

2018

Flexural Strength and Optical Characteristics of High Translucency Zirconia

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**FLEXURAL STRENGTH AND OPTICAL
CHARACTERISTICS OF HIGH TRANSLUCENCY
ZIRCONIA**

GOLSA AKBARIAN, D.D.S.

A Thesis Presented to the Faculty of the College of Dental Medicine of
Nova Southeastern University in Partial Fulfillment of the Requirements
for the Degree of
MASTER OF SCIENCE

June 2018

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By

GOLSA AKBARIAN, D.D.S

A thesis submitted to the College of Dental Medicine of Nova Southeastern

University in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

Department of Prosthodontics

College of Dental Medicine

Nova Southeastern University

June 2018

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DEDICATION

To my family for their endless love, support and encouragement.

ACKNOWLEDGEMENTS:

I would like to express my sincere gratitude to my mentor Dr. Jeffrey Thompson for his continuous support, patience, and immense knowledge. His guidance and advice on my research project have been invaluable.

I would like to acknowledge Dr. Marvin Golberg for serving on my committee and for all his very valuable comments on this thesis.

I would like to thank Dr. Rafael Castellon for serving on my committee and his contributions throughout this process.

I would like to acknowledge Dr. Patrick Hardigan for helping me with his expertise in statistics.

I would like to thank Ria Achong-Bowe for all her help in the research laboratory and her patience and guidance during this process.

I would like to thank Oral Arts Laboratory, Huntsville, AL for their generosity and providing my research materials.

ABSTRACT

FLEXURAL STRENGTH AND OPTICAL CHARACTERISTICS OF HIGH TRANSLUCENCY ZIRCONIA

DEGREE DATE: JUNE 18, 2018

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Objective: High translucency zirconia has been developed for esthetically demanding dental cases with the objective of improving light transmittance. The purpose of this in vitro study is to compare the translucency and flexural strength of high translucency zirconia with conventional zirconia and lithium disilicate ceramics, all of which are options for indirect dental restorative applications.

Methods: Three dental ceramic materials were selected: Group 1: conventional zirconia; group 2: high translucent zirconia; and group 3: lithium

disilicate glass-ceramic. Fifteen disk-shaped specimens (10mm in diameter) of each material were fabricated at 0.5mm, 0.75mm, and 1 mm thicknesses as subgroups A, B, and C respectively (n=135). The morphologies of the specimens were observed by a scanning electron microscopy at 5000X magnifications. Translucency and flexural strength of all specimens were measured using spectrophotometry and biaxial flexural testing respectively. The data was analyzed by ANOVA followed by post-hoc test ($p < 0.05$).

Results: Conventional Zirconia and lithium disilicate showed the highest and lowest mean flexural strength values respectively. Flexural strength of all 3 ceramic materials did not show significant difference with different material thicknesses. Lithium disilicate and conventional zirconia of equal thicknesses showed the highest and lowest translucency values respectively. Translucency of all 3 ceramic materials decreased with increasing material thickness.

Conclusion: High translucency zirconia did not show superior translucency compared to lithium disilicate. Thus, high translucency zirconia should be carefully used in the esthetic zone depending on the translucency needed for a successful restoration.

The flexural strength of conventional zirconia was significantly higher than high translucency zirconia and lithium disilicate. Therefore, the mechanical

properties of high translucency zirconia may be of concern when planning for more than three unit FPDs in the posterior area.

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CHAPTER 1: INTRODUCTION

Dental prostheses, such as crowns for single or multiple units, are fabricated from a variety of dental materials using different dental laboratory techniques. Porcelain fused to metal restoration has been the first choice of prostheses to satisfy requirements for esthetics, however it was known to cause graying of the gingival margin because of metal show-through. Increasing patient demand for enhanced esthetics, resulted in increased popularity of metal-free prostheses.¹

During the last few decades, dental ceramic materials such as glass-ceramics, poly-crystalline alumina, and zirconia-based ceramics have been successfully introduced into the dental profession, along with new processing technology, i.e. computer-aided design/computer-aided manufacturing (CAD/CAM).²

Lithium disilicate and zirconia are two of the most popular dental ceramic materials today. Lithium disilicate shows impressive esthetics and sufficient translucency, but its mechanical properties are less favorable compared to that of zirconia. On the other hand, zirconia is known for its superior mechanical properties but it remains clinically too white and opaque for highly esthetic cases. Therefore, it has been shown that using veneered porcelain on a zirconia framework improves esthetic results.^{3,4}

Several studies reported that chipping or fracturing of veneering porcelain occurs at a higher rate in PFZ (porcelain fused to zirconia) restorations than conventional PFM systems. Thermal coefficient mismatches of both materials is one of the factors which results in the mentioned failure in PFZ restorations. One ultimate solution to avoid the chipping of veneering porcelain is not to use porcelain. Therefore, the opacity of zirconia has been significantly improved and monolithic full-contour zirconia restorations developed for clinical use.⁵⁻⁸

In this study, the mechanical and optical properties of zirconia-based ceramics with enhanced translucency for more esthetic restorations are reviewed. In addition, these high translucency zirconia materials are compared with traditional zirconia and lithium disilicate glass-ceramics in regards to mechanical and optical properties.

1.1 Dental Ceramics:

1.1.1 Porcelain:

Dental ceramics that are close to natural teeth in regards to optical properties are predominantly glassy materials, and are derived principally from feldspar-quartz-kaolin triaxial porcelain compositions. However, current porcelains

contain mostly feldspar with no kaolin or quartz and have excellent translucency, but also low strength and resistance to crack propagation.

In 1962, a type of porcelain containing leucite was developed that could be fired directly onto dental alloys. Leucite is a crystalline mineral that is composed of potassium aluminosilicate. Leucite has a tetragonal symmetry at room temperature and changes to a cubic phase at 625°C. This phase transformation produces volume expansion of 1.2%, resulting in a high CTE ($20\text{-}25 \times 10^{-6}/\text{C}$). In contrast, feldspar glass has a low CTE ($8 \times 10^{-6}/\text{C}$). Thus, adding 17-25 mass% leucite to feldspar glass, allows a match between thermal expansion of the porcelain frits and that of dental alloys. Having porcelain with a slightly lower CTE compared to a metal substructure puts the porcelain in slight compression and helps control thermal stress on cooling from the firing temperature.⁹

In addition, leucite-containing feldspar glasses can be acid etched to create micromechanical features for resin bonding. Bonded restorations are generally more durable. Leucite feldspathic porcelain restorations have shown long-term clinical success rates when bonded to and supported by primarily enamel structures.^{9,10}

1.1.2 Glass Ceramics:

Glass-ceramics are much stronger when compared to porcelain because these ceramics undergo thermal processing (called ceraming), in which crystals are precipitated under controlled heat treatments from homogeneous glass through a nucleation and growth processes. Some current leucite-reinforced glasses are produced via the ceraming process. However, the most widely used dental glass-ceramics today are reinforced with lithium disilicate and are considered the strongest materials in this category.⁹

1.1.2.1 Lithium Disilicate Glass-Ceramic:

IPS e.max, Ivoclar Vivadent Schaan, Liechtenstein, contains a limited amount of lithium metasilicate and is produced via a ceraming process. IPS e.max has been shown to have flexural strength above other leucite reinforced dental ceramics due to refinement of the base glass composition and improved quality of the initial glass ingot with fewer defects and pores.

IPS e.max glass-ceramics come in two forms: Press and CAD. IPS e.max Press ingots are heat pressed at 920°C for 20 minutes. This form is slightly tougher than CAD because of higher crack propagation resistance by the larger grains. However, it also has slightly lower strength because these same grains introduce larger starting flaws into the structure.¹¹

The IPS e.max CAD ingots are first heat treated and partially crystallized to form the intermediate lithium metasilicate glass-ceramics, which are easier to mill. Once milling has been completed the restoration is subjected to a second round of heat treatment and tempering at 840°C for 7 minutes to form lithium disilicate glass-ceramic, which is more chemically stable and esthetic. This fully crystallized form of IPS e.max CAD has been shown to possess a recorded flexural strength of 360 ± 60 MPa and a fracture toughness of 2.02.5MPa.¹¹ Studies have shown high clinical success rate for using lithium disilicates as anterior or posterior single restorations and as short-span anterior FDPs.¹²

1.1.3 Polycrystalline Ceramics

Polycrystalline ceramics have no glassy phase, with all atoms packed into regular ordered arrays, which make them tougher and stronger than glassy ceramics.¹³

Zirconium dioxide (ZrO_2), known as zirconia is a white crystalline oxide of zirconium which is widely used as a restorative dental material due to its excellent mechanical properties. Unalloyed zirconia assumes three crystallographic forms depending on the temperature. At room temperature and upon heating up to 1170°C, it is monoclinic (in the form of a deformed

prism with parallelepiped sides). The structure is tetragonal (in the form of a straight prism with rectangular sides, which is the strongest and toughest phase) at 1170 - 2370°C and cubic (in the form of a straight prism with square sides) above 2370°C. The transformation from the tetragonal (t) phase to the monoclinic (m) phase upon cooling results in a significant volume increase (~4.5%). This change is about 2.3% in the case of a cubic to tetragonal transformation. While this imposes residual compressive stresses and consequent transformation toughening, it also results in microcracking and compromised mechanical properties. This transformation is reversible and begins at ~950°C on cooling.^{14,15}

Alloying pure zirconia with stabilizing oxides such as magnesium oxide (MgO), yttrium oxide (Y₂O₃, yttria), calcium oxide (CaO), and cerium(III) oxide (Ce₂O₃) allows the metastable retention of the tetragonal structure at room temperature.¹⁶

Most manufacturers of polycrystalline zirconia materials do not recommend grinding or sandblasting because these stress-generating surface treatments can trigger transformation from the tetragonal phase to the monoclinic phase. This matter is accompanied by a volume increase which results in surface compressive stresses and increases the flexural strength but also alters the

phase integrity of the material and increases the susceptibility to aging and premature failure.

