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Influence of fabrication methods on the flexural strength of two glass-ceramics with different chromas

Influência de métodos de fabricação na resistência flexural de duas vitrocerâmicas de diferentes cromas

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ABSTRACT

Objective: To investigate whether different methods of preparing the specimens for three-point flexural strength testing influence the flexural strength values of lithium disilicate (LD) and zirconia-reinforced lithium silicate (ZLS) of two chromas (A1 and A3). Also, to qualitatively assess differences in their fractographic patterns. **Material and Methods:** LD (IPS e.max CAD) and ZLS (Celtra Duo) specimens in chromas A1 and A3 were either cut using a diamond-coated disc directly to ISO 6872-specified dimensions (4 × 2 × 14mm) for three-point flexural strength tests, or cut with 1-mm extra thickness (4 × 3 × 14mm), and then subjected to thickness reduction with 45μm sandpaper in a polishing machine until reaching the same final dimension (8 groups, n=10). Flexural strength testing was conducted in an aqueous environment, and fractured surfaces were examined qualitatively. Flexural strength values (MPa) were analyzed using three-way ANOVA and Bonferroni post-hoc tests ($\alpha = 0.05$). **Results:** LD exhibited higher flexural strength than ZLS across all conditions. Thickness reduction decreased flexural strength in LD A1 but improved it in ZLS A3, with no significant effect on LD A3 or ZLS A1. No significant difference was observed between the strength of A1 and A3 specimens within the same material. Fractographic analysis revealed altered fracture patterns in LD A1 and ZLS A3 following thickness reduction. **Conclusion:** The method of specimen preparation can significantly influence flexural strength values depending on the material and shade. These findings highlight that even subtle variations in preparation can lead to inconsistent outcomes, emphasizing the urgent need for more detailed and rigorous standardization of mechanical testing protocols in ISO 6872.

KEYWORDS

Ceramics; Dental materials; Dental polishing; Dental porcelain; Flexural strength.

RESUMO

Objetivo: Investigar se diferentes métodos de preparo dos espécimes para o teste de resistência à flexão em três pontos influenciam os valores de resistência à flexão do dissilicato de lítio (DL) e do silicato de lítio reforçado por zircônia (SLZ) em dois cromas (A1 e A3). Além disso, avaliar qualitativamente as diferenças nos padrões fractográficos desses materiais. **Material e Métodos:** Espécimes de DL (IPS e.max CAD) e SLZ (Celtra Duo), nos cromas A1 e A3, foram preparados de duas formas: cortados com disco diamantado diretamente nas dimensões especificadas pela ISO 6872 (4 × 2 × 14 mm) para o teste de resistência à flexão em três pontos; ou cortados com 1 mm a mais de espessura (4 × 3 × 14 mm) e, em seguida, submetidos à redução de espessura com lixa de 45 μm em politriz, até atingirem as mesmas dimensões finais (8 grupos, n = 10). Os testes de resistência à flexão

foram realizados em meio aquoso, e as superfícies fraturadas foram analisadas qualitativamente. Os valores de resistência à flexão (MPa) foram analisados por ANOVA a três fatores e teste post-hoc de Bonferroni ($\alpha = 0,05$). **Resultados:** O DL apresentou maior resistência à flexão do que o SLZ em todas as condições testadas. A redução de espessura diminuiu a resistência à flexão do DL A1, mas a aumentou no SLZ A3, sem efeito significativo no DL A3 ou SLZ A1. Não foram observadas diferenças significativas entre os cromas A1 e A3 dentro de um mesmo material. A análise fractográfica revelou alterações no padrão de fratura em DL A1 e SLZ A3 após a redução de espessura. **Conclusão:** O método de preparação da amostra pode influenciar significativamente os valores de resistência à flexão, dependendo do material e da tonalidade. Essas descobertas destacam que mesmo variações sutis na preparação podem levar a resultados inconsistentes, enfatizando a necessidade urgente de uma padronização mais detalhada e rigorosa dos protocolos de ensaios mecânicos na norma ISO 6872.

PALAVRAS-CHAVE

Cerâmica; Materiais dentários; Polimento dental; Porcelana dentária; Resistência flexural.

