



EVALUATION AND COMPARISON OF THE CHARACTERISTICS OF THREE PRESSABLE LITHIUM DISILICATE GLASS CERAMIC MATERIALS

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ARTICLE INFO

Article History:

Received 19th August 2017
Received in revised form 10th September, 2017
Accepted 14th October, 2017
Published online 29th November, 2017

Key Words:

Lithium disilicate, Glass ceramics, Mechanical properties, Flexural strength, Wear test, Chemical solubility.

ABSTRACT

Objective: The objective of this study was to evaluate the biaxial flexural strength, wear resistance, and acid resistance of three different lithium disilicate glass ceramic materials.

Methods: Three lithium disilicate glass ceramic materials were used in this study: GC Initial LiSi Press (LiSi; GC), IPS e.max Press (e.max; Ivoclar Vivadent), and Vintage LD Press (LD; Shofu). Biaxial flexural strength and chemical solubility tests were conducted according to ISO 6872:2015. The biaxial flexural strength test was conducted using a universal testing machine. The wear test was performed as a two-body wear test, followed by analysis via measurement of the maximum wear depth (μm) with a laser microscope. The chemical solubility test with acid was followed by analysis via measurement of the mass loss ($\mu\text{g}/\text{cm}^2$). Finally, the results were analyzed via one-way ANOVA ($\alpha = 0.05$) and Bonferroni's multiple comparison tests.

Results: The pre- and post-thermal-stress flexural strengths of all the ceramic material groups did not differ significantly. The flexural strengths of LiSi and e.max were significantly higher than that of LD. In the two-body wear test, LiSi showed the lowest wear depth (significantly lower than that of the other ceramic material groups). The wear depth of e.max was significantly lower (resp. higher) than that of LD (resp. LiSi). The chemical solubility of LiSi was the lowest, and the dissolution amount of LD was not significantly different from that of e.max.

Significance: LiSi has better mechanical properties and chemical stability than other lithium disilicate glass ceramic materials.

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Citation: Katsura Ohashi, Yuka Kameyama, Yuuki Wada, Tomoyasu Midono, Kaori Miyake, Karl-Heinz Kunzelmann and Tomotaro Nihei. 2017. Evaluation and comparison of the characteristics of three pressable lithium disilicate glass ceramic materials. *International Journal of Development Research*, 7, (11), 16711-16716.

INTRODUCTION

Recent years have witnessed a growing demand for dental aesthetics accompanied by rising concerns regarding metal allergies, resulting in the increasing use of metal-free all-ceramic restorations, which are now expected to reproduce not only the shade and luster of natural teeth but also their natural translucency (Al Ben Ali, 2014; van den Breemer *et al.*, 2017; Alkadi, 2016). All-ceramic restorations have been widely used for crowns, bridges, inlays, onlays, laminates, and occlusal veneers in both anterior and posterior regions. Therefore, their mechanical and aesthetic properties are of critical importance (Salazar Marocho *et al.*, 2016; Della Bona, 2008; Conrad, 2007).

Currently, different processing methods, such as conventional porcelain build-up, press technique, and computer-aided design/manufacturing (CAD/CAM) technology, are adopted to process various ceramic materials. Lithium disilicate glass ceramic materials, which can be processed either by lost-wax pressing or by milling via CAD/CAM, are widely employed for anterior and posterior crowns, inlays, and onlays as well as for three-unit bridges in anterior and premolar regions (Al Ben Ali, 2014; Alkadi, 2016). They exhibit high translucency and provide good tooth shade reproduction either by staining or by conventional layering of additional ceramic materials, or both.