Several polycrystalline zirconia materials have been developed for dental applications, including zirconia toughened alumina (ZTA), partially stabilized zirconia (PSZ), tetragonal zirconia polycrystal (TZP), and fully cubic stabilized zirconia (CSZ).¹⁶

1.1.3.1 Tetragonal Zirconia Polycrystals (Y-TZP)

Monolithic 3 mol% yttria-doped tetragonal zirconia polycrystal (3Y-TZP) is the most widely used zirconia material for dental applications. Biomedical grade zirconia usually contains 2-4 mol% yttria (Y_2O_3) as a stabilizer which yields >98% tetragonal zirconia of fine grain size ($\sim 0.2-0.5\mu m$). While the stabilizing Y^{3+} cations and Zr^{4+} are randomly distributed over the cationic sites, electrical neutrality is achieved by the creation of oxygen vacancies.^{17,18}

The mechanical properties of 3Y-TZP are affected by its grain size. 3Y-TZP is less stable and more susceptible to spontaneous tetragonal to monoclinic phase transformation with grain sizes above $1\mu m$. However, transformation does not occur when the grain size is less than $0.2\mu m$, and as a result fracture toughness decreases. Higher sintering temperatures and longer sintering times creates larger grain sizes.^{19,20} In addition to larger grain sizes, stress, heat,

and/or moisture can also cause transformation to monoclinic zirconia, resulting in toughening or a compromise of mechanical properties.

TZP restorations milled by soft machining are sintered at a later stage. This process prevents stress-induced transformation from tetragonal to monoclinic phase and leads to a final product with no monoclinic phase. Because of the behavior, most manufacturers do not recommend grinding adjustments or sandblasting TZP to avoid both the $t \rightarrow m$ transformation and the formation of surface flaws that could compromise long-term performance. In contrast, when zirconia restorations are milled by hard machining fully sintered 3Y-TZP blocks, some amount of monoclinic zirconia is created. This process leads to surface microcracking, higher susceptibility to low temperature degradation and lower reliability. Flexural strength and fracture toughness of TZP are in the range of 800–1000MPa and $6\text{--}8\text{MPa}\cdot\text{m}^{1/2}$ respectively which are considered to be superior to the mechanical properties of other available dental ceramics.^{19,21}

1.1.3.2 Zirconia-Toughened Alumina (ZTA)

Another approach to advantageously utilize the stress-induced transformation capability of zirconia is to combine it with an alumina matrix, leading to a zirconia-toughened alumina (ZTA). In-Ceram[®] Zirconia[®] (Vita Zahnfabrik-

Germany) was the first ZTA developed for dental restorations, which was produced by adding 33 vol.% of 12 mol% ceria-stabilized zirconia (12Ce-TZP) to In-Ceram[®] Alumina.²² In-Ceram[®] Zirconia is made by either slip casting or soft machining. Initially this porous ceramic product is sintered at 1100°C for 2 hours and then infiltrated with glass which represents about 23% of the final product. One of the advantages of the slip-cast technique is that very limited shrinkage is produced. However, it has larger pore content (8-11%) compared to that of sintered 3Y-TZP which partially explains its generally inferior mechanical properties when compared to TZP.

Guazzato et al. reported that In-Ceram[®] Zirconia processed by slip casting showed significantly higher flexural strength ($630\pm 58\text{MPa}$) compared to the milled product ($476\pm 50\text{MPa}$), with no significant difference in fracture toughness. The microstructure of ZTA ceramic was shown to have large alumina grains together with clusters of small zirconia grains (less than $1\mu\text{m}$ in diameter).²²

1.1.3.3 Partially Stabilized Zirconia (Mg-PSZ)

The microstructure of this type of zirconia consists of tetragonal precipitates within a tetragonal stabilized zirconia matrix. 8-10 mol% MgO or 3 mol% Y_2O_3 is used to partially stabilize zirconia. In addition to a high sintering

temperature (1680-1800°C), the cooling cycle has to be controlled. This product has not been successful due to the presence of porosity, associated with a large grain size (30–60µm) that can induce wear.^{20,22}

1.1.3.4 Cubic Stabilized Zirconia (CSZ)

CSZ has an isotropic character, which can enhance light transmission compared to the other types of polycrystalline ceramics. When light reaches the object, some part is reflected or/and absorbed, but some part may also be transmitted. Light that transmits into the ceramic may experience interior reflection and refraction, which is known as scattering. This internal light scattering, which limits the translucency of a solid may result from several sources including pores, impurities that have a different refractive index than zirconia (e.g. alumina sintering additives), defects such as oxygen vacancies, and grain boundaries.^{23,24} It has been shown that pore sizes between 200 to 400nm (typical of the current dental zirconia) and pore populations as low as 0.05% can significantly relegate the translucency of Y-TZP.

Optimizing the sintering heat treatment condition is very crucial in order to reduce the oxygen vacancies, which reduces translucency, while avoiding the introduction of porosity.

Tetragonal zirconia crystals are birefringent and exhibit anisotropy of refractive index in different crystallographic directions. In polycrystalline zirconia, birefringence results in the discontinuity of the refractive index at the grain boundaries if adjacent grains do not have the same crystallographic orientation. This type of microstructure results in reflection and refraction at grain boundaries, leading to diversions in the incident beam and a reduction of light transmittance.^{21,24}

Regarding optimum translucency, cubic fully stabilized zirconia with 8 mol.% or more yttria has isotropic refractive index with no scattering from birefringent grain boundaries. However, the strength of cubic zirconia is significantly lower (about one-half to two-thirds) than that of partially stabilized tetragonal zirconia.²⁵ Studies have shown that nanocrystalline 3 mol.% Y-TZP has potentially both desirable translucency and mechanical properties. To achieve a better translucency, translucent Y-TZP has been modified by reducing porosity, decreasing grain size, and eliminating any alumina added as a sintering aid. Also, increasing yttria content results in a larger amount of cubic phase and thus greater translucency.²³

The current approach to develop translucent zirconia is to introduce optically isotropic cubic zirconia into an ordinarily tetragonal material. However, biphasic tetragonal/cubic zirconia has inferior mechanical properties

compared to tetragonal zirconia. For example, Katana ultra-translucent zirconia (Kuraray Noritake Dental Inc., Japan) has a flexural strength of 557 MPa, whereas their super-translucent and high-translucent zirconia have flexural strengths of 748 and 1125MPa, respectively.^{21,26}

1.2 Light Transmission and Translucency

Translucency is the relative amount of light transmitted through the material. There are two forms of transmittance including diffuse, in which measurement includes both the light passing through the material and that scattered in a forward direction, and specular, in which the measurement excludes the scattered light that does not reach the detector. There are two common parameters which are used in order to evaluate translucency: Contrast Ratio (CR) and Translucency Parameter (TP).²⁷

The CR values are calculated by using the following equation: $CR = Y_b/Y_w$, in which Y_b represents the reflectance of light of the specimen over a black background and Y_w over a white background. The CR value of a totally transparent material is 0, while the value of a totally opaque material is 1.

Translucency Parameter represents the color difference between a material over a black and a white background and is calculated using the following equation:

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

Pores in dental ceramics are the main cause of light scattering and can be either intragranular or intergranular. Intragranular pores locate are between two isotropic phases, whereas intergranular pores are between 2 or 3 crystalline grains of different orientations on grain boundaries. A large pore size may compromise both the mechanical and optical properties. Densification can partially or completely eliminate pores.²⁸

1.3 Purpose, Specific Aims, and Hypothesis:

1.3.1 Purpose:

The purpose of this study was to compare the relative translucency and flexural strength of different types of dental ceramic systems at different thicknesses.

