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Measuement of Translucency, Biaxial Flexural Strength, and Radiopacity of Different Lithium Disilicate Materials

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MEASUREMENT OF TRANSLUCENCY, BIAXIAL FLEXURAL STRENGTH, AND RADIOPACITY OF DIFFERENT LITHIUM DISILICATE MATERIALS

by

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A Thesis

Submitted to the graduate faculty of The University of Alabama at Birmingham, in partial fulfillment of the requirements for the degree of

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MEASUREMENT OF TRANSLUCENCY, BIAXIAL FLEXURAL STRENGTH AND RADIOPACITY AND OF DIFFERENT LITHIUM DISILICATE MATERIALS

PRESHTHA MANGLA

DENTISTRY

ABSTRACT

Objective: To measure translucency parameter, biaxial flexural strength, radiopacity and microstructure of various lithium disilicate materials used for dental restorations.

Materials and Methods: CAD/CAM lithium disilicate glass-ceramic blocks: IPS e.max CAD (Ivoclar Vivadent), CerecTessera (Dentsply Sirona), and Amber Mill (Hassbio America) were used (shade A2, HT, and MT). Blocks of each material were milled (PrograMill PM7, Ivoclar Vivadent) into cylinders (diameter = 14 mm) and cut using circular sectioning saw (IsoMET 1000 Precision Saw, Buehler) into disc-shaped specimens (thickness = 1.00 ± 0.05 mm) (n=5/group). They were hand polished to 1200 grit with SiC paper under water lubrication. The translucency parameter of uncrystallized (except IPS e.max CAD) and crystallized samples were tested against a white and black background (with glycerin gel) using a spectrophotometer (UltraScan VIS, HunterLab). A biaxial flexural strength test was performed following ISO 6872. Each specimen was placed centrally on three hardened steel balls (with a diameter of 3.2 mm, positioned 120° apart on a support circle with a diameter of 10 mm). The maximum load to fracture failure of each specimen was recorded using a universal testing machine (Instron, Model #33R4204 Norwood, MA) with a crosshead speed of 1 mm/min. Same specimens were tested for radiopacity using the aluminum wedge. SEM analysis was also done.

Results: Data were analyzed using one-way ANOVA and post hoc Tukey's HSD statistical analysis (p= 0.05). Materials with significantly different values are denoted with different superscripts. Under SEM imaging, emax CAD demonstrated long spindle shape crystals. Tessera and Amber Mill contained finer platelet-shaped crystals and Tessera contained a virgilite phase.

Conclusion: Once crystallized, all lithium disilicate materials produced similar translucency despite the smaller crystalline microstructure seen in Tessera and Amber Mill. Despite the presence of an additional Virgilite phase in Tessera, it did not produce increased biaxial flexural strength. Cerec Tessera showed the highest radiopacity amongst all groups.

Keywords: Lithium Disilicate, Biaxial Flexural Strength, Microstructure, IPS Emax, Tessera, Ambermill.

DEDICATION

I dedicate my dissertation work to my family and friends. A special feeling of gratitude for my loving parents, Deepti and Satish Mangla, and my maasi, Shashi Bala, whose words of encouragement and push for tenacity ring in my ears.

I also dedicate this dissertation to my pillars of strength, my brothers Dr. Shashideep Singhal, Dr. Shashikant Singhal, Dr. Ram Prasad, and Shreshth Mangla, who have never left my side. To my sister-in-laws Dr. Shilpa Jain, Dr. Kirty Pathak, and Dr. Jyoti Aggarwal who have supported me throughout the process. I will always appreciate all they have done, for helping me to master the leadership dots.

I dedicate this work and give special thanks to my best friend Aditya Pampana for being there for me and being my best cheerleader.

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Abbreviations Ceramic Translucency EHT IPS Emax High EMT | IPS Emax Medium THT (uTHT) Cerec Tessera (Unfired Cerec Tessera) High TMT (uTMT) Cerec Tessera (Unfired Cerec Tessera) Medium AMH (uAM) AmberMill (Unfired AmberMill) High AMM AmberMill Medium

LIST OF ABBREVIATIONS

1. INTRODUCTION

Based on a Practice-based Research Network study, 21% of dentists preferred LDS (lithium disilicate) for posterior and 54% for anterior restorations [1]. These material preferences demonstrate the current transition from the traditional metal-ceramic restorations to those composed entirely of ceramic. The use of LDS for dental restorations was introduced in 1984 by Corning Inc $[2,3]$. The first glass-ceramic introduced for use in dentistry was IPS empress II by Ivoclar Vivadent in the 1990s. This ceramic was fabricated using the lost wax technique. The second generation for LDS was IPS e.max Press, fabricated using the heat press technique. Later in 2005, IPS e.max CAD was introduced. It is a machinable block and uses milling for fabrication. This material is used in dentistry for over 20 years. In the current times, there are multiple LDS materials introduced by many companies.