INTRODUCTION

Glass-ceramics are among the most aesthetic faithfully materials for reproducing dental structures. Originally, their limited mechanical performance restricted their application to porcelain-fused-to-metal restorations [1,2]. Subsequent developments, especially heat treatment, improved their biomechanical behavior while preserving optical characteristics. These treatments enhance the crystalline content, thereby increasing flexural strength, fracture toughness, elastic modulus, and hardness, in addition to modifying optical properties such as opacity, opalescence, and color [2-4]. More recently, re-pressing techniques have been introduced to reduce internal defects and further improve mechanical properties [5,6].

Among the most clinically relevant dental glass-ceramics are those based on lithium disilicate (LD) and zirconia-reinforced lithium silicate (ZLS). LD ceramics exhibit flexural strength values between 340 and 450 MPa, which is over four times greater than feldspathic porcelain (86 to 102 MPa) [7-9], enabling their use in a wide range of restorative indications, including monolithic crowns and three-unit partial fixed prostheses in the anterior region [10,11]. ZLS ceramics present flexural strength around 210 MPa, which can be maximized up to 370 MPa after an additional firing protocol, supporting their use in similar indications, except for fixed dental prostheses [12,13].

Flexural strength values for dental glass-ceramics have been widely studied [4,7-9,14-30], but significant variability exists across the literature, particularly for LD ceramics (from 251 ± 30 MPa to 407 ± 45 MPa). This variation is often attributed to methodological differences in specimen preparation and testing procedures [4,7-9,14-30]. Additionally, translucency and chroma can affect crystal density, morphology, and size, all of which may influence mechanical properties [21,31].

Recognizing the need for test reproducibility [32], the International Standardization Organization (ISO) has developed the standard no. 6872 [33] with recommendations for more reproducible and valid flexural strength test protocols. While the standard outlines several parameters, it still lacks essential guidance on specimen fabrication techniques (e.g., cutting tools, polishing steps) and testing conditions (e.g., dry vs aqueous environments). This gap results in methodological inconsistencies across studies, compromising comparability [4,7-9,14-30].

Concerns about different fabrication methods come from the potential surface defects, which, albeit minute, can serve as a source of failure upon tensile stress by fostering crack propagation [24]. Based on this rationale, several studies have shown that different finishing and polishing procedures can influence on the flexural strength of different dental ceramics [25,27,34,35]. Yet, no study has specifically investigated whether the method used to obtain the final dimensions of test specimens itself affects flexural strength.

Therefore, the present study aimed to evaluate whether two preparation methods – direct cutting to ISO 6872 dimensions versus cutting followed by thickness reduction using a polishing machine – affect the flexural strength of LD and ZLS ceramics. It also investigated the influence of chroma (A1 and A3) and assessed fracture patterns through qualitative fractography.

The null hypotheses tested were: (1) there is no significant difference in flexural strength between LD and ZLS; (2) the preparation method does not influence flexural strength; and (3) chroma does not affect flexural strength.

MATERIAL AND METHODS

Experimental design

This in vitro study compared eight different experimental conditions, according to three factors (two levels each): (1) material; (2) fabrication method; and (3) shade. The dependent variable was the three-point flexural (bending) strength measured in MPa.

Sample acquisition

Specimens were obtained from two blocks of each material, LD and ZLS, each with a different shade: A1 and A3 (Vita Classical, Vita Zahnfabrik, Bad Säckingen, Germany). The LD (IPS e.max CAD lithium disilicate – C14 HT, Ivoclar Vivadent, Schaan, Liechtenstein) and ZLS (Celtra Duo zirconia-reinforced lithium silicate – C14 HT; Dentsply Sirona, York, PA, USA) blocks were stabilized in an Isomet 1000 cutting machine (Buehler, Lake Bluff, IL, USA). With a water-cooled diamond disc (Extec XL12205 High Concentration, Extec Corp, Enfield, CT, USA) at 275 rpm, the blocks were cut rectangularly in the dimensions of 4mm X 2mm X 14mm (width X thickness X length), totaling 10 specimens for each group (n = 10/group). This sample size was determined as the minimum recommended by ISO 6872:2015 for flexural strength testing [33] and simulates the diamond tip wear performed in the milling system.

Using the same parameters described above, two new blocks were cut rectangularly with extra thickness (4mm X 3mm X 14mm), totaling 10 specimens/group. A polishing machine (AutoMet 250, Buehler, Lake Bluff, IL, USA) with 45 μm diamond sandpaper, with pressure of 20 N and under refrigeration with deionized water was used only on the lower side of the specimen in order to reduce 1 mm of thickness. This resulted in specimens with the same measures proposed by ISO 6872:2015 [33]. Surface roughness measurements were not conducted after thickness reduction.