1.3.2 Specific Aims:

- 1) To assess flexural strength of high translucency zirconia and to compare it with flexural strength of conventional zirconia and lithium disilicate glass-ceramic.
- 2) To assess the translucency of high translucency zirconia and compare it with translucency of conventional zirconia and lithium disilicate glass-ceramic.

- 3) To evaluate the flexural strength of each of the three ceramic systems at different thicknesses.
- 4) To evaluate the translucency of each of the three ceramic systems at different thicknesses.

1.3.3 Specific Hypothesis:

- 1) Conventional zirconia will show significantly higher flexural strength compared to lithium disilicate glass-ceramic and high translucency zirconia of equal thicknesses.
- 2) High translucency zirconia and lithium disilicate will show significantly higher translucency compared to conventional zirconia of equal thicknesses.
- 3) Flexural strength of all three ceramic materials increases with increasing material thickness.
- 4) Translucency of all three ceramic materials decreases with increasing material thickness.

2 CHAPTER 2: MATERIALS AND METHODS

2.1 Material Selection

Three groups of available ceramic materials were selected for this study.

(Table 1)

Group 1: Conventional zirconia

Group 2: High translucency zirconia

Group 3: Lithium disilicate

Table 1. Ceramic materials evaluated.

Material	Type	Manufacturer
Cercon base	Conventional zirconia	DENTSPLY International, Inc. USA
Katana UTML	High translucency zirconia	KURARAY NORITAKE Dental Inc. USA
IPS e.max CAD	Lithium disilicate glass-ceramic	Ivoclar Vivadent, USA

2.1.1 Support Acknowledgement

This study was awarded a grant by the Health Professions Division at Nova Southeastern University. (#335083)

2.2 Sample Preparation

Pre-sintered milled rods of each dental ceramic material, having a 9.40mm diameter, were prepared by a large commercial dental laboratory (Oral Arts Dental Laboratory, Huntsville, Alabama) using Computer-Aided Design/Computer-Aided Manufacturing (CAD/CAM) technology. 135 disk-shaped specimens were prepared by sectioning the milled rods to obtain specimens with 3 different thicknesses (0.50mm, 0.75mm, 1.00mm) using sectioning blades (IsoMet Diamond Wafering Blades, Buehler, Illinois, USA) and precision low speed saws (IsoMet® 1000 Precision Cutter, Buehler GmbH, Dusseldorf, Germany) at 125 rotations/minute. (Fig 1)

Figure 1. Precision low speed saw.



15 disks of each ceramic material group were prepared with a thickness of 0.50mm as subgroup A. Subgroups B (n=15) and C (n=15) specimens were prepared with thicknesses of 0.75mm, and 1.00mm respectively. (Table 2, figure 2)

Figure 2. Study specimens.



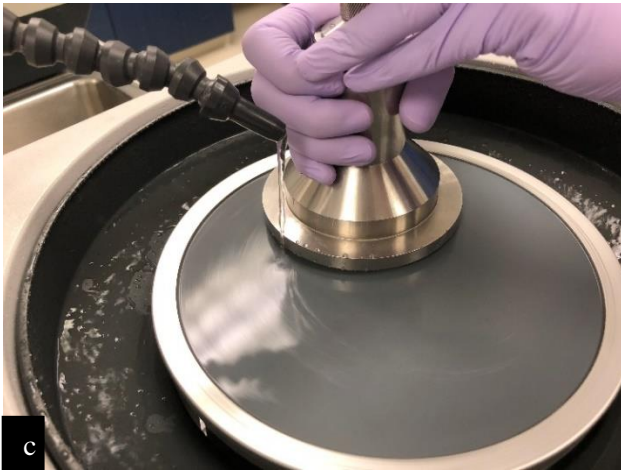
Table 2. Study groups.

Group	Thickness (mm) of subgroups	N
Group 1: Conventional zirconia	A = 0.50	15
	B = 0.75	15
	C = 1.00	15
Group 2: High translucency zirconia	A = 0.50	15
	B = 0.75	15
	C = 1.00	15
Group 3: Lithium disilicate	A = 0.50	15
	B = 0.75	15
	C = 1.00	15

The ceramic disks were polished with 400, 600, and 800-grit silicon carbide paper (CarbiMet™2, Buehler, Illinois, USA) under constant water rinsing in a polishing machine (MetaServ 2000 Grinder Polisher; Buehler GmbH, Dusseldorf, Germany) at 250 rotations/minute for 10 minutes. (Fig 3) The thickness of the specimens was controlled by measurement with a digital caliper (Pittsburgh, Camarillo, CA, USA) with an accuracy of 0.01mm. (Figure 3)

Figure 3a,b,c. Polishing machine and procedure.





2.3 Microstructural Assessment of Surface Topography:

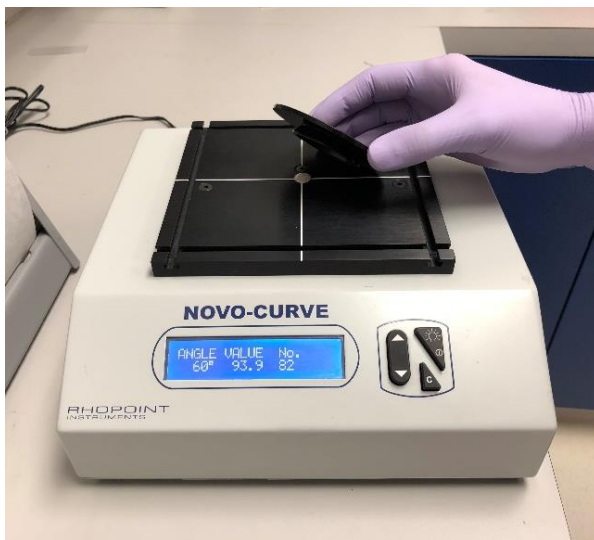
The morphologies of the specimens were observed by scanning electron microscopy. Three Ceramic disks with different thickness were randomly selected from each group. The selected samples were cleaned and rinsed with distilled water for 5 minutes. SEM images were obtained of each specimen using FEI Quanta 200 scanning electron microscope (FEI, Hillsboro, OR) at 500 \times , 1000 \times , and 4000 \times magnifications.

2.4 Surface Gloss Measurement

The specular reflection gloss of all the specimens was measured using a gloss meter (Novo-Curve™ Glossmeter, Rhopoint™ Instruments Ltd). The machine was calibrated before any measurement as per the manufacturer's instructions. The ceramic disks were cleaned with alcohol wipes and then placed over the reading aperture of the gloss meter. The specimens were covered by an opaque black plate in order to eliminate the effect of ambient light. (Figure 4)

The surface gloss measurement was collected (in degrees) as the amount of reflected light at an equal but opposite angle to the projected beam of light from the specimen. The measurements were conducted 3 times for each of 3 different spots on each specimen and then the gloss values were averaged.

Figure 4. Gloss meter used for surface gloss measurement.



2.5 Translucency Testing:

A spectrophotometer (Color-Eye 7000A; GretagMacbeth, New Windsor, NY, USA) was used to measure the translucency parameter for each specimen. (Figure 5) The color of each specimen was measured according to Commission Internationale de l'Eclairage (CIE) system based on three coordinates- $L^*a^*b^*$ in which L^* refers to the brightness, a^* to redness to greenness, and b^* to yellowness to blueness. The light source illumination corresponds to average daylight (D65). The CIE $L^*a^*b^*$ values of each specimen was measured on a black and a white background. (Figure 6)

For translucency measurements, translucency parameter (TP) was obtained by calculating the color difference between the values against a white background (w) and a black background (b), which is denoted by the following equation:

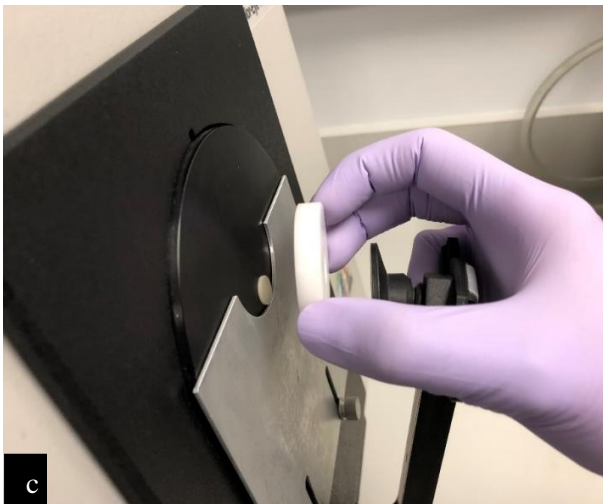
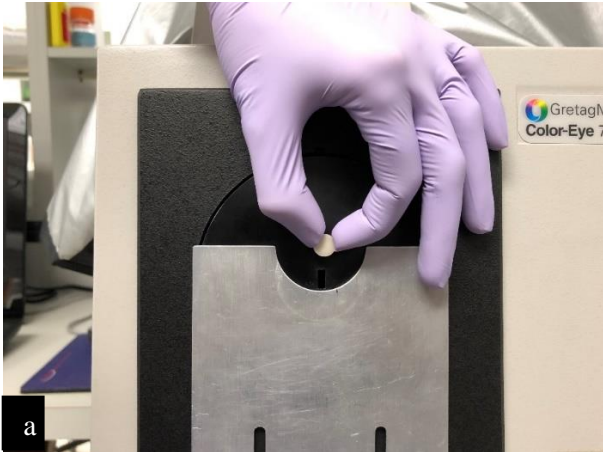
$$TP = [(L_b^* - L_w^*)^2 + (a_b^* - a_w^*)^2 + (b_b^* - b_w^*)^2]^{1/2}$$

A high TP value indicates high translucency and low opacity.

