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RESEARCH ARTICLE

Mechanical Properties and Microstructure of the Leucite-Reinforced Glass-Ceramics for Dental CAD/CAM

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The computer-aided design/computer-aided manufacturing (CAD/CAM) system was introduced to shorten the production time of all-ceramic restorations and the number of patient visits. Among these types of ceramic for dental CAD/CAM, they have been processed into inlay, onlay, and crown shapes using leucite-reinforced glass-ceramics to improve strength. The purpose of this study was to observe the mechanical properties and microstructure of leucite-reinforced glass-ceramics for dental CAD/CAM. Two types of leucite-reinforced glass-ceramic blocks (IPS Empress CAD, Rosetta BM) were prepared with diameter of 13 mm and thickness of 1 mm. Biaxial flexural testing was conducted using a piston-on-three-ball method at a crosshead speed of 0.5 mm/min. Weibull statistics were used for the analysis of biaxial flexural strength. Fracture toughness was obtained using an indentation fracture method. Specimens were observed by field emission scanning electron microscopy to examine the microstructure of the leucite crystalline phase after acid etching with 0.5% hydrofluoric acid aqueous solution for 1 minute. The results of strength testing showed that IPS Empress CAD had a mean value of 158.1±8.6 MPa and Rosetta BM of 172.3±8.3 MPa. The fracture toughness results showed that IPS Empress CAD had a mean value of 1.28±0.19 MPa · $m^{1/2}$ and Rosetta BM of 1.38±0.12 MPa · $m^{1/2}$. The Rosetta BM sample exhibited higher strength and fracture toughness. Moreover, the crystalline phase size and ratio were increased in the Rosetta BM sample. The above results are expected to elucidate the basic mechanical properties and crystal structure characteristics of IPS Empress CAD and Rosetta BM. Additionally, they will help develop leucite-reinforced glass-ceramic materials for CAD/CAM.

Key Words: Biaxial flexural strength, CAD/CAM, Fracture toughness, Leucite-reinforced glass-ceramic, Microstructure

Introduction

Dental ceramics are excellent dental restoration materials due to their excellent durability and esthetics, but they are susceptible to brittle fracture at a relatively low level of stress[1]). To improve their strength and sensitivity to brittle fracture, metal-ceramic restorations, with metal as the core material, are being widely used. However, they are vulnerable to problems such as increased opacity, exposed metal in the margins due to gingival recession, low bonding strength between metal and ceramic, and metal allergy. Consequently, there is heightened interest in all-ceramic restorations that can offer aesthetics, marginal integrity, and fracture resistance[2,3]).

In the early 1980s, Adair and Grossman found a way to improve all-ceramic system from crystallization by controlling glass, and since then, various types of glass-ceramic have been developed[4,5]). Glass-ceramic materials have excellent biocompatibility and improved strength over regular glass, while also satisfying the esthetic demands of both clinicians and patients.

Among them, leucite-reinforced glass-ceramic (LRGC), which contains potassium-aluminum-silicate ($KAlSi_2O_6$) crystalline mineral leucite within a glass matrix, allows the thermal expansion coefficient of glass-ceramic to be controlled according to the content of leucite crystals,

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which have high thermal expansion (>20×20^{-6}/°C). Moreover, it can increase the strength of glass-ceramic by absorbing the fracture energy to delay or suppress the propagation of cracking[6,7]. With increasing demand for restorations with natural tooth color, LRGC with excellent esthetics is being used as the material for all-ceramic restorations[8].

Meanwhile, the computer-aided design and computer-aided manufacturing (CAD/CAM) method have been introduced to shorten the time required to fabricate all-ceramic restorations and reduce the number of patient visits. CAD/CAM for fabrication of dental restorations was developed in the 1980s, and has evolved since then with improvements in hardware, software, and CAD/CAM materials, and as a result, its clinical application has gained much popularity[9]. Machinable ceramic blocks can be fabricated into different shapes using the CAD/CAM system, including inlays, onlays, and crowns. For fabrication of the restoration, the missing tooth is scanned and imaged through a computer, after which, the restoration is fabricated into the desired shape by a milling machine according to computer-controlled input. The disadvantages of such CAD/CAM restorations include imprecise fitting inside the mouth and the fabrication process being technically sensitive, and requiring expensive equipment[6]. On the other hand, their main advantages include the fact that they do not require impression taking, which can shorten treatment time and reduce the need for materials related to the impression process, while also reducing the number of patient visits, which can result in increased satisfaction level in the patients and clinicians[10].

