Flexural Strength of Various CAD/CAM Ceramic Materials

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ABSTRACT

**Aim and Hypothesis:** The aim of this in vitro study was to evaluate and compare the flexural strength of the recently introduced zirconia reinforced lithium silicate glass-ceramic with lithium disilicate and feldspathic ceramics, and to investigate the effect of various surface treatments on the fracture resistance of the tested materials.

**Materials and method:** 120 specimens of three types of CAD/CAM ceramic blocks were divided into three groups: zirconia-reinforced lithium silicate ZLS (Celtra Duo) for group (1), leucite-reinforced feldspar glass-ceramics LRF (IPS Empress1 CAD) for group (2), and lithium disilicate ceramics (LDS) (IPS e-max CAD) for group (3) (Ø14.5 x 12.5 mm, thickness 1.5 mm). Specimens were randomized into four subgroups for each group. The first subgroup (A) did not receive any surface treatment, the second subgroup (B) received polishing only, the third subgroup (C) received glazing only, and the fourth subgroup (D) received both the polishing and glazing surface treatments. Biaxial flexural strength test was performed at a rate of 0.5 mm/min until failure occurred and biaxial flexural strength was calculated in MPa.

**Results:** The study found that group (A, 2) showed the lowest value of biaxial flexural strength (FS) (89.34±25.30MPa) and group (D, 3) showed a significantly higher FS value of (365.38±52.52MPa) in comparison to control and polished A and B, which showed no statistically significant difference between each other (p=0.683), while subgroup C had no significant difference with subgroup D (p=0.145).
There was a statistically significant difference detected among the material groups. Material 3 showed the highest FS and was significantly different (p<0.001) from both materials (1 and 2).

**Conclusion:** For CAD/CAM materials, LDS has higher fracture resistance followed by ZLS, and the least mechanical strength was exhibited by LRF. Polished surface treatment was more prone to have a negative influence on the flexural strength. However, glazing combined with polishing had a significant effect on increasing the flexural strength of ceramics.
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Flexural Strength of Various CAD/CAM Ceramic Materials
Computer-aided design (CAD) and computer-aided manufacturing (CAM) technologies have given rise to a new era in modern dentistry. CAD/CAM technologies were first introduced in 1971 by Francois Duret. These technologies were developed to improve the strength and natural appearance of dental restorations and to achieve the same outcomes, using faster and easier means, without compromising accuracy. CAD/CAM technologies have facilitated machining materials like the high functioning ceramics and titanium with high accuracy.

Throughout dental history, the objective of clinicians and manufacturers has been to attain esthetically and functionally ideal restorations. Many of the esthetic materials for CAD/CAM machining have been introduced, developed, and advanced with the rapid evolution of the CAD/CAM technology. Also, patients’ demand for tooth-colored restorations, metal-free has continued with the restorative development.

Besides esthetic outcomes, permanent dental restorations require superior physical properties. The success of a dental restoration is determined by three main factors: marginal adaptation, resistance to fracture and esthetic value. Ceramics, for instance, are brittle in nature, which increases their susceptibility to fracture under tension. This brittleness behavior is an outcome of cracks initiated by stress concentration with subsequent catastrophic failure of the restoration. Clinical data on the survival rates of all-ceramic dental restorations show that the fracture vulnerability occurs from repetitive occlusal loading force. Flexural strength is the most
popular test for dental ceramics, cements, and polymers, due to its easy specimen preparation procedure and because no sophisticated sample grips are required.[11] Ceramic materials are weaker in tension than in compression; measuring the flexural strength of these ceramics is considered a valid way to evaluate their strength.[12] Flexural strength often symbolizes the capacity to tolerate chewing force, which is about 300 to 500 Newtons in healthy adults and is usually measured in MPa.[13,14] There are two major techniques that have been reported to determine the flexural strength of ceramics: the uni-axial and the biaxial flexure test.[12] 3-point and 4-point bending tests used to be the most common methods of testing. However, due to the significant drawback of difficulty in eliminating undesirable edge failures, a bi-axial flexure test is now frequently used as a substitute. [11]

There are a number of factors affecting the fracture resistance of all-ceramic restorations, surface finish and treatment, types of ceramic material, cement thickness, and type of luting agents.[15] All-ceramic materials undergo various fabrication procedures in the laboratory and some clinical modifications to permit proper occlusion, shape, or fitting. These adjustments or procedures likely create some surface roughness, which has been shown to diminish flexural strength by initiating subcritical flaws and even enormous defects. According to Mencik J surface roughness may cause higher stress concentration and the development of larger flaws among the microstructure of these materials; milling procedures roughen the surface and set small micro-cracks.[16-18] Generation of residual stress, along with the formation of a radial-lateral crack network, was noted from the contact forces that cause crushing, plastic
flow, and elastic recovery which, in place, may develop an unfavorable situation such as fracture of the material. These cracks most likely compromise the long-term prognosis of the restoration and lead to restorative failures.[16, 19] The presence of these cracks has been confirmed in fractographic studies by Rice and Mecholsky. [18, 20]

Mechanical polishing with ceramic burs and glazing are common methods used to achieve ceramics surface smoothness. The impact of the processing procedures, polishing, grinding and glazing on the mechanical properties of the all-ceramic restorations has been investigated by many researchers such as Bhamra et al.[21] Giordano et al.[22], Campbell et al.[23], and Rosenstiel et al.[24] Nonetheless, there is disagreement regarding the most appropriate method to achieve a strong and smooth surface necessary for both esthetics and the biological reasons.[19, 25] CAD/CAM ceramic materials tend to be over-glazed to strengthen their material properties and improve the esthetic appearance. Glazing can be applied either as an auto-glazing or by low-fusing glass overcoat, which is established by a certain time of firing with a maximum temperature.[19]

