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Effect of fatigue protocols on flexural strength of lithium disilicate bars with clamped-ends

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A B S T R A C T

The aim of this study was compare the effect of two in vitro ageing protocols to intraoral aging on the flexural strength of a lithium disilicate (LD) ceramic bars with clamped ends. After polishing and crystallization, the both ends of the bars were cemented to a metallic device and subjected to mechanical cycling, thermomechanical cycling, or intraoral ageing. Ten volunteers used an intraoral device - similar to an occlusal splint with a balanced contact condition on the occlusal surface of the ceramic bar - during 8 h night time / 30 days. Both in vitro and intraoral ageing decreased the flexural residual strength of LD, with the lowest values obtained after intraoral ageing. Thus, the in vitro ageing protocols tested in this study revealed to be less deleterious than intraoral ageing of LD.

1. Introduction

Lithium disilicate (LD) ceramics are composed by glass matrix ceramic with crystalline components, which provides better aesthetics when compared to crystalline ceramics, and higher strength than feldspathic ceramics. Due to good mechanical strength (flexural strength around 360 MPa) (Wiedhahn, 2007; Lien et al., 2015), lithium disilicate are indicated for diverse clinical situations, since veneers (Ritter, 2010) to even 3-unit fixed dental prosthesis (FDP) with abutments in the 1st pre-molar and 1st molar (Plengsombut et al., 2009; Wolfart et al., 2007, 2009).

In short periods of observation (until 48 months), LD clinical success rates are reported as 100% (Suputtamongkol et al., 2008; Esquivel-Upshaw et al., 2013; Wolfart et al., 2009). However, for multi-unit FDP, the survival rate reported after 37 months of observation was 89% (Wolfart et al., 2009), and decreasing to 63% after 72 months (Makarouna et al., 2011). Lithium disilicate FDP are indicated for the anterior region until the 2nd pre-molar, but failure rates are high in this configuration even when following the indication.

As a brittle material, LD is susceptible to fatigue (Zhang et al., 2013) by defect growth and their resulting cracks when subjected to loads under the critical value (Quin, 2007). The defects/cracks, which were not critical under a relatively low load, i.e. chewing load, grow until reaching a critical size for the applied load, and lead the material to fracture. Water plays an important role in crack propagation; the water molecules attack the silicate/oxide bonds at the crack tip, leading them to rupture and extending the crack (Quin, 2007).

Clinically, restorations are subjected to thermal and pH variations, and cyclic load application (Palmer and Barco, 1992; Youngson and Barclay, 2000). *In vitro* fatigue tests have the role of approximating *in vivo* ageing conditions, similar to chewing activity in oral temperature, or with temperature variation (Oyafuso et al., 2008; Vásquez et al., 2009; Komine et al., 2004; Stappert et al., 2008). The test setting is also important: for brittle materials, flexural strength is determinant for the clinical endurance of restorations, since these materials present lower tensile strength than compression strength (Della Bona and Anusavice, 2002; Della Bona et al., 2003). The three-point bending test is largely applied (Della Bona and Anusavice, 2002) for measuring a ceramic's strength, but when compared to a FDP, a bent bar is only supported by the inferior rods, while the FDP restoration is cemented at both ends on the abutment teeth. The flexural moment generated in both situations is different, and might influence the flexural properties of the ceramic.

Thus, the aim of this study was to compare the effect of two *in vitro* ageing protocols to intraoral aging on the flexural strength of a lithium disilicate ceramic bar with clamped ends. The null hypothesis is that the tested ageing protocols will not decrease the flexural strength of lithium disilicate bars.

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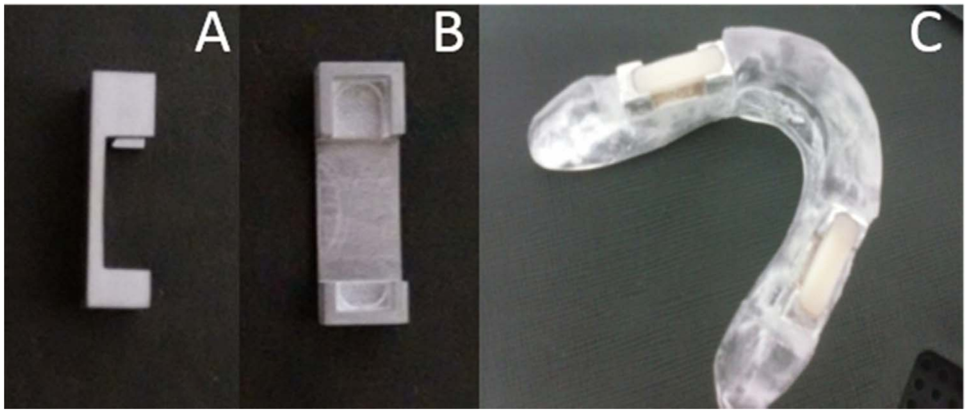