Along with the development of LRGC materials machinable through the CAD/CAM method, measurements of their elastic modulus, flexural strength, surface hardness, and fracture toughness are being performed to assess their physical properties. Typically, flexural strength indicates how well glass-ceramic, a brittle material, fares inside the mouth, while fracture toughness measures the sensitivity of glass-ceramic, which is strongly affected by defects[11,12].

Accordingly, the objective of the present study was to measure the biaxial flexural strength and fracture toughness of two types of LRGC material for CAD/CAM application, IPS Empress CAD (Ivoclar Vivadent, Schaan, Liechtenstein) and Rosetta BM (HASS, Gangneung, Korea), and to observe the microstructures of their crystals to identify their basic physical properties.

Materials and Methods

1. Materials

The two types of LRGC block for CAD/CAM application used in the present study were IPS Empress CAD and Rosetta BM (Table 1).

2. Methods

1) Biaxial flexural strength measurement

Millable blocks were cut to prepare 15 specimens of each type, with each disk-shaped specimen having a diameter of 13.0 mm and thickness of 1.0 mm. After initially polishing both surfaces of the specimens with #400～1,200 SiC sand paper, the final polishing was performed using diamond slurry (Buehler, Lake Bluff, IL, USA), 1 μm in size, to remove the polishing marks from the surface where the tensile load would be applied.

Using the polished disk-shaped specimens, biaxial flexural strength was measured in 15 specimens from each experimental group, in accordance with ISO 6872. The loading device for biaxial flexural strength testing was fixed on a universal testing machine (model 4201; Instron, Canton, MA, USA); a specimen holding device with a circumference maintaining a diameter of 10 mm and having steel balls (1.5 mm in diameter) placed 120° apart was loaded on the machine. Subsequently, a piston (1.6 mm in diameter) was used to apply compressive force at a load speed of 0.5 mm/min. A polyethylene film was placed between the piston and the disk-shaped specimen to ensure that the load was applied evenly, and the load at

Table 1. Leucite-Reinforced Glass-Ceramic Blocks for Dental CAD/CAM Used in This Study

Product name	Manufacturer
IPS Empress CAD	Ivoclar Vivadent (Schaan, Liechtenstein)
Rosetta BM	HASS (Gangneung, Korea)

the moment of fracture was measured (Fig. 1). The biaxial flexural strength was calculated using the following equation (σ)[13]. The mean value for each experimental group was derived, and the final value was calculated accordingly.

$$\sigma = -0.2387\ P\ (X - Y)/b^2$$

$$X = (1+\upsilon)\ \ln\ (r_2/r_3)^2 + \{(1-\upsilon)/2\}\ (r_2/r_3)^2$$

$$Y = (1+\upsilon)\ \{1 + \ln\ (r_1/r_3)^2\} + (1-\upsilon)\ (r_1/r_3)^2$$

σ: maximum flexural strength (MPa)
P: fracture load (N)
B: specimen thickness (mm)
υ: Poisson's ratio (set to 0.25)
r_1: support radius (mm)
r_2: circumferential radius of loading area (mm)
r_3: specimen radius (mm)

A statistical analysis is required when analyzing the fracture strength of brittle materials, which is highly sensitive to defects. Consequently, Weibull analysis was performed using OriginPro 8 (OriginLab, Northamton, MA, USA).

2) Fracture toughness measurement

For fracture toughness measurements, the Vickers indentation fracture (IF) method was used. For each experimental group, 15 Vickers indenters were fixed on a micro Vickers hardness tester (HM-124; Mitutoyo, Tokyo, Japan) and indentations were formed under the conditions of holding time 15 seconds and indentation load 9.8 N. The experiment was conducted under room temperature of 23±1°C and relative humidity of 50±10%, and to minimize measurement errors, the initial crack length (ICL) was measured within 5 minutes from forming the indentation. Ultimately, the following equation was used to calculate the fracture toughness value[14,15]. The mean value for each experimental group was derived, and the final value was calculated accordingly.

$$Kc = 0.016\ (E/H)^{1/2}(P/C^{3/2})$$

$$H = 0.4635\ P/a^2$$

Kc: fracture toughness value ($Pa.m^{1/2}$)
E: elastic modulus (Pa)
H: Vickers hardness (Pa)
P: indentation load (N)
C_0: ICL (m)
a: half-length of indentation diagonal (m)

For the values of E, elastic modulus, the values provided by the manufacturers (IPS Empress CAD: 62 GPa, Rosetta BM: 90 GPa) were used.