Reductions in depth and sharpness of surface flaws and porousness are believed to be a result of glazing; for that reason, this practice has been generally recommended for ceramic materials[26]. However, several studies show no effect on strength as a result of glazing [15, 19] The ability of polishing ceramic materials to remove the defects and flaws resulting from surface treatments or adjustments are considered to be responsible for increasing the flexural strength of the
material.[19] Polishing the restorations may be done by using polishing kits, disks, or cleaning-prophylaxis paste materials.[16] It has been hypothesized that the strength of the restoration increases more than 50% after polishing. The strength obtained by polish may result from the removal of larger surface flaws and a combination of compressive residual stress. Many studies have demonstrated the strengthening effect on ceramics after polishing.[16, 17, 20] Studies such as Giordano et al. [22] Campbell [27] and Levy et al.[20] tested the effect of polishing and glazing on the flexural strength of dental ceramics. Results showed no significant difference between treatments; however, polished glazed specimens had higher strength values. The problem in both studies was that the degree of polishing was not objectively quantified. Other studies, such as Rosenstiel et al.[24], contradicted these results by determining that a polished surface improved the fracture resistance of the ceramic in contrast to a glazed surface.[20]

Currently, there are two main groups of permanent, non-metal CAD/CAM restorative materials: ceramics and composites. Dental ceramics are recognized for being esthetically pleasing, biocompatible, and chemically durable and stable. [5] The ceramic group is divided into glass ceramics and polycrystalline.[8] Lithium disilicate and zirconia are the most common CAD/CAM ceramic materials used for single crowns. Both materials can be implemented either as substructures or as monolithic restorations, followed by the application of a porcelain veneer for esthetic improvement. These restorations are milled from prefabricated blocks of porcelain, either fully- or partially-crystallized or sintered.[1] However, recently, new materials have been
developed as alternatives to CAD/CAM single crown restorations, such as restorations zirconia-reinforced lithium silicate (ZLS) ceramics (e.g., CeltraDuo, DeguDent), resin-nano ceramics (e.g., Lava Ultimate, 3M Espe), and polymer-infiltrated ceramic-network materials (e.g., Enamic, Vita).[28]

Vita block Mark I (Vident), the first in-office ceramic material, was compressed into a block of fine grain feldspathic-based ceramic that was milled into a dental restoration. Around 1987, the original Mark I evolved into the current generation of blocks Vita Mark II (Vident). The blocks are fine feldspathic porcelains and are composed of silica oxide and aluminum oxide.[10, 29] Mark II blocks are fabricated from feldspar porcelain particles embedded in a glass matrix, and exhibit better mechanical properties than Mark I. Mark II blocks are used for single unit restorations, and are now available in polychromatic blanks.[30]

In early 1998, IPS ProCAD™ (Ivoclar Vivadent) was proposed as a leucite-reinforced glass ceramic and is similar to IPS Empress™ press-able materials (Ivoclar Vivadent) in physical structure and properties. In 2016, IPS Empress™ CAD (Ivoclar Vivadent) was introduced as the successor of IPS ProCAD™. It has 30-45% leucite-reinforced glass with a finer particle size than IPS Empress I™ of about 1–5 mm that helps withstand the machining damages.[31] Its flexural strength is about 160 MPa.[32] Feldspathic and leucite glass-ceramics are known for their high levels of aesthetics-translucency and simulation of natural tooth features. They are indicated for veneers, three-quarter crowns, onlays, and complete coverage crowns, that mostly restricted to
the anterior segment, mostly due to their strength limits. [33]

In 1998, a new material was presented to the glass ceramics field: lithium-disilicate glass ceramic, which is comprised of quartz, lithium dioxide, phosphor oxide, alumina, potassium oxide, and other components. In 2006, Ceramic IPS™ e.max CAD (Ivoclar-Vivadent) system was introduced as an innovative lithium disilicate CAD/CAM and is a chairside monolithic restorative material. [32] This product is supplied as blue blocks, which contain about 40% partially-crystallized lithium meta-silicate and lithium disilicate nuclei. At this state, the flexural strength is about 130 MPa; the rationale for this is that it makes the blocks easier to mill the restorations without damage to the material or excessive diamond bur wear, in comparison to fully-sintered blocks.[34] After milling, the firing process is conducted in a porcelain oven at 840°C for 20 to 25 minutes, to complete the crystallization by converting the lithium metasilicate crystal phase to lithium disilicate crystal phase; the ceramic is glazed at the same time[10]. The block changes from blue to the selected shade and desired translucency.[32, 34] At this step, all crystal particles increase in size to approximately 1.5 μm and 70% crystal volume, incorporated in a glass matrix. The flexural strength of the material is increased by 360 to 400 MPa[35]. This crystallization step is usually associated with a 0.2% shrinkage, accounted for by the designing software[36]. Lithium-disilicate glass ceramics can function adequately when restoring 3-unit FPDs in the anterior area and posterior segment for single crowns.[33]

Polycrystalline ceramics, such as alumina and zirconia, have all of their crystals
densely packed into regular arrays which are then sintered. Especially in zirconia, this
dense crystal lattice prevents crack propagation, or “transformation toughening,”
resulting in excellent mechanical properties.[32] Zirconia (crystalline zirconium dioxide)
also known as “ceramic steel,” is the prototype material for this process. Zirconium (Zr)
is a very strong metal and shares the same chemical and physical properties of titanium
(Ti); [37] it also exhibits some tooth color resemblance. [38] Zirconium has polymorphic
forms and characteristics that occur depending on the temperature; it adopts its
monoclinic crystal structure at room temperature and converts to a tetragonal and cubic
at higher temperatures. The volumetric changes resulting from the structure transitions
from tetragonal to monoclinic to cubic encourage significant stresses, leading it to crack
upon cooling from high temperatures. The tetragonal or cubic phases can be stabilized
when the zirconia is integrated with some other oxides. The most common form used in
dentistry is yttrium oxide (Y₂O₃, yttria). [39, 40] By adjoining small percentages of yttria,
these disruptive phase changes caused upon heating or cooling are removed, and the
arising material has superior mechanical, electrical and thermal characteristics. When
sufficient stress concentration develops in the tetragonal structure and a crack in the
area begins to propagate, the metastable tetragonal crystals (grains) or precipitates
next to the crack tip can convert to the stable monoclinic form, with the associated
volume expansion. This phase transformation can then place the crack under a state of
compressive stress and retard its growth. [41, 42] This mechanism (transformation
toughening) is extending the reliability and lifetime of the stabilized zirconia. Recently,
many companies have incorporated zirconia into their CAD/CAM workflow due to its
high properties. Some of these properties include high mechanical strength, fracture
toughness, radiopacity, low corrosion potential, and natural-tooth appearance.[7] In-Ceram Zirconia (Vident) was one of the first CAD/CAM systems that used zirconia.[36] Still, there are some limitations facing this material. One of the most frequently occurring complications is delamination or chipping of the veneering ceramic on bi-layered zirconia restorations. For this reason, monolithic zirconia restorations were introduced, which had some esthetic limitations due to high opacity and were restricted to posterior restorations. [18, 20]

