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



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#### ABSTRACT

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 ( $2 \times 4 \times 16 \text{ mm}$  - 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  $(16 \times 4.3 \times 2.3 \text{ mm})$ . 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  $\mu$ m, 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  $1.2 \times 10^6$  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.

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