Evaluation and Comparison of Mechanical Properties of Lithium Disilicate-Based CAD/CAM Blocks

(Penilaian dan Perbandingan Sifat Mekanikal Blok CAD/CAM Berasaskan Litium Disilikat)

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ABSTRACT

Lithium disilicate are commonly used in dental restoration due to its aesthetic and mechanical performance. However, the patent expiration of the IPS emax system has led to the emergence of other variations of the system. Data and studies concerning mechanical properties of these recent lithium disilicate-based CAD/CAM are scarce and it warrants for an investigation to provide scientific evidence to support its routine use. The aim of this study was to investigate and compare the mechanical properties of lithium disilicate-based CAD/CAM blocks from four different brands. Four CAD/CAM lithium disilicate brands were investigated; IPS emax, Mazic Claro, Cameo, and Tessera. Specimens (n=10) were prepared accordingly; for flexural strength ($16 \times 4 \times 1.2 \text{ mm}$) and microhardness test ($15 \times 13 \times 2 \text{ mm}$). One specimen from each brand was analysed for the microstructure, elemental composition and distribution before and after heat treatment using scanning electron microscope and energy dispersive x-ray spectroscopy. The three-point flexural strength test (n=10) and microhardness test (n=10) was performed. Data were analysed using one-way ANOVA and Dunnett's T3 test. The results showed that the highest mean flexural strength was from Group 4 Tessera (540.52 \pm 143.33 MPa). For microhardness, the highest mean was from Group 1 Mazic Claro ($667.70 \pm 9.41 \text{ HV}$). Within the four groups, statistically significant difference is noted for flexural strength and microhardness. As a conclusion, Tessera demonstrated significantly higher flexural strength than IPS emax and Cameo. All materials tested were above the threshold of 300 MPa.

Keywords: CAD/CAM; flexural strength; lithium disilicate; microhardness; microstructure

ABSTRAK

Litium disilikat kerap digunakan dalam rawatan pergigian disebabkan sifat mekanikal dan estetiknya yang memberangsangkan. Paten IPS emax CAD tamat tempoh membawa kepada kemunculan variasi lain. Walau bagaimanapun, data dan kajian mengenai sifat mekanikal CAD/CAM litium disilikat baru-baru ini adalah terhad dan ia memerlukan kajian demi menyediakan bukti saintifik untuk menyokong penggunaan hariannya. Matlamat kajian ini adalah untuk membandingkan sifat mekanikal blok CAD/CAM litium disilikat daripada empat jenama berbeza. Empat jenama CAD/CAM litium disilikat telah dikaji; IPS emax, Mazic Claro, Cameo dan Tessera. Spesimen (n = 10) disiapkan mengikut dimensi; untuk kekuatan lentur $(16 \times 4 \times 1.2 \text{ mm})$ dan ujian mikrokekerasan $(15 \times 13 \times 2 \text{ mm})$. Satu spesimen daripada setiap jenama dianalisis untuk struktur mikro, komposisi unsur dan pengedaran sebelum dan selepas rawatan haba menggunakan Mikroskop Elektron Pengimbasan dan spektroskopi sinar-x penyebaran Tenaga. Ujian kekuatan lentur tiga mata (n=10) dan ujian mikrokekerasan (n=10) telah dilakukan. Data dianalisis menggunakan ANOVA sehala dan ujian pasca hoc T3 Dunnett. Keputusan menunjukkan purata kekuatan lenturan tertinggi adalah daripada Kumpulan 4 Tessera (540.52 ± 143.33 MPa). Untuk kekerasan mikro, min tertinggi ialah daripada Kumpulan 1 Mazic Claro (667.70 ± 9.41 HV). Dalam empat kumpulan, perbezaan ketara secara statistik dicatatkan untuk kekuatan lentur dan kekerasan mikro. Secara kesimpulan, Tessera menunjukkan kekuatan lenturan yang lebih tinggi daripada IPS emax dan Cameo. Mazic dan Tessera menunjukkan kekerasan mikro yang lebih tinggi daripada IPS emax dan Cameo. Semua bahan yang diuji melebihi ambang 300 MPa.

Kata kunci: CAD/CAM; kekerasan mikro; kekuatan lentur; litium disilikat; struktur mikro

INTRODUCTION

Teeth play a vital role in a person's quality of life. Tooth loss negatively affects a person's systemic health, social function, and psychologic status. According to the European Prosthodontics Association, prosthodontics is one of the branches in dentistry that deals with the replacement of missing teeth and the associated soft and hard tissues by prostheses (crowns, bridges, dentures) which may be fixed, removable, or may be supported and retained by implants.