It can be surmised that the introduction of zirconia fillers into glass-ceramic has increased the flexural strength (900 MPa to 1,200 MPa) and the modulus elasticity. [12,18, 20]

Due to the increased demands of having a combination of high strength and adequate translucency for monolithic ceramics, a new group of CAD/CAM ceramic materials has recently been introduced: zirconia-reinforced lithium silicate ceramics (Celtra Duo, Dentsply DeTrey, Konstanz, Germany; Suprinity, Vita Zahnfabrik, Bad Säckingen, Germany). These materials offer enhanced flexural strength values equivalent to lithium disilicate and enhanced aesthetics, because of the microcrystalline structure and the increased glass content, ranging from 370 to 420 MPa. The values of mechanical properties are approximately three times higher than traditional leucite-reinforced glass ceramics (IPS Empress, Ivoclar Vivadent, Schaan, Liechtenstein). [6, 28]

Per the manufacturer, this newly developed generation of glass ceramic materials combines the advantageous characteristics of glass ceramics and zirconia, achieving
superior optical and mechanical properties. [43, 44]

This system contains a dual microstructure of fine lithium meta silicate and lithium disilicate crystals in a glassy matrix consisting of 10% zirconium dioxide by weight. [44] After the final crystallization process, the material exhibits very fine microstructures that provide a high percentage of glassy matrices and, at the same time, superior flexural strength. The material is as anatomically-contoured as monolithic restoration and offers different shades. Also, it can be easily finished by both glaze-firing and surface polishing. The manufacturer has claimed that, if the material is glazed, it attains 370 MPa flexural strength in 29:50 minutes; if manually polished, it attains 210 MPa, which is twice as strong as glass ceramics after milling. [6,28]

Research Aims and Hypothesis

The aim of this in vitro study was to evaluate and compare:

1) The flexural strength of the recently introduced zirconia reinforced lithium silicate glass-ceramic with lithium disilicate and feldspathic ceramics.

2) The fracture strength of the ceramic materials that are within the clinically acceptable range, around 408 N or higher. [45]

3) The surface treatments that will enhance the fracture resistance.

Hypotheses:

1) Fracture strength of lithium disilicate is higher than zirconia reinforced lithium silicate glass which is stronger than feldspathic ceramic.
2) The fracture strength of each material is within the clinically acceptable range

3) The combination of polishing and glazing of the surface will provide the highest fracture strength compared to the other surfaces.

**Clinical significance:**

This research will give the clinician an estimate of the fracture resistance of the ceramic material and how the surface treatment will affect the flexural strength.
Material and Methods:

1- Specimen Preparation and testing

120 rectangular shaped specimens were sectioned from blocks using a diamond saw (Isomet 1000, BUEHLER, Germany) underwater irrigation, and grinding-polishing machine (EcoMet® 250, BUEHLER, Germany) was used for optimal sample thickness preparations. (Figure 10,11).

Each specimen had a diameter of (14.5 x12.5 mm) and then each was trimmed to achieve 1.5mm thickness, to simulate the occlusal thickness of a crown. The dimension of each specimen was confirmed with a digital caliper (Dentagauge 2, Erskine dental) that is sensitive to 0.01 mm for standardization (Figure 2). The 120 plates were a collection of three types of materials according to the CAD/CAM blocks ceramic materials that were used: Leucite-reinforced feldspar glass-ceramics (IPS Empress I CAD, Ivoclar-Vivadent, Liechtenstein), zirconia-reinforced lithium silicate ZLS (Celtra® Duo, Dentsply, Germany) and lithium disilicate ceramics (IPS e-max CAD, Ivoclar-Vivadent, Liechtenstein). The IPS e-max CAD samples were fired at 820 °C and 20 minutes for full crystallization state using a porcelain oven (Programat P300, Ivoclar Vivadent, Liechtenstein). The samples of each type of the material group were randomly divided by Microsoft Excel 2017 (version 16.11.1) software, after numbering each material group from 1 to 30, into four subgroups [(A) no surface treatment (control), (B) polished only, (C) glazed only and (D) polished and glazed]. (Figure 1a,4a)
For subgroup (C) samples were glazed using the glaze material provided by the manufacturer (IPS Emax CAD Glazes, Ivoclar Vivadent, Liechtenstein), for both material IPS Empress CAD and IPS e.max CAD. IPS e.max CAD was fired for full crystallization combined with glazed on firing cycle at 820 for 20 minutes and for IPS Empress CAD on firing cycle 820 for 20 minutes then followed with 830 for 15 minutes, as specifically recommended by manufacturer for using IPS Emax CAD Glazes, and glaze material (Celtra™ Universal Glaze, Dentsply, Germany) used for Celtra Duo on firing cycle 932 for 20 minutes. For these subgroups, each specimen was cleaned using steamed air (Triton SLA, Bego, USA), and then two thin coats of a clear glaze were applied with porcelain brush. After that, specimens were fired in a furnace (Programat P300, Ivoclar Vivadent, Liechtenstein) for the appropriate period of time that described in figure (4b). The glaze was applied to non-polished and polished specimens.