Figure 5. Spectrophotometer.



Figure 6a,b,c. Translucency measurement of the ceramic disk.



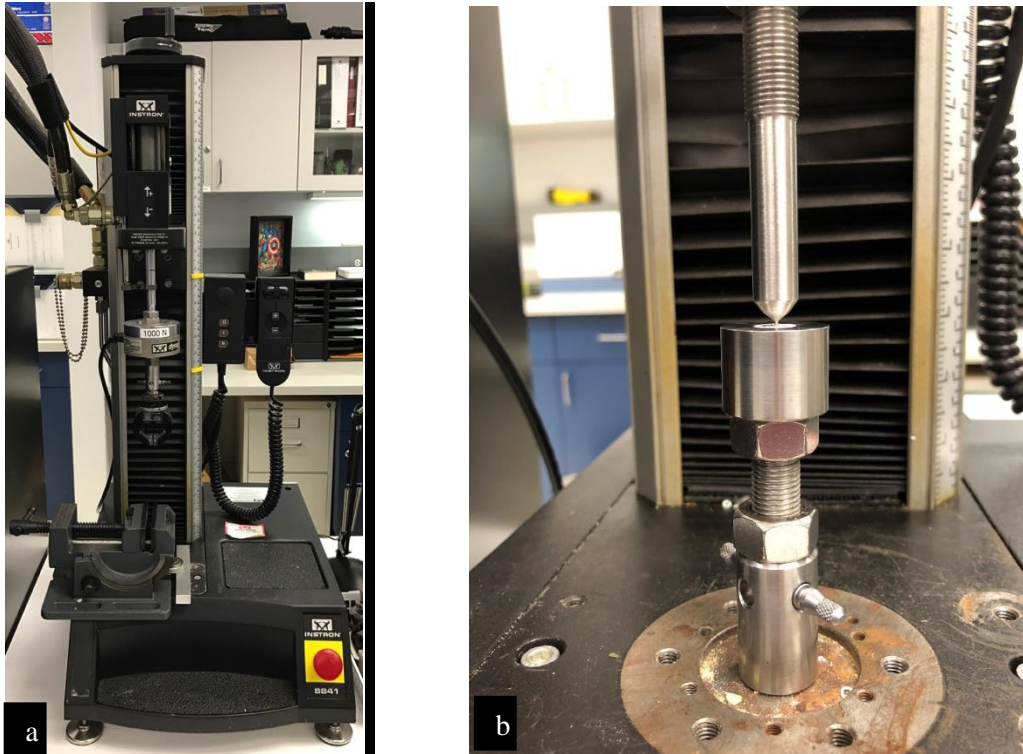
Samples with different thicknesses within each group were compared with each other regarding translucency. In addition, translucency of all three groups of specimens with the same thickness were compared together at 0.50mm, 0.75mm, and 1.00mm thickness.

2.6 Biaxial Flexural Strength Testing:

The piston-on-three-ball test (ASTM Standard F394-78) was used to determine the biaxial flexural strength of all specimens. The thickness of each specimen was measured with a digital caliper (Stainless Steel Digital Caliper, VWR[®] International, LLC, West Chester, PA, USA) before testing. Disc-shaped specimens were placed on three support balls (1.60mm diameter), which were arranged 120° apart from each other in a triangular position on the perimeter of a 10.00mm diameter support circle.

A flat end circular cylinder of hardened steel with a 0.72mm radius was used during loading perpendicular to the axis of the specimen center. The testing was conducted in a universal testing machine (Instron 8841, Canton, MA, USA) at a crosshead speed of 1mm/min until fracture. (Figure 7)

Figure 7a,b. Instron universal testing machine.



The load at the time of fracture of the sample was recorded and the biaxial flexural strength was calculated using the formula below:

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

S is the maximum center tensile stress (MPa) which corresponds to the biaxial flexure strength, P is the total load at fracture (N), and b is specimen thickness at fracture origin (mm).

$$X = (1 + \nu) \ln (r_2/r_3)^2 + [(1 - \nu)/2] (r_2/r_3)^2$$

$$Y = (1 + \nu) [1 + \ln (r_1/r_3)^2] + (1 - \nu) (r_1/r_3)^2$$

in which ν is Poisson's ratio, r_1 is the radius of the support circle (mm), r_2 is the radius of the tip of the piston (mm), r_3 is the radius of the specimen (mm).

Poisson's ratio was set at 0.25, the standard value for IPS e.max ceramics, and at 0.30 for zirconia.²⁹

The flexural strength of all specimens in different groups, but with the same thickness, was calculated and compared. Moreover, flexural strength of samples within a group but with different thicknesses were compared together as well.

2.7 Morphological Assessment of Surface Topography:

Specimens from each group were randomly selected for microscopic evaluation of both the polished and fractured surfaces. All specimens were thoroughly cleaned with acetone. The conventional and high translucency zirconia samples were thermally etched in a sintering furnace. The firing temperature was set 150°C below the sintering temperature for 20min.³¹ Lithium disilicate specimens were etched for 60s with 9.6% hydrofluoric acid (Porcelain Etch Gel, Pulpdent Corporation, Watertown, MA), and cleaned under running water. The specimens were ultrasonically cleaned in acetone for 5min, air dried and fixed onto an SEM Pin Stub Mount using graphite conductive adhesive (Electron Microscopy Sciences, FT.Washington, PA). Samples were coated with gold using Cressington sputter coater 108 Auto (Ted Pella, Inc. CA, USA) and imaged using a FEI Quanta 200 Scanning

Electron Microscope (FEI, Hillsboro, OR). SEM micrographs of specimens were taken at a 5000x original magnification.

2.8 Statistical Analysis:

Means and standard deviations were calculated for all continuous measures. To compare differences for the outcome measures gloss, translucency and flexural strength three general linear models (ANOVA) were created. The fixed effects were material (conventional zirconia vs. high translucency zirconia vs. lithium disilicate), thickness of material (0.50mm vs. 0.75mm vs 1.00mm), and the interaction of material by thickness. Post-hoc tests were conducted using a Holm adjustment. RStudio and R 3.2.2 were used for all statistical analysis, and significance was accepted at $p < 0.05$.

3 CHAPTER 3: RESULTS

Means and standard deviations were calculated for all continuous measures. To compare differences for the outcome measures gloss, translucency and flexural strength three general linear models (ANOVA) were created. The

fixed effects were material (conventional zirconia vs high translucency zirconia vs. lithium disilicate), thickness of material (0.50mm vs. 0.75mm vs 1.00mm), and the interaction of material by thickness. Post-hoc tests were conducted using a Holm adjustment. RStudio and R 3.2.2 were used for all statistical analysis, and significance was accepted at $p < 0.05$. Results are presented below:

3.1 Surface Gloss Results:

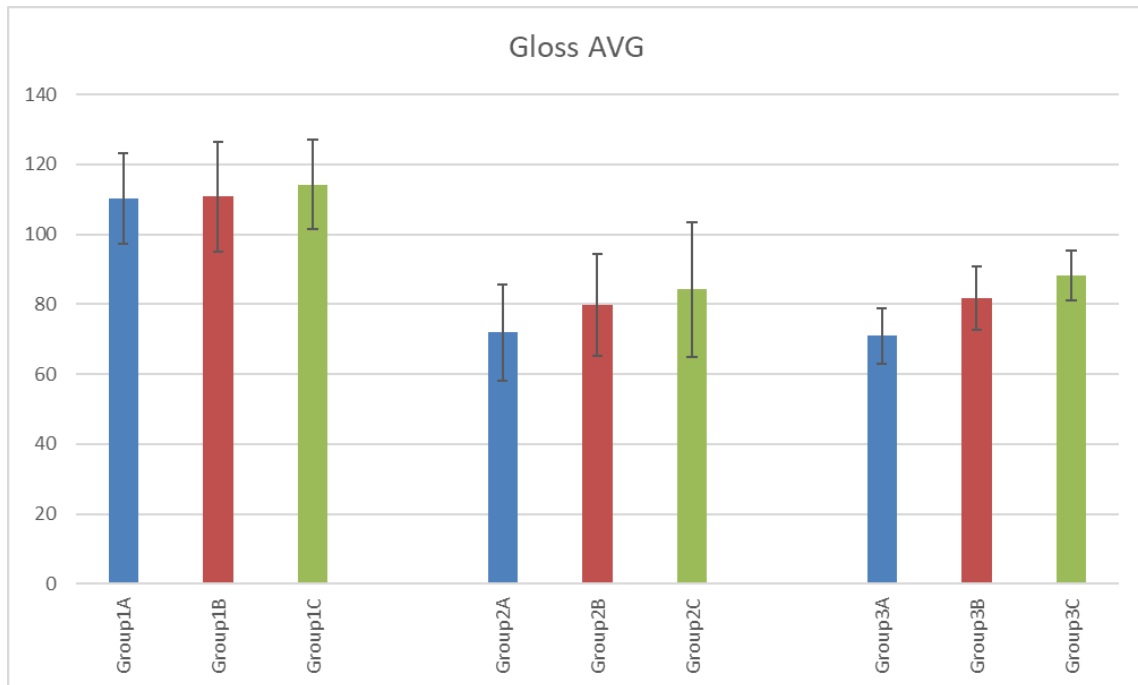
- There was a significant difference in the measurement of gloss by material $F(3,128) = 61.89$, $p < 0.001$, eta-squared = 55% - meaning 55% of the variability in gloss was accounted for by the differences in material].
- There was a significant difference in the measurement of gloss by thickness of material $F(3,128) = 16.52$, $p < 0.001$, eta-squared = 5% - meaning 5% of the variability in gloss was accounted for by the differences in the thickness of material].
- There was no significant difference in the measurement of gloss for material by thickness $F(2,128) = 2.00$, $p = 0.13$, eta-squared = 2% - meaning 2% of the variability in gloss was accounted for by the differences in material by thickness].

- Descriptive statistics are presented in Table 3 and Figure 9 and a plot of the pairwise comparisons are shown in Figure 10.

Table 3. Descriptive statistics for surface gloss.

Gloss						
Material	Thickness(mm)	N	M	SD	Min	Max
Conventional Zirconia	0.50	15	110.32	12.98	93.13	133.20
Conventional Zirconia	0.75	15	110.81	15.71	76.47	139.37
Conventional Zirconia	1.00	15	114.26	12.82	95.20	137.80
High Translucency Zirconia	0.50	15	71.91	13.71	59.67	110.83
High Translucency Zirconia	0.75	15	79.84	14.59	62.43	118.23
High Translucency Zirconia	1.00	15	84.19	19.16	60.00	126.37
Lithium Disilicate	0.50	15	71.01	7.90	57.87	83.23
Lithium Disilicate	0.75	15	81.70	9.13	69.20	94.80
Lithium Disilicate	1.00	15	88.25	7.07	68.70	95.13

Figure 8. Mean surface gloss readings.

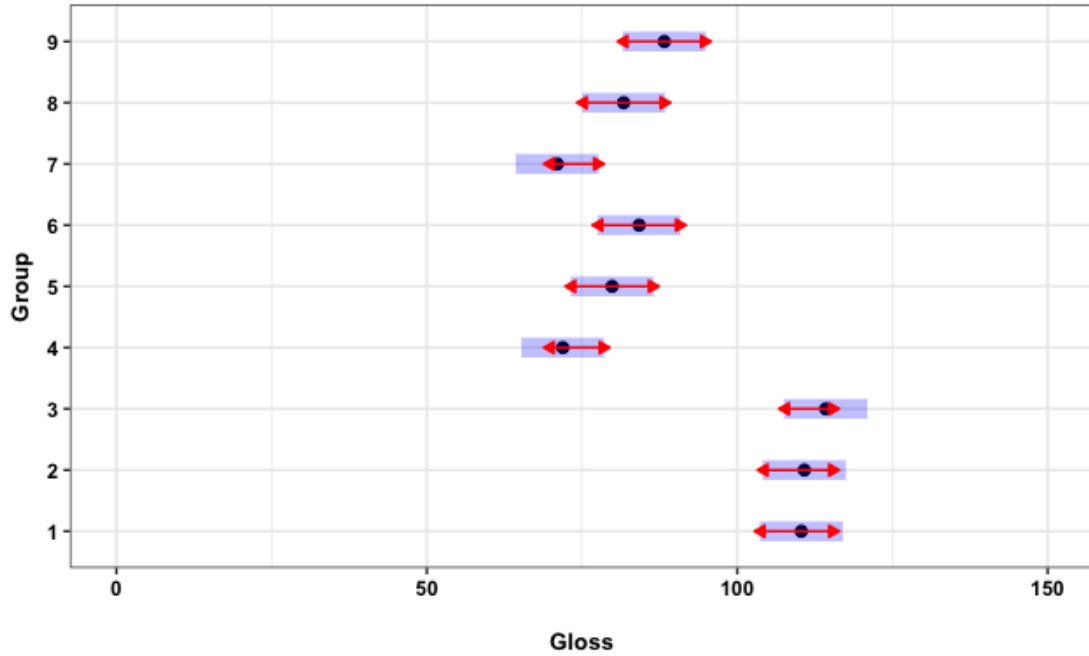


According to figure 10, group 1 (conventional zirconia) showed a significantly higher gloss value compared to groups 2 (high translucency zirconia) & 3 (lithium disilicate).

There was no significant difference within each group except for group 3, in which subgroup C (lithium disilicate with 1mm thickness) showed a higher gloss value compared to subgroup A (lithium disilicate with 0.50mm thickness).

Figure 9. Plot of Gloss by Group.

The blue bars are confidence intervals for the means, and the red arrows are for the comparisons among them. If an arrow from one mean overlaps an arrow from another group, the difference is not significant.



- G1 = Conventional Zirconia 0.50mm
- G2 = Conventional Zirconia 0.75mm
- G3 = Conventional Zirconia 1.00mm
- G4 = High Translucency Zirconia 0.50mm
- G5 = High Translucency Zirconia 0.75mm
- G6 = High Translucency Zirconia 1.00mm
- G7 = Lithium Disilicate 0.50mm
- G8 = Lithium Disilicate 0.75mm
- G9 = Lithium Disilicate 1.00mm

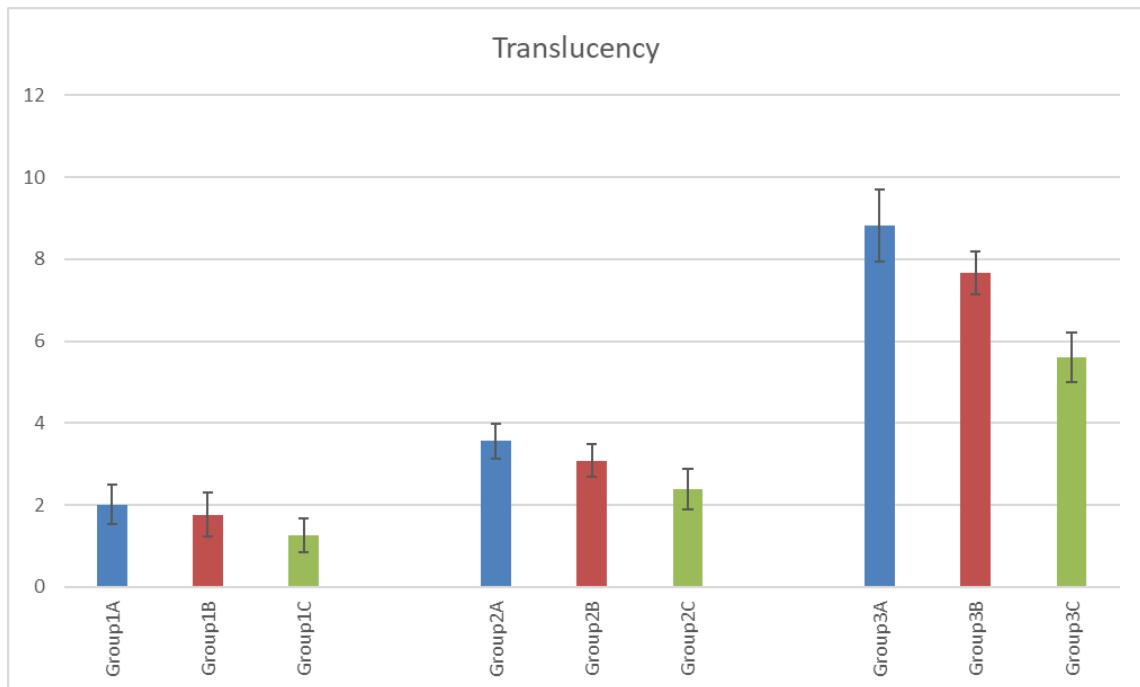
3.2 Translucency Parameter Results:

- There was a significant difference in the measurement of translucency by material $F(3,128) = 853.83$, $p < 0.001$, eta-squared = 86% - meaning 86% of the variability in translucency was accounted for by the differences in material].
- There was a significant difference in the measurement of translucency by thickness of material $F(3,128) = 211.74$, $p < 0.001$, eta-squared = 7% - meaning 7% of the variability in translucency was accounted for by the differences in the thickness of material].
- There was a significant difference in the measurement of translucency for material by thickness $F(2,128) = 41.45$, $p = 0.13$, eta-squared = 3% - meaning 3% of the variability in translucency was accounted for by the differences in material by thickness].
- Descriptive statistics are presented in Table 4 and Figure 11 and a plot of the pairwise comparisons are shown in Figure 12.