Thereafter, a single operator polished all specimens, regardless of their fabrication method, using appropriate polishers in a handpiece (HDZ2 and HDZ3, DHPro, Paranagua, Brazil). The four largest edges of each specimen were then chamfered by a single operator using a metallic device in conjunction with the polishing machine. The device featured a slot that exposed only one edge (0.1 mm) at a time to the sandpaper, ensuring consistent chamfering in accordance with ISO 6872:2015 [33]. Sandpapers were replaced after every three specimens.

Then, the LD specimens were placed in a ceramic furnace (Programat EP3000, Ivoclar Vivadent, Schaan, Liechtenstein) for final crystallization according to the manufacturer's recommendations to achieve their final mechanical strength. The crystallization protocol recommended by the manufacturer consisted of a starting temperature of 403 °C with a holding time of 6min, followed by a heating rate of 90 °C/min until reaching 820 °C, with a holding time of 10s. The temperature was then increased at 30 °C/min until reaching 840 °C, where it was maintained for 7min.

In brief, 80 bar-shaped specimens were obtained, equally divided among the experimental condition. Table I summarizes the different conditions/groups.

Table I - Material, classification and fabrication method to obtain the specimens

Material	Shade	Fabrication method	Sample size
Zirconia-reinforced lithium silicate (ZLS, Celtra Duo)	A1	Precisely cut	10
		Cut + thickness reduction	10
	A3	Precisely cut	10
		Cut + thickness reduction	10
Lithium disilicate (LD, IPS e.max CAD)	A1	Precisely cut	10
		Cut + thickness reduction	10
	A3	Precisely cut	10
		Cut + thickness reduction	10

Three-point bending test

In accordance with ISO 6872:2015 [33], the three-point bending test (σf) was performed by a single operator with a 500 N load cell at a constant speed of 0.5 mm/min using an Instron 3342 universal testing machine (Instron Co., Canton, MA, USA) with the aid of a metallic device. The specimens were stored in a dry environment for 7 days and then measured using a digital caliper before testing. The specimens were positioned on the metallic device, maintaining a distance of 12 mm between the lower cylindrical supports and the load applied in the center of the upper metallic rod. During the tests, the specimen and the metallic device were submerged in distilled water at a temperature of 37°C to simulate what occurs in the oral environment. For the polished specimens, the polished side was positioned facing down, subjecting this surface to to tensile stress. The flexural strength values were calculated according to the following equation, where P refers to the value of the fracture load in Newtons (N), I refers to the distance between the supports (standardized at 12mm), w refers to the specimen width (approximately 4 ± 0.2 mm) and b refers to the thickness of the specimen (approximately 2.0 ± 0.3 mm).

$$\sigma f = 3PI / 2wb^2 \quad (1)$$

Qualitative fractographic analysis

Three fractured specimens from each group were first cleaned by immersion in distilled water in an ultrasonic bath for 15 minutes. Then, a single operator analyzed the fractured surface of each specimen using Scanning Electron Microscopy (SEM) (JEOL-JSM 5600LV; Tokyo, Japan) to observe characteristic fracture surface features. Magnifications ranged from 50x to 200x.

Statistical analysis

Data were Log_{10} transformed and checked for normal distribution ((Shapiro-Wilk test, p = 0.681) and homogeneity of variances (Levene test, p = 0.139). Between-group comparisons were performed using a by three-way analysis of variance (3-way ANOVA), followed by post-hoc Bonferroni test. All statistical analyses were performed using IBM SPSS Statistics Software v.23 (IBM Corp, Armonk, NY). The level of significance was set at α = 5% and power (1-β) = 80%.

RESULTS

In general, results show statistical difference between the two tested materials, with LD being stronger than ZLS (Material, $p<0.001$). The fabrication method alone (Fabrication method, $p=0.043$) and its interaction with material (Fabrication method*Material, $p=0.003$) and shade (Fabrication method*Shade, $p=0.009$) were significant. No significance was seen for shade (Shade, p=0.979), as well as for the interaction between material and shade (Material*Shade, p=0.734) and the three-factor interaction (Material*Fabrication method*Shade, p=0.598).

