Microstructural and mechanical analysis of two CAD-CAM lithium disilicate glass-reinforced ceramics

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Abstract: The aim of this study was to analyze the structural, morphological and mechanical properties of two different lithium disilicate glass-reinforced ceramics for CAD-CAM systems (IPS e.max CAD and Rosetta SM). Five methodologies were used for both ceramics: microstructure (n = 2) was analyzed using x-ray diffraction (XRD); morphological properties (n = 2) were analyzed by scanning electron microscopy (SEM), with and without hydrofluoric etching; porosity (n = 3) was assessed using 3D micro-computed tomography (micro-CT); flexural strength was measured (n =1 0) using the three-point bending test; and bond strength was determined with self-adhesive resin cement (n = 10), using a microshear bond test. After performing all the tests, the data were analyzed using *t*-Student test and two-way ANOVA. All the tests used a significance level of $\alpha = 0.05$. High peak positions corresponding to standard lithium metasilicate and lithium disilicate with similar intensities were observed for both ceramics in the XRD analysis. Morphological analysis showed that the crystalline structure of the two ceramics studied showed no statistical difference after acid etching. Additionally, no significant differences were recorded in the number or size of the pores for the ceramics evaluated. Moreover, no differences in flexural strength were found for the ceramic materials tested, or in the bond strength to ceramic substrates for the resin cements. Based on the study results, no significant differences were found between the two CAD-CAM lithium disilicate glass-reinforced ceramics tested, since they presented similar crystalline structures with comparable intensities, and similar total porosity, flexural strength and bond strength.

Keywords: Ceramics; Dental Materials; Computer-Aided Design.

Introduction

The clinical use of ceramic restorations has grown substantially in the past years, owing to the increase in demand for aesthetic restorative procedures, and to the improvements in dental ceramic materials. Today, lithium disilicate glass-reinforced ceramic is one of the most frequently selected indirect restorative materials used for all-ceramic restorations.[1] Its excellent resistance to fracture (± 380 MPa),[2] good esthetics, and satisfactory bond strength to resin cements, when adequate surface

treatment is provided (± 18.00 MPa),[3] are factors favoring the growing acceptance of these ceramic materials. Additionally, the possibility of milling this ceramic using simplified fabrication methods, such as laboratory and chairside computer-aided design/computer-aided manufacturing (CAD-CAM) systems,[4,5,6] is another important advantage contributing to its excellent clinical approval.

Lithium disilicate glass-reinforced ceramics were introduced in 1998 as IPS Empress 2 (Ivoclar Vivadent, Schaan, Liechtenstein), an exclusive product released by Ivoclar. The e.max Press system (Ivoclar Vivadent, Schaan, Liechtenstein) emerged in 2006 as the new generation of heat-pressed ceramics, featuring improved mechanical and optical properties over the first-generation material.[7] However, the patent of this product recently expired, and other companies can now fabricate and market similar ceramic materials. Several manufacturers are now producing other lithium disilicate-based ceramics, including Rosetta (Rosetta, Hass, Gangneung, Korea), T-lithium (Talmax, Curitiba, Brazil), AIDITE (Shenzhen, Guangdong, China) and IRIS (Tianjin, Mainland, China). The manufacturers suggest that these new ceramics have mechanical, structural and morphological properties similar to the primary IPS e.max system (Ivoclar Vivadent, Schaan, Liechtenstein), but there are few available studies that compare these materials.[2]

The crystalline structure of ceramics influences the mechanical and morphological properties of these materials.[8] Structural properties are commonly investigated using x-ray diffraction analysis (XRD), which identifies the peaks of the crystals present in the ceramic and its crystalline phase, and determines the degree of crystallinity and the size of the crystals formed.[8,9,10,11] The morphological characteristics of dental ceramics can also be evaluated by scanning electron microscopy (SEM),[12] since high resolution emission field protocols enable the shape and size of the crystal grains to be observed. The percentage of pores and the ceramic characterization can also be determined by micro-computed tomography (μCT), which allows analysis without destroying the specimens.[13] Moreover, the flexural strength test is important to evaluate the maximum force to fracture and the flexural modulus of dental ceramics,

properties that can help characterize the load capacity of the material.[14] Additionally, the interaction of glass ceramics with resin cements, and their bonding capability to these cements, are important clinical parameters, modulated by the composition and susceptibility of these ceramics to surface treatment with hydrofluoric acid etching associated to silane coupling agents. The microshear bond strength test has been commonly used to measure the bond strength of resin cements to ceramic materials, because it is an easy operation to perform.[15]