For subgroup (B) samples were polished by using porcelain polishing burs that are recommended by the manufacturer (Celtra TwisTec® Polishing Kit, Dentsply, Germany) for Celtra duo, and (Optrafine® Polishing, Ivoclar Vivadent, Liechtenstein) for IPS Empress 1 CAD and IPS e.max CAD. The polishing steps were performed manually by a single operator using lab micro-motor hand-piece (Ultimate XI-K, NSK, Japan) in sweeping motion forward and backward for 30 seconds (12 strokes) for the assigned specimens on consistent pressure as shown in (Figure 6). For IPS Empress CAD and IPS e.max CAD, finishing was done with ‘Finisher F’ bur the light blue colored, followed by polishing with ‘Polisher P’ the dark blue colored, and lastly for the high gloss finish, high-gloss brush and diamond polishing paste HP were used. Recommended speed used at a maximum 15000 rpm for polishing burs, and 5,000 rpm for the high-
gloss polishing (Figure 6). For Celtra duo pre-polishing was done with the green wheel followed by surface smoothening with the blue wheel at recommended speed 10,000 rpm; after that high gloss polishing was completed with the brown wheel at 7,000 rpm speed.

For subgroup (D) samples were polished first and then glazed with the same materials and companies used in subgroups (B and C). (Figure 5, 7)

Specimens for this in-vitro study were randomly allocated. After that, each specimen was labeled on the back with a letter indicating the assigned group [(I) for Celtra duo, (II) for IPS Empress CAD and (III) for IPS e.max CAD], and a number (1-10) indicating the number of the specimen within the group using permanent pen marker. (Figure 1B)

2- **Biaxial Flexural Strength:**

Each specimen group was secured in the Universal Testing Machine with an appropriate sample holder (Model 5566; Instron Corp, Canton, MA, USA). A thin tape (50μm in thickness) was applied in the compression side of the plate between the upper surface of the specimen and the piston, to avoid spreading of the fragments and distribute the load uniformly until failure at a crosshead speed. The specimens center was placed upon the steel and the equilateral triangle center was aligned coaxially. After the positioning, the specimen’s center was loaded from above with a plunger through piston tip at a cross-head speed of 0.5 mm/min in air at room temperature until catastrophic failure occurred. Computer software (Bluehill 2, Instron, Canton, MA, USA) was used for displaying and controlling the results. The maximum compressive load (N)
and the flexural strength (MPa) were calculated as described in ISO 6872. (Figure 7,8)[46]

\[ \sigma = -0.2387P \frac{(X - Y)}{b^2} \]

\[ X = \left( \frac{1+v}{1} \right) \ln \left( \frac{r2}{r3} \right)^2 + \left( \frac{1-v}{2} \right) \left( \frac{r2}{r3} \right)^2 \]

\[ Y = \left( \frac{1+v}{1} \right) \left( 1 + \ln \left( \frac{r1}{r3} \right)^2 \right) + \left( 1-v \right) \left( \frac{r1}{r3} \right)^2 \]

- \( \sigma \): maximum tensile stress (MPa)
- \( P \): the total load causing fracture (N)
- \( b \): thickness at fracture origin (mm) = 1.5 mm
- \( v \): Poisson’s ratio (0.25)
- \( r1 \): radius of the support circle (mm) = 10 mm
- \( r2 \): radius of the piston (mm) = 1 mm
- \( r3 \): radius of the specimen (mm) = 12.5 mm

### 3- Sample Size Calculation

A sample size calculation was performed using nQuery Advisor (Version 7.0) software. Based on the means and the pooled standard deviation obtained in a pilot study*, a sample size of \( n=10 \) per group was found to achieve a power of 81% for the comparison of surface treatments and a power greater than 99% for the comparison of materials, using a Type I error rate of 0.05.

* The pilot study had a sample size of \( n=3 \) per group
4- **Statistical Analysis**

All analyses were performed using SPSS version 24. Descriptive statistics (mean, median, standard deviation [SD], and interquartile range [IQR]) were calculated for each combination of material (Celtra Duo, Empress I CAD, and Emax CAD) and surface treatment (control, polished, glazed, polished and glazed). Initially, a two-way analysis of variance (ANOVA) was conducted and the interaction between material and surface treatment was found to be statistically significant (p<0.001). Therefore, a separate comparison of materials was performed for each surface treatment, and a separate comparison of surface treatments was performed for each material. Normality was assessed using the Shapiro-Wilk test; homogeneity of variance was assessed using Levene’s test. In cases of significant non-normality, the Kruskal-Wallis test was used alongside Dunn’s test and the Bonferroni correction for post-hoc comparisons. In cases where the test of normality was not significant but the test of homogeneity of variances was significant, Welch’s test was used alongside the Games-Howell test for post-hoc comparisons. In cases where neither the test of normality nor the test of homogeneity of variances was significant, one-way ANOVA was used alongside Tukey’s HSD for post-hoc comparisons. Additionally, the count and percentage of samples with acceptable fracture strength were calculated for each combination of material and surface treatment. Fisher’s exact test was conducted alongside the Bonferroni correction for post-hoc comparisons. Fracture strength of at least 408 N was considered acceptable based on previous literature [45].
RESULTS:

Descriptive statistics for all groups are presented in Table 1. Group (A, 2) showed the lowest mean flexural strength (89.34±25.30MPa) while the highest mean value was recorded for group (D, 3) (365.38±52.52MPa).