In the 3-point bending test, the highest mean flexural strength was observed for the precisely cut LD A1 (279.6 ± 52.5 MPa), followed by cut and reduced LD A3 (275.0 ± 66.5 MPa), precisely cut LD A3 (249.2 ± 50.0 MPa), cut and reduced LD A1 (233.3 ± 40.2 MPa), cut and reduced ZLS A3 (145.7 ± 15.8 MPa), cut and reduced ZLS A1 (136.5 ± 26.3 MPa), precisely cut ZLS A1 (121.6 ± 31.6 MPa), and precisely cut ZLS A3 (107.3 ± 11.1 MPa). Statistical differences between groups are presented in Figure 1.

All LD specimens showed higher flexural strength than ZLS specimens, regardless of fabrication method or shade. The 1-mm thickness reduction reduced the strength of LD A1 specimens (from 279.6 ± 52.5 to 233.3 ± 40.2 MPa), while it improved that of ZLS A3 specimens (from 107.3 ± 11.1 to 145.7 ± 15.8 MPa). For LD A3 and ZLS A1 groups, the fabrication method did not result in a statistically significant change in flexural strength values. No differences between shades A1 and A3 within the same material were detected.

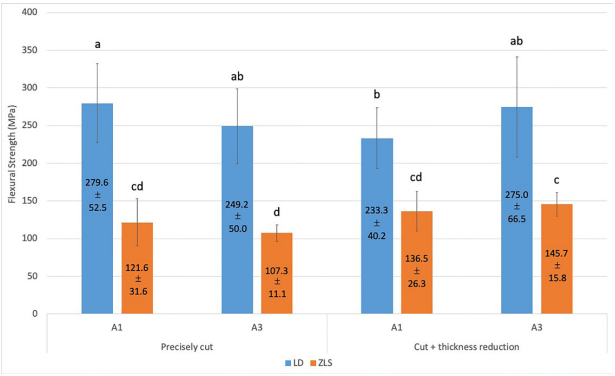

Figure 1 - Mean ± standard deviation of flexural strength values (MPa) presented by each group. Different lowercase letters indicate significant differences based on Bonferroni post-hoc test".

The qualitative fractographic analysis of LD A1 specimens highlighted that polishing changed the fracture pattern (Figure 2). For the polished specimens, the defect responsible for fracture was located on the surface subjected to tensile stress, while the non-polished specimens had their critical defect located in the bulk of the material.

The polished specimen (Figure 2A) showed a more defined failure pattern, with a mirror region (M) (large white arrows), mist (MI) (small white arrows) and Hackle lines (HL) (black arrows), indicating the direction of crack propagation with no signs of fracture plane change. The non-polished specimen (Figure 2B) showed a less classic failure pattern, with a less defined mirror region (M) (white arrows), Hackle lines (HL) (large black arrows) and arrest lines (AL) (large black arrows) indicating fracture plane change and the direction of crack propagation (DCP) (small black arrows).

For the ZLS A3 material, the defect was located on the surface for both the polished and non-polished specimens (Figure 3). The polished specimen (Figure 3A) showed a failure pattern with a mirror region (M) (large white arrows), mist (MI) (small white arrows) and Hackle lines (HL) (large black arrows), arrest lines (AL) (large black arrows) and twisted arrest lines indicating the direction of crack propagation (DCP) (small black arrows), with signs of fracture plane change. A compressive curl (CC) (large black arrow) was observed on the compression side. The non-polished specimen (Figure 3B) showed a flatter fractured surface, with a less defined mirror region (M) (large white arrows) and mist (MI) (small white arrows). A compressive curl (CC) (black