LDS glass-based ceramics consist of $Li₂Si₂O₅$ crystals and lithium orthophosphate (Li3PO4) crystals (in minor amounts) that are randomly oriented and uniformly dispersed in a glassy matrix $[4]$. The presence of P₂O₅ promotes volume nucleation of the lithium silicate phases by acting as a heterogeneous nucleating agent $^{[4]}$. After nucleation, crystal growth occurs which imparts improved mechanical and physical properties by maximizing the presence of crystals and the generation of compression stress around the crystals ^[5].

There are two types of interfaces in glass-ceramics: the interface between crystalline phases and the interface between the crystalline phase and the glassy matrix [6]. The translucency of an LDS ceramic can be adjusted by varying the refractive index of the crystalline phase and the glassy matrix, and the light path at the interface between these two phases ^[8]. The crystalline phases of the ceramic can affect its strength. Crystals have discrete structural plans that cause deflection, branching, or splinting of cracks. The presence of these cleavage planes and grain boundaries prevents fracture propagation^[7].

When preparing LDS ceramics for oral use, they require the additional processing steps of milling and post-mill crystallization [9,10]. Initially, there are small size crystals, which on exposure to heat enlarge in size. The enlarged crystals further form colonies that inter-tangle with each other. This gives this material a meshwork-like structure. Postmilling crystallization results in dendritic (tree-like or sheaf-like with significant branching) or Spherulitic (subparallel needlelike crystallites radiating from the center and forming spherical mass) morphology. These acicular structures help to achieve high strength and fracture toughness in glass-ceramics ^[7]. Ceramic machinability during milling is essential as low milling accuracy can cause errors in dental prostheses leading to crown-tooth margin discrepancies and clinical failure $[11, 12]$.

1.1 Ambermill

AmberMill (AM) is a nanocrystalline LDS ceramic dental material $^{[13]}$. It allows multiple different translucencies to be achieved based on the post-milling crystallization parameters. This property allows clinicians to achieve translucency to match the clinical scenario with a smaller inventory of raw materials. Nucleation is controlled to limit the size of crystals, which helps control translucency $[7]$.

1.2 IPS e.max CAD

IPS e.max CAD (IEC) is composed of 58-80% SiO \Box , 11-19% Li \Box O, 0-13% K \Box O, 0-8% ZrO \Box , 0-5% Al \Box 0 \Box ^[4]. It is easier to mill this material when it is partially crystallized. It is purchased and milled in a partially crystallized, "blue state" [14]. As it crystallizes, the intermediate phase which consists of 40% platelet-shaped lithium metasilicate crystals embedded in a glassy phase changes to 70% fine-grain LDS crystals embedded in a glassy matrix post crystallization $[15]$. High strength and toughness values demonstrated by IEC $^{[16,17,18]}$ are due to the high crystal content (of more than 60 vol%) of LDS ceramic and interlocking microstructure.

1.3 Cerec Tessera

Introduced in March 2021, Cerec Tessera (CT) is a recent innovation in glassceramics technology. It contains both LDS and virgilite crystals. Virgilite is the only naturally occurring representative of the solid-solution series between β-quartz (Qz) and LiAlSi₂O₆ (Sp) with a stuffed β-quartz structure ^[19]. The virgilite crystals usually occur in parallel bands resembling flow structures ^[19]. Virgilite crystals are activated through the matrix firing process which adds strength and contributes to improved esthetics.

Glass-based ceramic biomaterials should be able to fulfill the functions of human teeth $[20,21]$. Since data regarding the translucency and biaxial flexural strength of IEC, CT, and AM are limited, this in vitro study aims to assess and compare the translucency, and biaxial flexural strength (BFS) and microstructure of recently introduced LDS materials.

2. OBJECTIVES

2.1 To Evaluate the translucency parameter of glass-ceramic materials.

The objective is to measure and compare the translucency before and after crystallization of IPS emax, Cerec Tessera, and Amber Mill.

2.2 To Evaluate the Biaxial flexural strength of glass-ceramic materials.

The objective is to measure and compare the biaxial flexural strength of Cerec Tessera and Amber Mill to IPS emax.