The difference between surface treatments when using material 1 was statistically significant (Table 2). Specifically, in post-hoc tests, comparisons of subgroups A and B with subgroups C and D were significant (p<0.001). However, there was no significant difference between subgroups A and B (p=0.683) or between subgroups C and D (p=0.145). For material 2, there was no significant difference between the groups, except between subgroups B and D (p=0.028), with subgroup D exhibiting a higher mean. Finally, for material 3, comparisons were not statistically significant, except that subgroup B exhibited lower flexural strength in comparison with subgroups C (p=0.012) and D (p=0.001).

For subgroups C and D, statistically significant differences were found between all materials (Table 3). For each of these subgroups, material 3 exhibited the highest flexural strength, and material 2 exhibited the lowest. For subgroups A and B, material 3 again exhibited the highest flexural strength, and material 2 again exhibited the lowest; there was a significant difference between materials 1 and 3 and between materials 2 and 3, but not between materials 1 and 2.

Table 4 displays the count and percentage of samples with acceptable fracture strength (at least 408 N), as well as the mean and standard deviation of fracture strength, for each combination of material and surface treatment. Material 3 exhibited
100% acceptable fracture strength when in combination with subgroups A and D. Material 1 exhibited 0% acceptable fracture strength when in combination with subgroups A and B; Material 2 exhibited 0% acceptable fracture strength when in combination with all subgroups. For materials 2 and 3 there was no statistically significant difference between the surface treatments (p=1.00, p=0.167, respectively) and material 1 showed statistical significance in the global test (p=0.002). However, in the post-hoc tests, there was no statistically significant difference when using the more stringent Bonferroni correction (p>0.0083) (Table 5). For each of the surface treatments, the global test comparing the materials was statistically significant (p<0.001). Table 6 shows that no surface treatments displayed a statistically significant difference between material 1 and material 2 except for subgroup D (p=0.011). However, when comparing material 2 and material 3 there were statistically significant differences for all surface treatments. In addition, there were statistically significant differences between material 1 and material 3 except for subgroup D (p=0.087).
DISCUSSION:

Ideally, a clinically reliable dental ceramic should withstand high chewing forces during a long service period. The bend test is a reliable, simple, and sensitive method for testing the comparative strength of dental ceramic materials. Many novel CAD/CAM restorative materials have been introduced to attain functionally and aesthetically ideal restorations. The fracture strength and longevity of these restorations are influenced by various factors, for instance their mechanical properties, intraoral conditions, luting agents, and fabrication techniques. Clinical studies tend to require extensive time and costs. Therefore, preclinical and in vitro studies should be conducted to assess the durability of these restorations. The number of experimental conditions influences the flexural strength, such as geometry, design, size, and thickness of specimens, in addition to the position and direction of the force applied. Rectangular ceramic plates were adopted as the simple experimental design. These plates are used as an alternative to disc-shaped specimens in the biaxial test, which demonstrates the suitability for materials that are supplied in blocks. Optimizing standardized geometry will reduce the incorporation of defects, and results in a more homogeneous material sample. [15, 47, 48]

This in vitro study was conducted to investigate flexural strength and the effect of surface treatments on various CAD/CAM ceramic restoration strengths. The statistical analysis showed that the flexural strength values of the IPS Empress 1 CAD material has the lowest flexural strength (89.34±25.30MPa) with no surface treatment, and the
IPS e.max CAD shows the highest value (365.38±52.52MPa) with glazed and polished surface treatment. Overall, our results indicate that IPS e.max CAD had the highest flexural strength. Results also show that flexural strength value was at its peak with a combination of polished and glazed surface treatments. According to the results of the present investigation, there were statistically significant differences between the ceramic materials and the surface treatments. Additionally, the maximum force that was applied and broke all the samples varied in their percentage range.

Regarding the amount of flexural strength in relation to the clinically minimum acceptable range of fracture resistance of the materials tested, the limit number used was set to 408 N, based on the Miura et al. literature. This number shows that the material should withstand the median molar bite force of healthy individuals in the posterior region. The results of this study show that the percentage value of the most of e.max CAD surface treatments fall within the acceptable force range, and both of other materials—Empress CAD and some of Celtra Duo—exhibited lower percentages in the acceptable force range. From this result, e.max CAD could be possible for restoring single posterior crowns. However, Empress CAD and Celtra Duo may be suggested to be restricted to restoring anterior crowns.

Unlike the tested ceramics, IPS e.max CAD was tested after firing in all subgroups. This might have contributed to the its increased flexural properties. According to the manufacturer, the partially crystallized lithium disilicate material has a 40 vol% of 0.2–1.0 mm crystal size and increases to 70 vol% of 1.5 mm grain size after complete crystallization. Through this process, the particle size increases from 0.5 μm to
5 μm. During the conversion processes, prismatic glass-ceramics are established and distributed over the glassy matrix. Because of this alteration, the restoration flexural strength increases 1.7-fold. This high crystal content, and the high degree of interlocking crystals, is attributed to the development of compressive stresses within the material leading to crack deflection, a condition that resists crack propagation.[49]

In general, the IPS Empress CAD obtained the lowest flexural strength in all its subgroups, although not all differences were significant. Leucite is added to porcelains by manufacturers to improve resistance to crack propagation, obtaining a higher fracture toughness. The study performed by Chen et al.[4] found that by optimizing the microstructure of the fine-grained leucite content, the biaxial flexural strength of glass-ceramics improved. Yet, in our data this material still obtained the lowest strength.

