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Evaluation of resin composites for dental restorations

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Abstract

In the production of dental restorations, there are, currently, two main types of materials: ceramics and resin composites. These latter kinds are typically suggested because of their quick fabrication, easy reparation and increased crossed link *density* compared with conventional light-cured materials. However, it is not clear for the specialist what is the best option among the many commercially available materials for each precise clinical case. For that reason, this work aims to clarify the real mechanical performance of resin-based composites for indirect dental restorations obtained by material removal processes and their most suitable application.

Two kinds of resin CAD/CAM blocks were selected: Lava™ Ultimate and Cerasmart™, which were tested under two conditions: in the as received by the manufacturer state and after storage in artificial saliva during 30 days. The mechanical properties of both materials were analysed (*density*, *hardness, flexural strength, fracture toughness*) but also the influence on the degradation of the mechanical performance due to the contact with the saliva.

Results indicate a better mechanical performance of the Lava Ultimate material in the as-received condition, despite its coarser microstructure. However, Cerasmart shows a stabilised microstructure with a smaller degradation of the mechanical properties in contact with the artificial saliva; in other words, improved durability inside the mouth.

Keywords: resin composites; mechanical properties; CAD/CAM dental materials; microstructure

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1. Introduction

During the last decade, dental materials have experimented a significant evolution. The manufacturers have the constant need for innovating and creating novel materials that fulfil the mechanical but also aesthetical properties required by both dentists and clients. Also, new manufacturing technologies have been incorporated ininto the dental world, mainly in the field of oral restoration. For this reason, it is possible to regularly find in the market various materials that require from both, clinic and laboratory investigation. This is highly important to decide which the best option is in clinical practice and obtain the desired results in the patients.

Currently, the assistant computer-aid design and manufacturing technologies (CAD/CAM) are one of the most popular to produce different restorations. Dental materials have experimented a significant evolution and can be divided into two big groups: ceramics and resin-based composites. Ceramics have the advantages of high abrasion resistance, excellent colour stability and high biocompatibility, however, they accelerate the abrasion of the opposite tooth, and there are have low *fracture toughness* also susceptibility to fracture due to the formation of flaws or defects in the surfaces [1][2]. Alternatively, resin-based composites are known for their superior aesthetics, smooth cutting, simple repairing in the oral cavity and less abrasive effect toward opposing dentition [3]. Despite this, there is some concern about possible allergic reactions in both dental personnel and patients [4].

In a first approach, this study was focused on the determination of the mechanical properties of the dental materials, by comparing them with the information about material composition, microstructure and mechanical properties (when provided). However, a more in-depth study had developed, including microstructural analysis and X-Ray fluorescence to obtain the precise composition of the materials because the information provided by the manufacturers are often incomplete or labelled with misleading terminologies regarding phase constituents.

2. Methodology

2.1 Materials and specimen preparation

The materials of this study were selected over the full range of chairside CAD/CAM commercial materials used nowadays for indirect dental restoration. Both of them are resin-based composites: Lava™ Ultimate Restorative (3M ESPE) and Cerasmart™ (GC Dental Product) with differences mainly in the dispersed nanoparticles (Table 1).

Table 1. Composition and manufacturers´ information on tested materials.

Material	Manufacturer	Abreviation	Composition
Lava™ Ultimate	3M ESPE	LU	Composite resin material (BisGMA, UDMA, BisEMA, TEGDMA) with 80 wt.% silica and zirconia nanoparticles and zirconia/silica nanoclusters
Cerasmart™	GC Dental Product	CS	Composite resin material (BisMEPP, UDMA, DMA) with 71 wt.% silica and barium glass nanoparticles

BisGMA: bisphenol A diglycidyl ether dimethacrylate; UDMA: urethane dimethacrylate; BisEMA: ethoxylated bisphenol A dimethacrylate; TEGDMA: trimethylene glycol dimethacrylate; BisMEPP: 2,2-bis (4-methyacryloxy polyethoxy phenyl) propane; DMA: dodecyl dimethacrylate.

The manufacturer provided CAD/CAM material (Fig. 1) only in small blocks of approximately 18 x 16 x 18 mm^3 (C16 blocks), and due to their brittleness, they were embedded before cutting in epoxy resin. First, to cut slices of 1.5 mm thickness, and after that, embedded again to cut their nominal final dimensions 1.5 x 1.5 x 17 mm^3. This cut-off to obtain the beam specimens was performed with an Accutom-50 (Struers, Denmark) using a diamond disk under water refrigeration in several steps. As the last step, they were cleaned in distilled water for 10 minutes by ultrasounds and dried.