Thus, the aim of this study was to compare two CAD-CAM lithium disilicate glass-reinforced ceramics using different methodologies, specifically, XRD, SEM, porosity test (μ-CT), microshear bond strength and three-point bending test. The null hypothesis tested was that no differences would be detected in the microstructural and mechanical properties of the glass ceramic material evaluated.

Methodology

Two CAD-CAM lithium disilicate glass-reinforced ceramics, IPS e.max CAD (Ivoclar-Vivadent, Schaan, Liechtenstein) and Rosetta SM (Hass, Gangneung, Korea), were evaluated using HT-A2 C14 blocks. The specimens for each ceramic material were prepared according to the respective methodologies, as follows:

X-ray diffraction (XRD)

X-ray diffractogram patterns were performed (n = 2) at room temperature (25°C) using a diffractometer (XRD-6000, Shimadzu Corp., Tokyo, Japan) with monochromatic Cu-K$_1$ (λ = 1.54056Å) radiation. XRD scanning was carried out using the Cu-K1 emission (λ = 1.54056Å), generating a current of 15mA, 30kV, a wavelength equal to 1.5406Å, and a continuous scanning interval of 2θ (20–80), with a step of 0.02 s. The XRD patterns were compared with the JCPDS (Joint Committee on Powder Diffraction Standard) to identify the type of crystal and crystalline phase of both ceramic materials.[2,3,4,5,6,7,8] Two specimens were selected from each group (pre-crystallized and crystallized) to perform the structural analysis. Crystallization of the specimens was performed using a special furnace (Programat P300, Ivoclar Vivadent, Schaan,

Liechtenstein), together with the P91 program. This furnace reaches a maximum temperature of 845°C, and then stabilizes for a period of 7 minutes, after which it starts to cool slowly to prevent thermal shock.

Traditionally, the structural analysis test requires that the specimen be reduced to powder, but the ceramic block was too rigid. Alternatively, the specimens were cut to a size of approximately 1 cm^3 using a diamond saw (Isomet 1000, Buehler, Lake Bluff, USA). Then, they were placed on a metal device filled with aluminum particles to start the test, which lasted an average of 1 hour and 40 minutes, for each specimen. Two graphs (diffractograms) were obtained for each group, and interpreted qualitatively.

Scanning electron microscopy (SEM)

The morphological structures of both ceramics were analyzed after crystallization. Initial preparation consisted of obtaining specimens (n = 2) with dimensions of approximately 3.0 mm^3 using a diamond saw (Isomet 1000, Buehler, Lake Bluff, USA). Next, they were sectioned from the ceramic blocks at the pre-crystallized stage, and submitted to the crystallization process as described above. All the ceramic surfaces were polished sequentially using silicon carbide paper (#600, 800, 1200, Norton, Guarulhos, Brazil) for 20 s, and then etched with 10% hydrofluoric acid (HF) (Condac Porcelana, FGM, Joinville, Brazil) for 20 s. The specimens were washed using 70% alcohol and distilled water, followed by an ultrasonic bath with distilled water for 10 min, to remove the HF etching residues. Afterwards, the specimens were mounted on coded brass stubs and sputter-coated with gold palladium for 60 s at 45 mA (QR 150ES, Quorum Technologies, Laughton, Lewes, UK) to obtain images of better quality that allowed visualization of the crystals. Three images of each ceramic material with and without HF etching were obtained using SEM (VEGA 3, TESCAN, LMU, Kohoutovice, Czech Republic), with an accelerating voltage of 20.0 kV and original magnification of 20,000×. The images were obtained and analyzed by a skilled operator.