Table 4. Descriptive statistics for translucency.

Translucency						
Material	Thickness(mm)	N	M	SD	Min	Max
Conventional Zirconia	0.50	15	2.01	0.48	1.47	3.01
Conventional Zirconia	0.75	15	1.76	0.54	1.06	3.01
Conventional Zirconia	1.00	15	1.25	0.42	0.72	2.01
High Translucency Zirconia	0.50	15	3.56	0.43	3.03	4.73
High Translucency Zirconia	0.75	15	3.08	0.40	2.63	3.90
High Translucency Zirconia	1.00	15	2.39	0.50	1.27	3.31
Lithium Disilicate	0.50	15	8.82	0.87	7.66	10.35
Lithium Disilicate	0.75	15	7.66	0.52	7.06	8.65
Lithium Disilicate	1.00	15	5.61	0.60	4.57	6.80

Figure 10. Mean translucency readings.

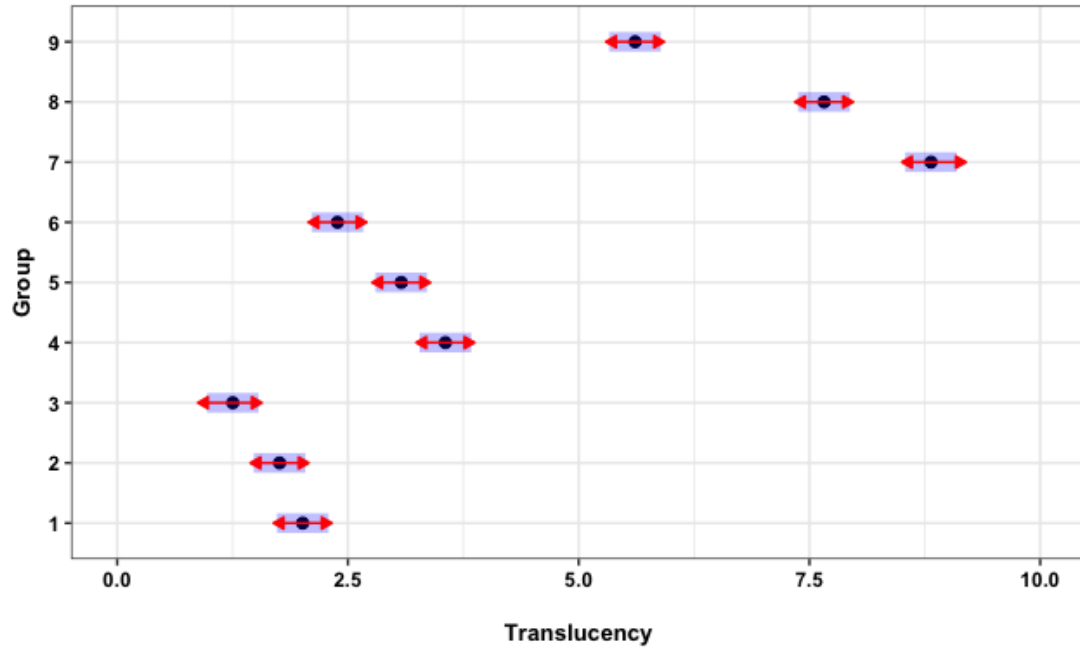


According to figure 12, group 3 (lithium disilicate) showed significantly higher translucency compared to groups 1 (conventional zirconia) and 2 (high translucency zirconia) of equal thicknesses. In addition, group 2 showed significantly higher translucency compared to group 1 of equal thicknesses.

According to figure 12, when comparing translucency of the specimens within each group, group 1A (0.50mm thickness conventional zirconia) showed significantly higher translucency compared to group 1C (1.00mm thickness conventional zirconia). Also group 2A (0.50mm high translucency zirconia) showed significantly higher translucency compared to group 2C (1.00mm thickness high translucency zirconia). All three subgroups in group 3 (lithium disilicate) showed significant differences with each other. Overall, translucency decreased with increasing the material thickness.

Figure 11. Plot of Translucency by Group.

The blue bars are confidence intervals for the means, and the red arrows are for the comparisons among them. If an arrow from one mean overlaps an arrow from another group, the difference is not significant.



- G1 = Conventional Zirconia 0.50mm
- G2 = Conventional Zirconia 0.75mm
- G3 = Conventional Zirconia 1.00mm
- G4 = High Translucency Zirconia 0.50mm
- G5 = High Translucency Zirconia 0.75mm
- G6 = High Translucency Zirconia 1.00mm
- G7 = Lithium Disilicate 0.50mm
- G8 = Lithium Disilicate 0.75mm
- G9 = Lithium Disilicate 1.00mm

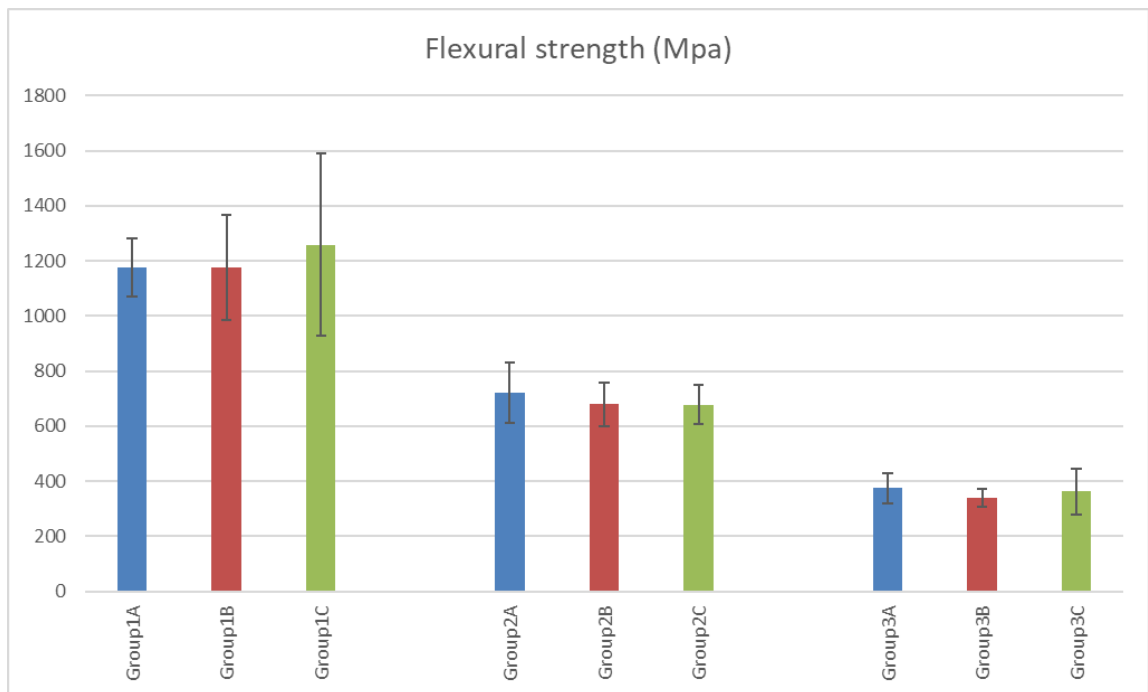
3.3 Biaxial Flexural Strength Results:

- There was a significant difference in the measurement of *flexural strength* by material $F(3,128) = 257.10$, $p < 0.001$, eta-squared = 85% - meaning 85% of the variability in *flexural strength* was accounted for by the differences in material].
- There was no significant difference in the measurement of *flexural strength* by thickness of material $F(3,128) = 0.04$, $p = 0.830$, eta-squared = 0.01% - meaning 0.1% of the variability in *flexural strength* was accounted for by the differences in the thickness of material].
- There was no significant difference in the measurement of *flexural strength* for material by thickness $F(2,128) = 1.53$, $p = 0.220$, eta-squared = 0.03% - meaning 0.3% of the variability in *flexural strength* was accounted for by the differences in material by thickness].
- Descriptive statistics are presented in Table 5 and Figure 13 and a plot of the pairwise comparisons are shown in Figure 14.

Table 5. Descriptive Statistics for flexural strength.

