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EVALUATION OF BIAXIAL FLEXURAL STRENGTH AND SHEAR BOND STRENGTH OF A NOVEL CHAIR-SIDE CAD/CAM PRE-CRYSTALIZED ZIRCONIA REINFORCED LITHIUM SILICATE (IN-VITRO STUDY)

AbdulRahman A. AlShahalii BDs [a*], Sanaa H. Abel Kader BDs, MSc, PhD [b], Rewaa G. AboElhassan BDs, MSc, PhD [c].

ABSTRACT

BACKGROUND. Lithium disilicate has been the gold standard for esthetic restorations, but several materials have been introduced to improve the strength without compromising esthetics.

OBJECTIVE. The objective of this study was to compare between chair-side pre-crystalized zirconia reinforced lithium silicate containing virgilite crystals and lithium disilicate ceramic material (LDS) regarding biaxial flexural strength and shear bond strength.

MATERIALS AND METHODS. For BFS, 20 disc-shaped specimens, measuring 10 mm in diameter and 1.5 mm in thickness, were manufactured from both Tessera and LDS, with each group consisting of 10 specimens (n=10). BFS was evaluated using the piston-on-ring method on a universal testing machine. For SBS, 20 cuboid specimens (3x3mm and 5 mm thickness) were fabricated from Tessera and LDS (n=10). Specimens of both groups were bonded to natural human teeth with dual cured resin cement after appropriate surface treatment for both human natural teeth and specimen discs. SBS was assessed using a universal testing machine, and the testing was conducted at a crosshead speed of 1 mm/min.

RESULTS. The data obtained was statistically analyzed using appropriate tests. As for BFS, difference in results was statistically significant. Mean BFS value was higher for Tessera group (274.58±22.26) compared to the LDS group (243.31±24.98), however the difference in SBS values were not statistically significant between Tessera group (20.97±1.59), and LDS group (19.18±2.99). The independent t test was used as the test of significance among the groups.

CONCLUSION. Zirconia reinforced lithium silicate material performed better overall than the more time-consuming lithium disilicate ceramic.

KEYWORDS. Ceramics, Computer-Aided Design, CEREC, resin cements, lithium disilicate, zirconia, reinforced glass ceramics.

RUNNING TITTLE. Evaluation of a cad/cam pre-crystalized zirconia reinforced lithium silicate

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a- Graduate student, Division of Fixed Prosthodontics, Department of Conservative Dentistry, Faculty of Dentistry, Alexandria University, Egypt.

b- Professor of Fixed Prosthodontics, Department of Conservative Dentistry, Faculty of Dentistry, Alexandria University, Egypt.

c- Lecturer of Fixed Prosthodontics, Department of Conservative Dentistry, Faculty of Dentistry, Alexandria University, Egypt.

Corresponding Author:
abdulrahman.alshahalii.dent@alexu.edu.eg

INTRODUCTION

Lithium disilicate-based glass ceramics (LDS) have long been regarded as the gold standard for esthetic restorations in the field of restorative dentistry. This reputation is owed to their ability to combine esthetic qualities with sufficient strength, making them suitable for a wide range of applications. As a result, these materials have gained significant popularity among dental professionals.(1) Lithium disilicate based glass ceramics provide excellent esthetic properties but lack strength and even though zirconia-based restorations have high strength, one of the main disadvantages of zirconia is its poor bonding ability and opaqueness.(2)

To overcome these disadvantages of zirconia, many new materials, zirconia-reinforced lithium silicate (ZLS) were introduced in an attempt to increase strength while maintaining excellent aesthetics for fabrication of monolithic anterior and posterior restorations.(3) At the beginning, Celtra Duo was introduced, it contains 10% zirconia dissolved in the glass matrix, it improved the translucency and machinability of the material but did not add extra strength.(4, 5) Another modification of the material was the recently introduced CEREC® Tessera, the new composition of CEREC Tessera blocks made of two crystal types in addition to the 10% dissolved zirconia: lithium disilicate ($Li_2Si_2O_5$)

and virgilite (Li0.5Al0.5Si2.5O6), an LAS (Lithium Aluminum Silicate) type of crystal.(1, 6)
Moreover, during the heating process, minute virgilite crystals develop at a nano scale. These needle-shaped crystals, measuring roughly 0.5 µm in length, become embedded within a glass matrix enriched with zirconia. These components come together to produce a resilient, high-density restorative material. (1, 2, 4)

The manufacturer claims that the dense interwoven crystal composition of CEREC Tessera is the reason for their increased strength and that it eliminates microcracks and crack propagation. The null hypothesis for this study states that there will be no significant difference in either biaxial flexural strength or shear bond strength between the milled Tessera and e-max restorations.