Total porosity

Three specimens were obtained from each ceramic material, with a cubic format (± 4.0mm^3)

for pore evaluation, and 3D reconstruction using μCT scanner (Skyscan 1272, Bruker, Kontich, Belgium). The specimens were also sectioned from the ceramic blocks at the pre-crystallized stage, and then submitted to crystallization as described earlier. The following scanning parameters were used: filter Al 0.5 & Cu 0.038; source voltage 90 kV; source current 111A; image pixel size 10.0m; 81 slices; lower gray threshold 60; upper gray threshold 190.[13] The total porosity percentage was measured, and 3D images contrasting the dense mass of the ceramic and the void spaces of the pores were collected. The reconstructed 3D data sets were quantified using a CTAn automated image analysis system (Brunker, Kontich, Belgium). The block images were obtained virtually, without destroying the specimens. The values were analyzed using the *t*-Student test (α =.05).

Three-point bending test

The three-point bending test was performed as recommended by ISO standard 6872.[14,15] Both ceramic materials were sectioned into rectangular bars using a diamond saw mounted on a low speed precision cutting machine (Isomet 1000, Buehler, Lake Bluff, USA). The bar dimensions were approximately 20.0mm long, 4.0 mm wide and 1.2 mm thick, and all the edges were chamfered, leaving a 0.1-mm-wide chamfer, following ISO 6872:2008. The dimensions were checked with a Digimatic caliper (Mitutoyo Absolute Digimatic Caliper, Tokyo, Japan) and recorded. The bars from each group (n = 10) were crystallized in a special furnace (Programat P310, Ivoclar Vivadent, Schaan, Liechtenstein). Afterwards, the bar surfaces were polished with a polishing machine (EXAKT 400 CS, EXAKT Technologies, Oklahoma City, USA), using silicon carbide papers (#600, 800, 1000, and 1200-grit; EXAKT Technologies, Oklahoma City, USA) under running water at 300 rpm. Lastly, the specimens were washed and stored dry until testing.

In the testing process, the specimens were placed in a mechanical testing machine (DL2000, EMIC, São José dos Pinhais, Brazil) over two rods approximately 2.0 mm in diameter, with a 16.0-mm span length. The three-point bending test consisted of applying a compressive force with a 50.0 kgf load cell over the center of the ceramic bar, and using a piston approximately

2.0 mm in diameter, at a 0.5 mm/min crosshead speed all the way to fracture. The values were analyzed using the *t*-Student test (α =.05). The three-point bending test consisted of calculating flexural strength ($\sigma_{3\text{-pt}}$), where σ is the distance between the supports (16.0 mm), and w and b are the width and thickness of the specimen, respectively, measured immediately prior to testing. The following formula was used:

$$\sigma_f = \frac{3\,Pl}{2wb^2}$$

where P is the fracture load (N), l is the span size (16 mm), w is the specimen width (mm) and b is the thickness of the specimen (mm).

Bond strength test

Ten CAD-CAM blocks from each ceramic material were selected for the microshear bond strength test. One 3.0 mm slice was removed from each block using a diamond saw (Isomet 1000, Buehler, Lake Bluff, USA), and crystallized using the same protocol described earlier. The ceramic slices were embedded in polystyrene resin (Aerojet, Santo Amaro, Brazil). Next, all the ceramic surfaces were polished sequentially using silicon carbide paper of (#600, 800, 1200; Norton, Guarulhos, Brazil) for 20 s.

Afterwards, the test surface of each ceramic slice was etched with 10% HF (Condac Porcelana, FGM, Joinville, Brazil) for 20 s, followed by rinsing with water for 30 s and air-drying for another 30 s. One layer of a silane-coupling agent (Prosil, FGM, Joinville, Brazil) was actively applied to the ceramic surface for 20 s, and left to react for 60 s. A self-adhesive resin cement (RelyX U200, 3M ESPE, St. Paul, USA) was prepared according to the manufacturer's directions, and inserted into silicon molds of Tygon® bore tubbing (1.0 mm in diameter and height) on the ceramic surfaces.[16] After 5 min of preparation, the resin cement was activated for 40 s using a monowave LED light-curing unit (Radii Cal, SDI, Victoria, Australia). Next, the silicone mold was removed using scalpel blades. Six cylinders were made on the surface of each ceramic block, spaced at 3.0 mm intervals.[16,17] The specimens were stored at 100% relative humidity, at 37°C for 24 h previous to testing.