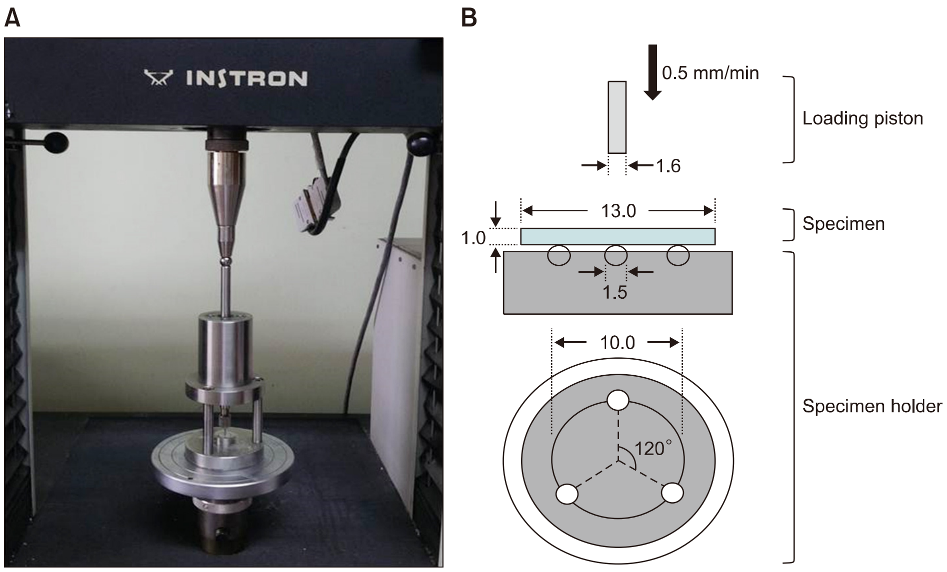

Fig. 1. (A) Biaxial flexural test device. (B) Schematic illustration of the piston-on-three ball biaxial flexural test (unit: mm).

3) Structural observation

For identification of leucite crystalline microstructures after surface polishing, observations were performed using a field emission scanning electron microscope (S800; Hitachi, Tokyo, Japan) after 1 minute of acid etching in aqueous 0.5% hydrofluoric acid (HF) solution. Moreover, indentation created by fracturing the Vickers indenter was observed using an optical microscope (DM 2500; Leica Microsystem, Wetzlar, Germany).

4) Statistical analysis

Statistical analysis was performed on the measured values using SPSS 12.0 (SPSS Inc., Chicago, IL, USA). One-way ANOVA was performed for significance testing, and for variables that showed significant differences, post-hoc testing was performed by Tukey's test (p=0.05).

Results

1. Measurement results on biaxial flexural strength of veneering ceramic

Fig. 2 shows the flexural strength measurement results. LRGC materials IPS Empress CAD and Rosetta BM had mean values of 158.1±8.6 MPa and 172.3±8.3 MPa, respectively, where Rosetta BM had a significantly higher value ($p < 0.05$).

Fig. 3 shows the Weibull analysis results on flexural strength. The table within the figure shows the values that demonstrate the distribution characteristics. Weibull distribution tended to show good consistency in single mode ($r^2 > 0.968$), Weibull coefficient (m) and characteristic strength (σ_0) were higher in Rosetta BM than in IPS Empress CAD.

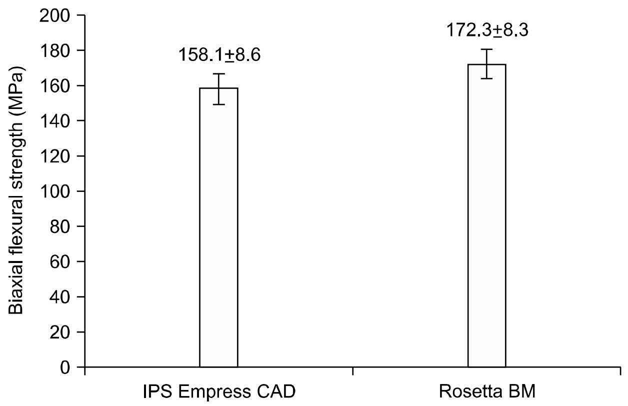

Fig. 2. Biaxial flexural strength of the two leucite-reinforced glass-ceramics for dental computer-aided design/computer-aided manufacturing (CAD/CAM) (IPS Empress CAD, Rosetta BM) (n=15).