Ceramics have since become the restorative material of choice for its aesthetics, inertness, and biocompatibility. The dental community's increased interest in glass ceramics is related to its combination of good physical and chemical properties, such as excellent aesthetics, translucency, low thermal conductivity, biocompatibility, adequate strength, wear resistance, and chemical durability (Ritzberger et al. 2010). Over the past few years, many studies had been conducted towards advancement and evolution of dental ceramics in regard to mechanical properties and manufacturing technology. Amongst the prominent advancements and evolution is the introduction of lithium disilicate glass ceramics which was first introduced in 1998, and marketed as IPS Empress 2 (Ivoclar Vivadent, Lichtenstein), which composed of approximately 65% crystalline lithium disilicate filler and 35% glass (Guazzato et al. 2004). Few years later, Ivoclar Vivadent released and patented an improved version of their lithium disilicate glass ceramics called IPS emax (Ivoclar Vivadent, Lichtenstein) which composed of fine-grain lithium disilicate crystals (~70%) that are embedded in the glass matrix (Ivoclar Vivadent 2011). It is available in two forms; press ingot that utilises the lost wax technique (IPS emax Press) and machinable block that utilizes CAD/CAM (Computer Aided Design/Computer Aided Manufacturing) milling technique (IPS emax CAD). As digital dentistry became increasingly popular and there were promising advances in CAD/CAM methods, IPS emax CAD was introduced in 2006 as a lithium disilicate glass ceramic which utilizes the CAD/CAM workflow (Zarone et al. 2016). The CAD/CAM workflow has many advantages such as reduced chairside time, more predictable treatment outcome, and is cost-effective.

The patent expiration of IPS emax press and IPS emax CAD, which are considered the gold standard, led to the emergence of other variations of the being made available in the market. Chairside Economical Restoration of Aesthetic Ceramic (CEREC) TesseraTM CAD, Mazic Claro CAD and Cameo Aidite CAD are amongst the products that entered the market ever since year 2016 and have been utilised by clinicians for the fabrication of restorations. The manufacturers of each material have claimed that they have similar mechanical properties as the IPS emax system. However, data and studies concerning mechanical properties of these recent lithium disilicate-based CAD/CAM blocks are scarce and it warrants for an investigation to provide scientific evidence to support its routine use as mechanical properties are important factors for the clinical success and longevity of a restoration.

The aim of this study was to investigate and compare the mechanical properties of four different lithium disilicate-based CAD/CAM blocks. The objectives of this study were to compare the flexural strength and microhardness of the tested materials and to analyse and compare the microstructure, elemental composition and distribution of the tested materials before and after heat-treatment. The null hypothesis was that there is no significant difference in the flexural strength and microhardness of the four lithium disilicate-based CAD/ CAM blocks.

MATERIALS AND METHODS

SPECIMEN PREPARATION

The four brands of lithium disilicate-based CAD/ CAMblocks were included in this study are IPS e-max CAD, Mazic Claro CAD, Cameo CAD, and Tessera CAD. Specimens were obtained by cutting the blocks with a low-speed precision water-cooled diamond saw, Micracut 125 (metkon, Greenville, USA). The final crystallization of the lithium disilicate CAD/CAM blocks were performed using Programat EP 5000 (Ivoclar Vivadent, Schaan, Liechtenstein). The specimens (n=40) that were tested for flexural strength were polished and finished with water-cooled silica carbide papers using BETA Twin Variable Speed Grinder-Polisher (Buehler, Illinois, USA) up to #1200 grit, until the rectangular dimension stated by ISO 6872:2015 is obtained; $16.0 \pm$ 0.2 mm in length, 4.0 \pm 0.2 mm in width, and 1.2 \pm 0.2 mm in thickness. A 45° edge chamfer will be made at each sharp edge of the specimens. As for the appraisal of microhardness (n=40), the materials were prepared according to dimensions which are approximately 15 mm in length, 13 mm in width, and 2 mm in thickness. All the specimens were measured using digital calliper, Mitutoyo ABSOLUTE (Mitutoyo, Kanagawa, Japan).

FLEXURAL STRENGTH TEST

Ten specimens of each material that were prepared according to ISO 6872:2015 were subjected to a threepoint bending test using the universal testing machine (AG-X Series Shimadzu, Kyoto, Japan). The specimens were subjected to a loading tip that is operating at a crosshead speed of 1 mm/min. Test was carried out in dry conditions and at room temperature. The fracture load was recorded in N and the flexural strength (σ) was calculated in MPa by using the following equation:

$$\sigma = 3Pl/(2wb^2) \tag{1}$$

where P is the fracture load in N; l is the span (distance between the centre of the supports) in mm; w is the width in mm; and b is the height in mm of the specimen.

Microhardness test

Ten specimens of each glass-ceramic that has undergone heat-treatment were prepared for this Vickers indentation technique test. Vickers microhardness testing machine (HMV-FA, Shimadzu, Kyoto, Japan) with a load of 19.62 N and a dwell time of 10 s was utilized to determine the surface microhardness of glass ceramics. The two indent diagonals were measured immediately after the load is released. The average value of the indent diagonals was used to calculate the Vickers hardness (HV, GPa) according to ASTM C 1327-08, using the following formula:

$$HV=1.854F/d^2$$
 (2)

where d is the area of indentation (mm²); and F is the applied load (kgf).