IPS e.max Press and IPS e.max CAD have been employed as lithium disilicate glass ceramic materials, and several manufacturers are now introducing other lithium disilicate glass ceramic materials for use with the press technique. However, these materials suffer from poor clinical reliability because various data on their physical properties are separately published by the manufacturers. Many studies have reported on the physical properties of IPS e.max Press and IPS e.max CAD (van den Breemer, 2017; Nawafleh *et al.*, 2016; Kern *et al.*, 2012; Etman, 2010). On the other hand, few studies have investigated or compared the physical properties of other lithium disilicate glass ceramic materials. Therefore, the objective of this study is to evaluate and compare three pressable lithium disilicate glass ceramic materials fabricated by different manufacturers in terms of their flexural strength, wear resistance, and acid resistance. Toward this end, we hypothesize that these materials do not differ in terms of the above-mentioned physical properties.

MATERIALS AND METHODS

Sample Preparation

Table 1 summarizes the details of the three pressable lithium disilicate glass ceramic materials investigated in this study, namely GC Initial LiSi Press (LiSi; GC), IPS e.max Press (e.max; Ivoclar Vivadent), and Vintage LD Press (LD; Shofu). These ceramic materials were prepared according to each manufacturer's instructions. More specifically, for LiSi, the acrylic resin patterns were invested using LiSi Press Vest (GC), while Panamat Press (GC) was used as a press furnace for press molding the LiSi specimens with the heating and pressing programs recommended by the manufacturer. Similarly, for the e.max specimens, IPS Press VEST Speed (Ivoclar Vivadent) and Programat EP 5000 (Ivoclar Vivadent) were used for investing and press molding, respectively, according to the manufacturer's instructions. For LD, Ceravety Press and Cast (Shofu) and Panamat Press (GC) were used for investing and press molding, respectively, according to each manufacturer's instructions to complete the processing. The size of each ceramic specimen to be subjected to each test was adjusted as described below.

Scanning Electron Microscopy (SEM)

For the observation of lithium disilicate crystals in the lithium disilicate glass ceramic materials, each specimen was prepared by press molding acrylic resin patterns (diameter, 8mm; thickness, 3mm) that were invested according to the manufacturer's instructions. Subsequently, the surfaces of each specimen were polished with waterproof sandpaper (up to #4,000 grit) to obtain a mirror finish. Then, Pt-Pd particles were vapor-deposited onto the specimen surfaces using a sputtering apparatus (MSP-1S, VACUUM DEVICE) to produce a 1-nm-thick coating layer in order to observe lithium disilicate crystals under a field-emission scanning electron microscope (FE-SEM, SU-70, HITACHI).

Mechanical Properties

The biaxial flexural strength test was conducted for each lithium disilicate glass ceramic specimen according to ISO 6872:2015 (Dentistry—Ceramic materials) (ISO 6872: 2015. Dentistry – Ceramic materials; 2015.).

The specimens were prepared by press molding acrylic resin patterns (diameter, 12 mm; thickness, 1.2 mm) that were invested according to the manufacturer's instructions. Then, all the specimens surface were polished with waterproof sandpaper (up to #1,000 grit). A universal testing machine (AG-X plus Shimadzu) was used to perform the biaxial flexural strength test at a crosshead speed of 1.0 mm/min. The biaxial flexural strength σ (MPa) was calculated using the following equation:

$$= -0.2387P \left\{ (1+v) \ln \left(\frac{r_2}{r_3} \right)^2 + \left[\frac{(1-v)}{2} \right] \left(\frac{r_2}{r_3} \right)^2 \right. \\ \left. - \left\{ (1+v) \left[1 + \ln \left(\frac{r_1}{r_3} \right)^2 \right] + (1-v) \left(\frac{r_1}{r_3} \right)^2 \right\} \right\} / b^2,$$

where P is the total load causing fracture (N), v is Poisson's ratio (v=0.25), b is the specimen thickness (mm), r_1 is the radius of the supporting circle (mm), r_2 is the radius of the piston (mm), and r_3 is the radius of the specimen (mm). The biaxial flexural strength test was repeated using specimens thermo cycled in a water bath for 10,000 cycles between 5°C and 55°C with a dwell time of 30 s.