Flexural Strength						
Material	Thickness(mm)	N	M	SD	Min	Max
Conventional Zirconia	0.50	15	1176.73	104.85	979.84	1352.22
Conventional Zirconia	0.75	15	1177.32	191.08	925.68	1627.56
Conventional Zirconia	1.00	15	1257.49	331.00	897.56	2364.81
High Translucency Zirconia	0.50	15	721.11	109.98	546.89	997.86
High Translucency Zirconia	0.75	15	679.02	78.86	527.85	803.28
High Translucency Zirconia	1.00	15	677.38	71.55	561.11	825.40
Lithium Disilicate	0.50	15	379.67	54.57	287.73	502.85
Lithium Disilicate	0.75	15	341.47	32.93	269.35	384.59
Lithium Disilicate	1.00	15	362.36	83.54	270.48	548.47

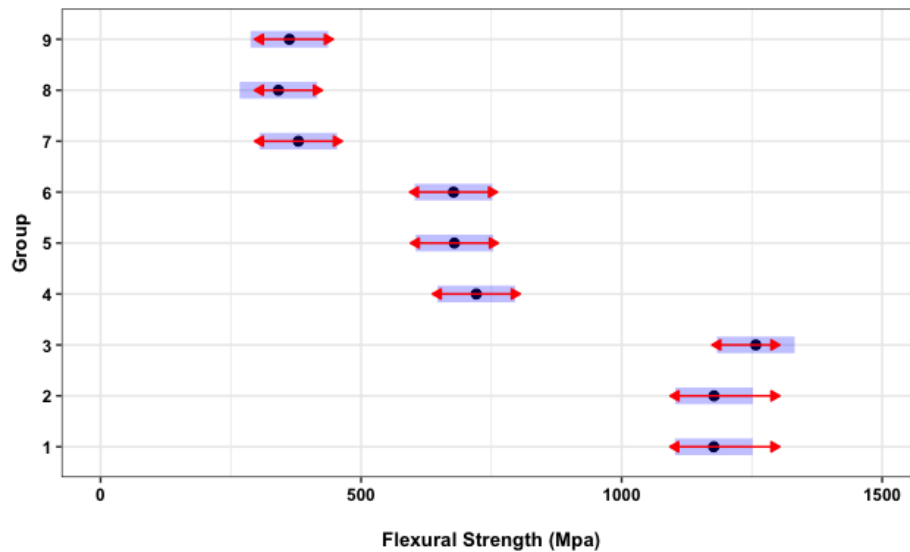
Figure 12. Mean flexural strength readings.



According to figure 14, Group 1 (conventional zirconia) showed significantly higher flexural strength value compared to group 2 (high translucency zirconia) and group 3 (lithium disilicate). In addition, group 2 showed significantly higher value compared to group 3. There was no significant difference within each group.

Figure 13. Plot of Flexural Strength by Group.

The blue bars are confidence intervals for the means, and the red arrows are for the comparisons among them. If an arrow from one mean overlaps an arrow from another group, the difference is not significant.



- G1 = Conventional Zirconia 0.50mm
- G2 = Conventional Zirconia 0.75mm
- G3 = Conventional Zirconia 1.00mm
- G4 = High Translucency Zirconia 0.50mm
- G5 = High Translucency Zirconia 0.75mm
- G6 = High Translucency Zirconia 1.00mm
- G7 = Lithium Disilicate 0.50mm
- G8 = Lithium Disilicate 0.75mm
- G9 = Lithium Disilicate 1.00mm

3.4 SEM Evaluation of Ceramic Structure:

Figures 14-16 demonstrate SEM images of all treatment groups from randomly selected specimens of all three ceramic materials. The images displayed are of 5000x magnification.

Figure 14. Conventional zirconia 5000x magnification.

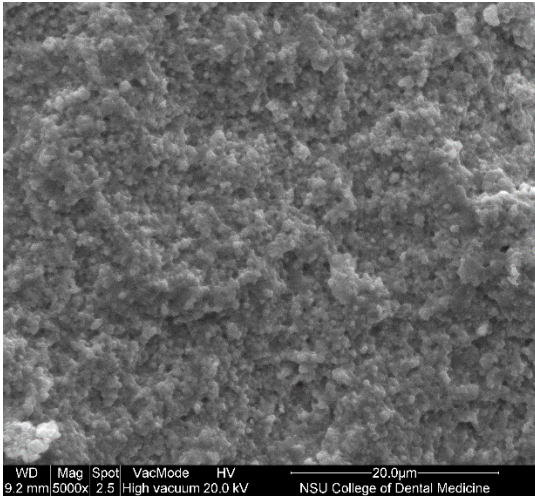


Figure 15. High translucency zirconia 5000x magnification.

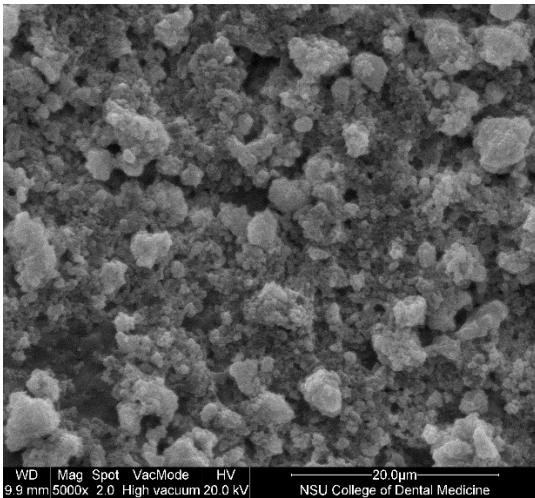
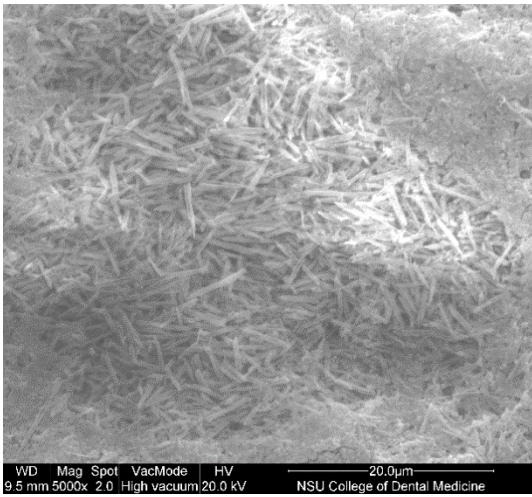


Figure 16. *Lithium disilicate* 5000x magnification.



4 CHAPTER 4: DISCUSSION

This study was conducted to compare the translucency and flexural strength of three ceramic materials including conventional zirconia, high translucency zirconia, and lithium disilicate glass-ceramic. The specimens were prepared as 9.40mm diameter discs with three different thicknesses (0.50, 0.75, and 1.00mm). The size is assumed to more closely mimic the dimensions of dental ceramic restorations. Each specimen underwent the same polishing protocol to ensure uniformity. The specimens were divided into 9 groups based on the ceramic system and the thickness as shown in table 2. The specimens were subjected to optical and mechanical testing using standardized testing protocols. Following testing, statistical analysis was performed to analyze the results and to determine whether the proposed hypothesis were supported or rejected.

4.1 Surface Gloss:

Gloss is an optical phenomena that represents the amount of specular reflection from a surface and is responsible for a lustrous or mirror-like appearance. Gloss is calculated by comparing the magnitude of incident light traveling toward a surface at a 60° angle, as indicated by ISO 2813 for

specimens of medium gloss, to the magnitude traveling away from the surface at an equal and opposite angle.³⁰

Achieving a smooth and glossy ceramic surface for dental restorations is very important in order to obtain an enhanced esthetic result. In addition, staining and plaque retention are more pronounced on rough surfaces which increases the chance of gingivitis or caries. Thus, a smooth restoration results in patient comfort and optimum biological outcome. Rough ceramic restorations are also abrasive and can cause greater wear of opposing teeth.^{31,32}

Surface gloss, color, and translucency are three main factors that determine the esthetic of an all-ceramic restoration. All three parameters should be in balance in order to achieve a natural looking restoration with optimum esthetic result.

In the present study, conventional zirconia showed a significantly higher gloss value compared to high translucency zirconia and Lithium disilicate. This could be because zirconia allows the material to be more efficaciously polished.³⁰ Also, conventional zirconia is very opaque compared to the other two materials which results in minimum light transmission.