MATERIALS AND METHODS

The sample size was determined based on an assumed 5% alpha error and 80% study power. The mean (SD) biaxial flexural strength of Lithium Silicate was 416.1(7) MPa and 502.46 MPa for Zirconia reinforced lithium silicate. (8) The mean (SD) shear bond strength of Lithium Silicate was 18.7 MPa (9) and 13.3 MPa for Zirconia reinforced lithium silicate. (9) The sample size calculation was based on the difference between independent means, and it was determined that a minimum of 9 specimens per group would be needed. To account for potential processing errors, the sample size was increased to 10 specimens per group. Therefore, the total sample size for the study was calculated as follows: 10 specimens per group multiplied by 4 groups, resulting in a total of 40 specimens.

BIAXIAL FLEXURAL STRENGTH

Ten disk-shaped specimens, each measuring 10 mm in diameter and 1.5 mm in thickness, were created for both CEREC Tessera (Group IB) and IPS e.max CAD (Group IIB). These specimens were designed using dental CEREC CAD software to meet the specified dimensions and were milled using the MCXL milling machine (Dentsply Sirona).

Following the milling process, the specimens underwent heat treatment for their final crystallization and glazing. This heat treatment was conducted in a furnace (Programat® - P300, Ivoclar Vivadent) in accordance with the manufacturer's instructions.

Starting Temperature of 400°c with 2 minutes closing time and 2 minutes preheating time then a heating rate of 55°/min till a final temp of 760°c and held for 2:00

The piston-on-ring test method was employed to assess the biaxial flexural strength of the specimens. This testing was performed using a Universal Testing Machine (Tinus Olsen, model 5ST). Each specimen was carefully positioned on a 10-mm diameter knife-edge and centrally loaded by a 5 mm diameter sphere indenter, with the test conducted at a crosshead speed of 1 mm per minute until fracture occurred (as shown in Figure 1). Throughout the testing process, all samples were oriented in a manner where the grounded side faced the direction of the applied load. To ensure an even distribution of the load, a small rubber sheet was inserted under the disc.(10, 11) The bi-axial flexural strength was calculated using the formula below:

$$\sigma \max = P/h2 \{(1+v) [0.485 \times \ln (a/h) +0.53]+0.48\}$$

In the provided equation:

σ max represents the maximum tensile stress

P is the calculated fracture load

'a' is the radius of the knife-edge support

'v' is the Poisson's ratio for the material (typically substituted with 0.25 for ceramics)

'h' represents the thickness of the disc and is measured using a digital caliper

'ln' stands for the natural logarithm. (12)

The data obtained was statistically analyzed using appropriate tests. Difference in results was statistically significant.

SHEAR BOND STRENGTH

Ten cuboid specimens (3x3mm and 5 mm thickness) were fabricated from both CEREC Tessera (Group IS) and IPS e.max CAD (Group IIS) and bonded to the tooth substrate with dual cure resin cement.

A total of 20 freshly extracted human maxillary premolars were randomly gathered for this study. These premolars were obtained from individuals who had undergone orthodontic treatment and had minimal to no caries or prior dental restorations. Upon extraction, the teeth were subjected to a disinfection process using chloramines-B-hydrate. Subsequently, they were stored in distilled water at room temperature from the day of extraction until they were utilized in the experiments. (13)

The teeth were affixed into auto-polymerizing acrylic resin blocks (Acrostone ,WHW plastic, Packed by Anglo Egyptian Lab) that extended up to the cementoenamel junction (CEJ). The specimens, now securely mounted, were randomly allocated into two distinct groups for the experimental procedure. In Group I, the specimens were designated for bonding with CEREC Tessera blocks (Dentsply Sirona). Group II specimens, on the other hand, were designated for bonding with IPS e.max CAD blocks (Ivoclar Vivadent). To ensure uniformity, the occlusal surfaces of the mounted specimens were reduced by 2 millimeters, creating a flat and parallel surface to the base of the mold. This reduction was accomplished with the use of a diamond disk