Microstructure evaluation

The lithium disilicate CAD/CAM blocks were evaluated before and after furnace-mediated heat treatment process. Specimens (n=8, 2 from each group) were polished and finished with water-cooled silica carbide papers of #600, #800, and #1200. Then, they were etched for 20 s (except CEREC Tessera specimens, which were etched for 30 s according to manufacturer's instructions) with 4.9% hydrofluoric acid (IPS Ceramic Etching Gel, Ivoclar Vivadent AG, Schaan, Liechtenstein), rinsed out with running water for the acid removal, ultrasonically vibrated with a 95% alcohol solution for 3 min, and air dried with an oil-free stream. Specimens were platinumcoated in a vacuum sputter coater (JEC-3000FC, Auto Fine Coater, JEOL USA, Inc.) and secured to scanning electron microscope (SU8030, Hitachi, Tokyo, Japan). Surfaces were observed at x10,000 magnifications for crystals morphology and orientation. Scanning electron microscope (SEM) images were then transferred to an image processing software (ImageJ, National Institutes of Health, USA) to measure the grain size. Randomisation of grain selection was performed by drawing two diagonal lines across the SEM images, and the grains that touched the lines drawn were selected for

measurement taking. Fifteen grain size measurements were taken for each SEM image, and the average grain size was recorded.

ELEMENTAL COMPOSITION AND ELEMENTAL DISTRIBUTION

In conjunction with SEM analysis, each specimen's surface was scanned using energy dispersive spectroscopy (EDS) device, SU8030 (Hitachi, Tokyo, Japan). Elemental composition was obtained for each specimen, evaluating the whole area displayed at x10,000 magnification. A typical chemical elements composition summary was recorded for each material before and after heat treatment. Then, EDS mapping was performed at the same magnification and eight frames resolution to obtain representative maps of the chosen elements, which are Silica (Si) and Zirconia (Zr).

DATA ANALYSIS STRATEGY

The statistical analysis was carried out using SPSS version 27.0 (IBM SPSS Inc, Chicago, IL, USA). Shapirowilk test was done to check for normality of data. The data obtained will be analysed by One-way ANOVA and post-hoc Dunnett's T3 test to compare the flexural strength and microhardness of the four brands of lithium disilicate-based CAD/CAM blocks.

RESULTS AND DISCUSSION

The flexural strength (MPa) of the tested materials is plotted in Figure 1.

According to one-way ANOVA, the p-value between the groups is 0.001, which means that there is a significant difference between the groups. Post-hoc test showed that there was a significant difference (p<0.05) between Group 4 (Tessera), and Group 2 (IPS emax) as well as between group 4 (Tessera) and Group 3 (Cameo) with a mean difference of 159.402 and 182.905, respectively. Results showed no significant difference (p>0.05) between Group 1 (Mazic Claro) and Group 4 (Tessera) despite the high mean difference of 166.579 due to the large standard deviation in both groups. Based on the mean difference, the most significant difference in flexural strength is between Group 4 (Tessera) and Group 3 (Cameo). The microhardness (HV) of the tested materials is plotted in Figure 2.

According to one-way ANOVA, the p-value between the groups is <0.001, which means that there is a significant difference between the groups. Post-hoc test showed there was a significant difference (p<0.05) between Group 1 (Mazic Claro), with Group 2 (IPS emax) and Group 3 (Cameo) with a mean difference of 35.2 and 25.6, respectively. Another set of groups that shows

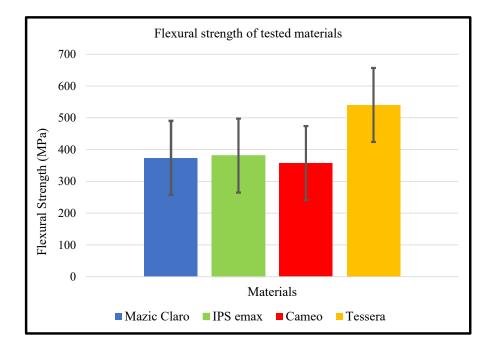


FIGURE 1. Flexural strength of tested materials

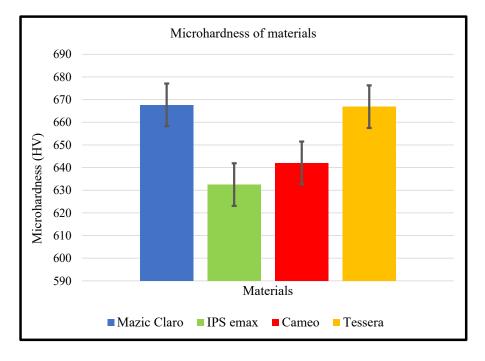


FIGURE 2. Microhardness of tested materials

a significant difference (p<0.05) is between Group 4 (Tessera), with Group 2 (IPS emax) and Group 3 (Cameo) with a mean difference of 34.4 and 24.8, respectively.

Table 1 shows the SEM images of the specimens before and after heat treatment. The SEM images for

Mazic Claro showed a homogenous and interlocked irregular-shaped crystals before and after heat treatment. The microstructure of the Mazic Claro specimen after heat treatment appears to have lesser porosity and more interlocking between crystals was observed. SEM images for IPS emax showed that there is a mixture of rodshaped and platelet-shaped crystals that are interlocking with each other before and after heat treatment. The microstructure of the IPS emax after heat treatment appears to be denser and less porosity was noted. SEM images for Cameo showed that there is a mixture of rodshaped and platelet-shaped crystals that are interlocking with each other before and after heat treatment.