Fig. 1. Metallic support. A: lateral view; B: top view, with the spaces for cementation of the ceramic bars; C: intraoral device with the sample (metallic device + ceramic bar).

2. Material and methods

This research evaluated three methods of ageing applied to lithium disilicate ceramic. It was approved by the ethical committee of the Institute of Science and Technology – Sao Paula State University (# 45703315.0.0000.0077).

LD blocks (IPS emax CAD, Ivoclar Vivadent) were sectioned in a precision cutting machine (ISOMET 1000, Buehler Ltd.) with a diamond disc to obtain ceramic bars (2 × 4 × 16 mm - ISO 6872/2008). The bars were polished (sandpaper 320- to 1200-grit size) under water cooling, and 1 mm chamfers were performed on the bars' edges. Bars were then crystalized (1100 °C, 45 min) in a specific furnace (Programat EP 3000, Ivoclar Vivadent). Bars were divided into 3 groups (n = 20) according to fatigue procedures: control (C); mechanical cycling (MC); thermo mechanical cycling (TMC); and intraoral aging (IO).

A specific device (Fig. 1A and B) was manufactured in stainless steel with rectangular configuration (16 × 4.3 × 2.3 mm). The device presented a socket on each end (2.3 mm deep, 4.3 mm width) for cementation of the ceramic bar ends, with 0.3 mm space for cement layer. The length of these sockets was 4 mm and 2 mm, thus simulating the support provided by a molar and a pre-molar, respectively, with a 10 mm *span*.

Both ends of the lithium disilicate bars were etched with 10% hydrofluoric acid (Condac Porcelain, FGM) for 20 s, subsequently dried and received silane application (Rely X Ceramic Primer, 3 M ESPE). The sockets of the metallic device were air abraded with silica modified aluminum oxide particles (110 µm, Rocatec Plus, 3 M ESPE). A dual cured resin cement (Variolink Dual II, Ivoclar Vivadent) was applied onto treated surfaces of the metallic device, and ceramic bars were attached to the respective sockets. Excess cement was removed and the assembly received light activation (Radii Cal, SDI) for 40 s on each end.

Samples were then subjected to the following ageing protocols: *Control (C)* – no ageing: after cementation, samples were stored for 24 h in water at 37 °C and tested for flexural strength; *Mechanical Cycling (MC)* – samples were subjected to mechanical cycling (Erios Equipamentos) with axial load (45 N) applied to the center of the bar by a loading nose - a total of 1.2×10^6 cycles at 3.8 Hz. After aging, samples were tested for flexural strength; *Themo Mechanical Cycling (TMC)* – samples were simultaneously subjected to mechanical cycling as previously described and to thermal cycles. Samples under load application received water irrigation (2550 thermal cycles) between 5 °C and 50 °C, with 30 s of irrigation time in each temperature and a 15 s interval. After aging, samples were tested for flexural strength; *Intraoral Ageing (IO)* – 10 volunteers were selected from the undergraduation and graduation courses of the Institute of Science and Technology of the Sao Paulo State University (10 women, 25 years old). Inclusion criteria were: good general health, absence of caries, lesions and periodontal disease; volunteers should also be classified as occlusion Class I according to Angle's classification; and did not currently have/use any orthodontic devices or removable dentures. Parafunction was not considered exclusion criteria. Volunteers were informed about the research and they signed a consent term. Oral impressions were made from the superior and inferior arch of each volunteer with alginate (Hydrogum, Zhermack) and plaster models were produced (Durone IV, Denstply). Models were set to an articulator (Bio Art Dental equipment, São Paulo, Brazil) and a superior occlusal splint was fabricated for each volunteer with thermal cured acrylic resin (JET, Dental Product Crassic Ltda). Two samples were attached to each superior molar region, 4 mm high (Fig. 1C) inside the free functional space (Okeson, 2000). Occlusal adjustment was performed on the occlusal splint to obtain homogeneous bilateral contacts (Fig. 2).