The tooth surface that had been prepared was subjected to etching using a 37% phosphoric acid solution (Meta Etchant by Meta Biomed) for a duration of 20 seconds. Following the etching process, the surface was meticulously rinsed and

dried with oil-free air spray. Subsequently, a bonding agent (All Bond Universal by BISCO) was administered using a microbrush onto the etched surfaces in two separate layers to ensure thorough penetration of the bonding agent. This was then light-polymerized for a period of 40 seconds, adhering to the instructions provided by the manufacturer

The inner surfaces of the ceramic specimens underwent surface treatment as per the manufacturer's guidelines. This involved the application of 9.5% hydrofluoric acid etching gel (Porcelain Etchant 9.5%, BISCO) for a duration of 30 seconds. Following this, thorough rinsing was carried out, and the surfaces were dried using oil-free air spray. Subsequently, a silane coupling agent (Porcelain Primer, BISCO) was administered using a microbrush on the ceramic-treated surfaces required for adhesive bonding. The silane was allowed to permeate for 60 seconds and then gently air-dried using oil-free air spray.

Ceramic blocks were bonded to human teeth using resin cement (Duo-Link Universal, BISCO) and a static loading device applied a force of approximately 2 kilograms. Subsequently, the specimens were subjected to light-polymerization for a duration of 40 seconds, in accordance with the manufacturer's recommendations.

The cemented specimens underwent shear testing until debonding using a universal testing machine (model 5ST, Tinus Olsen). The blade of the universal testing machine was positioned against the interface between the ceramic and the tooth. Shear force was incrementally applied to each specimen at a crosshead speed of 1 mm per minute until the point of failure was reached. The load, measured in kilograms, at which the ceramic specimen detached from the tooth surface, was recorded on a digital monitor, and reported in megapascals (MPa). The shear bond strength (SBS) was determined by dividing the fracture load (measured in kilograms) by the surface area, which was calculated as the square of the side length (3mm x 3mm). To express the SBS in megapascals (MPa), the obtained values were then multiplied by 0.09807.

The data obtained was statistically analyzed using appropriate tests. The difference in SBS values were not statistically significant between Tessera group (20.97±1.59), and LDS group (19.18±2.99), so the second part of the null hypothesis was accepted. The independent t test was used as the test of significance among the groups.

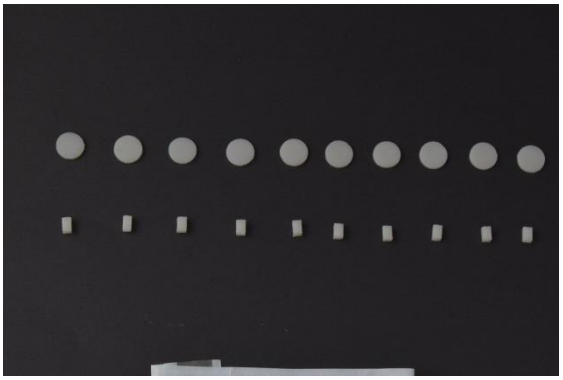

Figure 1: CEREC Tessera Disc (above) and IPS e.max Disc (Below) on Universal Testing Machine (Piston-on-ring test).

RESULTS

BIAXIAL FLEXURAL STRENGTH

Table 1 provides a summary of the mean, standard deviation, and confidence interval for the results of Biaxial Flexural Strength (BFS) in each study group, along with the associated P values.

The BFS values of Tessera and e.max specimens were calculated then statistically analyzed. On examining the BFS of both groups: the mean BFS values were higher in the Tessera (IB) group (274.58±22.26) than LDS (IIB) group (243.31±24.98).

The independent t test was used as the test of significance among both groups which showed the presence of a significant difference in the BFS mean values in favor of Tessera (Group IB).

All statistical tests were conducted with a two-tailed approach, and the predetermined significance level for the analysis was set at a p-value of less than or equal to 0.05. The data were subjected to analysis using IBM SPSS, version 23, based in Armonk, NY, USA.

SHEAR BOND STRENGTH

Table 2 presents a summary of the mean, standard deviation, and confidence interval for the results of Shear Bond Strength (SBS) in each study group, as well as the corresponding force values. Additionally, the table includes the associated P values for these results.