SEM images for Tessera showed a homogenous platelet-shaped crystals that are interlocking with each other before and after heat treatment. The microstructure of the Tessera specimen after heat treatment appears to be denser and less porosity was observed.

Materials	Before heat treatment	After heat treatment
Mazic Claro	NAZIE AFTE 5 000 420mm x10 9k SEUL)	Mitzle APTE 6 0KV-48pm gto gik SE(UL)
IPS emax		EMAXAMENTER 5 (KV/4 Seminar) DK SE(U)
Cameo	entres de la futur que operate de la futur de	CAMEO AFTES 00 V 40mm ×10 0k. SE(UL)
Tessera		Estates are a regeneral United (1)

TABLE 1. SEM images of all the tested materials before and after heat treatment

As can be noted in Figures 3 and 4, all the tested specimens have an increase in average crystal width and length after undergoing heat treatment.

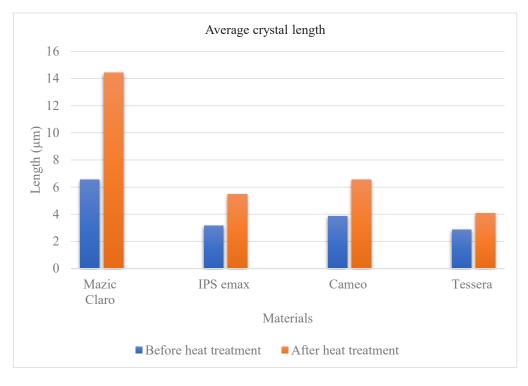


FIGURE 3. Average crystal length before and after heat treatment

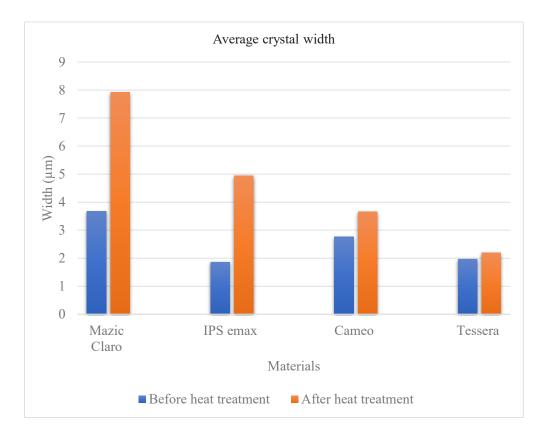


FIGURE 4. Average crystal width before and after heat treatment

The EDS analysis (Table 2) was performed to show the elements present in all the tested material. The EDS analysis confirmed that certain elements are present as claimed by the manufacturers, with an addition of other elements that were not mentioned by the manufacturer. However, Tessera is the only brand that has no zirconia element in it. In regard to the elemental distribution, the EDS mapping of all the tested materials (Table 3) showed no measured changes before and after heat treatment.

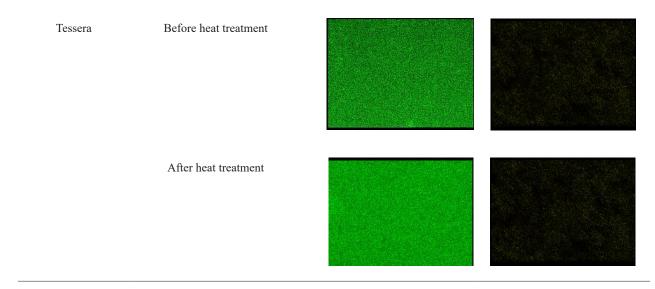
Materials –	E	Before heat treatm	nent		After heat treatme	ent
	Element	Weight %	Atomic %	Element	Weight %	Atomic %
	СК	7.57	12.69	C K	11.37	18.95
	O K	49.10	61.77	O K	44.71	55.93
	Na K	0.48	0.42	Al K	1.22	0.91
Mazic Claro	Mg K	0.17	0.14	Si K	28.44	20.26
	Al K	1.43	1.06	KK	2.77	1.42
	Si K	28.43	20.38	Ca K	1.38	0.69
	S K	0.14	0.09	Zr L	6.36	1.39
	ΚK	2.80	1.44	Ce L	1.18	0.17
	Ca K	0.86	0.43	Tb L	0.93	0.12
	Zr L	4.68	1.03	Pt M	1.65	0.17
	Ce L	1.67	0.24			
	Tb L	1.42	0.18			
	Pt M	1.25	0.13			
	Total	1	00	Total	1	00
	СК	8.70	14.95	СК	12.68	20.96
	O K	45.40	58.58	O K	41.44	51.42
	F K	0.19	0.21	F K	4.81	5.03
IPS emax	Na K	0.36	0.32	Na K	0.47	0.41
n 5 chiax	Mg K	0.19	0.16	Mg K	0.13	0.11
	Al K	1.61	1.23	Al K	1.44	1.06
	Si K	27.32	20.08	Si K	24.41	17.25
	КК	3.29	1.74	K K	2.21	1.12
	Ca K	1.91	0.98	Ca K	1.47	0.73
	Zr L	3.64	0.82	Zr L	4.95	1.08
	Ce L	3.39	0.50	Ce L	3.04	0.43
	Pt M	4.00	0.42	Pt M	1.79	0.18
				S K	0.18	0.11
				Tb L	0.98	0.12
	Total	1	00	Total	10	00

