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# Influence of substrate, cement and aging on the biaxial flexural strength of lithium disilicate

Influência do substrato, cimento e envelhecimento na resistência a flexão biaxial do dissilicato de lítio

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## ABSTRACT

**Objective:** To evaluate the biaxial flexural strength (BFS) of lithium disilicate (L), cemented on different substrates (epoxy resin - E and metal - M) with dual-cure resin cement (Rc) and zinc phosphate cement (Zc), not aged, thermally aged (TC) or thermo-mechanical aged (TC/MC). **Material and Methods:** Disks of L, E, and M were fabricated, and the cementation was performed according to the following groups: ERc (L+E+Rc); MRc (L+M+Rc); MZc (L+M+Zc); EZc (L+E+Zc). Ten samples from each described group were tested in BFS, ten more samples were subjected to TC ($1\times10^4$ cycles between 5 °C and 55 °C water), and the last 10 samples were subjected to TC/MC (MC: $1.2\times10^6$ cycles, 50 N, 3.8 Hz). The BFS test was performed and scanning electron microscopy (SEM) was performed to evaluate the failure mode. The effect of the cementation strategy (cement/substrate) was compared in each aging method and the effect of the aging method was evaluated for each cementation strategy by one-way ANOVA and Tukey post-hoc test ($\alpha$=0.05). **Results:** The strength values were highest to M (237.8 ~ 463.9 MPa), in comparison to the E (41.03 ~ 66.76 MPa), despite aging and luting agent. Flexural strength data decreased after TC and TC/MC in groups cemented with Zc, but was stable when cemented with Rc. SEM analysis indicated that failure origins were located at the tensile surface of the L. **Conclusion:** Lithium disilicate discs cemented to the metallic substrate presented the highest biaxial flexural strength. The cementation with dual-cure resin cement did not decrease BFS after aging.

## KEYWORDS

Aging; Cementation; Ceramics; Flexural strength; Lithium disilicate.

## RESUMO

**Objetivo:** Avaliar a resistência à flexão biaxial (BFS) do dissilicato de lítio (L), cimentado sobre diferentes substratos (resina epóxi - E e metal - M) com cimento resinoso dual (Rc) e cimento de fosfato de zinco (Zc), não envelhecido, submetido ao envelhecido térmico (TC) ou ao envelhecido térmico-mecânico (TC/MC). **Material e Métodos:** Foram confeccionados discos de L, E e M, e a cimentação foi realizada de acordo com os seguintes grupos: ERc (L+E+Rc); MRc (L+M+Rc); MZc (L+M+Zc); EZc (L+E+Zc). Dez amostras de cada grupo descrito foram testadas em BFS, mais dez amostras foram submetidas à TC ($1\times10^4$ ciclos de imersão em água entre 5 °C e 55 °C), e as últimas 10 amostras foram submetidas à TC/MC (MC: $1.2\times10^6$ ciclos, 50 N, 3.8 Hz). Foram realizados os testes de BFS e a microscopia eletrônica de varredura (MEV) para avaliar o modo de falha. O efeito da estratégia de cimentação (cimento/substrato) foi comparado em cada método de envelhecimento e o efeito do método de envelhecimento foi avaliado para cada estratégia de cimentação por ANOVA a um fator e teste post-hoc de Tukey (α=0,05). **Resultados:** Os valores de resistência foram maiores para M (237.8 ~ 463.9 MPa), em comparação com E (41.03 ~ 66.76 MPa), independentemente do envelhecimento e do agente cimentante utilizado. Os dados de resistência à flexão diminuíram após TC e TC/MC nos grupos cimentados com Zc, mas se mantiveram estáveis quando cimentados com Rc. A análise MEV indicou que a origem das falhas estava localizada na superfície de tração do L. **Conclusão:** Os discos de dissilicato de lítio cimentados ao substrato metálico apresentaram maior resistência à flexão biaxial. A cimentação com cimento resinoso dual não diminuiu o BFS após o envelhecimento.