2.3 To Evaluate the Radiopacity of glass-ceramic materials

The objective is to measure and compare the radiopacity of Cerec Tessera and Amber Mill to IPS emax.

2.4 To analyze and evaluate the microstructure of glass-ceramic materials.

The objective is to measure and compare the microstructure of Cerec Tessera and Amber Mill to IPS emax.

3. NULL HYPOTHESES

- 1. There will be no difference in the **translucency of the three materials**
- 2. There will be no difference between the **flexural strength of the three materials**.
- 3. There will be no significant difference between the **radiopacity of the three materials**.

4. MATERIALS AND METHODS

4.1 Materials Used

4.1.1 Translucency Parameter: Three contemporary glass-ceramic materials with medium and high translucency

- 1. IPS emax Ivoclar Vivadent
- 2. Cerec Terrasa Dentsply Sirona
- 3. Amber Mill Hassbio America

Table 1: Trade names and pictorial representation of lithium disilicate materials used in the study for measuring translucency.

4.1.2 Biaxial Flexural Strength : Three contemporary glass-ceramic materials with medium and high translucency

- 1. IPS emax Ivoclar Vivadent
- 2. Cerec Terrasa Dentsply Sirona
- 3. Amber Mill Hassbio America

Table 2: Trade names and pictorial representation of lithium disilicate materials used in the study for measuring Biaxial flexural strength.

4.1.3 Radiopacity : Three contemporary glass-ceramic materials with medium and high translucency

- 1. IPS emax Ivoclar Vivadent
- 2. Cerec Terrasa Dentsply Sirona
- 3. Amber Mill Hassbio America

Table 3: Trade names and pictorial representation of lithium disilicate materials used in the study for measuring radiopacity.

4.2 Method

4.2.1 Specimen preparation

The experimental groups included LDS glass-ceramics: IPS e.max CAD (Ivoclar Vivadent), Cerec Tessera (Dentsply Sirona), and AmberMill (Hassbio America) in this study (Table.1). Translucencies compared were High Translucency (HT) and Medium Translucency (MT). A2 shade was used for all the ceramics. Blocks of size C14 were milled into cylinders of a diameter of 14 mm using PrograMill PM7 (Ivoclar Vivadent). Disc-shaped test specimens with a diameter of 14 mm and a thickness of 1 ± 0.05 mm were fabricated from $14\times12\times18$ mm cylinders using the sectioning saw from IsoMET 1000 Precision Saw, Buehler.

Figure 1: Specimen Preparation a) Milled Block b) Specimen placed in saw c) Disc Shaped specimen

4.2.2 Surface treatment

All samples were hand polished using silicon carbide sandpaper on a polishing wheel, up to 1200 grit on one side and 320 grit on the other side for translucency parameter analysis. For BFS, samples were polished up to 600 grits on one side and 320 grits on the other. They were polished under water lubrication.

Figure 2: Polishing under water lubrication

4.2.3 Translucency measurement

Color measurements were taken using a spectrophotometer (UltraScan PRO Spectrophotometer HunterLab) before and after crystallization (except). Crystallization was done as per the manufacturer's recommendation using a Programmat furnace (Ivoclar Vivadent). L*a*b* color parameters were recorded in the spectrophotometer against black and white tiles. A thin layer of glycerine was placed between the specimens and the tiles. The difference in color recorded against the black and white backgrounds was used to calculate the translucency parameter (TP), and the contrast ratio (CR) using the formula:

$$
TP = [(L_b - L_w)^2 + (a_b - a_w)^2 + (b_b - b_w)^2]^{1/2}
$$

CR = L_B / L_W

Figure 3: Translucency measurement a) glycerine gel b) specimens placed against white tile c) specimens placed against black tile d) final position of specimen on tile

Figure 4: Spectrophotometer used.

4.2.4 Biaxial Flexural strength

Figure 5: Metal block used for placing disc specimen.

The BFS was performed according to DIN EN ISO 6872:2019[11]. Each specimen was placed centrally on three hardened steel balls [with a diameter of 3.2 mm (figure 2), positioned 120° apart on a support circle with a diameter of 10 mm. A 1.8 mm tip steel indenter applied force to the center of the disc at a rate of 1 mm/min. The maximum load to fracture failure of each specimen was recorded using a universal testing machine (Instron, Model #33R4204, Noorwood, MA).