TABLE 2. EDS analysis for all the tested materials

	C K	4.26	7.66	C K	9.88	16.51
Cameo	O K	46.38	62.65	O K	47.18	59.16
	F K	0.65	0,73	F K	0.27	0.28
	Na K	1.48	1.39	Na K	0.79	0.69
	Al K	1.18	0.95	Al K	0.75	0.55
	Si K	27.86	21.44	Si K	25.04	17.89
	S K	0.15	0.10	S K	0.18	0.11
	K K	3.82	2.11	K K	2.37	1.21
	Ti K	0.28	0.12	Ti K	0.15	1.45
	Zn L	2.17	0.72	Zn L	1.21	0.06
	Zr L	4.51	1.07	Zr L	5.15	0.37
	Ce L	5.67	0.87	Ce L	3.58	1.13
	Pt M	1.60	0.18	Pt M	0.56	0.51
				Ca K	2.90	0.06
	Total	1	00	Total	1	00
	СК	3.64	6.25	СК	2.20	3.79
	O K	50.55	65.19	O K	52.02	67.25
	Na K	1.334	1.20	Na K	0.54	0.49
Tessera	Mg K	0.07	0.06	Mg K	0.13	0.11
	Al K	5.04	3.86	Al K	5.34	4.09
	Si K	27.72	20.36	Si K	29.03	21.38
	КК	3.90	2.06	ΚK	3.80	2.01
	Zr L	-0.05	-0.01	Zr L	-0.18	-0.04
	Ba L	3.77	0.57	Ba L	3.35	0.50
	Tb L	1.68	0.22	Tb L	1.37	0.18
	Pt M	2.34	0.25	Pt M	2.41	0.26

Materials	Condition of material	EDS 'Si' map	EDS 'Zr' map
Mazic Claro	Before heat treatment		
	After heat treatment		
IPS emax	Before heat treatment		
	After heat treatment		
Cameo	Before heat treatment		
	After heat treatment		

TABLE 3. EDS mapping for all the tested materials



As for discussion, flexural strength technique is a basic test to determine the resistance of ceramics and glasses to fracture (Quinn et al. 2009). The three-point bending test has been used as a standard test for measuring flexural strength (Leung et al. 2015). In order to quantify flexural strength using three-point bending tests, the ISO 6872 for dental ceramics permits samples as small as 12 mm to be used in three-point bending tests (ISO 2015). The specimens for flexural strength test in this study were prepared according to the dimensions stated in the ISO 6872 for three-point bending test.

In this study, Vickers indentation method was used to determine the microhardness of the tested materials. The application of the Vickers indentation technique in studying the behaviour and properties of brittle materials is specifically appropriate because only small dimensional specimens are required and the crack growth parameter is similar to those cracks expected in clinical conditions (Quinn, Sundar & Lloyd 2003). This approach is widely used to estimate microhardness and fracture toughness since it is simple to use, time-saving, non-destructive, and requires only a small sample with a flat and smooth surface (Serbena et al. 2015).

The introduction of lithium disilicate as a restorative material in dentistry has shown great potential as it exhibits satisfactory aesthetics and good mechanical properties. Flexural strength and microhardness are the mechanical properties that are frequently studied and are essential in aiding decision-making for selection of restorative material.

Flexural strength usually represents the ability to tolerate chewing forces (Kang, Chang & Son 2013). In the present study, the flexural strength for IPS emax CAD reported is 381.12 ± 56.72 MPa, which is similar to other studies that reported a flexural strength of 376.85 ± 39.09 MPa (Sedda et al. 2014), $(367 \pm 43.3 \text{ MPa})$ (Lien et al. 2015), $(381.04 \pm 42.02 \text{ MPa})$ (Fonzar et al. 2017), and $(364.64 \pm 66.51 \text{ MPa})$ (Al-Thobity & Alsalman 2021). It also confirms the manufacturer's claim that IPS emax CAD has a flexural strength of $(360 \pm 60 \text{ MPa})$ (Ivoclar Vivadent 2011). Previous study had shown that IPS emax CAD and IPS emax Press had no significant difference in flexural strength, suggesting that a difference in material's composition and manufacturing process may not have a significant impact on the mechanical property of the material (Fonzar et al. 2017). Dentsply Sirona claimed that Tessera has a flexural strength of more than 700 MPa (Sirona 2021), which is higher than the flexural strength reported in this study (540.52 ± 143.33 MPa). The discrepancy between the reported flexural strength and the ones claimed by the manufacturer can be due to the method of testing.

