly, our objective was to develop an oral simulator that would be able to mimic the oral environment with all its aspects, whether physical or mechanical, this device

constructed to evaluate dental materials, would provide valuable information about the actual performance of these materials under relevant physical and mechanical stresses.

The conception, design and configuration settings of this device were presented in our study (Development of a chewing simulator for testing dental materials: a pilot study). The chewing simulator would include other parameters that are missing from other oral simulators, which tend to focus more on the mechanical wear of dental materials.

The final objective would be to correlate the results from the chewing simulator with the results obtained clinically, thus reducing the gap between *in vivo* and *in vitro* results. The chewing simulator would represent a more relevant method for *in vitro* evaluation than other isolated methodologies.

The Chewing Bench offers many benefits to final material evaluation methods. Material behavior would be considered under relevant stress, which closely resembles actual intraoral occlusal forces. Moreover, it allows the testing of different materials with varying structure and composition under the same conditions. The proposed device would evaluate the effect of mechanical, physical and chemical stresses on increased elution of noxious molecules from dental materials. Likewise the mastication bench allows for monitoring the interface region over time, and examining the thermal or physical stresses effect on mechanical properties.

The mastication bench would thus introduce a new step to dental material evaluation that would help to reduce the gap between *in vitro* and *in vivo* results obtained with conventional methods.

However, combining many parameters in this device present the difficulty of identifying the origin of failure whether an initial material defect or as a result of degradation factors.

Moreover, this kind of *in vitro* evaluation has to be combined with biological studies in order to be able to specifically evaluate biomaterials cytotoxicity or other adverse effects.

In an overall view of the above mentioned studies, a wider appreciation of the methods used to examine dental materials was developed.

The knowledge obtained through these studies and of similar studies performed in abundance throughout the dental scientific literature, made it obvious that effectively, restoring dental tissues form and function depend on a multitude of complex factors, and that, a case by case, analysis have to be performed in order to be capable of effectively restore the lost tissue.

We have considered the material choice not only regarding direct or indirect technique, but also all corresponding properties required for each case. Moreover, dental material evaluation has to be considered from a broader perspective, and numerous properties of a restoration or prosthesis are combined to resist numerous types of stresses and strains encountered once in function.

7. Conclusion

A complete appraisal of the parameters affecting the longevity of dental restorations was too ambitious.

However deeper knowledge was obtained about the factors affecting the material behavior during laboratory testing and during function.

Moreover, our transversal approach shows that the aging of resin composite materials is very difficult to understand, particularly considering studies including only few parameters. It also shows how *in vitro* tests could be enhanced and that the *in vitro* simulations fail to simulate the *in vivo* behavior of these materials.

Our various studies demonstrated the standardized testing ability to provide valuable information about dental materials properties. As a prerequisite, standardized testing provides the advantage of comparing results between different institutes and gives a preliminary evaluation of the dental material to be used.

Conversely, it is obvious that there is a need to constantly develop these testing methods, to adapt to the new materials chemistry and formulations that are developed continually.

The second step in the assessment of restorative dental materials could be an *in vitro* chewing simulator including simultaneously a large variety of aging factors. It would eventually allow us to save time, to save money and to improve security.

Pairing the Chewing Bench aging with *in vitro* testing would help reveal important interactions between different properties, and provide us with more detailed information about the causes of failure.

At the end, clinical trials remain, as explained by Ferracane, essential as a final step but regarding cost, safety and time delay to collect the results, only materials showing valid results through in vitro testing will be indicated in such clinical evaluation.

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Sommaire

La littérature scientifique révèle que les résultats *in vitro* ne sont pas toujours le reflet exact de la façon dont les matériaux dentaires se comportent dans la situation réelle. Les tests standardisés sont capables de fournir des informations précieuses sur les propriétés des matériaux dentaires, et permettent la comparaison des résultats entre les différents instituts. Inversement, la chimie des nouveaux matériaux et les formulations qui sont développées en permanence, rendent nécessaire de développer constamment nouvelles méthodes d'essai. Le travail en cours a examiné le processus d'évaluation et de test des matériaux dentaires avant l'insertion dans la cavité buccale. Les études incluses dans ce travail ont utilisé des méthodes de test normalisées, mais parfois certaines modifications ont été apportées à la méthodologie de ces tests; pour explorer davantage les aspects cachés de différents matériaux, de simuler plus la situation réelle, ou de provoquer des contraintes potentielles qui pourraient conduire à une défaillance matérielle *in vivo*. Une autre section du travail a examiné les effets nocifs de certains constituants du matériau dentaire tels que le bisphénol A et le développement d'un simulateur de mastication (banc de mastication) qui simule l'environnement buccal et permet de surveiller la fuite de substance des restaurations dentaires, ainsi que l'évaluation physique et mécanique de diverses restaurations et prothèses dentaires. Le nouveau dispositif permettrait de répliquer de différents paramètres chimiques, physiques et mécaniques de l'environnement buccal, permettant également de réduire l'écart entre les tests *in vitro* et *in vivo* des matériaux dentaires.

Mots clés: Matériaux dentaires. Essais normalisés. Essai *in vitro*. Composite dentaire. Simulateur de mastication. Banc de mastication.

Summary

Scientific literature reveals that *in vitro* results are not always the exact reflection of how dental materials behave in the clinical situation. ISO standardized testing is able to provide valuable information about the dental materials properties, and enables result comparison between different institutes. Conversely, new materials chemistry and formulations that are developed continually, makes it necessary to constantly develop these testing methods. The current work reviewed the process of dental materials evaluation and testing before placement into the oral cavity. The studies included in this work used standardized testing methods, yet occasionally some modifications were made to the methodology of these tests; to explore further the concealed aspects of different materials, to simulate more the real situation, or to provoke potential stresses that could lead to material failure *in vivo*. Another section of the work examined the noxious effects of some eluted materials from dental restorations like Bisphenol A, and the development of a chewing simulator that would simulate the oral environment and enable monitoring substance leakage from dental restorations, as well as physical and mechanical evaluation of various restorations and prostheses. The new simulator would enable reproduction of different chemical, physical and mechanical parameters of the oral environment, thus permitting to bridge the gap between *in vitro* and *in vivo* testing of dental materials.

Key words: Dental materials. Standardized testing. *In vitro* test. Dental materials. Chewing simulator.