## PALAVRAS-CHAVE

Cerâmica; Cimentação; Dissilicato de lítio; Envelhecimento; Resistência à flexão.

## INTRODUCTION

As an aesthetic smile has an important role in life quality and personal relationships, metal-free restorations, such as those made with ceramic materials, are increasingly used. Even with the improvement in ceramic's physical properties, some of these materials are still subject to splints and cracks [1]. For these reasons, the vitreous lithium disilicate ceramic (L) is now widely used, since it can resist stressing conditions, such as masticatory forces, mimics the natural tooth color, and has a wide indication [2].

Currently, the L restorations are adhesively luted on the tooth structure (enamel or dentin) presenting satisfactory bond strength to the tooth structure [3]. Adhesive luting agents are usually compared to conventional cement, such as zinc phosphate [4], since it is considered a standard luting agent [5] due to the lengthy clinical history [6]. In addition, L can be a suitable material for restorations over metal substrates, such as cast metal post/cores, or metal components of implant prostheses [2], but the behavior of the L when luted with different agents on metallic substrate needs to be more explored.

The luting agent used is an important component for restoration longevity, since it can create a link between the dental substrate and the ceramic, reducing the stress, protecting the substrate from saliva absorption, and reinforcing the ceramic strength [7]. As the L is a vitreous material, an adhesive cementation with resin cement is recommended, mainly because this material has silica and can be etched by the hydrofluoric acid, thus obtaining satisfactory adhesion due to the micromechanical and chemical bonds [8]. However, their use for the metallic substrate has some limitations, given the minimal chemical affinity between luting agents and metallic alloys [9], which stimulated the establishment of different protocols for metal to create micromechanical and chemical retention between those substrates. As an example, acid etching, air abrasion with aluminum oxide and the use of chemical components, such as metal primer [10].

To investigate the performance of ceramic restorations, studies were made evaluating their optical [11] and mechanical behavior [12], the luting process and how the cracks propagate in this material. However, information about the longevity of the L restorations luted with different type of luting agents when submitted to aging or fatigue is rare in the literature.

Within this context, the aim of this study was to evaluate the biaxial flexural strength of lithium disilicate ceramic discs, luted on different substrates (epoxy resin and metal) with dual-cure resin cement or conventional zinc phosphate cement, thermally or thermo-mechanically aged or not-aged. The null hypotheses tested were that the different luting agents, the type of substrate and the aging protocol would not influence the biaxial flexural strength of lithium disilicate.

## MATERIALS AND METHODS

### Specimens preparation

Prefabricated lithium disilicate blocks (IPS e.max CAD, Ivoclar Vivadent, Schaan, Liechtenstein) were cut with a Diamond trephine drill into 12 mm diameter cylinders. Cylinders were sectioned (Extec High Concentration, Extec) into discs of 1.2 ±0.2 mm thickness, according to ISO 6872 [13], with a precision saw machine (Isomet 1000, Buehler, Plymouth, MN, EUA). All discs were polished (Politriz, Buehler) with increasing grit silicon carbide paper (400 to 1200 grit, Norton), obtaining 120 lithium disilicate discs that were randomly distributed into twelve groups (n = 10).

The crystallization process was made in specific furnace (Programat EP 3000, Ivoclar Vivadent) with a maximum temperature of 850ºC for 10 min as recommended by the manufacturer.

The epoxy resin (Nema Grade G10, International Paper, Hampton, USA), a material with the elastic modulus analogue to dentin [14], which had a cylindrical shape (12 mm), was also cut (Isomet 1000) and polished (Politriz) with increasing grit silicon carbide paper (400 to 1200 grit), obtaining 60 epoxy resin discs (1.2 ±0.2 mm thick) randomly distributed into six groups (n = 10). In previous study [15], the epoxy resin has been already used as a substrate to evaluate the biaxial flexural strength of a ceramic material.