A microshear bond strength test was performed after positioning and fixing the ceramic slices in a mechanical testing machine (OM100, Odeme Dental Research, São José dos Pinhais, Brazil). The resin cement cylinders were then aligned in the direction of the force application. A 0.2 mm diameter orthodontic wire (NiCr, Morelli, Sorocaba, Brazil) was used to load the cement cylinders perpendicular to the ceramic surface. The crosshead speed was set at 0.7 mm/min.[18] The procedure for the specimens that were tested immediately (24h - T0) consisted of loading three cylinders, and then obtaining the mean values. The remaining resin cylinders from each group were then submitted to aging by storing the ceramic specimens at relative humidity, at 37°C for 30 days. After storage, the three remaining resin cylinders were loaded (30 days - T1) using the same protocol described above. Bonding strength values from T0 and T1 were compared within the groups and between both ceramics. Statistical analyses were performed using two-way ANOVA followed by the Tukey test. The bond strength of each specimen (MPa) was calculated using the following equation:

R = F / A

where R is the bond strength in MPa; F is the force required for specimen rupture (N); and A is the adhesive area of the specimens (mm²).

The specimens were submitted to a microshear bond strength test, and the fractured surface of the specimens was evaluated by optical microscopy (Axiocam, Mitutoyo, Absolute, Tokyo, Japan) at 40× magnification to determine the failure mode, classified into: a) adhesive failure, b) cohesive failure, and c) mixed failure.

Results

X-ray diffraction (XRD)

The XRD results are shown in Figure 1. Both ceramics presented comparable, narrow diffraction peaks, which confirm the formation of crystalline compounds of similar intensity. The diffraction peaks detected in the XRD patterns are characteristic of lithium metasilicate (ICCD 029-0829) and lithium disilicate (ICCD 040-0376) crystals, confirming the formation of these crystalline compounds for both ceramic materials tested.
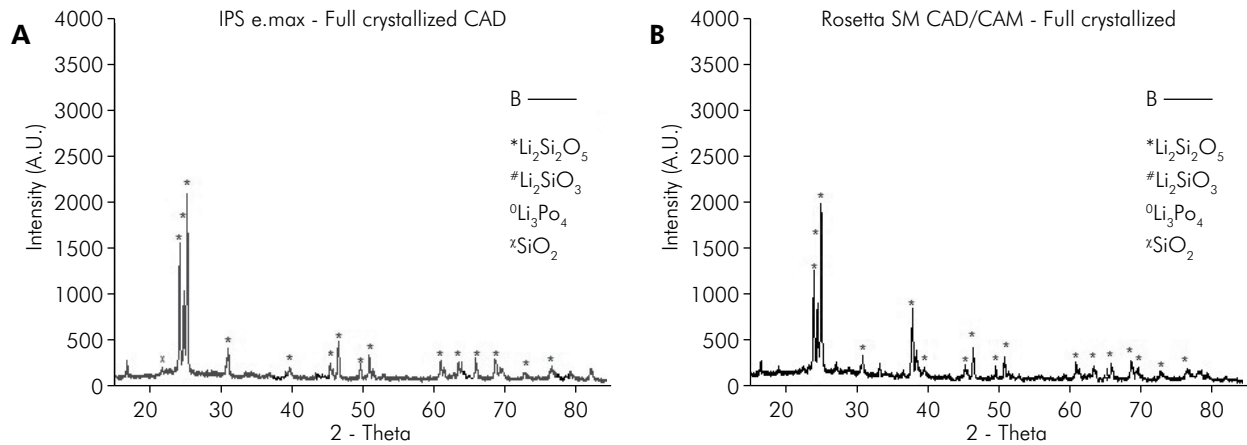
Figure 1. XDR patterns: A: IPS e.max CAD group; B: Rosetta SM group.

Scanning electron microscopy (SEM)

After crystallization, the crystalline microstructure of the crystals became denser, and the surface etching caused changes in their superficial morphology. SEM images of the etched ceramic surfaces of both systems are shown in Figure 2. The 10% HF etching caused IPS e.max CAD and Rosetta SM ceramics to form elongated spindle-shaped lithium disilicate crystals surrounded by a sparse glass matrix. The shape and size of these crystals were very similar for both ceramic systems analyzed.