In our investigation, Celtra Duo exhibited lower strength than IPS e.max CAD, which is not as claimed by the manufacturers. However, compared with leucite feldspathic, it was reported to have a significantly higher flexural strength, which is in agreement with our test. Some studies, such as the Nathaniel et al [51] study, reported Celtra Duo as having equivalent flexural strength to e.max CAD, and its strength was suppressed after firing. Nathaniel et al. tested sectioned bars of Celtra Duo (fired group) and partially crystalized e.max CAD (then fired). The bars were then stored in distilled water at 37°C for 24 h. After storage, the specimens were tested in a 3-point bending fixture. Their numbers were: 300.1 MPa for the unfired Celtra Duo and the fired value reached 451.4 MPa; the e.max FS had a value of 376.9 MPa. Such results are in
disagreement with our results. The study also showed a difference in methodology by using distilled water. Another study, conducted by Badawy et al. [52], showed different outcomes. In their study, they demonstrated that Celtra Duo had lower flexural properties when compared to IPS e.max. Their flexural strength data for fired IPS e.max CAD was 359.87 MPa and 177.32 MPa for Celtra Duo. This study is in agreement with our data, however, their FS values were higher than our corresponding values. Also, a study recently published by Wendler et al.[47] reported that both materials had variations in strength values, with Celtra Duo at 565.80 MPa and e.max CAD at 609.80 MPa. When comparing our results to those of completed studies, the insufficient research and testing of these materials makes arriving at a significant conclusion challenging.

Many authors, such as Aurélioa and Giordano [53,20], consider overall glazing procedures, which generally influences the mechanical strength of all-ceramic restorations. These positive impacts include reducing porosity, blunting the flaw tips, and sharpening and/or deepening the surface flaws. In the present study, the FS data of the glazed group in each type of material was significantly higher than the polished and control groups. This result was in agreement with those of some previous studies. Oh et al. [19] investigated the effect of heat pressing and subsequent heat firing on the flexural strength of Empress 2. They found that heat pressing had a positive influence on the flexural strength, but Cima and Pober [53] found that the flexural strength of conventional feldspathic ceramic reduced after the heat treatment.
The utilized heat treatment has a remarkable elevation in ceramic strength. As Giordano et al. [20] reported in their study, different thermal schemes and treatments should be engaged for ceramics in order to control residual stresses. Nathaniel et al. [51] suggested that the increased strength noted with a firing cycle is due to the healing of any defects that might be generated during the fabrication process. These treatments are conducted at the temperatures in which glass transition occurs. This causes the material viscosity to be reduced, and the structural rearrangements to promote the relaxation of internal stresses in the ceramic material.[53]

Previous studies have researched the effects of surface treatments, such as finishing and polishing on dental ceramics, and their effect on flexural strengths. However, multiple reports had limited detail of polishing procedures or a lack of standardization—they were difficult to correlate with our study. Furthermore, some investigators have expressed that the adoption of manufactured polishing techniques is not clinically applicable. Enhancing the strength of glass-ceramic by adopting a polishing procedure has generally shown to eliminate the deformation zone surrounding surface defects that are subsequent to any surface damage. [19, 51] Since the compared studies' polishing parameters were not quantified, the contradicting results, such as increasing or decreasing in strength, may root from the polishing process itself. In this study, polishing alone showed different results, as there was no significant difference when compared to the control group. It is assumed that polishing had either a negative effect or no effect on the sample thickness. This result is in agreement with some studies such as Rohana et al. [54], who reported in their results that polishing with
the three abrasive systems at the manufacturer’s recommended speed (10,000 rpm) had no effect on the flexural strength of ceramics. They assumed that these polishing procedures have limited strengthening effect as the magnitude of the compressive stresses that are produced is small. Also, they reported that a higher polishing speed produced significantly weaker specimens, in spite of the smoother surface they produced. Surface crack development could cause a reduction in ceramic strength—as the polishing wheels showed noticeable chatter during higher speed polishing.

Polishing is a mechanical chip-removal process that involves friction, which creates a glossy and smooth finish. Ahmad et al. described the actual polishing mechanism in their study: rubbing of the grain along the surface will cause friction. During the polishing operation, the grain produces wear flat and rubs along the polished surface. The energy dissipates mainly in the state of heat due to friction. The surface properties are adversely affected by the temperature rise during polishing, as residual stresses are induced on the workpiece.[55] The polishing process has to be a piecemeal approach, which will introduce finer scratches to the substrate surface in order to methodically eliminate the deeper scratches.

It could be speculated that the most practical method of increasing the flexural strength of all ceramic material is by applying a surface glaze as the final finish after polishing. As shown in our study, the flexural strength increase was approximately twice that of the polished surface only. Giordano et al. reported that a glaze, in which the thermal coefficient of expansion is slightly less than the underlying material, could
significantly improve the flexural strength of dental porcelains by shrinking the underlying material more than the glaze during cooling, compressing the glaze. Also, Giordano et al. [20] properly indicated that larger defects are eliminated during grinding and polishing procedures, so some flaws that may become cracks are removed and increase flexural strength. In another study, they reported that the flexural strength of dental ceramics decreased by 11% to 18% after re-firing the ground and polished samples. However, the flexural strength increased by 15% to 30% when over-glazing, grinding, and polishing is used. Levy et al [54] also reported no significant difference on flexural strength among treatments such as polishing with pumice, etching dental ceramics after air, vacuum glazing, and over-glazing; however, polished and glazed specimens had higher strength values. Hence, to help improve the ceramic restoration strength and increase their resistance to failure, careful polishing of the ceramic surface followed by glazing is recommended.

This study showed the fracture strength and resistance of various CAD/CAM ceramic restorations and the role of combining surface treatments in flexural strength. Significant information was obtained through a standardized comparison of flexural strength between IPS e.max CAD, IPS Empress CAD, and Celtra Duo ceramics with the following surface treatments: no treatment, polished, glazed, and combination (polished and glazed). The study does, however, have its limitations, and results are only valid for CAD/CAM ceramics processed under the same conditions and polished by the described protocols mentioned in the current study. Limitations include the in vitro design of the study, which does not evaluate the influence of the complex factors
present in the oral cavity such as parafunctional habits, dynamic occlusal load, neuromuscular forces, thermos-cycling, and abrasive food. In clinical situations, the material will be subjected to chemical changes, mechanical changes, and aging. The anatomical crown shape can be considered as another limitation as well as the influence of bonding agents on fracture strength, which can affect the flexural and compressive strengths. It is recommended for further studies in the future to compare the surface treatments and flexural strength of different luting or bonding agents while considering the effect and interaction of other factors, such as cyclic loading. It is also recommended that prospective clinical studies of these materials are conducted. Such procedures would have different results and conclusions.
CONCLUSIONS:

Under the conditions and limitations of the present study and based on findings presented, the following conclusions can be drawn:

- **Surface treatment** may significantly affect the flexural strength of the materials. The combination of polishing and glazing surface treatment was significantly higher than polishing alone for all three materials. No significant difference was found between the following surface treatments: control and polished alone surface treatments, and glazed alone and the combination of polished and glazed surface treatments of ceramic restorations.