The 60 metal discs were initially waxed (GEO Crowax, Renfert) and fused with a Co-Cr (cobalt-chrome) alloy (DeguDent Ind. And Com. Ltda.). After polishing with sandpaper #120 #400 and #600, the final thickness of the discs was 1.2 ±0.2 mm. All metal discs were sandblasted with aluminum oxide (50$\mu$m, BioArt) on the cementation surface. Then they were randomly distributed into six groups (n = 10). All materials used in the present study, respective commercial information, and elastic modulus [14,16-18] are described in Table I.

The groups were defined according to the type of luting agent (dual-cure resin cement - Rc or conventional zinc phosphate – Zc), substrate (epoxy resin - E or metal - M), initial testing, thermal cycling (TC) and thermal cycling followed by mechanical cycling (TC/MC). Twelve groups were formed: ERc; ERc-TC; ERc-TC/MC; EZc; EZc-TC; EZc-TC/MC; MRc; MRc-TC; MRc-T/MC; MZc; MZc-TC; and MZc-TC/MC.

## Cementation process

Before any surface treatment, all discs (ceramic, metallic and epoxy dentin) were cleaned for five minutes in ultrasonic bath (Cristófoli Ultrasonic Washer) with isopropyl alcohol. The surface treatments performed on each material surface according to luting agent are described in Table II.

For cementation, the dual-cure resin cement was mixed, placed on the center of the lithium disilicate treated surface, and immediately bonded to the treated surface of the substrate material disc (E or M). A 750-g load was applied for 60 s to the top of lithium disilicate disc to allow cement excess removal and to obtain

**Table I -** Description of materials used

| Type of material<br>Elastic modulus | Trade mark/ Manufacturer | Composition |
|---|---|---|
| Lithium disilicate 95 GPa [18] | IPS e-max CAD / Ivoclar Vivadent, Schaan, Liechtenstein | $SIO_2$, $LI_2O$, $K_2O$, $P_2O_5$, $ZRO_2$, ZNO, $AL_2O_3$, MGO |
| Dual Cure Resin Cement 8.3 GPa [18] | Panavia F/ Kuraray,Tokyo, Japan | *Paste A:* MDP; Aromatic dimethacrylate; Silanized silica; Catalysts and Initiators.<br>*Paste B:* 124/5000<br>Aromatic dimethacrylate; Particles of silanized barium glass; Sodium fluoride; Catalysts; Accelerators and Pigments |
| Conventional zinc phosphate cement 22.4 GPa [16] | Cement LS / SS White, Rio de Janeiro, Brazil | Powder: zinc oxide (90%) and magnesium oxide (10%);<br>Liquid: orthophosphoric acid, water, aluminum and zinc. |
| Metal substrate 203 GPa [17] | Fit Flex / Degudent Dentsply, São Paulo, Brazil | cobalt – chrome alloy |
| Epoxi Resin substrate 18 GPa [14] | NEMA Grade G10 / St. Louis, Missouri, EUA | Epoxi resin |
| Hydrofluoric etching acid | Condac Porcelain 5% / FGM, Joinville, Brazil | 5% hydrofluoric acid |
| Phosphoric etching acid | Condac 37% / FGM, Joinville, Brazil | 37% fluoridric acid |
| Aluminum oxide particles | Kota Knebel / KOTA, Cotia, Brazil | 320μm aluminum oxide particles |
| Silane bonding agent | Monobond S / Ivoclar Vivadent, Schaan, Liechtenstein | Silane methacrylate alcohol solution |
| Metal Primer | Alloy Primer / Kuraray, Tokyo, Japan | VBATDT, 10-MDP and acetone |