Volunteers were oriented regarding hygiene and the use of intraoral devices for 8 h during night time for 30 days, thus yielding a total of 240 h. Volunteers gave back the intraoral devices after 30 days, when the samples were removed and tested for flexural strength.

Maximum biting force was also measured for each volunteer: a digital dynamometer was positioned on the occlusal surface of all teeth of each volunteer, who were then requested to bite with maximum force. Each volunteer was requested to repeat the procedure for 3 times with a 1-min interval. Maximum biting forces were recorded in Newtons for posterior analysis.

For flexural strength test, a compressive load was applied (0.5 mm/

Fig. 2. Intraoral device installed in one of the volunteers, with occlusal adjustment performed. A: frontal view; B: right lateral view and; C: left lateral view, showing the occlusal contact of the bars with antagonist pre-molars.

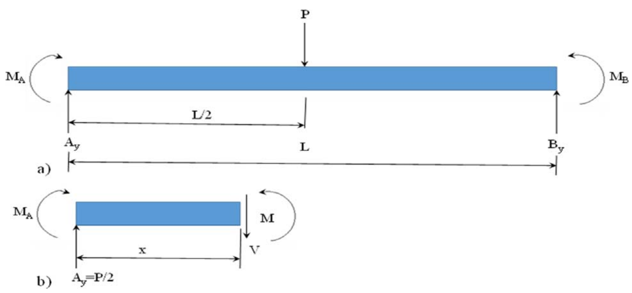

Fig. 3. Diagram of free body of clamped ceramic.

min), by a universal testing machine (DL-1000, EMIC, Equipments and Systems Ltda.), to the upper surface of the ceramic bar cemented to the metallic device until its fracture. The maximum load-to-failure (N) was recorded. Flexural strength (MPa) was calculated according to the diagram of a free body (Fig. 3). The problem is statically undetermined, which requires relating the elastic line equation, including flexural formula and Hook's law, as seen in Eq. (1).

$$\frac{d^2\vartheta}{dx^2} = \frac{M(x)}{EI} \tag{1}$$

where υ is the elastic line, x is the reference of the elastic line deformation, M is the applied moment, E is the bending modulus, and I is the inertia moment. By applying the sum of the moments in the right side of Fig. 3 and replacing it in Eq. (1), it yields Eq. (2) by successive integration. Then the maximum moment is obtained by replacing the distance L/2.

$$M(x) = \frac{P}{2}x - \frac{PL}{6} \tag{2}$$

Therefore, flexural strength is given by Eq. (3) by substituting the maximum moment in the flexural strength formula in which y is the coordinate from the neutral line. Hence, the maximum flexural strength is obtained at the outer surface of the beam given by y = -b/2.

$$\sigma_f = -\frac{M_{max}Y}{I} \tag{3}$$

Substituting all the literal dimensions of the beam, the maximum flexural strength can be calculated by Eq. (4).

$$I = \frac{bd^3}{12} \tag{4}$$

$$\sigma_{fmax} = \frac{1}{2}\frac{PL}{bd^2} \tag{5}$$

Where P is the applied force, L is the span length, and d is the specimen thickness.

After the flexural strength test, the fractured surfaces of ceramic bars were observed via scanning electron microscopy (Inspect S50, Fei Company) with 1000 × magnification in order to investigate the fracture origin site. Data obtained from the flexural strength test were tested for normal distribution and homoscedasticity, and also subjected to one-way analysis of variance (ANOVA) followed by Tukey post-hoc test ($\alpha = 0.05$).

3. Results

The ageing protocols influenced the flexural strength of clamped LD bars ($p < 0.001$). The control group presented the highest values of flexural strength (Table 1). Groups subjected to mechanical cycling and thermo mechanical cycling presented similar intermediate results of flexural strength. Intraoral ageing was the most degrading procedure, providing the lowest values of flexural strength. Average maximum biting force recorded from the volunteers was 59 ± 12 N.

No ceramic bar debonded during ageing or strength test. Ceramic bars were posteriorly mechanically removed from supports for fracture analysis: all groups presented bars fractured into two fragments with the origin at the tensile side of the bar, and they subsequently propagated until the compression side of the bar forming the compression curl. In addition, a second origin took place with less fracture signs displaced from the center and at the compression side of the bar (Fig. 4).