Two-body Wear Test

Each lithium disilicate glass ceramic specimen was prepared using acrylic resin patterns (diameter, 8mm; thickness, 3mm). The prepared specimens were polished with waterproof sandpaper (#600 grit) and then subjected to a two-body wear test performed in a wear tester (K655, Tokyo Giken), where by the specimens were immersed in water at 37°C and subjected to a load of 4 kg at a distance of 2 mm for 10,000 cycles. Diamond balls (diameter, 1.5 mm) were used as the antagonist material. Following the wear test, the maximum wear depth of the worn area was determined using a laser microscope (VR-3100, Keyence). Then, as described in Section 2.2, Pt-Pd particles were vapor-deposited onto the specimen surfaces using a sputtering apparatus to observe the worn surfaces under an FE-SEM (SU-70, HITACHI).

Chemical Solubility Test

The chemical solubility test with acid was performed for each lithium disilicate glass ceramic specimen according to ISO 6872:2015 (Dentistry—Ceramic materials) (10). Disk-shaped ceramic specimens were prepared by press molding acrylic resin patterns (diameter, 15 mm; thickness, 1mm) according to the manufacturer's instructions. Eventually, nine disc-shaped specimens having a surface area of 30cm² or more were prepared and subjected to the chemical solubility test after all their surfaces were polished with waterproof sandpaper (up to #1,000 grit). In the chemical solubility test, the specimens were washed with distilled water, dried at 150 °C for 4 h, and subsequently immersed in 4v/v% acetic acid solution at 80 °C for 16 h. Then, the specimens were removed from the acetic acid solution, rinsed with distilled water, and dried thoroughly at 150 °C. Each specimen was weighed with an electronic balance; then, its chemical solubility was determined from the acid-induced mass loss. Finally, as described in Section 2.2, Pt-Pd particles were vapor-deposited onto the surfaces of the specimens using a sputtering apparatus to observe the surfaces under an FE-SEM (SU-70, HITACHI).

Statistical Analysis

The means and standard deviations were calculated from the numerical results of each test, and the data were statistically

analyzed via one-way ANOVA ($\alpha=0.05$) and Bonferroni's multiple comparison tests.

RESULTS

Microstructure

Fig. 1 shows typical SEM images of each lithium disilicate glass ceramic specimen. The back-scattered electron (BSE) micrographs of the specimens showed different levels of contrast.

In the LiSi specimens, fine lithium disilicate crystals (size, 1.0–1.5 μm) were densely distributed. In the e.max specimens, many larger lithium disilicate crystals (size, 1.0–4.0 μm) were observed. The size of the crystals in the LD specimens (1.0–3.0 μm) was comparable to that of the crystals in the e.max specimens, but their distribution was sparser than that in both LiSi and e.max.

Flexural Strength

Fig. 2 shows the biaxial flexural strength of each lithium disilicate glass ceramic material.

Ceramic material	Code	Manufacturer	Lot No.	Investment material	Press furnace
GC Initial LiSi Press (LT-A)	LiSi	GC	1601191	LiSi PressVest	PANAMAT PRESS
IPS e.max Press (LT-A2)	e.max	Ivoclar Vivadent	U53476	IPS PressVest Speed	Programat EP5000
Vintage LD Press (MT-A2)	LD	Shofu	91501	Ceravety Press and Cast	PANAMAT PRESS