Porosity – (μCT)

The total porosity values found for both ceramics using μCT scanning ranged from 0.05 to 0.11% for IPS e.max CAD, and from 0.07 to 0.13% for Rosetta SM
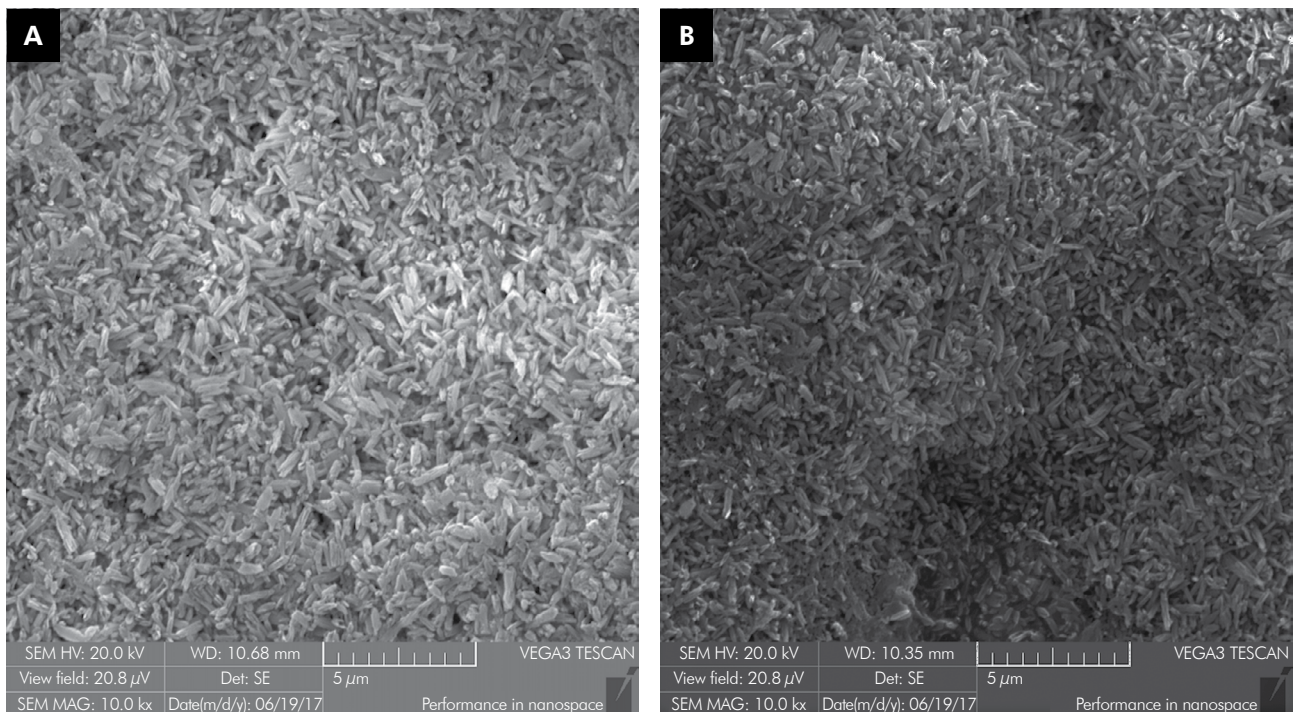

Figure 2. SEM images: A: IPS e.max CAD group after etching, 10,000× magnification; B: Rosetta SM group after etching, 10,000× magnification.

(Table 1). The *t*-Student test revealed no significant differences between the two ceramic systems for total porosity (p = 0.473). Uniform pore distribution was observed for both ceramics by µCT evaluation (Figure 3).

Three-point bending test

Mean flexural strength (MPa) values and standard deviation for both ceramic systems are presented in Table 2. The flexural strength was found to be between 340 and 350 MPa for the two ceramic systems, and the *t*-Student test showed no significant differences for the materials tested (p = 0.652).

Microshear bond strength test and failure mode

Mean microshear bond strength (MPa) values and standard deviation of the self-adhesive resin cement for the ceramic substrates tested immediately and after 30 days are shown in Table 3. Bond strength values of the resin cement were almost similar for both ceramic systems, regardless of the different storage times (± 17.0 MPa). Two-way ANOVA revealed no significant differences between the ceramic materials for bond strength (p = 0.881), at either storage time (p = 0.712).