The predominant strengthening mechanism of lithium disilicate glass ceramic is correlated to the interlocking effect of the crystals in the glass ceramic. This would retard the crack progression in glass ceramics as the crack propagates in a zigzag path (crack deflection) instead of propagating in a direct path, which effectively consumes the energy of cracks. This resulted in effective strengthening (Hallmann, Ulmer & Kern 2018; Zhang et al. 2014).

Generally, glazing surface treatment reduces porosity, reduces the depth and sharpness of surface flaws, and blunts the flaw. Furthermore, the elevated temperature during glaze firing cycles leads to crack bridging by viscous flow of glass content (Denry, Holloway & Tarr 1999). Previous studies reported that glazing increases the overall mechanical strength of all-ceramic restorations (Hirao & Tomozawa 1987; Schweitzer et al. 2020), and this may be associated with the fact that Tessera exhibits the highest flexural strength and is the only brand that requires glazing in order to achieve its maximum flexural strength (as instructed by the manufacturer). However, a study that compared the flexural strength of IPS emax CAD that was unglazed and glazed according to manufacturer's instructions, has shown that there was no significant difference between the two groups (Aurélio et al. 2018). Furthermore, the SEM image of Tessera specimen after heat treatment exhibits a more dense and less porous microstructure compared to the SEM image before heat treatment, which might contribute to the superior flexural strength of that material.

Hardness is defined as the resistance to a permanent surface indentation. The microhardness reported in this study are between (632.50 ± 21.92 HV) and (677.70 ± 9.41 HV). The microhardness for the IPS emax CAD that was reported in this study is slightly lower (632.50 ± 21.92 HV) than another study which reported microhardness value of 731.63 ± 30.64 HV (Leung et al. 2015). However, all the tested materials reported microhardness value that is higher than the manufacturers' claims. The observed discrepancy might be associated with the differences in the preparation of the tested specimens.

In ceramics, hardness affects the polish-ability, wear resistance, and ease of milling (Etman 2013). An ideal restorative material should not wear the opposing dentition, as it defeats the whole purpose of dental treatment, which is to provide good oral health to the patient. Therefore, a ceramic with very high hardness value might not be desired, as it may wear the opposing tooth or it might lead to the catastrophic fracture of the tooth instead of the fracture of restoration if they are not well-polished. The hardness value of enamel reported is about 611.8 HV (Braly et al. 2007; Cuy et al. 2002), which is slightly lower to the microhardness reported for all the tested materials in this study. Besides that, ceramic with very high hardness value may make it unfavourable for prosthesis fabrication as it increases the wear rate of the milling bur, thereby, requiring frequent replacements leading to increase cost of fabrication. It is important to note that Mazic Claro, IPS emax, and Cameo are in the 'blue state' form or lithium metasilicate phase during the milling process, which makes it favourable for milling as the hardness value is lower than the fully crystallized lithium disilicate phase.

The mechanical properties of the tested materials should be considered during selection of restorative material as it influences the longevity of the restoration. However, material selection should not be solely based on the mechanical properties as there are many other aspects that should be considered such as aesthetics, marginal fitting of restoration, and others. When a ceramic dental restoration is cemented, the mechanical performances also change.

The strengthening mechanism for glass ceramics is closely associated with the interlocking microstructure which results in crack deflection that effectively retards crack propagation. The SEM images of the tested specimens after heat treatment showed an increase in the average crystal size that are interlocking with each other. The dimensions of the crystals for the fully crystallized IPS emax CAD that was reported by other studies ($\approx 1 \ \mu m$ in length and $\approx 0.4 \ \mu m$ in width) (Denry & Holloway 2004) and manufacturer's claim (0.2 to 1.0 μm) (Ivoclar Vivadent 2011) is lower to the one reported in the present study (5.48 \pm 1.10 μm in length and 4.95 \pm 1.99 μm in width).

The incorporation of ZrO₂ particles in the material's composition aims to positively affects the mechanical properties of the lithium disilicate material (Elsaka & Elnaghy 2016; Riquieri et al. 2018) by slowing down the crack propagation via transformation toughening of the zirconia particles. However, the tested materials in this study that contain Zirconium (Zr) element (IPS emax, Mazic Claro and Cameo) recorded a lower flexural strength compared to Tessera which Zr element was not detected. The Zr element is not detected probably due to weak resolution of the EDS mapping. This can be explained by a study which emphasized the role of the content of ZrO₂ towards the material's mechanical property. It was reported that a ZrO₂ content 10 wt% or below will not positively affect the strength of the lithium disilicate, as the ZrO₂ will only act as a nucleating agent or induce the precipitation of lithium metasilicate, which hinders the crystal growth. This leads to the spheroidization or spherical morphology of the lithium disilicate crystals formed, instead of rod-like

crystals (Huang et al. 2017; Lubauer et al. 2022). This can be related to the energy-dispersive x-ray analysis which shows that the Zr content in IPS emax, Mazic Claro and Cameo is below 10 wt%.