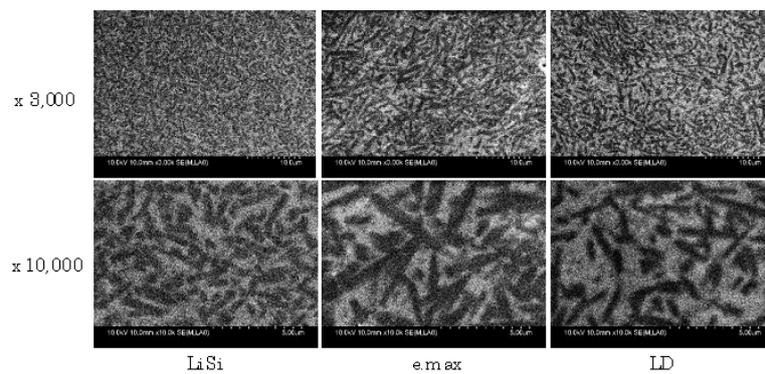


Fig. 1 Representative SEM micrographs ($\times 3,000$ and $\times 10,000$) of each lithium disilicate ceramic material. The back-scattered electron (BSE) micrographs of the specimens showed different contrast levels. The darker parts represent lithium disilicate crystals and the brighter represent the glassy matrix.

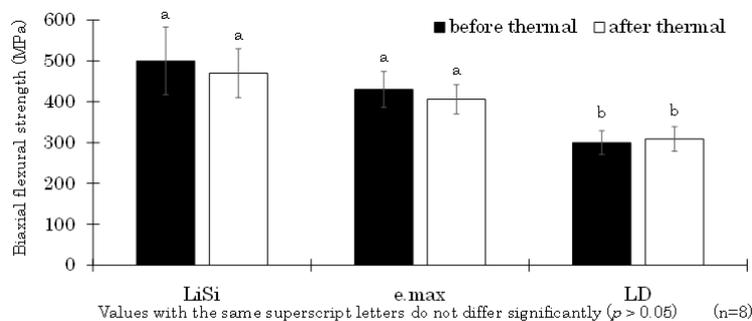


Fig. 2 Biaxial flexural strength of each lithium disilicate ceramic material before and after thermocycling for 10,000 cycles. The pre- and post-thermal-stress biaxial flexural strengths of all the ceramic groups did not differ significantly. The flexural strengths of LiSi and e.max were significantly higher than that of LD.

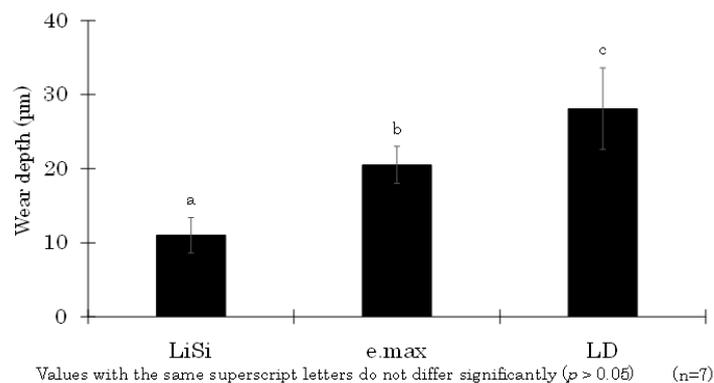


Fig. 3 Maximum wear depth of each lithium disilicate ceramic material after wear test. LiSi showed the lowest wear depth, which was significantly lower than that of the other ceramics. The wear depth of e.max was significantly lower than that of LD and significantly higher than that of LiSi.

The biaxial flexural strengths of LiSi and e.max were 500 MPa and 430 MPa, respectively, with no significant difference between them; however, the biaxial flexural strength of LD was significantly lower (300 MPa; $p < 0.05$). The biaxial flexural strengths of LiSi, e.max, and LD after the thermal cycling test were 470, 406, and 309 MPa, respectively, and the pre- and post-thermal-cycling-test flexural strengths of each ceramic material did not differ significantly.

lower than that of e.max ($13.2 \mu\text{g}/\text{cm}^2$) and LD ($14.1 \mu\text{g}/\text{cm}^2$) ($p < 0.05$). Compared with the SEM images of LiSi, those of e.max and LD showed a larger number of pits and cavities, indicating dissolution.

DISCUSSION

Ceramic materials used for aesthetic restorations are brittle. Hence, they are subject to the risk of fracture under external forces such as occlusal forces.