**Table II -** Surface treatments applied to each material according to the cement used

| Cement | Material | Surface treatment |
|---|---|---|
| Dual cure resin cement (Rc) | Lithium disilicate (L) | 5% hydrofluoric acid etching (20s) + washed by air-water spray (40s) + air dried (30s) + silane (30s) + air dried (30s) |
| | Epoxi resin (E) | 37% phosphoric acid etching (15s) + washed by air-water spray (30s) + air dried (30s) + mixture of adhesive primers (A and B) from resin cement |
| | Metal (M) | Sandblasting with aluminum oxide particles (10mm, 45°, 2.8 bar, 15s) + application of the metal primer |
| Conventional Zinc Phosphate Cement (Zc) | Lithium disilicate (L) | No treatment |
| | Epoxi resin (E) | No treatment |
| | Metal (M) | Sandblasting with aluminum oxide particles (10mm, 45°, 2.8 bar, 15s) |

uniform distribution of cement throughout the interface. The excess of cement material was removed, and each side of the discs was light cured for 40 s with 1200 mW/cm$^2$ LED (Radii Cal, SDI). The light intensity was measured on a radiometer (Kondortech-Kondentech). After the bonding, all specimens were immersed in distilled water and stored at 37°C for 24 hours.

The cementation with the zinc phosphate cement was performed with carefully staged additions of powder to the liquid, on a glass slide, as recommended by manufacturer. At reaching the recommended consistency, after 1 min manipulation, the cement was placed on the lithium disilicate disc, and the cementation was performed as described before. In this case, samples (substrate, conventional cement, and ceramic) remained under the 750-g load for 10 minutes to respect this material chemical cure time.

Ten samples from each cementation strategy (n=10) were tested by biaxial flexural strength test, other ten samples from each cementation strategy (n=10) were subjected to $1\times10^4$ thermocycles (Nova Ética), between two water baths of 5 °C and 55 °C, with a time of 30s each. And the last ten samples from each cementation strategy (n=10) were subjected to TC as described before, followed by $1.2 \times 10^6$ mechanical cycles (ERIOS, Model: ER-11000), under 50N load, at 3.8 Hz. Samples were immersed in 37°C water during MC.

## Biaxial flexural strength test – BFS

The biaxial flexural strength test (n = 10) was performed in a universal testing machine (EMIC DL-1000, EMIC) according to ISO 6872 [13]. The dimensions of each sample were measured with digital caliper (model 500-195-20B, Mitutoyo America) before the test. Lithium disilicate disc was positioned on the top, for simulation of occlusal load on a flat occlusal restoration, and an increasing load (1 mm/min) was applied to the center of the disc with a piston (3-mm radius) until fracture of lithium disilicate – catastrophic failure. Biaxial flexural strength was calculated according to the equation described for computations for multilayers [13,19], as follows:

$$S = -0.2387\,\frac{P(X-Y)}{d^2} \tag{1}$$

In this formula, S (expressed in Pascals) means the maximum tensile stress; P (expressed in Newtons) means the amount of load needed to fracture the material and d means the specimen thickness (expressed in mm). X and Y were calculated based on the ceramic's Poisson's ratio (v), the radius of support circle (r1), of loaded area (r2) and of the specimen (r3) as follows:

$$X = (1+v)\,\mathrm{In}\left(\frac{r2}{r3}\right)^2 + \left[\frac{(1-v)}{2}\right]\left(\frac{r2}{r3}\right)^2 \tag{2}$$

$$Y = (1+v)\left[1+\mathrm{In}\left(\frac{r2}{r3}\right)^2\right] + (1-v)\left(\frac{r1}{r3}\right)^2 \tag{3}$$

The Poisson ratio considered were: 0.25 to L [18], 0.3 to epoxy resin [20] and 0.3 to metal [21]. Figure 1 is a schematic illustration of the sample dimensions and test setup.

## Statistics

Data were subjected to descriptive statistical analysis (mean and standard deviation). The effect of cementation strategy (cement/substrate) were compared in each aging method (no aging, thermocycling and thermocycling + mechanical cycling), and the effect of aging method was evaluated for each cementation strategy by one-way ANOVA followed by Tukey post hoc test ($\alpha$=0.05).