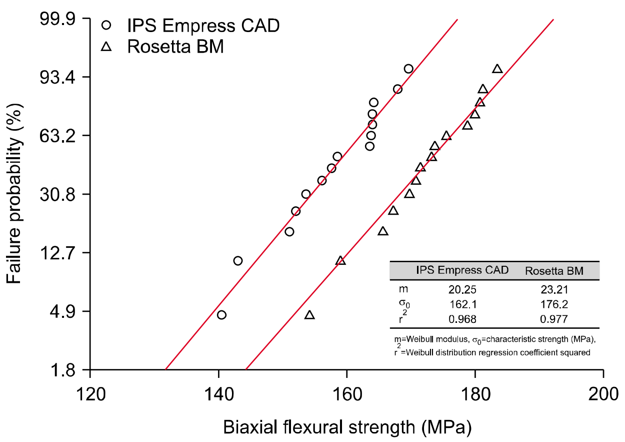

Fig. 3. Weibull analysis of the biaxial flexural strength.

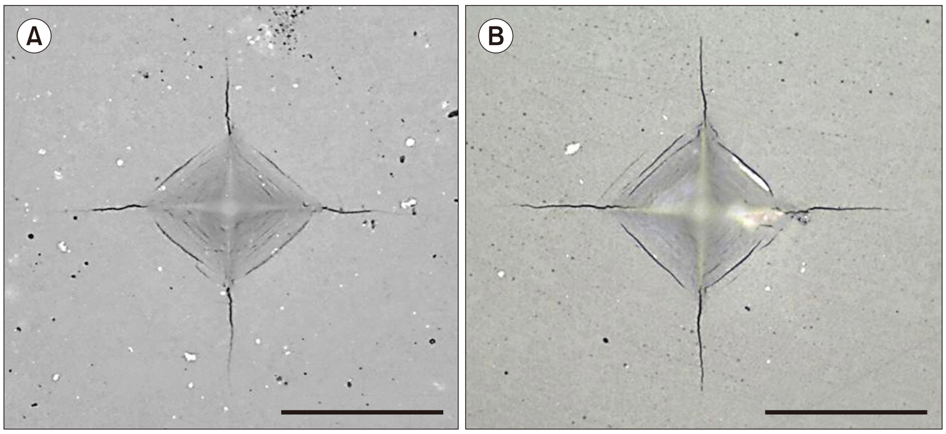

Fig. 4. Vickers produced indentation crack pattern of the two leucite-reinforced glass-ceramics: (A) IPS Empress CAD, (B) Rosetta BM. Scale bars=50 μm.

2. Fracture toughness measurement results

Fig. 4 shows the results of observations using optical microscopy after fracturing of Vickers indenters by applying an indentation load of 9.8 N (1 kg). In the IF area, indentation mark on the Vickers indenter and radial crack growing in diagonal direction from the indentation were observed.

Fig. 5 shows the fracture toughness values derived by the Vickers IF method, where Rosetta BM (1.38±0.12 MPa·$m^{1/2}$) had a significantly higher value than IPS Empress CAD (1.28±0.19 MPa·$m^{1/2}$; $p<0.05$).

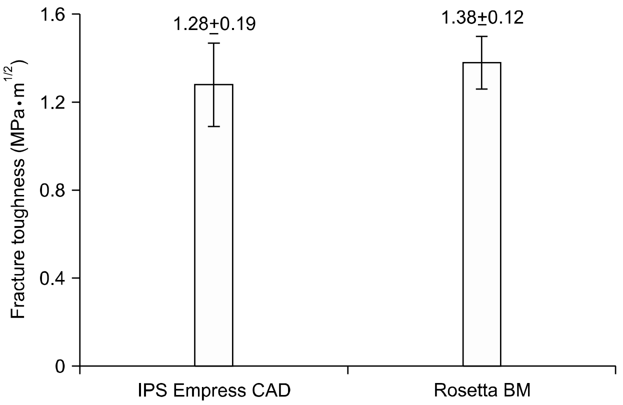

Fig. 5. Fracture toughness of the two leucite-reinforced glass-ceramics for dental computer-aided design/computer-aided manufacturing (CAD/CAM) (IPS Empress CAD, Rosetta BM) (n=15).