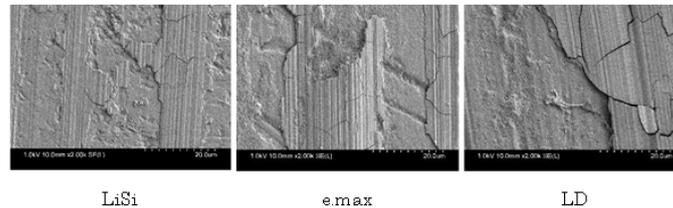


Fig. 4 Representative SEM micrographs (x 2,000) of each lithium disilicate ceramic material surface after wear test.

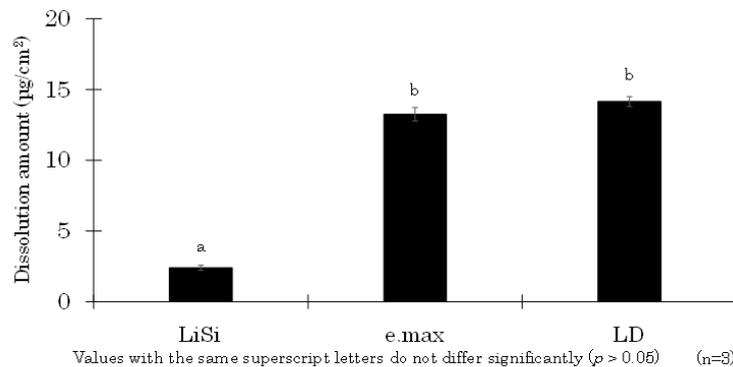


Fig. 5 Dissolution amount of each lithium disilicate ceramic material after chemical solubility test. The dissolution amount of LiSi in acid was the lowest, while those of e.max and LD did not differ significantly.

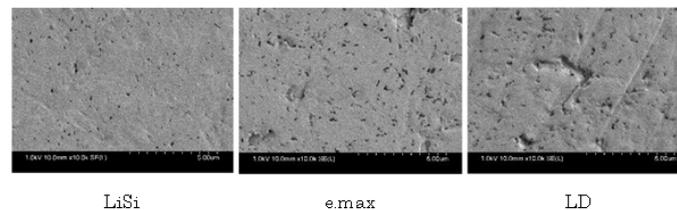


Fig. 6 Representative SEM micrographs (x 2,000) of each lithium disilicate ceramic material surface after chemical solubility test.

Wear Resistance

Fig. 3 shows the maximum wear depth of each lithium disilicate glass ceramic material after the two-body wear test, while Fig. 4 shows the SEM images of the worn surfaces after the wear test. The maximum wear depths of LiSi, e.max, and LD after the wear test differed significantly (11.0, 20.5, and $28.1 \mu\text{m}$, respectively < 0.05). Both worn and unworn surfaces were commonly present in the SEM image of each ceramic specimen after the wear test, and large and small cracks were observed on the worn surfaces. The level difference between the worn and unworn surfaces was greater in LD than in LiSi and e.max.

Acid Resistance

Figs. 5 and 6 show the dissolution amounts and SEM images, respectively, of each lithium disilicate glass ceramic material after the chemical solubility test. The dissolution amount of LiSi was $2.4 \mu\text{g}/\text{cm}^2$, which was significantly