The SBS values of Tessera and e.max specimens were calculated then statistically analyzed. On examining the SBS of both groups: the difference in mean SBS values was statistically insignificant between the Tessera (IS) group (20.97±1.59), which was slightly higher, and the LDS (IIS) group (19.18±2.99).

The independent t test was used as the test of significance among the two groups which showed insignificant difference in the SBS mean values. All statistical tests were conducted with a two-tailed approach, and the predetermined significance level for the analysis was set at a p-value of less than or equal to 0.05. The data were

subjected to analysis using IBM SPSS, version 23, based in Armonk, NY, USA.

Table 1: Comparison of biaxial force between E-max and Tessera

	E-max (n=10)		Tessera (n=10)		Test (*p* value)
	Mean± SD	95% CI	Mean±SD	95% CI	
Force	407.96 ±41.88	378.00,437.92	456.59± 37.01	430.12, 483.07	2.752 (0.013*)
BFS	243.31 ±24.98	225.44,261.18	274.58± 22.26	258.66,290.50	2.955 (0.008*)

*Statistically significant difference at *p* value≤0.05

Table 2: Comparison of shear bond strength between E-max and Tessera

	E-max (n=10)		Tessera (n=10)		Test (*p* value)
	Mean± SD	95% CI	Mean± SD	95% CI	
Force	172.58 ±26.91	153.33, 191.83	188.76 ±14.32	178.52, 199.90	1.679 (0.116)
SBS	19.18± 2.99	17.04,21.31	20.97± 1.59	19.84,22.11	1.679(0.116)

*Statistically significant difference at *p* value≤0.05

DISCUSSION

The tests conducted in this study were chosen based on their significance for the clinical application of materials. Specifically, the biaxial flexural strength test was selected because it simulates the forces and conditions that dental restorations are subjected to in the oral cavity. This makes it highly relevant for evaluating a material's performance in a clinical setting thus providing a more relevant insight into clinical use compared to compressive strength. It provides a standardized and reproducible method for assessing material strength and offers valuable data for both material development and clinical decision-making in dentistry. Additionally, the shear bond strength test was included because the tested materials are often used in situations where bonding is crucial, such as inlays, onlays, and partial coverage restorations, it helps assessing how well a dental material adheres to the tooth structure, which is crucial for the longevity and success of the restoration. While milling IPS e.max blocks, the material hasn't yet achieved its maximum strength, measuring at approximately 130 MPa in terms of flexural strength. However, it is sufficiently robust for milling into the desired shape of the final restoration. Subsequently, once the milled restoration is subjected to a temperature of 850°C for a duration of 20 to 31 minutes, its strength significantly increases.(14)

As a result, the lithium metasilicate crystals will undergo a solid-state reaction with the surrounding silica, leading to the formation of compact, rod-like crystals of lithium disilicate, measuring approximately 1.5 µm in length, which interlock with one another.(14-16)

In the fully crystallized material, the final lithium disilicate phase ($Li_2Si_2O_5$) occupies as much as 70% of the total volume.(17) The remaining part of the volume consists of the glassy matrix and a small quantity of lithium orthophosphate (Li_3PO_4) crystals.(18)

CEREC Tessera is a novel material recognized for its exceptional esthetic qualities. According to the manufacturer's assertions, it exhibits remarkable strength, surpassing other glass ceramics by over 30% with a reported strength of 700 MPa. This enhanced strength is attributed to the inclusion of lithium disilicate crystals, approximately 0.5 µm in length, integrated into a glassy matrix. Additionally, the material features platelet-like lithium aluminosilicate crystals, known as virgilite, measuring between 0.2 and 0.3 µm.(6) The most favorable development of virgilite crystals takes place in the temperature range between 800°C and 850°C.

The densely interwoven crystal structure, along with the disparity in thermal expansion between virgilite crystals and lithium disilicate crystals, gives rise to residual stresses and microcracks during the cooling process. Surprisingly, these microcracks also serve as crack tip shielding, ultimately enhancing the material's toughness.(6, 19)

The manufacturer also claims high strength with fine margins, less chipping risk, and more conservative preparation. Also, Tessera has the advantage of Fast Firing: 4:30 in the SpeedFire Furnace. Up to 44% Faster total processing (Grinding + Firing) than leading glass ceramics, while also being able to be fired in normal furnaces in 9.5-12.5 minutes.