The failure mode observed for the specimens was predominantly mixed failures (Table 4), followed by adhesive failures, irrespective of the ceramic material or storage time (Figure 4).

Table 1. Total porosity percentage (SD) for CAD-CAM lithium disilicate glass ceramic systems.

Ceramic system	Total porosity
IPS e.max CAD	0.08 (0.030) A
Rosetta SM	0.10 (0.034) A

*Same uppercase letters indicate no significant difference between groups; *t*-Student test (p < 0.05).

Table 2. Mean flexural strength (MPa) values (SD) for CAD-CAM lithium disilicate glass ceramic systems (MPa).

Ceramic system	Flexural strength
IPS e.max CAD	341.45 (61.44) A
Rosetta SM	352.39 (36.77) A

*Same uppercase letters indicate no significant difference between groups; *t*-Student test (p < 0.05).

Table 3. Mean microshear bond strength (MPa) values (SD) of a self-adhesive resin cement to CAD-CAM lithium disilicate glass ceramic systems immediately (T0) and after 30 days (T1).

Ceramic system	T0	T1
IPS e.max CAD	17.89 (6.3) Aa	17.11 (5.9) Aa
Rosetta SM	17.27 (3.0) Aa	17.60 (4.7) Aa

*Same uppercase letters indicate no significant difference between groups in rows; same lowercase letters indicate no significant difference between groups in columns; two-way ANOVA and Tukey HSD test (p < 0.05).
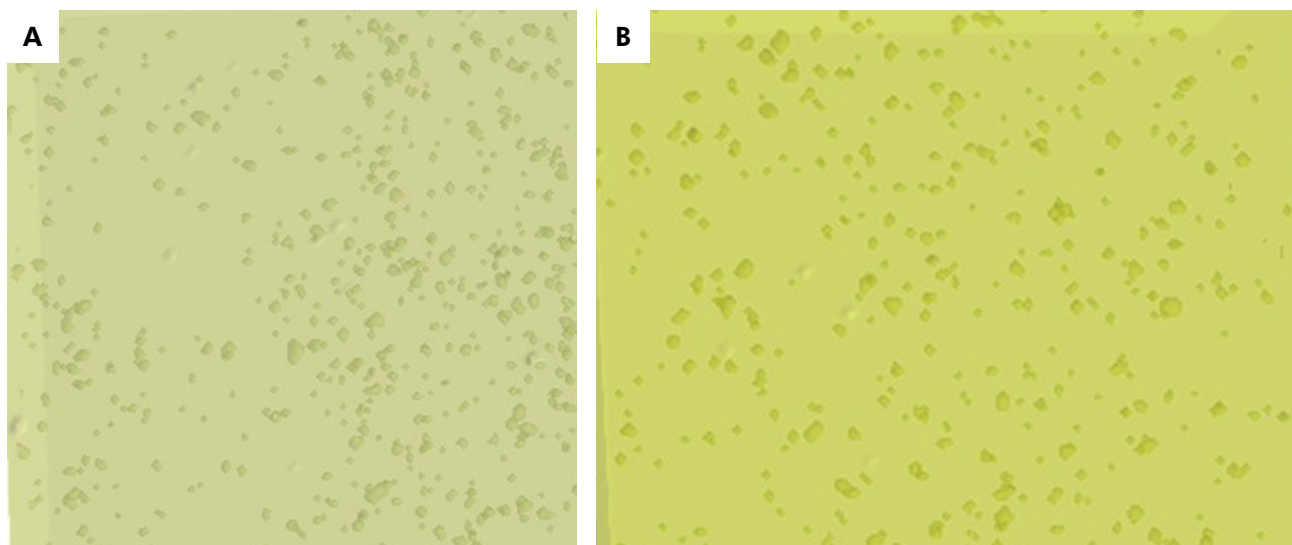

Figure 3. 3D reconstruction: A: IPS e.max CAD group using micro-CT scan; B: 3D reconstruction of Rosetta SM group using micro-CT scan

Table 4. Failure mode percentage (%) following microshear bond strength test for CAD-CAM lithium disilicate glass ceramic systems immediately (T0) and after 30 days (T1).