Therefore, ISO has defined requirements for the flexural strength and chemical solubility of dental ceramics according to their intended use with specific examples (ISO 6872: 2015. Dentistry – Ceramic materials; 2015.). Lithium disilicate glass ceramics, where lithium disilicate crystals precipitate in the glassy matrix, exhibit greater strength than conventional porcelain or leucite crystal-reinforced glass ceramics (van den Breemer, 2017; Alkadi, 2016). In the 1950s, lithium disilicate glass ceramic material was discovered to offer superior resistance to heat and impact. Since then, it has been employed in micro fabrication applications and as a substrate for hard disks (Pinckney, 2000). Lithium disilicate glass ceramic materials were commercialized as dental materials in 1998. Since then, they have been widely employed and clinically integrated as stronger and more aesthetic materials than conventional ceramics, and they have been extended to metal-free aesthetic restorations. Recently, several manufacturers have marketed novel lithium disilicate glass ceramic systems, thereby promoting their clinical

application. Although many studies have reported on e.max (Pinckney, 2000; Nawafleh, 2016; Kern, 2012; Etman, 2010), no reports on newly developed lithium disilicate glass ceramic materials are currently available. Therefore, we evaluated and compared the mechanical properties of three pressable lithium disilicate glass ceramics.

The size of lithium disilicate crystals observed in the SEM images of the e.max specimens was comparable to that reported previously (length, 3–6 μm ; width, 0.5–0.8 μm) (Denry, 2010). It has been reported that IPS e.max Press consists of approximately 70vol% lithium disilicate crystals ($\text{Li}_2\text{Si}_2\text{O}_5$), which translates into 30–35 vol% glassy matrix ($\text{SiO}_2\text{-Li}_2\text{O-K}_2\text{O-ZnO-P}_2\text{O}_5\text{-Al}_2\text{O}_3\text{-La}_2\text{O}_3$) (Höland *et al.*, 2016; Hench, 2010). The major physical properties reported for IPS e.max Press include fracture toughness (2.5–3.0MPa $\text{m}^{1/2}$), modulus of elasticity (95GPa), Vickers hardness (5.9GPa), and density (2.5g/cm³) (Denry, 2010). Furthermore, the following physical properties of IPS e.max CAD have been reported: fracture toughness (1.8–2.1MPa $\text{m}^{1/2}$), three-point flexural strength (320–380MPa), modulus of elasticity (60GPa), and Vickers hardness (5.5GPa) (Elsaka, 2010). In this study, the biaxial flexural strength test and the chemical solubility test were conducted according to ISO 6872:2015 (Dentistry – Ceramic materials) (ISO 6872: 2015. Dentistry – Ceramic materials; 2015) in order to evaluate the mechanical strength and chemical stability, respectively. The biaxial flexural strengths of LiSi and e.max did not differ significantly, but that of LD was significantly lower. This might be explained from the SEM observation of lithium disilicate crystals. Although the crystals of LiSi and e.max differ in size, both have a similar volume ratio of crystal to glassy matrix. On the other hand, the crystals are sparser in LD, suggesting a higher proportion of the glassy matrix. Such a difference might affect the extension of cracks. Presumably, in the biaxial flexural strength test, lithium disilicate crystals can offer resistance against crack propagation, which might be facilitated in the glassy matrix. Thus, the densely distributed lithium disilicate crystals in LiSi and e.max can inhibit crack growth more strongly than in LD, which may, in turn, contribute to their higher flexural strengths.

The biaxial flexural strengths of all three lithium disilicate glass ceramics did not decrease significantly after the thermal cycling test, which contradicted our assumption that the thermal cycling test accelerates the degradation of the glass components (Joshi *et al.*, 2014). Thus, in this study, the thermal cycling test did not affect the biaxial flexural strength. In the chemical solubility test with acid, LiSi showed significantly lower solubility than e.max and LD. Rather than the lithium disilicate crystals, the glassy matrix is considered to be responsible for chemical solubility. SEM observations of the lithium disilicate crystals revealed a higher proportion of the glassy matrix in LD; by contrast, LiSi exhibited densely distributed lithium disilicate crystals in a relatively small volume of the glassy matrix. Accordingly, this could result in LD showing higher chemical solubility than LiSi. Furthermore, the SEM images of LD showed a large number of pits and cavities on the surface, which may be attributed to dissolution. By contrast, few pits and cavities were observed in the case of LiSi. Although the SEM images of lithium disilicate crystals in LiSi and e.max were similar, indicating comparable proportions of the glassy matrix, the solubilities of LiSi and e.max in acid differed significantly.