Figure 6: Measurement of biaxial flexural strength a) specimen on supports b) indentor placed on top of specimen

4.2.5 Radiopacity

The radiopacity of the specimens was compared by measuring the radiodensity of the materials on digital radiographs. The specimens were radiographed with a digital x-ray device (Planmeca; Prostyle Intra) set at 70 kVp for 0.32 seconds along with an aluminum step wedge. A standardized radiograph of the specimens will be made using an occlusal film 41.15×30.99 mm and the processed film will then be scanned with a digital imaging system (Scan-X 1/0; Air Techniques Inc). Each step of the aluminum wedge represents an equivalent 1-mm thickness of pure aluminum that will be converted into values of radiopacity. Image analysis software (Photoshop; Adobe Systems Inc) will be used to measure the gray scale levels of the ceramics using the Histogram function and to compare intensity values to the step wedge. The material should have an equivalent radiopacity to 2mm of aluminum in order to be considered radiopaque.

Figure 7: Measurement of Radiopacity using Aluminium Wedge a) specimen placed on digital film with aluminum step wedge, b) x-ray positioned above specimen, c)processor

4.2.6 Microstructure analysis

The specimens were cleaned in ethanol, secured to tabs with gold conducting tape, and gold-coated in a vacuum sputter coater. The specimens were examined in a scanning electron microscope (SEM) (Quanta FEG 650, FEI) using the secondary electron imaging mode.

Scanning electron microscope (SEM) (Quanta FEG 650, FEI)

Figure 8: Microstructure analysis a) specimen cleaning b) placement on tray, c) gold coating specimens, d) specimens examined in SEM

5. STATISTICAL ANALYSIS

Differences between groups were analyzed by one-way analysis of variance (ANOVA), and Tukey's test was used for multiple comparisons in the case of a difference among groups. For all tests, the level of significance was set at $p < 0.05$, and SPSS Statistic 27.0.1 software (IBM Inc., Armonk, NY, USA) was used.

6. RESULTS

There were significant differences in the TP and BFS of experimental samples $(p<0.05)$. Tukey's analysis showed that some study groups were statistically significant from others. Detailed results are shown in Table 2. Materials with significantly different values are denoted with different superscripts. Under SEM imaging, IEC demonstrated long spindle shape crystals. CT and AM contained finer platelet shaped crystals and CT contained a virgilite phase.

6.1.1 Values

Table 4: Results

*values with different superscripts in each column are statistically different

6.1 Results for Translucency

Table 5: Results for translucency parameter.

Graph 1: Translucency parameters of ceramic.

6.1.3 Statistical analysis

ANOVA

Figure 9: Anova output for translucency parameter of ceramics.

TP

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 5.000.

Figure 10: Tukey output for translucency parameter of ceramics.

6.2 Results for Biaxial Flexural Strength

6.2.1 Values

Table 6a: Biaxial flexural strength for IPS emaax

6	1.04	1 $\mathbf{1}$	1. 4	$\mathbf{1}$ 4	0.25	5.75 271	1.110 105	267.3	404.8435
$\overline{7}$	1.05	1 $\overline{1}$	1. 4	$\mathbf{1}$ 4	0.25	5.75 271	1.110 105	419.67	623.5686
8	1.05	1 $\mathbf{1}$	1. 4	$\mathbf{1}$ 4	0.25	5.75 271	1.110 105	327.73	486.9591
9	1.01	$\mathbf{1}$ $\mathbf{1}$	1. 4	$\mathbf{1}$ 4	0.25	5.75 271	1.110 105	219.69	352.795
10	0.98	1 $\mathbf{1}$	1. 4	1 4	0.25	5.75 271	1.110 105	185.69	316.7315
Average									406.5272
Standard Deviation									90.23863

Table 6b: Biaxial flexural strength for AmberMill

Table 6c: Biaxial flexural strength for Cerec Tessera

6.2.2 Graph

Graph 2: Biaxial Flexural strength of ceramics

6.2.3 Statistical analysis

ANOVA

Figure 11: Anova Output for biaxial flexural strength of ceramics.

Biaxialflexuralstrength

Tukey HSD^a

Ceramic N N Subset for alpha = 0.05

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 10.000.

Figure: 12: Tukey test output for biaxial flexural strength of ceramics.

6.3 Results for Radiopacity

6.3.1 Values

Table 7a: Radiopacity results for AmberMill

Figure 13a Ceramic specimens (AmberMill) with aluminium wedge for comparing radiopacity.

Figure 13b Ceramic specimens (IPS e.max CAD) with aluminium wedge for comparing radiopacity.

Figure 13c Ceramic specimens (Cerec Tessera) with aluminium wedge for comparing radiopacity.