### Scanning Electron Microscopy (SEM)

Fractured surfaces were examined under an optical microscope (Discovery V20, Carl Zeiss Microscopy) with 100× magnification, for origin and failure propagation pattern identification. Representative specimens were sputter coated with gold and evaluated under scanning electron microscopy (SEM; Inspect S50, FEI) for illustrative images.

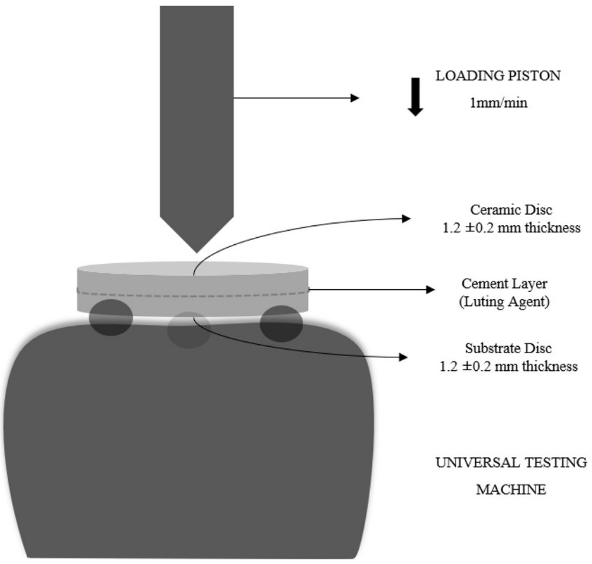


**Figure 1 -** Schematic illustration of the sample dimensions and test setup.

## RESULTS

Four samples from MZc-TC and three from EZc-TC groups failed during thermocycling, and were not considered for statistical analysis, because they were considered outliers (unrepresentative). Seven samples from MZc-TC/MC and nine samples from EZc-TC/MC failed after thermal and mechanical cycling; these groups were excluded from statistical analysis. Flexural strength data with statistical analysis and number of tested samples per group are showed in Table III.

The highest strength values were found in groups cemented to the metallic substrate, despite aging and luting agent. Flexural strength data decreased after TC and TC/MC in groups cemented with conventional zinc phosphate cement but was stable for lithium disilicate cemented with dual-cure resin cement.

No substrate disc presented fracture after test. Thus, the values presented were regarding the strength of lithium disilicate. The fractographic analysis showed that all lithium disilicate discs fractured from the cementation surface toward the piston contact point (Figure 2A – 1F).

## DISCUSSION

In the present study, the biaxial flexural strength of lithium disilicate ceramic was evaluated when cemented on different substrates with different protocols. Additionally, aging was performed to simulate stress conditions that occur in the oral cavity. The highest strength values were found in groups cemented to metallic

**Table III -** Means and respective standard deviations of biaxial flexural strength data (values in MPa) obtained for the specimens, according to the experimental group

| Substrate material | Cement | Without aging | With aging (TC) | With aging (TC + MC) | ANOVA 1-way (aging method) |
|---|---|---|---|---|---|
| E | Rc | 63.84 (17.09)$^{B}$ (n=10) | 64.73 (13.55)$^{B}$ (n=10) | 66.76 (15,80)$^{B}$ (n=10) | p=0.920 |
| | Zc | 53.15 (6.25)$^{B}$ (n=10) | 41.03 (10.09)$^{B}$ (n=6) | 65,25 (----) (n=1)* | p=0.013 |
| M | Rc | 321.9 (116.04)$^{A}$ (n=10) | 290.9 (81.20)$^{A}$ (n=10) | 330,05 (65,5)$^{A}$ (n=10) | p=0.598 |
| | Zc | 463.9 (161.70)$^{A}$ (n=10) | 237.8 (126.6)$^{A}$ (n=7) | 296,32 (151,50) (n=3)* | p=0.095 |
| ANOVA 1-way (cementation strategy – cement/substrate) | | p<0.001 | p<0.001 | p<0.000 | |

*Groups not included in statistical analysis. Different uppercase superscript letters indicate statistical difference in the same column.