Table 1
mean ± standard deviation of residual flexural strength (MPa) obtained for each tested group, with statistical significance given by Tukey's test. Identical superscripted letters indicate statistical similarity.

Group	Mean ± Standard deviation (Mpa)
C	257.50 ± 29.79^{A}
MC	202.09 ± 29.59^{B}
TMC	191.12 ± 31.02^{B}
IO	159.20 ± 18.32^{C}

4. Discussion

This study evaluated the effect of three ageing protocols on the flexural strength of LD. The null hypothesis was rejected. Ageing protocols decreased the maximum flexural strength of lithium disilicate. The control group with no ageing presented the highest flexural strength – 257.5 MPa (Table 1). This material contains 65% volume fraction of lithium disilicate (Guazzato et al., 2004), and the cracks mainly propagate into the 34% volume fraction of residual glass (Guazzato et al., 2004) and deflect when they reach crystals (Apel et al., 2008). Crack deflection requires more energy and results in enhanced strength measured for the material (Quin, 2007). Since there is no previous crack growth (i.e. as promoted by ageing), the strength values are maximum.

Ageing was simulated by mechanical cycling, which was performed by applying a 45 N load to the lithium disilicate bars (Oyafuso et al., 2008; Komine et al., 2004; Stappert et al., 2008), similar to the load applied in intraoral ageing during night time (Okeson, 2000). The application of loads bellow the critical load can promote crack nucleation and slow crack growth in brittle materials (Quin, 2007). The cracks grow and reach critical size, thus leading the ceramics to fracture in fatigue (Studart et al., 2007), or as in the present research, these propagated cracks represent the initial flaws, much larger than those present in the control group.

The combination of mechanical cycling and thermal ageing was also performed as ageing simulation. This protocol comprises the simultaneous application of both cyclic load and thermal variation (in water) and is less employed in literature. It was reported to decrease fracture strength of aluminum oxide ceramic posterior crowns, cemented with resin cement (Komine et al., 2004). Values presented decrease when compared to mechanical cycling only (Table 1), however, this decrease was not statistically significant. Ceramics and glasses are susceptible to thermal shocks caused by the different water temperatures (5–55 °C), which may cause strains between portions of the bar (Quin, 2007). Sudden cooling of the bar surface can generate substantial tensile stresses (Quin, 2007), decreasing strength or even leading to fracture. Additional cycles could have evidenced this thermal effect (Mollazadeh et al., 2015).

The intraoral ageing protocol decreased the flexural strength of LD to the lowest values (Table 1). The mean maximum biting force registered for an individual using the IO device was 59 N, but different and non-measurable stresses received by samples when in a human mouth for 8 h/30 days may have contributed to the decrease in strength; chippings and fractures of the ceramic were only observed in the intraoral ageing. Furthermore, the alkaline pH of saliva during night-time is deleterious to the mass loss of glass based systems, breaking up the silica glass framework (Esquivel-Upshaw et al., 2013), leading to a