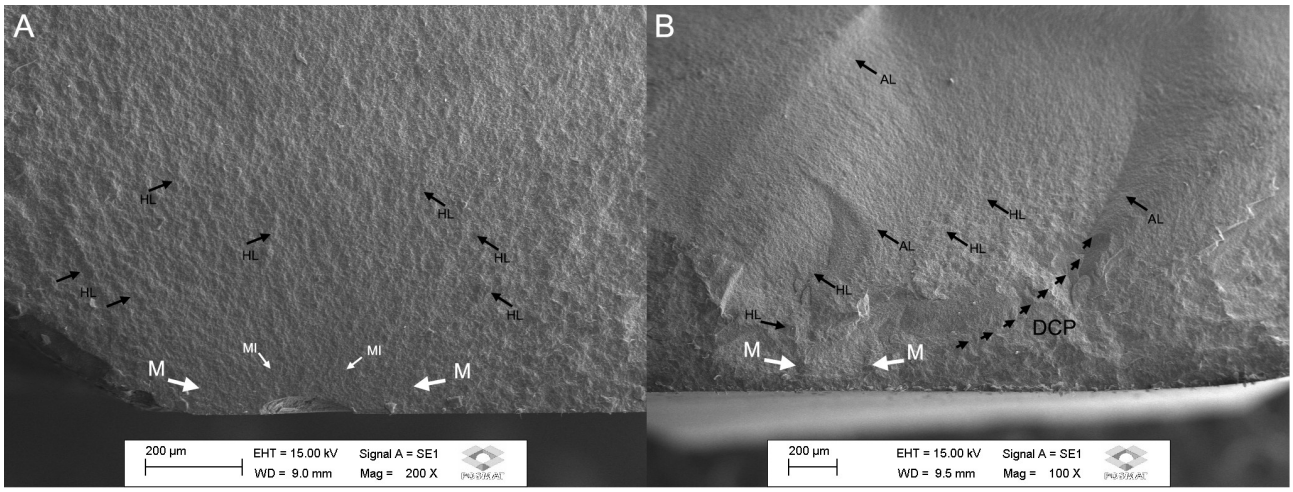

Figure 2 - Fractographic analysis of the LD A1 specimens. When the specimens were polished, the critical defect was located on the surface of the specimen (A), while the non-polished specimens presented defects in the bulk of the material (B).

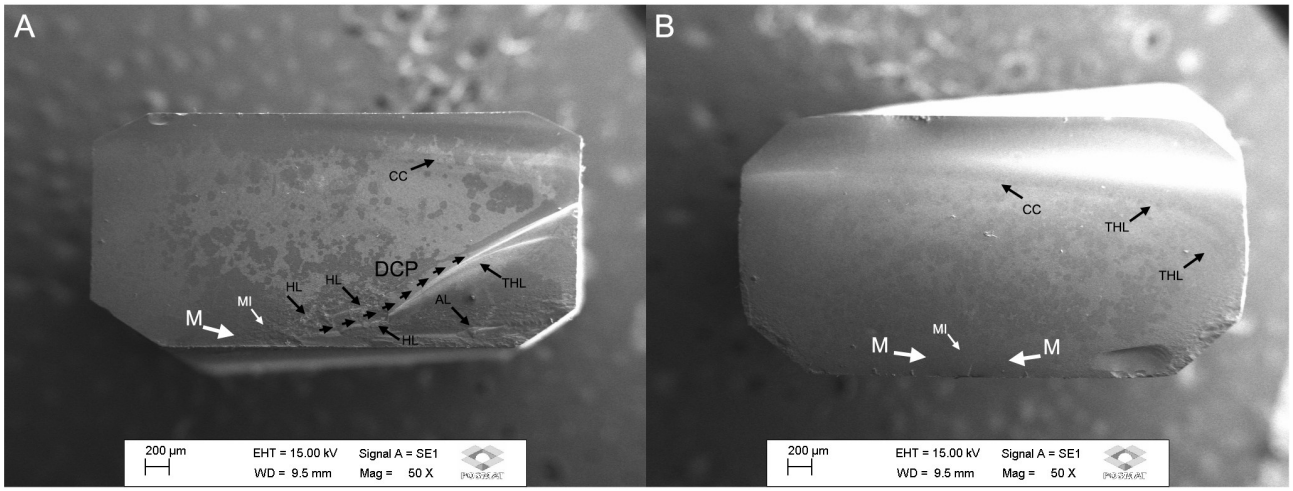

Figure 3 - Fractographic analysis of the ZLS A3 specimens. The critical defect was located on the surface for both polished (A) and non-polished specimens (B).

arrow) is evident and twist Hackle lines (THL) (black arrows) signal fracture plane change.

For the LD A3 and ZLS A1 materials, polishing did not change the fracture pattern, and the critical defects were located on the surface subjected to tensile stress.