Group	Adhesive failures	Cohesive failures	Mixed failures
Rosetta T0	60%	0%	40%
Rosetta T1	70%	0%	30%
E.max T0	100%	0%	0%
E.max T1	80%	0%	20%

Discussion

The null hypothesis tested was accepted, since both CAD-CAM lithium disilicate glass-reinforced ceramics tested presented similar crystalline structures and morphological and mechanical properties. Lithium disilicate ceramics are commonly used for dental purposes, owing to their favorable properties, such as good fracture strength and satisfactory aesthetics.[4] The company that first developed and patented this material held the exclusive right to its production, limiting the option for the lithium disilicate-based ceramics available in the market. After the patent expired, other companies started to produce glass dental ceramics reinforced with lithium disilicate as well. However, little knowledge is available on whether these recently introduced lithium disilicate-based ceramics have the same quality as the precursor system first introduced.

Another important advantage reinforcing the overwhelming clinical approval of lithium disilicate ceramics is that they can be milled by simplified fabrication methods, such as laboratory and chairside CAD-CAM systems.[4,5,6] The lithium disilicate ceramics tested have been reported to have more lithium metasilicate when treated at temperatures below 780°C. Conversely, when temperatures above 780°C are used, three strong peaks (23.9, 24.6, and 30.1) of lithium disilicate have shown a greater amount of crystallization in previous studies.[19] An investigation using XRD revealed that the transformation of lithium metasilicate into lithium disilicate was dependent on the heating temperature, irrespective of the overall heating time.[8] In the present study, XRD results confirmed that the main crystalline components of Rosetta SM were similar to those of IPS e.max CAD, at the pre-crystallized and crystallized stages (Figure 1). This finding indicates that the ceramic materials evaluated presented close conversion of lithium metasilicates into lithium disilicate crystals when submitted to the heat treatment program
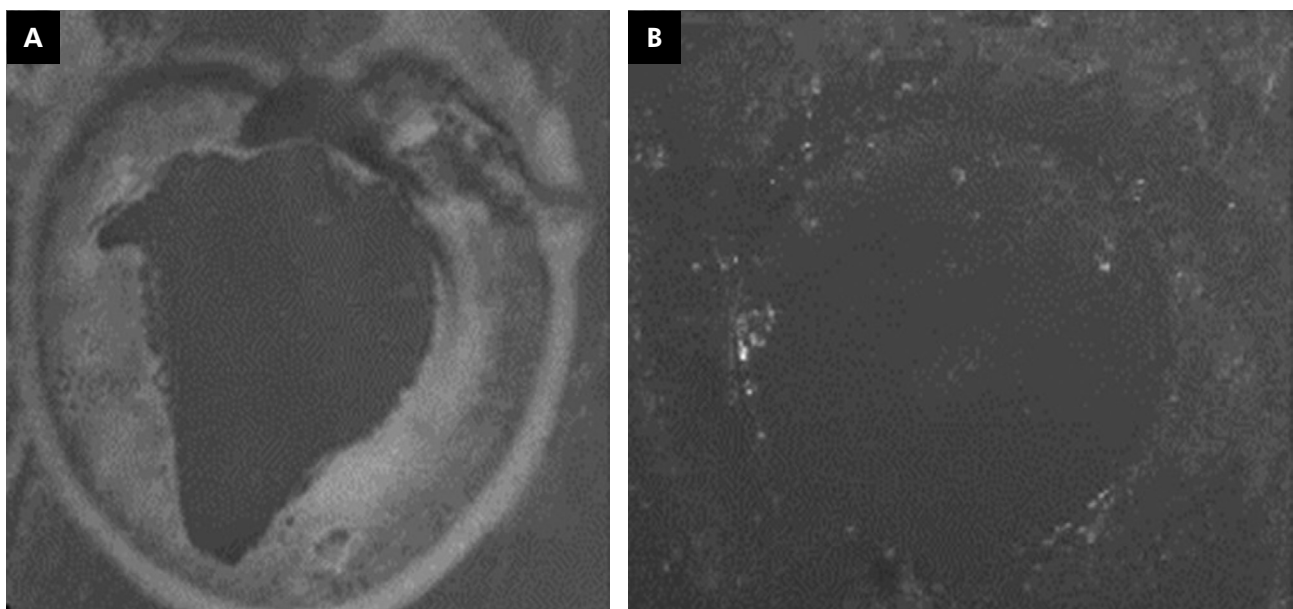

Figure 4. Optical microscopy images: A: Mixed failure mode after microshear bond strength testing. 40× magnification; B: Adhesive failure mode after microshear bond strength testing. 40× magnification.

used in this investigation, which is the same as that suggested by the processing instructions of both manufacturers.