- **Celtra Duo** showed significantly lower flexural strength than IPS e.max CAD. However, it displayed higher flexural strength than IPS Empress I CAD, although the difference was only significant for glazed and the combination of polished and glazed.

- A greater percentage of IPS e.max CAD samples were within the acceptable range of fracture resistance in comparison with IPS Empress I CAD and Celtra Duo.
APPENDIX A
Table 1: Descriptive data (MPa)

<table>
<thead>
<tr>
<th>Surface treatment</th>
<th>Material (Ceramic)</th>
<th>Mean</th>
<th>SD</th>
<th>Median</th>
<th>IQR</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>Celtra</td>
<td>125.79</td>
<td>32.01</td>
<td>136.40</td>
<td>53.47</td>
</tr>
<tr>
<td>Control</td>
<td>Empress</td>
<td>89.34</td>
<td>25.30</td>
<td>85.94</td>
<td>55.79</td>
</tr>
<tr>
<td>Control</td>
<td>Emax</td>
<td>317.45</td>
<td>43.35</td>
<td>300.74</td>
<td>73.88</td>
</tr>
<tr>
<td>Polished</td>
<td>Celtra</td>
<td>111.80</td>
<td>26.75</td>
<td>109.78</td>
<td>43.00</td>
</tr>
<tr>
<td>Polished</td>
<td>Empress</td>
<td>92.54</td>
<td>18.42</td>
<td>90.28</td>
<td>25.35</td>
</tr>
<tr>
<td>Polished</td>
<td>Emax</td>
<td>268.15</td>
<td>48.34</td>
<td>288.74</td>
<td>86.52</td>
</tr>
<tr>
<td>Glazed</td>
<td>Celtra</td>
<td>210.53</td>
<td>25.15</td>
<td>205.90</td>
<td>41.64</td>
</tr>
<tr>
<td>Glazed</td>
<td>Empress</td>
<td>110.00</td>
<td>7.68</td>
<td>108.30</td>
<td>8.79</td>
</tr>
<tr>
<td>Glazed</td>
<td>Emax</td>
<td>341.01</td>
<td>70.12</td>
<td>372.04</td>
<td>127.79</td>
</tr>
<tr>
<td>P&amp;G</td>
<td>Celtra</td>
<td>238.03</td>
<td>27.89</td>
<td>241.77</td>
<td>55.31</td>
</tr>
<tr>
<td>P&amp;G</td>
<td>Empress</td>
<td>114.54</td>
<td>11.83</td>
<td>116.54</td>
<td>24.18</td>
</tr>
<tr>
<td>P&amp;G</td>
<td>Emax</td>
<td>365.38</td>
<td>52.52</td>
<td>363.10</td>
<td>98.25</td>
</tr>
</tbody>
</table>

Table 2: P-values of tests comparing flexural strength of different surface treatments for each material type

<table>
<thead>
<tr>
<th>Materials Group</th>
<th>Celtra (1)</th>
<th>Empress (2)</th>
<th>Emax (3)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control polished</td>
<td>0.683</td>
<td>0.988</td>
<td>0.141</td>
</tr>
<tr>
<td>Control Glazed</td>
<td>&lt;0.001</td>
<td>0.122</td>
<td>0.293</td>
</tr>
<tr>
<td>Control P&amp;G</td>
<td>&lt;0.001</td>
<td>0.059</td>
<td>0.072</td>
</tr>
<tr>
<td>Glazed P&amp;G</td>
<td>0.145</td>
<td>0.742</td>
<td>0.456</td>
</tr>
<tr>
<td>Glazed polished</td>
<td>&lt;0.001</td>
<td>0.071</td>
<td>0.012</td>
</tr>
<tr>
<td>P&amp;G polished</td>
<td>&lt;0.001</td>
<td>0.028</td>
<td>0.001</td>
</tr>
</tbody>
</table>

- For Celtra Duo the global test was one-way ANOVA and the post-hoc tests were Tukey’s HSD. The p-value of the global test was <0.001.
- For Empress I the global test was Welch’s test and the post-hoc tests were Games-Howell. The p-value of the global test was 0.010.
- For Emax the global test was Kruskal-Wallis and the post-hoc tests were Dunn’s test with Bonferroni correction (the cutoff for significance was α≈0.0083 for post-hoc tests). The p-value of the global test was 0.007.
Table 3: P-values of tests comparing flexural strength of different material types for each surface treatment

<table>
<thead>
<tr>
<th>ST Group</th>
<th>Control (A)</th>
<th>Polished (B)</th>
<th>Glazed (C)</th>
<th>P&amp;G (D)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Celtra</td>
<td>Empress</td>
<td>0.063</td>
<td>0.264</td>
<td>&lt;0.001</td>
</tr>
<tr>
<td>Celtra</td>
<td>Emax</td>
<td>&lt;0.001</td>
<td>&lt;0.001</td>
<td>&lt;0.001</td>
</tr>
<tr>
<td>Empress</td>
<td>Emax</td>
<td>&lt;0.001</td>
<td>0.001</td>
<td>&lt;0.001</td>
</tr>
</tbody>
</table>

- For control the global test was one-way ANOVA and the post-hoc tests were Tukey’s HSD. The p-value of the global test was <0.001.
- For polished the global test was Kruskal-Wallis and the post-hoc tests were Dunn’s test with Bonferroni correction (the cutoff for significance was α=0.0167 for post-hoc tests). The p-value of the global test was <0.001.
- For glazed the global test was Welch’s test and the post-hoc tests were Games-Howell. The p-value of the global test was <0.001.
- For polished and glazed the global test was Welch’s test and the post-hoc tests were Games-Howell. The p-value of the global test was <0.001.