Fig. 1. (a) C16 blocks of (a) LU and (b) CS

Two types of beam specimens were used to perform the tests. Beam specimens as finished after the cut-off process for measuring all the properties except the fracture toughness, and beam specimens with a notch for measuring fracture toughness. In these last specimens, notches were introduced using ultra-short laser ablation, so-called single edge laser-notch beam (SELNB) method [5] and can be considered, therefore, as a real crack. The novel technique, which was successfully tested in brittle metals, produce very sharp notches in the material with speed, high accuracy, good reproducibility and precision for reliable *fracture toughness* testing.

2.2 Aging procedure

Half specimens from each material were immersed and stored in artificial saliva (AS) at room temperature during 30 days, which was observed to be enough time to reach a stationary state in the immersed samples. Tests performed with the material after storage in AS compared with the ones performed with the materials as received by the manufacturer (AR) state and the data provided by the manufacturer (MN) itself to check the influence of the direct contact of the saliva with the tooth.

2.3 Density

The experimental *density* was measured via Archimedes' method with immersion in high purity ethanol at room temperature.

2.4 Hardness tests

The *microhardness* was obtained by microindentation tests at room temperature with a durometer AKASHI MVK-EIII (Japan), by using a Vickers indenter and following the ASTM-E92-17 [6]. Two applied loads (3 and 9.8 N) were used for tests performed in the AR state, whereas tests for samples stored in AS were performed only with an applied load of 9.8 N. This way, the results for the tests in materials tested AR and materials stored in saliva can be compared with the load of 9.8 N to check its influence in both materials.

The nanoindentation tests were performed at room temperature using a NanoIndenter XP from former MTS Systems Corporation (United States). They were performed using a standard Berkovich tip calibrated with fused silica. Tests were completed under load control and two applied loads: 0.25 and 0.5 N and continuous measuring of the displacement. Based on the load-displacement data obtained, the average values (with their standard correspondent error) of *nanohardness* and elastic modulus were calculated according to the Oliver and Pharr method [7], [8].

2.4 Three-point bending tests

Miniaturised three-point bending (TPB) tests were performed on smooth and SELNB specimens to determine the *flexural strength* and the *fracture toughness*, respectively (Fig. 2). For both types of results, tests were performed with samples in the AS state with a span (L_s) of 12 mm and with samples immersed in AS with a span of 8.5 mm. The standard span of 20-40 mm cannot be used due to the reduced dimensions of the C16 blocks.

From the force-displacement data obtained from the TPB samples without a notch, the *flexural strength* was obtained at the fracture point by using the standard material strength formulas [9]:

(1)

where σ_f is the *flexural strength*, F_{max} the maximum applied a load, L_s the support span and B and D, the width and height of the specimen, respectively, which were measured for each sample.

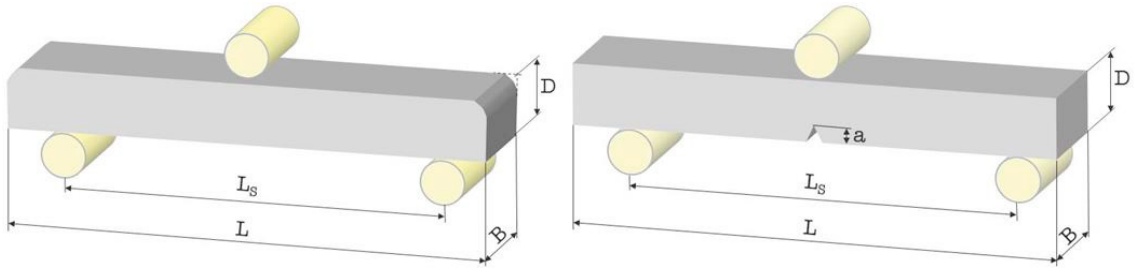

Fig. 2. (a) smooth specimen to obtain *flexural strength* and (b) SELNB specimen to obtain *fracture toughness* of the samples.

Besides that, SELNB specimens were used to calculate the fracture toughness. From each specimen, the initial notch length was measured using a FESEM and the maximum applied load recorded during the test. Then, using the appropriate formula [10], the *fracture toughness* was calculated for each specimen. At least three tests were performed for each condition (AR state and after AS storage).