Thermal residual stress that arises upon cooling of glass ceramics due to the coefficient of thermal expansion mismatch between the crystalline and glass phase, also plays an important role in the mechanical property of glass ceramic. The mechanical properties of glass ceramics are dependent not only on their composition and microstructure but also on the type (tension or compression) and the magnitude of these thermal residual stresses. Thermal residual stress slows down crack propagation as the propagating crack will deviate from the crystalline phase, resulting in crack deflection (Serbena & Zanotto 2012). Dentsply Sirona has claimed that Tessera contains virgilite or Lithium Aluminosilicate (LAS) Li₂O-Al₂O₃-SiO₂-P₂O₅ (Sirona 2021). It is the only brand amongst the tested material in this study that contains virgilite. The LAS glass ceramics containing the beta-spodumene crystalline phase showed extremely low to negative thermal expansion coefficient (K^{-1}) ranging from -0.0029×10^{-6} to 0.3227×10^{-6} (Kumar et al. 2019). The coefficient of thermal expansion of glass matrix was estimated to be $12.2-12.8 \times 10^{-6}$ /K (Li et al. 2016; Serbena & Zanotto 2012). Therefore, the mismatch of coefficient of thermal expansion between the crystalline and glass phase may contribute to the superior mechanical property of Tessera, secondary to the phenomenon of thermal residual stress development. However, this requires further study as the mismatch between coefficient of thermal expansion should not be too excessive as it can lead to crack formation which negatively affects the mechanical property of a material.

The limitation of this study is the lack of information given by the manufacturer in regard to the composition of the materials. Besides that, the result of this study cannot be extrapolated as is to clinical situation and should be supplemented with clinical studies.

The clinical relevance of this study are: a) All the materials tested were above the threshold of 300 MPa, thus they meet the ISO 6872:2015 requirements for construction of veneers, inlays, onlays, monolithic ceramic single anterior or posterior crowns, and monolithic ceramic three-unit prosthesis not involving the molar restoration, b) All the four materials possess hardness that is quite similar to the reported hardness value of enamel, which makes it a desirable restorative material as it protects the opposing dentition from excessive wear (although wear is a complex process and is not solely related to material hardness), c) Tessera has a very dense microstructure with spherically shaped crystal compared to the other materials. The dense microstructure might render the material more opaque, which indirectly affects the aesthetic of the material.

CONCLUSIONS

The null hypothesis that there is no significant difference in the flexural strength and microhardness of the four lithium disilicate-based CAD/CAM blocks has been rejected. Tessera demonstrated significantly higher flexural strength than IPS emax and Cameo. Mazic Claro and Tessera demonstrated significantly higher microhardness than IPS emax and Cameo. There was a difference in the crystal size after the heat treatment of all four lithium disilicate CAD/CAM blocks. There were no measured changes observed in elemental composition and distribution of the four lithium disilicate CAD/CAM blocks before and after heat treatment.

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REFERENCES

- Al-Thobity, A.M. & Alsalman, A. 2021. Flexural properties of three lithium disilicate materials: An *in vitro* evaluation. *The Saudi Dental Journal* 33(7): 620-627.
- Aurélio, I.L., Prochnow, C., Guilardi, L.F., Ramos, G.F., Bottino, M.A. & May, L.G. 2018. The effect of extended glaze firing on the flexural fatigue strength of hard- machined ceramics. *The Journal of Prosthetic Dentistry* 120(5): 755-761.
- Braly, A., Darnell, L., Mann, A., Teaford, M. & Weihs, T. 2007. The effect of prism orientation on the indentation testing of human molar enamel. *Archives of Oral Biology* 52(9): 856-860.
- CEREC TesseraTM: Advanced Lithium Disilicate. 2021. chrome-extension://efaidnbmnnnibpcajpcglclefindmkaj/ https://www.dentsplysirona.com/content/dam/flagship/ sv-se/products/restorative/NORTH-BROCHURE-CEREC-TESSERA.pdf
- Cuy, J.L., Mann, A.B., Livi, K.J., Teaford, M.F. & Weihs, T.P. 2002. Nanoindentation mapping of the mechanical properties of human molar tooth enamel. *Archives of Oral Biology* 47(4): 281-291.