Table 4: Count and percentage of samples in each group with acceptable fracture strength, and mean (SD) fracture strength

<table>
<thead>
<tr>
<th>Material type and surface treatment</th>
<th>Count of acceptable range</th>
<th>Mean (SD) fracture strength (N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Celtra control</td>
<td>0 (0%)</td>
<td>212.94 (54.19)</td>
</tr>
<tr>
<td>Celtra polished</td>
<td>0 (0%)</td>
<td>189.27 (45.29)</td>
</tr>
<tr>
<td>Celtra glazed</td>
<td>2 (20%)</td>
<td>356.40 (42.57)</td>
</tr>
<tr>
<td>Celtra P&amp;G</td>
<td>6 (60%)</td>
<td>402.96 (47.21)</td>
</tr>
<tr>
<td>Empress control</td>
<td>0 (0%)</td>
<td>151.25 (42.84)</td>
</tr>
<tr>
<td>Empress polished</td>
<td>0 (0%)</td>
<td>156.66 (31.19)</td>
</tr>
<tr>
<td>Empress glazed</td>
<td>0 (0%)</td>
<td>186.22 (13.01)</td>
</tr>
<tr>
<td>Empress P&amp;G</td>
<td>0 (0%)</td>
<td>193.91 (20.03)</td>
</tr>
<tr>
<td>Emax control</td>
<td>10 (100%)</td>
<td>537.41 (73.38)</td>
</tr>
<tr>
<td>Emax polished</td>
<td>7 (70%)</td>
<td>453.95 (81.83)</td>
</tr>
<tr>
<td>Material Type</td>
<td>Group</td>
<td>Celtra (1)</td>
</tr>
<tr>
<td>---------------</td>
<td>-------</td>
<td>------------</td>
</tr>
<tr>
<td>Emax glazed</td>
<td>9 (90%)</td>
<td>577.30 (118.70)</td>
</tr>
<tr>
<td>Emax P&amp;G</td>
<td>10 (100%)</td>
<td>618.55 (88.91)</td>
</tr>
</tbody>
</table>

Table 5: P-values of tests comparing fracture strength of different surface treatments for each material type

<table>
<thead>
<tr>
<th>Materials Group</th>
<th>Celtra (1)</th>
<th>Empress (2)</th>
<th>Emax (3)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control polished</td>
<td>1.00</td>
<td>NA*</td>
<td>NA</td>
</tr>
<tr>
<td>Control Glazed</td>
<td>0.474</td>
<td>NA</td>
<td>NA</td>
</tr>
<tr>
<td>Control P&amp;G</td>
<td>0.011</td>
<td>NA</td>
<td>NA</td>
</tr>
<tr>
<td>Glazed P&amp;G</td>
<td>0.170</td>
<td>NA</td>
<td>NA</td>
</tr>
<tr>
<td>Glazed polished</td>
<td>0.474</td>
<td>NA</td>
<td>NA</td>
</tr>
<tr>
<td>P&amp;G polished</td>
<td>0.011</td>
<td>NA</td>
<td>NA</td>
</tr>
</tbody>
</table>

* NA: not applicable

Table 6: P-values of tests comparing fracture strength of different material types for each surface treatment

<table>
<thead>
<tr>
<th>ST Group</th>
<th>Control (A)</th>
<th>Polished (B)</th>
<th>Glazed (C)</th>
<th>P&amp;G (D)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Celtra Empress</td>
<td>1.00</td>
<td>1.00</td>
<td>0.474</td>
<td>0.011</td>
</tr>
<tr>
<td>Celtra Emax</td>
<td>&lt;0.001</td>
<td>0.003</td>
<td>0.005</td>
<td>0.087</td>
</tr>
<tr>
<td>Empress Emax</td>
<td>&lt;0.001</td>
<td>0.003</td>
<td>&lt;0.001</td>
<td>&lt;0.001</td>
</tr>
</tbody>
</table>


APPENDIX B
Figure 1A: Experimental design of the study.

Figure 1B: Randomization and labeling of specimens. Specimens were randomized equally into four groups and each specimen was labeled with a letter and a number on the back-unglazed nor polished surface.
Figure 2: Checking the diameter and thickness of specimens with digital caliper.

Figure 3: IPS e.max CAD plates before sintering (right) and after sintering (left).
Figure 4(a): Firing of specimens according to the manufacturer’s instructions after glaze was applied.

Figure 4(b): Firing program according to the manufacturer’s instructions after glaze was applied. 1) IPS e.max and Empress (1\textsuperscript{st}), 2) IPS empress (2\textsuperscript{nd}), 3) Celtra duo.

Figure 5 (a): Bench vacuum- based vise used to hold the specimens securely during finishing and polishing. (b): The straight electric hand-piece was aligned with the flat edge of the vise during grinding and polishing for standardization.
Figure 6: Polishing rotary instruments with Optrafine fine polishing wheels used to polish group B and D for IPS Emax CAD and IPS Empress CAD. Step 1: light blue. Step 2: dark blue. Step 3: Nylon brush wand high gloss polishing paste.

Figure 7: Polishing rotary instruments. Twistec celtra set, polishing wheels used to polish group B and D. Step 1: Green Wheel. Step 2: Blue Wheel. Step 3: White Wheel and diamond shape
Figure 8: Instron machine used for biaxial flexural strength test.

Figure 9: Fractured specimen at the end of the biaxial flexural strength test.
Figure 10: (Isomat 100) Saw Machine
Figure 11: (EcoMet® 250), grinding-polishing machine.
References