2.5 Microstructure and fracture surfaces

For the microstructural analysis of the materials, the surfaces of LU and CS were grinded and polished. Then, to reveal the microstructure, they were etched with 5 % hydrofluoric acid (HF) during 30 s and finally coated with Au to ensure the conductivity for the field-emission scanning electron microscopy (FESEM) observation. After this preparation, the microstructure was analysed in an Auriga column FESEM from Zeiss (Germany).

After TPB tests fracture, surfaces were also examined with the FESEM to analyse the fracture mechanisms that produce the breakage of the specimens.

2.6 X-Ray fluorescence

A sample for each material was analysed by X-Ray Fluorescence (XRF) technique to ascertain element concentration. Tests were performed in tiny samples since in this technique small spots are exposed to X-Rays to obtain their characteristic energy and therefore, by analyzing the peaks energy, precise element concentration can be determined.

3. Results and discussion

3.1 Microstructure

LU, according to the manufacturer, is a typical resin composite (Table 1) with 80 wt.% of dispersed nanometric particles which are monodisperse, non-aggregated and non-agglomerated [11]. It contains two types of nanoparticles: silica nanomers of around 20 nm diameter, and zirconia spherical nanomers of 4 to 11 nm diameter. This data is in accordance with the composition obtained by X-ray fluorescence (Table 2), where a high concentration of silica (66.31%) and zirconia (31.63%) was identified. However, although FESEM micrograph and the results of the energy dispersive X-ray (EDX) spectroscopy line-scan analysis (Fig. 3) exhibits a homogeneous distribution of the particles, the observed grain size of the silica particles is of micrometre range.

Table 2. Element concentration (%) obtained from the X-Ray fluorescence analysis.

Material	SiO_2	ZrO_2	BaO	Al_2O_3	HfO_2	F	K_2O	Other
LU	66.31	31.63	-	0.29	0.57	-	0.37	0.83
CS	61.43	-	28.65	8.59	-	0.58	0.02	0.71

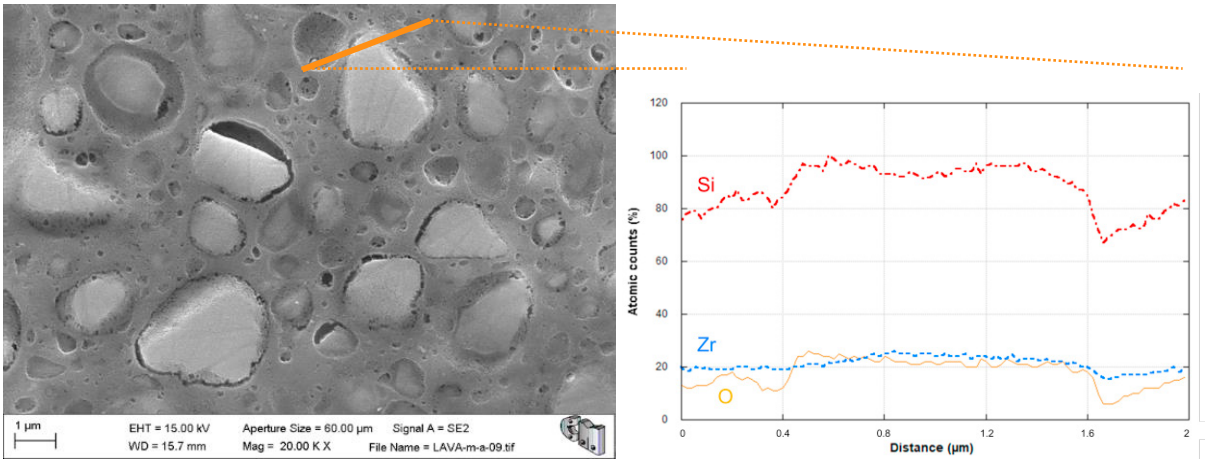

Fig. 3. (a) FESEM micrograph of a polished and etched section of LU, with (b) the result of the EDX line-scan analysis of the line indicated in (a).

CS has a matrix with dispersed barium glass under 300 nm and silica under 20 nm. Although the manufacturer composition indicates a content around 71 wt.% of silica and barium glass, the X-Ray fluorescence analysis (Table 2) exhibits values significantly higher than this: 61.43 % of silica and 28.65% of barium. Barium content was also complicated to detect with the EDX analysis, although silica was observed in higher concentrations in the sample (Fig. 4). It should be noted that unlike the LU, the microstructure of CS is of nanometer range with a homogeneous distribution of the particles.