DISCUSSION

This study demonstrated that the fabrication method significantly influences the flexural strength of specimens made with two widely used dental glass-ceramic: LD and ZLS. In all test conditions, LD specimens outperformed ZLS specimens, leading to the rejection of the first null hypothesis. The main contribution of this study lies in showing that the method used to prepare specimens for the three-point bending test can affect the mechanical performance of some materials, depending on their composition and intrinsic characteristics. Consequently, the second null hypothesis was also rejected. These findings underscore the importance of standardized testing protocols, particularly regarding specimen preparation methods. In contrast, since no statistically significant difference in flexural strength was found between A1 and A3 shades, the third null hypothesis was not rejected.

The flexural strength values are consistent with those reported in the previous literature [5,20,36], reinforcing the superior mechanical performance of LD-based glass-ceramics compared to ZLS, regardless of shade. This difference is attributed to their distinct microstructures, particularly in terms of crystalline content and morphology. During heat treatment of LD specimens, lithium metasilicate (Li_2SiO_3) dissolves and is followed by controlled crystallization and growth of lithium disilicate ($Li_2Si_2O_5$) crystals, resulting in a high crystal volume fraction (~70%). These crystals form an interlocked, needle-like structure that effectively deflects cracks and enhances fracture toughness [28]. In contrast, ZLS contains a lower crystalline content (~40-50%), composed of smaller, rounded lithium silicate (Li_2O-SiO_2) crystals and dispersed zirconia particles [28], which may inhibit further crystal growth. These features result in a less interlocked microstructure and reduced crack deflection capability, thereby compromising mechanical performance [29].

The results also indicated that the fabrication method may influence flexural strength differently depending on the material and shade. Previous studies have reported that polished surfaces have increased strength values by eliminating surface defects that can act as a source of cracks and catastrophic failures under tensile stresses [37-40]. This could explain the increased strength observed in ZLS A3 specimens following thickness reduction. Conversely, the same process led to reduced strength in LD A1 specimens. A plausible explanation is that the polishing machine (applying 20 N of pressure) may have induced microdefects in the softer, metastable lithium metasilicate phase, particularly since LD specimens were crystallized only after thickness reduction and chamfering, unlike ZLS specimens, which were tested in their final form (unfired).

As previously reported, the crystallization protocol is capable of affecting the porosity, hardness, and strength of the glass-ceramic [4], as well as generating a very fine and resistant to polishing microstructure [41]. In the case of LD, thickness reduction and chamfering were performed while the material was in its softer, pre-crystallized state, potentially introducing surface flaws. For ZLS, however, its higher hardness and wear resistance may have resulted in a more uniform surface following thickness reduction.

The finding that the fabrication method influenced the mechanical strength of ceramics in a shade-dependent manner is noteworthy and deserves further explanation. In glass-ceramic system, colorants are commonly used to achieve the desired shade and translucency of glass-ceramics. In glass-ceramic systems, colorants are commonly used to achieve desired shades and translucencies. These additives include metal oxides with 3d orbitals (e.g., Fe, Ti, Cr, V, Mn) and rare-earth elements with 4f orbitals (e.g., Ce), which interact with light to generate specific optical effects [31]. LD glass-ceramics are often doped with colorants such as $AgNO_3$, $FeCl_3$, TiO_2, Cr_2O_3, V_2O_5, CeO_2, and MnO_2 in varying concentrations, depending on the desired optical characteristics (shade, chroma, hue, and translucency).

Some studies have reported that the type and concentration of such dopants can influence nucleation and crystallization processes during heat treatment, which in turn affect the material's microstructure and mechanical properties [21,31,42-44]. However, it is important

to emphasize that the present study did not include any analytical techniques (e.g., spectroscopy) to assess the chemical state or influence of these colorants. Therefore, while these findings align with theoretical frameworks and prior literature, the current data do not permit definitive conclusions on this point.

Although unlikely, it cannot be entirely ruled out that particles from abrasive procedures or the heat generated during polishing may have altered the chemical state of certain oxides, as hypothesized by Kim et al. [31]. Such modifications could theoretically impact the crystallization behavior and contribute to variations in mechanical performance. However, no shade alteration was visually observed in the specimens, and further research involving specific chemical and microstructural analyses is necessary to test this hypothesis. These considerations highlight the importance of including specimen preparation as a variable in flexural strength studies, as also discussed by Wang et al. [30], who emphasized the influence of factors such as porosity, crystal size and density, coloring agents, heat treatments, and fabrication methods.