The morphological analysis provided by SEM enabled evaluation of the surface topography of the dental ceramics tested. After the heat treatment (crystallized stage), the crystalline microstructures became denser in both ceramic systems tested, further indicating close conformation of the lithium disilicate crystals (Figure 2). A previous study has shown that these microstructures can become even denser and more homogeneous when restorations are subject to masticatory forces.[2] Lithium disilicate crystals have an average length of 0.5 μm in their pre-crystallized phase, and the average crystal size increases up to 3.0 μm after crystallization, as observed in the present study (Figure 2).[8,12,20]

The μ-CT scanning showed that both ceramics tested presented similar total porosity (p = 0.473). Pores were observed in the specimens of both ceramics (Figures 3A and 3B), and this porosity may interfere in the mechanical properties of these materials. The crystals present in dental ceramics have isotropic characteristics that play a significant role in modifying their properties, such as material hardness, flexural strength, modulus of elasticity and fracture toughness.[3,21] However, the presence of the pores may interfere with stress distribution, since they act as stress concentrating areas, and influence the mechanical proprieties by favoring mechanical failures. Therefore, it was clear that the porosity found similarly in both ceramic materials had to be reduced in order to optimize the microstructure of the different lithium disilicate ceramics evaluated.

High flexural strength is commonly observed for lithium disilicate glass-reinforced ceramics, compared with conventional feldspathic porcelain or leucite glass-reinforced ceramics.[8] The flexural strength of ceramic materials is hard to determine, because multiple factors may influence its measurement, such as polishing procedures, stress rates, testing method and environmental conditions, as well as specimen dimensions.[22-24] The results of the present study showed similar mean flexural strength values (±346.92MPa) for both ceramics evaluated (p = 0.652),

corroborating previous investigations that tested the same materials.[2,8]

The bond strength of resin cements to lithium disilicate ceramics is an important factor for the longevity of these dental restorations.[17] The stress transferred through the ceramic restoration to the remnant tooth hinges on an adequate bonding interface, which may prevent failures.[18] According to the present study, no differences were found between the values for the bond strength of resin cement to both lithium disilicate ceramics (p = 0.881). The mean values for the bond strength of the resin cement to the ceramic substrates found in this study corroborate those of previous reports,[3] and are almost similar to each other (± 17.0 MPa). The specimens were stored in distilled water, which may cause bond degradation; however, the oral environment is even more challenging for ceramic/resin cement bonding interfaces.[25] Even after 30 days of water storage, no differences were observed for the bond strength values of the two ceramics (p = 0.712).

Additionally, no cohesive failures were found in this study, demonstrating that the microshear bond strength test was probably performed correctly, despite the limitations involving laboratory adhesion tests to dental ceramics. The most prevalent failure modes detected were adhesive and mixed failures for both ceramic materials, irrespective of the storage time. The IPS e.max CAD system showed more adhesive failures in the immediate testing period (T0), whereas both ceramic systems showed similar failure distribution after 30 days of storage (T1). Data obtained from laboratory bond strength assessments should be analyzed with caution, and the direct relationship to clinical outcomes is limited. Thus, this study has focused on discussing only the bond strength and failure modes between the resin cements tested, as well as different ceramic materials under distinct storage conditions, without extrapolating the results to clinical situations.

As seen, lithium disilicate-reinforced ceramics are one of the most important and versatile materials for dental rehabilitations, because of their excellent thermal and physical stability, as well as good resistance and aesthetic properties. Despite the limitations of an *in vitro* study, the results showed that both ceramics showed

similar characteristics in the parameters tested, thus increasing the alternatives made available to laboratory technicians and dental practitioners.

Conclusions

Based on the results of the present study, both CAD-CAM lithium disilicate glass-reinforced ceramics tested showed similar characteristics, considering that they presented comparable crystalline structures with similar intensities, total porosity, flexural strength and bond strength.

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