In contrast to IPS e.max CAD, which necessitates a crystallization process, CEREC Tessera, as per the manufacturer's guidelines, only requires an extra glaze firing (matrix firing) lasting 4.5 to 12 minutes at 760°C, and this can lead to an enhancement in the material's strength.(20)

The results in this study demonstrated superior flexural strength of CAD Tessera blocks (274.58±22.26) compared to IPS e.max CAD blocks (243.31±24.98). The difference was significant thus rejecting the first part of the null hypothesis. Wang et al found BFS values for LDS to be 248.5 MPa, close to those found in the present study.(21) It was found in the literature that BFS values for previous ZLS types were 261.5±31.89 MPa which also come close to the results in the present study.(22)

While the scientific literature has suggested that IPS e.max Press exhibits greater strength compared to IPS e.max CAD (7, 16, 21, 23-34), The primary aim of this study was to draw

a comparison between the CAD type and the Tessera material, noting that Tessera is exclusively accessible in the form of CEREC Blocs.

Drawing direct comparisons between the strength results of this study and those found in various literature sources proved challenging due to the wide variability in testing methods, specimen dimensions, and surface treatments. It's noteworthy that the flexural strength values we determined for lithium disilicate glass ceramics and zirconia-reinforced glass ceramics in this study are consistent with the range of values (typically falling between 251±30 MPa and 407±45 MPa) documented in the existing scientific literature. (7, 16, 21, 23-34)

Ensuring the successful adhesion of indirect restorations to tooth structure relies on the establishment of a reliable bond between the restorative material and the tooth structure, facilitated by the use of an effective luting agent.(9) In this study, we assessed the Shear Bond Strength (SBS) values of two distinct CAD/CAM restorative materials, both bonded using the same resin cement. Surprisingly, the results we obtained do not align with the research hypothesis.

This study followed the manufacturer's guidelines for surface treatment of both materials. The specimens of LDS (e.max) and ZLS (Tessera) were subjected to a 30-second etching process using 9.5% HF, followed by thorough rinsing, drying with oil-free air spray, and then the application of silane on the treated surfaces intended for adhesive bonding. The silane was allowed to soak for 60 seconds and was subsequently gently air-dried with oil-free air spray. The bonding to the tooth structure was achieved using dual-cure resin cement. It's worth noting that dual-cure resin cement is commonly used for partial coverage restorations, as opposed to self-cured or light-cured cement types, which have more specific applications in certain clinical scenarios. Although insignificant, there were differences between the SBS values of the LDS and ZLS CAD/CAM restorative materials. Secilmis et al. found that previous ZLS materials having lower shear bond strength than LDS (18.7 MPa for LDS and 13.3 MPa for ZLS)(9), In contrast to the findings of this study, the shear bond strength values of the specimens were lower for the LDS group. In some other studies, the difference was not that significant, Ataol et al stated found that LDS had a SBS of 10.8 MPa and previous types of ZLS had 10.5 MPa.(35) Kalavacharla et al. stated that The concentration and duration of exposure to hydrofluoric acid (HF), and to a lesser extent, the application of silane, play a substantial role in influencing the shear bond strength (SBS) between the tooth structure and CAD/CAM restorative material when using dual-cured self-adhesive resin cement, even when a universal bonding agent is employed.(36) Prior research has indicated that etching with 4.9% hydrofluoric acid (HF) for a period exceeding 90 seconds seconds(36, 37), or with 9% HF for 120 seconds(38) decreases the strength of the lithium disilicate. So, employing this etching method can offer time savings while maintaining the integrity of the lithium disilicate's strength.(36) To evaluate the adhesion between the CAD/CAM restorative material and the tooth structure, resin cement was directly administered onto the surfaces of the CAD/CAM restorative material. It's crucial to recognize that the outcomes may display some diversity because of the microstructural distinctions within the tooth structure.

CONCLUSIONS

Based on the findings of this in vitro study, the following conclusions were drawn:

1. ZLS restorations (CEREC Tessera) showed superior biaxial flexural strength compared with LDS (IPS e.max) of the same shade and translucency.
2. ZLS (CEREC Tessera) showed slightly higher shear bond strength with natural teeth using dual-cure resin cement than LDS (IPS e.max) but the difference was not statistically significant.

Statement of conflict of interest

The authors declare that they have no conflict of interest.

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