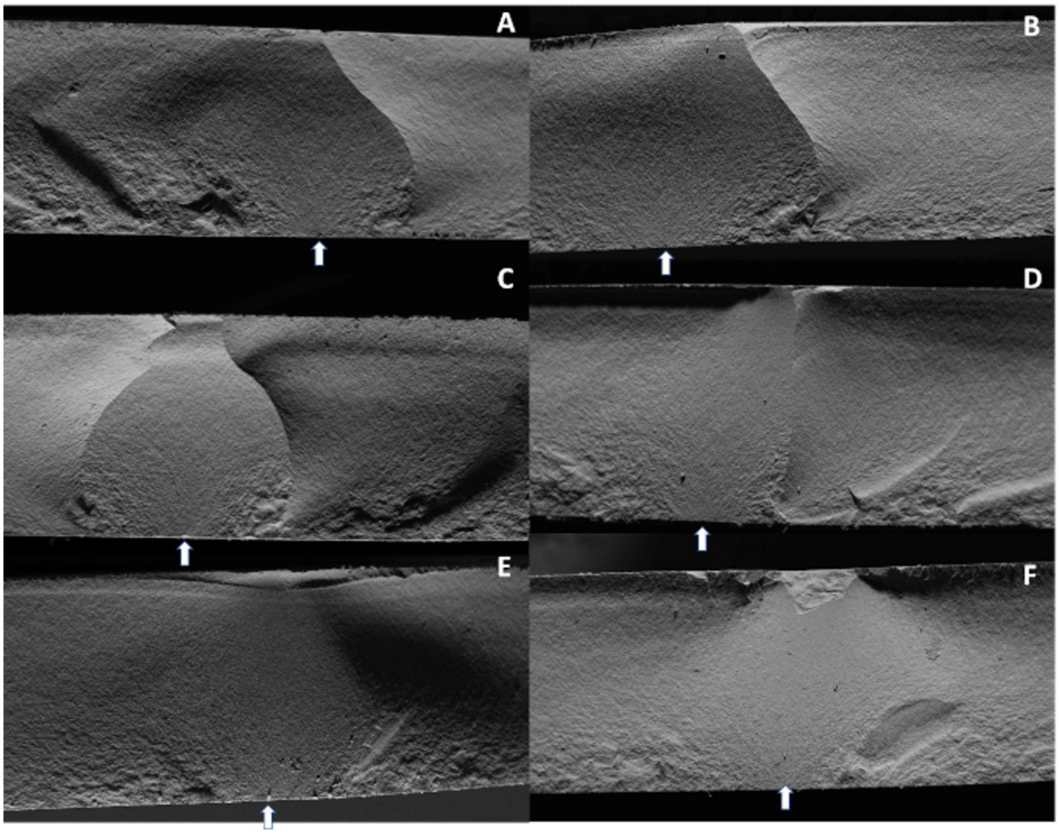


**Figure 2 -** A-F: Representative images of the fractographic analyzes (100× magnification) performed on samples indicating that the fracture originated on the tensile surface (white arrows). Figure 2A - ERc-TC; Figure 2B – EZc; Figure 2C - EZc-TC; Figure 2D – MRc; Figure 2E - MZc-TC and Figure 2F – MZc-TC.

substrate, despite aging and luting agent. Flexural strength data decreased after TC and TC/MC in groups cemented with conventional zinc phosphate cement but was stable for lithium disilicate cemented with dual-cure resin cement.

Previous literature suggested that the thermocycling reduced the adhesion between metal and ceramics [22]. In the present study, the samples format favored the contact of cement with water during the thermal aging which allows a potential degradation. But, in the groups conventionally cemented with zinc phosphate, lower strength values were presented because this material has higher solubility, no adhesion proprieties and lower mechanical property when compared with resin cements [23].