Graph 3 : Radiopacity of ceramics

6.3.3 Statistical Analysis

ANOVA

Figure 14: One-way ANOVA output for radiopacity of ceramics.

Radiopacity

Tukey HSD^a

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 9.000.

Figure 15 : Tukey test output for radiopacity of ceramics.

7. NULL HYPOTHESES REJECTION

There were statistically significant difference between the translucency, biaxial flexural strength, and radiopacity of the three ceramic materials. Thus the null hypothesis is rejected.

8. DISCUSSION

The development of lithium disilicate, zirconia, and alumina-reinforced ceramics has allowed the substitution of metallic infrastructures in diverse clinical situations, due to their high flexural and compressive strength $[22]$. The present study was undertaken to compare various commercially available contemporary glass ceramics for their translucency and flexural strength. High and medium translucencies for the A2 shade of IEC, CT, and AM were compared. The result of this study leads to the rejection of the null hypothesis.

Calculation of the translucency parameter is one of the most common approaches to evaluating light interactions in a dental restorative material $^{[23]}$. Heffernan et al $^{[24,25,26]}$ showed that different crystalline compositions result in a range of translucencies of ceramics at clinically relevant thicknesses. Our study confirms these results as ceramics with different microstructures showed variation in the translucencies. In AM, crystals are nano size and small crystal size gives high translucency. Limiting the grain size increases the density of the crystals and decreases the grain boundaries to increase translucency $[27]$. CT contains virgilite crystals which has been proposed to improve its translucency. But based on the current study, CT showed the lowest translucency amongst all groups. The high translucency of IEC is credited to a similar refractive index of the lithium disilicate crystalline phase and the glassy phase $[28]$. This interface is responsible for the light scattering properties of this material. Increasing the crystallinity percentage of the

ceramic improves mechanical properties but compromises translucency and color of the material [14,28].

Figure 16: (left to right) IPS e.max CAD, Cerec Tessera, AmberMill As per ISO standards 6872, uniaxial tests (three-point bending and four-point bending) and biaxial flexure (piston-on-three-ball) are accepted for measuring flexural strength. Uniaxial flexural strength tests use beam-shaped specimens which often have edge flaws $[29]$. These flaws act as the site for stress concentration and lead to undesirable edge failures. Therefore, the fracture is due to edge failure instead of a fracture that originates from the intrinsic flaw of the material $[30,31]$. Studies have shown biaxial flexural strength test is more sensitive than uniaxial flexural strength tests (ceramics $[32]$, glass ionomer cements $[33]$, composites $[34,35]$). Thus, biaxial flexural strength test was selected for this study.

Microstructure analysis of IEC gave a meshwork-like appearance. The crystals enlarge from nano-size crystals to form colonies. These colonies form layers, eventually resulting in highly intertwined colonies. This complex structure is difficult to break and is the main reason for its high flexural strength. A material with high flexural strength can chip during the milling procedure. Therefore, it is available partially crystalized for easy

milling. After full crystallization, it achieves high flexural strength. CT showed lower flexural strength than mentioned by the manufacturer. It is fired at a lower temperature (760ºC) which might have hindered the crystal growth eventually affecting its flexural strength. AM has nano-sized crystals which may have allowed a crack to propagate resulting in lower flexural strength.

Various studies have analyzed the translucency and flexural strength of IEC as it is one of the oldest CAD/CAM ceramics available. Studies by Eldwakhly et al [36] and Liebermann et al $[37]$ showed high translucency of IEC as compared to other contemporary ceramics. Apel et al. $[38]$ and Stawarczyk et al $[39]$ have shown flexural strength values of IEC around 430 MPa using the biaxial flexural test. These values are similar to our study. Alberto et al reported a flexural strength for IEC as 271.6 ± 64.7 MPa using a 3-point bend flexural strength testing conditions $[40]$. Further conclusions regarding the results achieved are hampered by the fact that detailed information on the compositions was not available for CT or AM. There is only one comparable investigation of the tested CAD/CAM ceramics (AM and CT) in the literature [37].

9. CONCLUSION

Once crystallized, all lithium disilicate materials produced similar translucency despite the smaller crystalline microstructure seen in CT and AM Despite the presence of an additional virgilite phase in CT, it did not produce increased biaxial flexural strength. The general ranking of the TP for the fired glass ceramics was

EHT > EMT > THT > AMM > AMH > TMT

BFS was

EHT > EMT > TMT > AMM > THT > AMH

Radiopacity was

TMT>AMM>EMT

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