The SEM image of e.max showed less evidence of dissolution compared to that of LD, but more pits and cavities were observed than those in the case of LiSi. This finding might explain the differences in solubility. Thus, these results indicate the difference between LiSi and e.max in terms of the solubility of the glassy matrix. In the two-body wear test for evaluating the wear properties, we measured the maximum wear depths of the lithium disilicate glass ceramics and found that they differed significantly. The SEM observations after the wear test showed that the level difference between worn and unworn areas was greater and clearer in LD, with a greater maximum depth value, than in LiSi. Moreover, the percentage of unworn area was smaller in LiSi, which had the lowest maximum depth value. The findings from the SEM observation suggest that the wear during the two-body wear test occurred through surface delamination caused by the propagation of cracks generated on the surface. In LD, the larger cracks on the worn surface were assumed to have caused larger and deeper abrasion, eventually leading to greater wear depth. By contrast, in LiSi, which had a smaller wear depth value, higher surface resistance to crack initiation during the two-body wear test was considered to limit crack growth as well as the abrasion area (if any cracks were generated), ultimately resulting in reduced wear.

Dupriez *et al.* and Peng *et al.* observed large cracks on the worn surface in their wear study of e.max, and they reported that trans granular fracture, fragmentation, and pulverization of crystals occurred in the wear process (Peng *et al.*, 2016; Dupriez, 2015). Similarly, in our study, observations after the wear test revealed large and small cracks as well as a delamination-like appearance on the surface. Owing to the increased surface roughness of the ceramic material through wear, antagonists, such as opposing teeth substance and other materials softer than the ceramic material, may be subject to a higher risk of abrasion (Hudson, 1995; Jagger, 1995; Olivera, 2008; Gore, 1997; Saiki *et al.*, 2016). However, in another study that compared the wear quantities of the enamel antagonists in the case of clinically placed metal-ceramic crowns and e.max crowns, e.max was found to undergo less wear than the metal-ceramic material and caused less wear of the antagonists, indicating that a material with superior physical properties does not necessarily have negative effects on the antagonists. This suggests that highly polished ceramic surfaces that remain intact are more likely to cause less damage to the antagonists. Among the lithium disilicate glass ceramics examined in this study, LiSi demonstrated flexural strength equal to or greater than that of e.max, greater resistance to wear, and smaller level difference between worn and unworn surfaces than the other materials. In addition, LiSi showed lower solubility in acid as well as less surface roughness than the other materials. Thus, we can conclude that LiSi has superior physical properties and chemical stability, and it is capable of reducing antagonist wear. Furthermore, based on these results, we can reject the initial hypothesis that there is no difference among materials provided by various manufacturers in terms of their flexural strength, wear resistance, and acid resistance. In summary, this study focused on evaluating the mechanical properties and chemical stability of novel lithium disilicate glass ceramic materials. Further evaluation of these ceramics will require basic studies for evaluating the adhesion between the

ceramics and dental substrates with resin cements, as well as clinical studies.

Conclusion

This study was conducted to evaluate and compare three pressable lithium disilicate glass ceramic materials provided by various manufacturers in terms of their flexural strength, wear resistance, and acid resistance. The following results were obtained.

- All three lithium disilicate glass ceramics exhibited different sizes and distributions of lithium disilicate crystals.
- The biaxial flexural strengths of LiSi and e. max did not differ significantly, but that of LD was significantly lower.
- The wear depth after the two-body wear test was the smallest in the case of LiSi, followed by e.max, and LD.
- The dissolution amount of LiSi in acid was significantly lower than that of e.max and LD.

The results presented above confirm that the physical properties of the three lithium disilicate glass ceramic materials differed significantly. In addition, they indicate that Li Si possesses superior physical properties and chemical stability as a dental material.

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