Despite the cement variable did not present statistically significant differences in the non-aged samples, which make us partially accept the first null hypothesis, when an adhesive cementation was made, regardless of the type of substrate, the mechanical strength of the ceramic was stable after aging compared to the ceramic that was conventionally cemented with zinc phosphate material. This was expected because it is known that an adhesive cementation can help to cure some flaws and microcracks of the restoration, promoting better mechanical proprieties to the ceramic material and being an important part of a multilayered restoration [24]. Different surface treatments were used for lithium disilicate, to better simulate the clinical practice, which requests different protocols according to the type of substrate and/or cement.

Although the chemical adhesion of resin cements to metallic substrates is not established, the use of cement with 10-MDP functional monomers in its composition could be the responsible to promote better bonding on the metal substrate and consequently a greater mechanical behavior of the samples [25]. This type of performance has already been noticed when, in a previous study [26], zirconia ceramics, which have an elastic modulus closer to the metals, were cemented with an adhesive system that contained MDP.

Finally, the null hypothesis that the type of substrate would not influence the mechanical behavior of the samples was rejected. This is due to the superior results obtained by groups cemented on metal, regardless of the aging process. The possible explanation for this is that the metallic substrate (Co-Cr) has a satisfactory hardness value, with a tensile strength of 1389 MPa and an elastic modulus of 203 GPa [17], while the epoxy resin present lower values, with a tensile strength of 450 MPa and an elastic modulus of 18 GPa [27]. The substrate has influence on the mechanical behavior of the restorative material: substrates with elastic modulus higher than the restorative material led to a more resistant assembly [28].

Failure analysis indicated that fracture always started on the tensile (cementation) surface of the ceramic, as previously reported in literature [11,12,29]. This fact explains the higher amount of failures occurred during TC/MC ageing, where the cementation surface was affected, resulting in loss of retention of L discs. The load application on the restorative material generates compression near the contact point and tensile stresses at the cementation surface. Brittle materials, in general, present higher strength to compression, and they are more prone to fracture under tensile stress. In addition, the crystallization process was performed with a maximum temperature of 850ºC, and this sintering parameter can decrease the mechanical properties, as a higher amount of porosity can be observed, when compared with 900ºC and 950ºC [30].

It should be emphasized that the data found in the present study are the result of an in vitro experiment, which has certain limitations, such as the use of lithium disilicate discs instead of complete restorations, which require prior prosthetic preparation of the substrate, followed by molding steps, and thus more accurately simulate the clinical reality. But even with these limitations, the study proved useful because it was able to suggest that the adhesive resin cement is an adequate option for luting the lithium disilicate both on dentin and on a metal substrate.

## CONCLUSIONS

Based on findings of this study, it can be concluded that: (1) The biaxial flexural strength of the lithium disilicate ceramic was the highest when cemented under a high elastic modulus substrate, despite the cement used; (2) Zinc phosphate cement presented the highest initial results but after aging, high decrease in strength and debonding were recorded, while adhesive cementation presented stable strength results; (3) Aging methods promoted decrease in flexural strength of lithium disilicate discs.

## Author's Contributions

LCL, JSM, RLAC, ETK: Conceptualization. LCL, JSM, RLAC, ASPB, ETK: Methodology. LCL, JSM, RLAC, ETK: Software. LCL, JSM, RLAC, ETK: Validation. LCL, JSM, RLAC, MA, ETK: Formal Analysis. LCL, JSM, RLAC, ASPB, ETK: Investigation. LCL, JSM, RLAC, ETK: Resources. LCL, JSM, ETK: Data Curation. LCL, JSM, MA, ETK: Writing – Original Draft Preparation. LCL, JSM, MA, ETK: Writing – Review & Editing. JSM, RLAC, ASPB, ETK: Supervision. LCL, JSM, RLAC, ASPB, MA, ETK: Project Administration. LCL, ETK: Funding Acquisition.

## Conflict of Interest

The authors have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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## Regulatory Statement

Not applicable.

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