3. Structural observation results

Fig. 6 shows the microstructure images of IPS Empress CAD (Fig. 6A) and Rosetta BM (Fig. 6C), where glassy matrix and leucite crystals were observed. Because of treatment with 0.5% HF for microstructure observations, leucite crystals were dissolved before the glassy matrix and they were distributed along the boundaries of those crystals. The leucite crystals of IPS Empress CAD were evenly and densely distributed at a volume of 35～45%. Moreover, the crystal size varied with a range of 1～5 μm, while formation of small beads appeared as well (Fig. 6B). Rosetta BM also showed similar crystalline patterns, but the size and ratio were higher than that of IPS Empress CAD (Fig. 6D).

Discussion

Early use of dental CAD/CAM was limited to just ceramic inlays, but in recent times, its range of use has expanded to include veneers, all-ceramic crowns, and ceramic frameworks[9,16,17]. Consequently, advances in dental CAD/CAM technology have had a major impact on the clinical utilization of glass-ceramic materials, which has also resulted in major advancement in all-ceramic prostheses through the ability to use a diverse range of materials[18]. In particular, LRGC materials have been

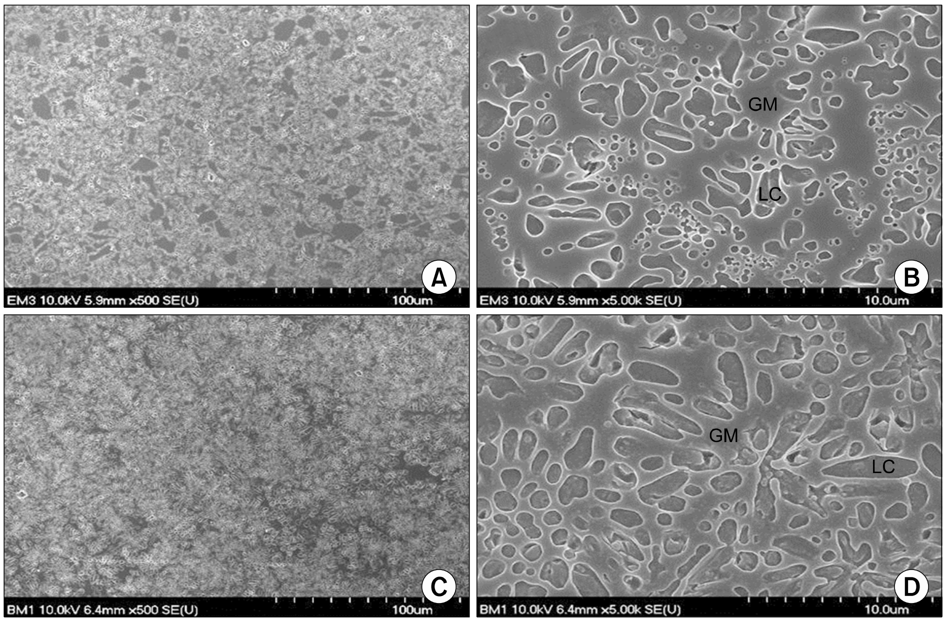

Fig. 6. Microstructure of leucite-reinforced glass-ceramics for dental computer-aided design/computer-aided manufacturing (CAD/CAM), which was etched with 0.5% hydrofluoric acid for 60 seconds (field emission scanning electron microscope images). (A) IPS Empress CAD (×500), (B) IPS Empress CAD (×5,000), (C) Rosetta BM (×500), (D) Rosetta BM (×5,000). GM: glassy matrix, LC: leucite crystal.

commercialized as CAD/CAM ceramic blocks and their range of use has become diversified in various clinical cases to include veneers, inlays, onlays, and anterior and posterior crowns[19,20]. In addition, the overall flexural strength of LRGC (73～182 MPa) is higher than that of traditional feldspathic porcelain (55～87 MPa), while the ability to fabricate ceramic crowns with excellent fracture resistance and marginal integrity has been greatly enhanced[20,21].