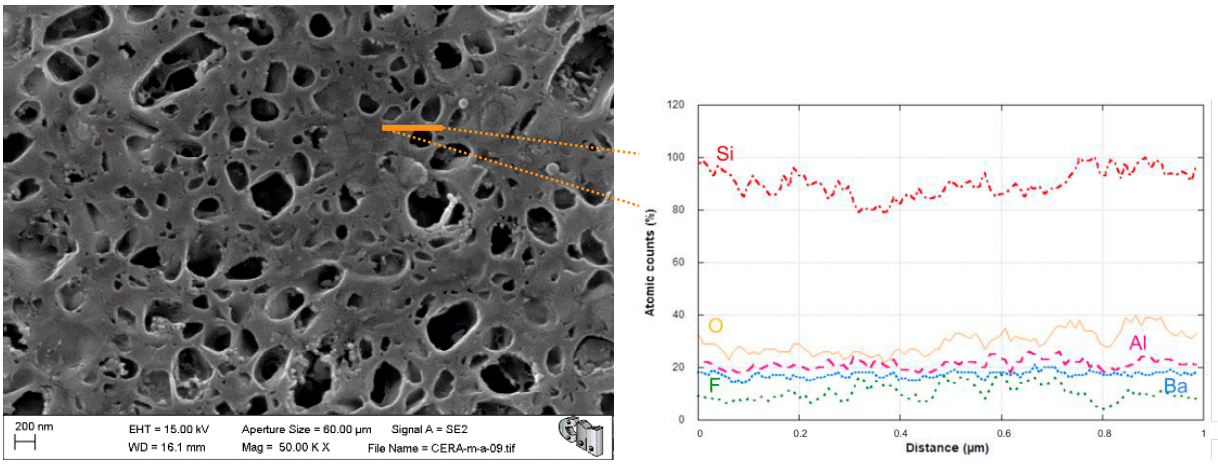

Fig. 4. (a) FESEM micrograph of a polished and etched section of CS, with (b) the result of the EDX line-scan analysis of the line indicated in (a).

3.2 Mechanical properties and fracture surfaces

Density for both materials is very similar, data for CS from the manufacturer was not found. However, published data for LU is slightly higher than experimental results (Table 3). Neverthelees, results are in accordance with results from other authors [12]. Regarding *young modulus* obtained from nanoindentations, results are in accordance too with results from other authors.

Table 3. Mean *density* values with their standard deviation measured (D) and provided by the manufacturer (D_{MN}) and nano elastic modulus (nE) along with elastic modulus value obtained by nanoindentation (nE).

Material	D (g/cm^3)	D_{MN} (g/cm^3)	nE (GPa)
LU	1.935 ± 0.006	2.1 ± 0.1	12 ± 1
CS	1.9068 ± 0.0004	-	8 ± 1

Regarding *hardness* test results, both materials exhibit similar values from instrumented Berkovick tests, but a slight disagreement from Vickers tests (Table 4). There is, therefore, a small influence of the applied load, that taking into account, the dispersion can be considered marginal since results are in a range over 1 GPa and comparable with literature and the manufacturer [12][13]. LU displays higher values than CS, which can be associated with the higher content of ceramic nanoparticles (Table 1). The artificial saliva produces an earlier degradation in both materials with a reduction of around 9% and 6% of the *hardness* values in LU and CS, respectively.

Table 4. Mean Vickers (HV) and *nanohardness* (nH) test results in GPa with their standard deviation for tests performed with the materials in the AR-state, after storage in AS and data obtained from the manufacturer (MN).

Material	nH_{AR} (0.25 N)	nH_{AR} (0.5 N)	HV_{AR} (2.9 N)	HV_{AR} (9.8 N)	HV_{AS} (9.8 N)	HV_{MN}
LU	1.23 ± 0.09	1.18 ± 0.03	1.30 ± 0.07	1.04 ± 0.07	0.95 ± 0.03	1.05 ± 0.01
CS	0.72 ± 0.02	0.72 ± 0.02	0.80 ± 0.01	0.76 ± 0.03	0.71 ± 0.02	-

As shown in Table 5, the results of *flexural strength* after TPB tests on beam specimens result significantly lower than the ones provided by the manufacturer for both materials. In the case of LU, a 25% of disagreement was observed, but in the case of CS, the divergence is even higher, reaching a reduction of around 40%. As for the *hardness* values, the saliva has a detrimental effect by reducing the *flexural strength*, more significant in the case of LU than in CS. LU has an improved *flexural strength* in the AR-state; however, since it is highly degraded by the saliva, the mechanical behaviour is slightly superior for CS.