- Denry, I. & Holloway, J. 2004. Effect of post-processing heat treatment on the fracture strength of a heat-pressed dental ceramic. *Journal of Biomedical Materials Research Part B: Applied Biomaterials* 68B(2): 174-179.
- Denry, I., Holloway, J., & Tarr, L. 1999. Effect of heat treatment on microcrack healing behaviour of a machinable dental ceramic. *Journal of Biomedical Materials Research* 48(6): 791-796.
- Elsaka, S.E. & Elnaghy, A.M. 2016. Mechanical properties of zirconia reinforced lithium silicate glass-ceramic. *Dental Materials* 32(7): 908-914.
- Etman, M. 2013. Wear properties of dental ceramics. In Nonmetallic Biomaterials for Tooth Repair and Replacement, edited by Vallittu, P. Woodhead Publishing. pp. 161-193.
- Fonzar, R.F., Carrabba, M., Sedda, M., Ferrari, M., Goracci, C. & Vichi, A. 2017. Flexural resistance of heat-pressed and CAD-CAM lithium disilicate with different translucencies. *Dental Materials* 33(1): 63-70.
- Guazzato, M., Albakry, M., Ringer, S.P. & Swain, M.V. 2004. Strength, fracture toughness and microstructure of a selection of all-ceramic materials. Part I. Pressable and alumina glass-infiltrated ceramics. *Dental Materials* 20(5): 441-448.
- Hallmann, L., Ulmer, P. & Kern, M. 2018. Effect of microstructure on the mechanical properties of lithium disilicate glass-ceramics. *Journal of the Mechanical Behaviour of Biomedical Materials* 82: 355-370.
- Hirao, K. & Tomozawa, M. 1987. Dynamic fatigue of treated high-silica glass: Explanation by crack tip blunting. *Journal* of the American Ceramic Society 70(6): 377-382.
- Huang, S., Li, Y., Wei, S., Huang, Z., Gao, W. & Cao, P. 2017. A novel high-strength lithium disilicate glass-ceramic featuring a highly intertwined microstructure. *Journal of the European Ceramic Society* 37(3): 1083-1094.
- ISO 2015. 6872: 2015. *Dentistry-Ceramic Materials*. Helsinki: Suomen Standardoimisliitto.
- Ivoclar Vivadent. 2011. IPS e. max CAD Scientific Documentation. In: Lichtenstein.
- Kang, S.H., Chang, J. & Son, H.H. 2013. Flexural strength and microstructure of two lithium disilicate glass ceramics for CAD/CAM restoration in the dental clinic. *Restorative Dentistry & Endodontics* 38(3): 134-140.
- Kumar, A., Chakrabarti, A., Shekhawat, M.S. & Molla, A.R. 2019. Transparent ultra- low expansion lithium aluminosilicate glass-ceramics: Crystallization kinetics, structural and optical properties. *Thermochimica Acta* 676: 155-163.
- Leung, B.T., Tsoi, J.K., Matinlinna, J.P. & Pow, E.H. 2015. Comparison of mechanical properties of three machinable ceramics with an experimental fluorophlogopite glass ceramic. *The Journal of Prosthetic Dentistry* 114(3): 440-446.

- Li, D., Guo, J., Wang, X., Zhang, S. & He, L. 2016. Effects of crystal size on the mechanical properties of a lithium disilicate glass-ceramic. *Materials Science and Engineering*: A 669: 332-339.
- Lien, W., Roberts, H.W., Platt, J.A., Vandewalle, K.S., Hill, T.J. & Chu, T.M. 2015. Microstructural evolution and physical behavior of a lithium disilicate glass- ceramic. *Dental Materials* 31(8): 928-940.
- Lubauer, J., Belli, R., Peterlik, H., Hurle, K. & Lohbauer, U. 2022. Grasping the lithium hype: Insights into modern dental lithium silicate glass-ceramics. *Dental Materials* 38(2): 318-332.
- Quinn, G.D., Sparenberg, B.T., Koshy, P., Ives, L.K., Jahanmir, S. & Arola, D.D. 2009. Flexural strength of ceramic and glass rods. *Journal of Testing and Evaluation* 37(3): 222-244.
- Quinn, J., Sundar, V. & Lloyd, I.K. 2003. Influence of microstructure and chemistry on the fracture toughness of dental ceramics. *Dental Materials* 19(7): 603-611.
- Riquieri, H., Monteiro, J.B., Viegas, D.C., Campos, T.M.B., de Melo, R.M. & Saavedra, G.d.S.F.A. 2018. Impact of crystallization firing process on the microstructure and flexural strength of zirconia-reinforced lithium silicate glass- ceramics. *Dental Materials* 34(10): 1483-1491.
- Ritzberger, C., Apel, E., Höland, W., Peschke, A. & Rheinberger, V.M. 2010. Properties and clinical application of three types of dental glass-ceramics and ceramics for CAD-CAM technologies. *Materials* 3(6): 3700-3713.
- Schweitzer, F., Spintzyk, S., Geis-Gerstorfer, J. & Huettig, F. 2020. Influence of minimal extended firing on dimensional, optical, and mechanical properties of crystalized zirconia-reinforced lithium silicate glass ceramic. Journal of the Mechanical Behavior of Biomedical Materials 104: 103644.
- Sedda, M., Vichi, A., Del Siena, F., Louca, C. & Ferrari, M. 2014. Flexural resistance of Cerec CAD/CAM system ceramic blocks. Part 2: Outsourcing materials. *American Journal of Dentistry* 27(1): 17-22.
- Serbena, F., Mathias, I., Foerster, C. & Zanotto, E. 2015. Crystallization toughening of a model glass-ceramic. Acta Materialia 86: 216-228.
- Serbena, F.C. & Zanotto, E.D. 2012. Internal residual stresses in glass-ceramics: A review. *Journal of Non-crystalline Solids* 358(6-7): 975-984.
- Zarone, F., Ferrari, M., Guido Mangano, F., Leone, R. & Sorrentino, R. 2016. "Digitally oriented materials": Focus on lithium disilicate ceramics. *International Journal of Dentistry* 2016(1): 9840594.
- Zhang, P., Li, X., Yang, J. & Xu, S. 2014. The crystallization and microstructure evolution of lithium disilicate-based glass-ceramic. *Journal of Non-crystalline Solids* 392: 26-30.

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