Methods that measure the flexural strength and fracture toughness of dental glass-ceramic materials are used to assess their mechanical properties. Among the methods for measuring flexural strength, biaxial flexural strength measurement is used to examine the fracture characteristics of ceramic materials, where the piston-on-three-balls method recommended by ISO 6872 is generally used[22,23]. Meanwhile, fracture toughness measurement methods include single edge precracked beam, compliance, and IF methods[14]. Among them, the IF method is often used, as it involves a simple measurement method and there are no constraints due to the shape and appearance of the specimen, while allowing repeated measurements from a single small specimen[24].

To examine the fracture characteristics of IPS Empress CAD and Rosetta BM, two types of LRGC material used in CAD/CAM restorations, which were the test materials in the present study, the biaxial flexural strength of these materials was measured. Moreover, the IF method was applied to derive the indentation load and ICL, after which fracture toughness was measured.

Experimental results showed that the biaxial flexural strength of IPS Empress CAD and Rosetta BM was 158.1±8.6 MPa and 172.3±8.3 MPa, respectively, where Rosetta BM had a significantly higher value (Fig. 2). Among papers that showed similar results, Ritzberger et al.[2] reported that the biaxial flexural strength of IPS Empress CAD was 160 MPa.

After measuring the flexural strength, Weibull analysis was performed on the brittle materials, which are sensitive to defects. Ceramic materials with higher Weibull coefficient (m) values can be viewed as materials with higher structural stability, and materials with relatively high Weibull coefficient ($m \geq 20$) can be considered to have a small range of error and high clinical reliability[22,25,26]. In the Weibull analysis, IPS Empress CAD and Rosetta BM showed 20.25 and 23.21, respectively (Fig. 3). Therefore, the two materials showed biaxial flexural strength values with small range of error and high clinical reliability, where Rosetta BM can be viewed as the ceramic material with greater structural stability. However, for more accurate comparison of structural stability between different materials, other factors such as strength level and fracture toughness must be considered together[27]. The fracture toughness results in the present study showed that IPS Empress CAD had a value of 1.28±0.19 $MPa \cdot m^{1/2}$ and Rosetta BM had a higher value of 1.38±0.12 $MPa \cdot m^{1/2}$ (Fig. 5). Such results were similar to the fracture toughness value of IPS Empress CAD (1.3 $MPa \cdot m^{1/2}$) reported by Ritzberger et al.[2] through measurement by the IF method.

Leucite glass-ceramics consist of a glass phase and a crystal (inorganic) phase. The glass phase has the typical characteristics of glass, such as brittleness, non-directional fracture pattern, transparency, and high surface tension in the liquid state. The crystal phase consists of leucite (potassium aluminosilicate) with high thermal expansion ($>20 \times 20^{-6}/^{o}C$), where the thermal expansion coefficient of ceramic can be controlled by the amount present, which can contribute to the strength[6]. The images of microstructures of LRGC used in the present study showed a glassy matrix and leucite crystals (Fig. 6), where Rosetta BM showed higher leucite crystal content than IPS Empress CAD, which was consistent with literature reporting that it has an impact on increase in strength[6]. Moreover, Della Bona et al.[28] also reported that greater amount and bigger size of leucite crystals in leucite ceramics can increase the fracture toughness of the materials, which can increase their strength.

The present study used LRGC materials, where the mechanical characteristics of recently developed Rosetta BM were identified by comparison to the previously developed IPS Empress CAD. As a result, clinically reliable values were obtained. In particular, Rosetta BM showed excellent biaxial flexural strength and fracture toughness, factors that have a major impact on the clinical applicability of the material being used, and together with the results of IPS Empress CAD, the findings in the present study may be used as basic information in the development of LRGC material for CAD/CAM restorations and material selection during prostheses fabrication.

The limitations of the present study were as follows. First, when prostheses are fabricated with ceramic material, the success of the prostheses depends on numerous factors including the moisture environment inside the mouth and fatigue. Second, flexural strength measurements may be affected by various factors, such as the specimen polishing process, load speed, and the area of the specimen receiving the stress. Surface quality has a very strong influence on the strength of the specimen. When cracks or porosity occur on the surface during specimen preparation, subsequent measurements may show appreciably lower flexural strength values[29]. Therefore, additional studies are needed to assess the effects of such factors. Last, clinical assessment is needed on issues that may arise during prostheses fabrication according to leucite glass-ceramic preparation methods (heat-pressed or CAD/CAM).

Future studies should conduct additional experiments to overcome the previously mentioned limitations, while focus should also be placed on studies related to the esthetic characteristics of ceramic prostheses.

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