Similarly, *fracture toughness* values calculated from TPB tests on SELNB specimens on LU samples reveal values over results from CS in the AR-state, but below the obtained after the storage in AR (Table 5). Despite this, differences in the *fracture toughness* results are not very meaningful.

Table 5. Mean *flexural strength* (σ) and mean *fracture toughness* (KIC) with their standard deviation in the AR-state, after storage in AS and data obtained from the manufacturer (MN).

Material	σ_{AR} (MPa)	σ_{AS} (MPa)	σ_{MN} (MPa)	KIC_{AR} (MPa·m$^{1/2}$)	KIC_{AS} (MPa·m$^{1/2}$)
LU	150 ± 10	112 ± 4	200 ± 20	1.04 ± 0.03	0.71 ± 0.01
CS	139 ± 3	120 ± 10	231 ± 5	0.93 ± 0.02	0.8 ± 0.03

Although both materials have a resin matrix, they exhibit brittle behaviour. There is no evidence of ductility in the analysis of the stress-strain curves from the TPB tests or the morphology of the fracture surfaces (Fig. 5).

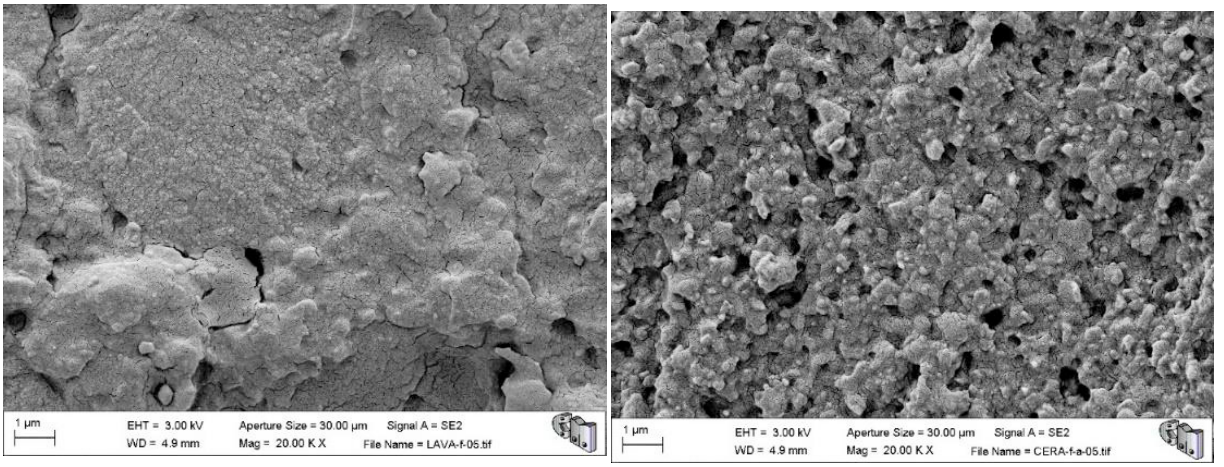

Fig. 5. (a) Facture surfaces after TPB tests for (a) LU and (b) CS.

4. Conclusions

Element concentration of Lava Ultimate and Cerasmart, two resin composites used for indirect dental restoration, and their nanoparticle size, slightly differs from the manufacturer data as obtained from the X-ray fluorescence and EDX spectrometer analysis.

The manufacturing process of both materials have been efficient because pores were not found in the samples. However, during that process, grain size of CS achieved a considerably finer microstructure than LU, and therefore, its mechanical performance was more reduced (considering *hardness*, *flexural strength* and *fracture toughness*) in the AR-state. In addition, it was observed that the saliva has less influence on the degradation in the CS than in the LU, and these changes in the mechanical performance, should also be taken into account during the manufacturing of the materials. This preliminary evaluation may represent an enhanced behaviour of the CS inside the mouth since it suffers a reduce detriment of *flexural strength* and *fracture toughness* values, which means enhanced durability inside the mouth of the patients.

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