Despite the evidence from Fabian Fonzar et al. [21] and Martins et al. [45], who reported that different translucency levels can influence flexural strength, the results of this study suggest that chroma-related differences in the material composition (e.g., A1 vs. A3) are not sufficient on their own to significantly alter flexural strength. These findings are in accordance with Santos et al. [26]. However, oxides of different hues (e.g., A1 vs. C1) may introduce more substantial compositional and optical differences, which should be explored in future studies.

According to the manufacturer [13], the flexural strength of ZLS is 370 MPa when glazed, but only 210 MPa in polished, unfired condition. In the study by Schwindling et al. [12], ZLS specimens subjected to the glaze firing protocol exhibited flexural strength values similar to LD. In addition, Badawy et al. [46] reported significantly higher fracture toughness for fired ZLS compared to unfired specimens. Therefore, the results of this study should not be extrapolated to glazed ZLS materials.

For LD specimens, the flexural strength values found in this study are close to the lower limit of those reported in the literature (251 ± 30 MPa to 407 ± 45 MPa) [4,7-9,14-30], while the flexural strength values of ZLS specimens are lower than those reported by Lawson et al. [20]. One contributing factor may be the testing environment: in this study, the three-point bending test was performed in an aqueous medium [17,47,48].

As discussed by Cattel et al. [17], mechanical testing in aqueous environments typically leads to an approximately 25% reduction in the material strength due to subcritical crack growth. This phenomenon occurs as water molecules weaken the silicate bonds (Si-O-Si) at the crack tip, facilitating crack propagation. Specifically, hydrolysis of the silicate network forms Si-OH groups and leads to stress corrosion, compromising the structural integrity of glass-ceramics [17,47,49].

The fracture path was analyzed using fractography, as shown in Figures 2 (LD) and 3 (ZLS). The arrows indicate potential failure origins due to residual tensile stresses, typical of three-point bending tests. The origin of failure, known as the critical defect according to the recommended nomenclature [39,50,51], is where the failure initiated. The fracture marks identified in the aforementioned figures were: M (mirror region), which is the smoothest region around the possible critical defect; MI (mist), which are surface markings usually observed between the mirror region and the hackle lines, observable initially as a misty appearance and with increasing velocity revealing a fibrous texture, elongated in the direction of the crack; AL (arrest lines), which are lines that indicate the direction of propagation; HL (hackle lines), which are lines on the surface that run in the direction of the crack, separating parallel but non-planar portions of the crack surface, formed commonly when the crack moves quickly; THL (twist hackle lines), which are hackles that separate portions of fractured surface that was rotated from the original crack plane; and CC (compression curl), which are the marks left before the fracture and are located right next to where compressive stresses were applied.

In view of this, future studies should include reliability assessments (e.g., Weibull analyses) and evaluate the mechanical behavior of different hues from the Vita scale. Additionally, surface roughness measurements following thickness reduction should be conducted, as well as testing of specimens subjected to high-quality mirror

polishing (e.g., diamond disks and suspensions up to 1 μm) to minimize the risk of surface flaws acting as fracture origins. These efforts are essential to improve test reproducibility, especially considering the variability in mechanical properties across studies—much of which stems from the lack of standardization in protocols as per ISO 6872:2015. Lastly, it is recommended that manufacturers report flexural strength values alongside detailed descriptions of the test conditions employed (e.g., three-point, four-point, or biaxial tests), since the test method significantly impacts the results

CONCLUSION

Within the limitations of the present study, it can be concluded that lithium disilicate (LD) exhibits higher flexural strength than zirconia-reinforced lithium silicate (ZLS), regardless of chroma or the specimen fabrication method. Although chroma (A1 vs. A3) did not significantly affect flexural strength overall, the method used to achieve the final specimen dimensions influenced the mechanical performance of specific groups. These findings highlight the importance of standardizing specimen preparation, and it is recommended that future revisions of ISO 6872 include more detailed guidance on specimen fabrication and test execution to reduce variability in reported values.

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Author's Contributions

VM: Conceptualization, Methodology, Formal Analysis, Writing – Original Draft Preparation. BMF: Conceptualization, Methodology, Investigation, Formal Analysis. FCNS: Conceptualization, Methodology, Investigation, Formal Analysis. RFS: Methodology, Investigation, Formal Analysis. LJAS: Methodology, Investigation, Formal Analysis. AFSB: Conceptualization, Writing – Review & Editing, Visualization, Project Administration.

Conflict of Interest

No conflicts of interest declared concerning the publication of this article.

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Regulatory Statement

Non applicable.

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