The factors that have been reported to affect gloss include optical properties (refraction index) of the material, angle of incident light, and surface topography.³⁰

4.2 Translucency:

The second null hypothesis of this study was accepted. According to the present study, translucency of conventional zirconia was significantly less than high translucency zirconia and lithium disilicate. This was in accordance with some other studies.^{27,28,31,33-36} In addition, translucency of high translucency zirconia was significantly less than that of lithium disilicate, thus its use for monolithic anterior restorations will still be limited for highly translucent restorations.^{31,33,37}

Selecting an appropriate material with the right amount of translucency is a critical factor in order to achieve a natural looking dental restoration.^{38,39}

Zirconia is one of the all-ceramic dental materials that has been widely used for esthetic zone especially in the last decade. However, one of the disadvantages of zirconia restorations is the relatively opaque nature of the material compared to other ceramics such as lithium disilicate, which has superior esthetic properties. The opacity of zirconia is due to the large size of the crystalline particles and the presence of porosity, which results in greater light scattering and less translucency. In addition, dopants such as alumina which are added to improve the phase stability, reduce ageing and prevent low temperature degradation (LTD) of polycrystalline materials, has an adverse effect on translucency because of its different refractive index compared to

zirconia. The refractive index is measured as the amount of reduction in the speed of light when passing through a medium.^{23,27,28,33}

High translucency zirconia is a relatively new dental restorative material that manufacturers introduced it as an alternative for conventional zirconia due to its more satisfactory esthetic characteristics. This improved translucency is attributed to the significantly reduced frequency and size of the porosity within the material. In addition, high translucency zirconia presents more uniform grain size and configuration than conventional zirconia, and has a lower alumina content. As mentioned previously, adding alumina to zirconia is effective for the prevention of low temperature degradation (LTD). LTD is a chemical aging process which can occur in the presence of water and is associated with the spontaneous transformation of tetragonal phase to monoclinic phase as a result of penetration of saliva into surface microcracks in the oral environment. Thus, the hydrothermal stability is lowered by decreasing the amount of alumina.²⁴ To counter LTD, the amount of Y_2O_3 is increased (from 3 mol% to mol8%) in the high translucency zirconia which results in an increased amount of cubic phase zirconia. The cubic phase of zirconia is isotropic in different crystallographic directions, which decreases light scattering that occurs at grain boundaries and results in improved translucency.^{28,31,33} However, this approach reduces the flexural strength and

fracture toughness as a result of decreased transformation toughening effect of the tetragonal phase of zirconia.²⁴

There are other factors that can also impact the translucency of zirconia such as sintering temperature and atmospheric conditions during sintering which determine the density, porosity, and grain size of zirconia.^{40,41}

Lithium disilicate material has high transmittance values because of the refractive index of the lithium disilicate glass crystals which matches that of the glassy matrix. Also the linear well-organized and regular arrangement of the crystalline structure within the glass-ceramic improves its transmittance values as comparison to conventional zirconia.³³

The present study shows that translucency of each ceramic system decreased with increasing the material thickness. Thus, the fourth null hypothesis was accepted. Corresponding results were shown by Church et al. and Wang et al. who found that translucency significantly decreased as the thickness of each ceramic material increased.^{27,42}

Church et al conducted a study to evaluate the translucency and strength of highly translucent zirconia ceramic materials and compare those with a high-translucency, lithium disilicate glass-ceramic.²⁷ They sectioned the materials into 0.50, 1.00, 1.50, and 2.00mm thick specimens and calculated the translucency parameter using spectrophotometry. The results of their study

showed that at 0.50mm thickness, high translucency zirconia (BruxZir Shaded 16, inCoris TZI C, Lava Plus, and BruxZir HT) was less translucent than IPS e.max CAD HT at the same thickness but similar to or more translucent than IPS e.max CAD HT at 1.00mm thickness. Cherch et al concluded that their studied high translucency zirconia materials could replace dentin within a restoration in terms of translucency.²⁷ However, according to the present study, even 1mm thick IPS e.max CAD showed significantly higher translucency compared to 0.50mm high translucency zirconia.

4.3 Flexural Strength:

Flexural strength estimates the resistance of a material under bending, which is a common form of stress in prosthetic dentistry and can be calculated using bi-axial flexure testing. In this method, a disc shaped specimen is subjected to a bi-axial moment in its central region. The maximum stress occurs at the center of the surface opposite to load application and fracture happens as soon as the most critical of the flaws starts to grow.^{27,42}

The results of the current study supports the first null hypothesis that conventional zirconia shows significantly higher flexural strength value compared to high translucency zirconia and lithium disilicate. In addition, high translucency zirconia showed significantly higher biaxial flexural

strength values compared to lithium disilicate, which was in accordance with other studies.^{27,31,37}

Church et al. evaluated translucency and flexural strength of highly translucent monolithic zirconia ceramics and compared these properties to that of high-translucency, lithium disilicate monolithic glass-ceramic material.²⁶ They revealed that all high translucency zirconia materials showed significantly higher flexural strength value (855.2 – 953.9MPa) compared to lithium disilicate (387.4MPa) at 1.3mm thickness. Some other studies have shown that high translucency zirconia materials demonstrated approximately two thirds more flexural strength than lithium disilicate.³³

Conventional dental zirconia (TZP) contains 3 mol% yttria to stabilize its tetragonal phase at room temperature. 3Y-TZP can undergo transformation toughening, in which a transformation zone forms and shields the growing crack and enhances the fracture toughness of the material. However, 3Y-TZP is very opaque due to the presences of Al_2O_3 . Alumina helps prevent the formation of pores during sintering and helps stabilize the tetragonal phase. Thus, in order to improve the translucency of 3Y-TZP, the alumina content was decreased from 0.25wt% to 0.05wt% in some variants. However, the new material is more susceptible to low-temperature degradation (LTD) because there is less alumina to stabilize the tetragonal phase.^{28,37,44}

More recently, dental zirconia has been fabricated with increased yttria content in order to create materials with more cubic phase of zirconia which results in more translucency. The examples of these materials are completely stabilized cubic phase (doped zirconia with 8 mol% yttria), and partially stabilized zirconia with approximately 50% cubic phase zirconia (doped zirconia with 5 mol% yttria) such as Katana UTML. The drawback of stabilized cubic zirconia is its lack of transformation toughening and possible low-temperature degradation, which causes reduced mechanical properties.^{28,37}

Chen et al. mentioned in their study that all-ceramic materials for posterior teeth require a minimum biaxial flexural strength of 150MPa. Thus, based on this recommendation, all the ceramic systems used in the current study could be used for posterior tooth restorations.

The results of our study suggest that according to the ISO 6872:2015 table, high translucency zirconia could be used as a monolithic ceramic for three-unit prostheses involving molar restoration (ISO class 4). However, the decision in regards to clinical indications of 5Y-ZP should be made carefully, as 5Y-ZP does not have the same potential to undergo transformation toughening and is likely less durable than conventional zirconia.^{37,45}

It was observed in the present study that flexural strength values within each group for all three thicknesses were not significantly different, and therefore, the third null hypothesis was rejected. However, some studies have shown that biaxial flexural strengths for ceramic materials are higher when using thicker test specimens.¹⁰ The fact that no statistically significant relationship between material thickness and flexural strength was noticed in this study could be due to the minimum thickness difference (0.25mm) between subgroups.

Lowering the thickness of the restoration makes the material more translucent, but minimal indicated thickness should always be considered in order to avoid the risk of short-term material fracture failure. Thus, the minimal thickness provided by the manufacturer should be investigated carefully.³¹

Table 6. Clinical recommendation by ISO 6872:2015 for dental ceramics.

	Class Recommended clinical indications Flexural strength minimum (mean) MPa	
1	(a) Ceramic for coverage of a metal framework or a ceramic substructure. (b) Monolithic ceramic for single-unit anterior prostheses, veneers, inlays, or onlays.	50

2	(a) Monolithic ceramic for single-unit, anterior or posterior prostheses adhesively cemented. (b) Partially or fully covered substructure ceramic for single-unit anterior or posterior prostheses adhesively cemented.	100
3	(a) Monolithic ceramic for single-unit anterior or posterior prostheses and three-unit prostheses not involving molar restoration adhesively or non-adhesively cemented. (b) Partially or fully covered substructure for single-unit anterior or posterior prostheses and for three-unit prostheses not involving molar restoration adhesively or non-adhesively cemented.	300
4	(a) Monolithic ceramic for three-unit prostheses involving molar restoration. (b) Partially or fully covered substructure for three-unit prostheses involving molar restoration.	500
5	Monolithic ceramic for prostheses involving partially or fully covered substructure for four or more units or fully covered substructure for prostheses involving four or more units.	800

CHAPTER 5: CONCLUSIONS

Within the limitations of this in-vitro study, it was found that lithium disilicate glass-ceramic has a significantly higher translucency in comparison with conventional and high translucency zirconia. Thus, the transmittance value of high translucency zirconia materials is still inferior to lithium disilicate, which

should be carefully considered when selecting it as a single restoration in the esthetic zone depending on the translucency needed for a successful restoration.

According to the present study, the flexural strength of conventional zirconia was significantly higher than high translucency zirconia and lithium disilicate glass-ceramic. Therefore, the mechanical properties of high translucency zirconia may be of concern when planning for more than three units FPDs in the posterior area.

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