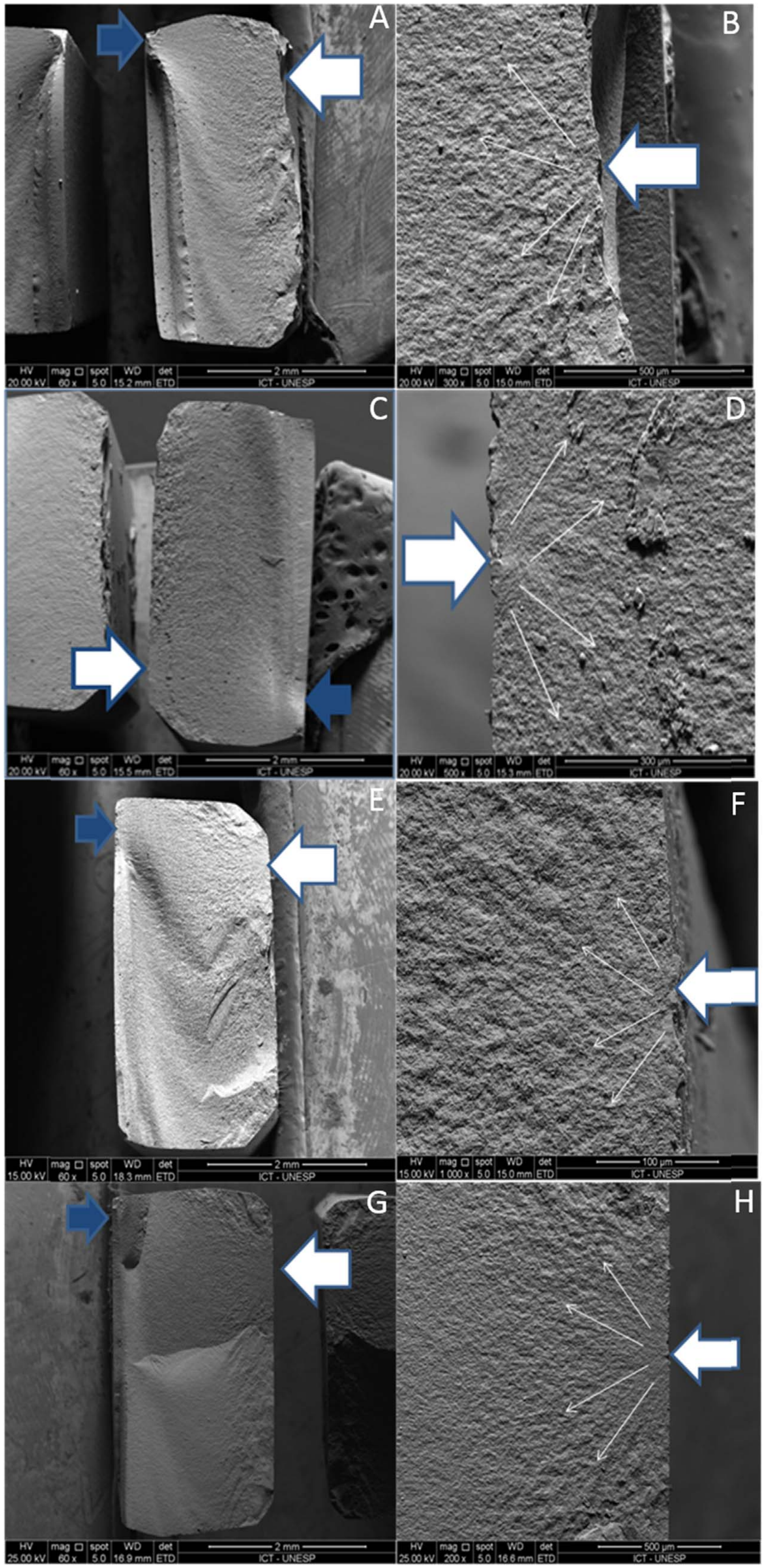

Fig. 4. Images of SEM of the fractured surface of the sample from (A,B) control, (C,D) mechanical cycling, (E,F) thermomechanical cycling, and intraoral ageing (G,H) groups. Left: characteristic signs of the fracture origin: compression curls on the left side, main origin indicated by the white arrow, and a second event possibly indicating torsion, pointed by the blue arrow. Right: closest view of the fracture origin, thin white arrows indicate the origin of fracture propagation (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.).

decrease in strength. The uncontrolled intraoral loads and alkaline pH may interact with intrinsic defects and decrease the flexural strength of the material, which facilitate nucleation and crack propagation. The load incidence in an oblique direction and in multiple points in intraoral ageing may have also contributed to the lowest values of residual strength. The protocol applied for the intraoral ageing is experimental, since no clinical data is available in literature regarding the degradation suffered by dental ceramics. The group of volunteers is homogeneous and represent a very specific population. More embracing and well designed clinical trials are necessary to specify the clinical significance of *in vitro* ageing protocols.

Fracture analysis of all groups have similar patterns (Fig. 4) with failure origin located at the tensile side, which is similar to previous studies (Quin, 2007). A second fracture origin was observed in the compression side (small blue arrow, Fig. 4). This second fracture origin presents less defined signs of fracture, such as no compression curl on the opposite side of the sample, or less sharp hackle lines, and may be resultant of a torsion effect in the final stages of fracture, being associated to the bar's clamped-end characteristic.

5. Conclusion

The mechanical and thermomechanical cycling in vitro ageing protocols decreased the flexural strength of lithium disilicate bars when compared to non-aged specimens. Intraoral ageing promoted the lowest values of flexural strength for clamped-end LD bars. Thus, the *in vitro* ageing protocols tested in this study revealed to be less deleterious to the ageing effect than night-time intraoral ageing, considering the limitations of this study.

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