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Effect of Multiple Firing Cycles and Temperatures on Mechanical Properties of Lithium Silicate

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Aim: This study was designed to assess the effect of multiple firing cycles with different firing temperatures on the Flexural strength and Micro shear bond strength of the lithium silicate.
Materials and methods: Specimens of Lithium Silicate (obsidian) were divided into 2 groups according to the tests done. The first group, Obsidian blocks were sectioned into 36 bar-shaped samples for the three-point bending test, according to ISO 6872 standard. While the second group, the blocks were used to prepare 32 slices for measuring microshear bond strength. Each group was then divided into four subgroups according to the number of the firing cycles which were 1, 2, 3 and 5 firing cycles respectively. The flexural strength and microshear bond strength were measured separately.
Results: For the first group, the highest flexural strength value was found in control samples, while the lowest value was found in 3 cycles' samples. As for the second group, the highest micro shear bond strength value was found in samples subjected to 3 cycles, while the lowest value was found in 2 cycles' samples. This shows that there was no significant difference between values of each group.
Conclusion: Obsidian can be fired up to 5 firing cycles with no adverse effect on bond strength or flexural strength.

Keywords: Bond strength, Flexural strength, Firings, Obsidian, Lithium silicate.

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Introduction

Dental ceramics in the previous decades have gone through huge revolution. In order to meet the increasing demand of patients and dentists for a greatly esthetic, biocompatible and long lasting restorations, numerous forms of all ceramic restorations have been established.

Early mechanical failure of ceramics was almost entirely attributed to brittle fracture that in combination with surface flaws led to low ceramic strength. In order to increase flexural strength of ceramics, the crystalline filler content was increased within the glass matrix with even distribution of particles and finer size which had a major improving effect. Techniques applied to overcome the brittleness of porcelain fall into two categories: strengthening of ceramic and modifying of component.[1]

Glass ceramics containing lithium silicate had been presented in the dental market in the former few years. Glass ceramics are made of glass and crystals and according to their ratios; their esthetic and physical properties are detected.[2]

In the previous few years, a glass ceramic made of lithium silicate was introduced known as Obsidian. Obsidian is a lithium silicate glass ceramic manufactured combining around twenty unique elemental oxides that were selected from the periodic table (including Zirconia). Furthermore, the Obsidian Blocks have its remarkable properties due to a great content of ultra-nanometer-size lithium silicate and lithium phosphate crystals. This may be used to formulate numerous monolithic restorations with enhanced esthetics, natural translucency, and high strength to attain full-contour crowns, posterior crowns, anterior crowns , inlays, onlays, and veneers and partial crowns.[3]

So as to attain better color, contour and esthetics, multiple firing in procedures are needed for construction of all-ceramic restorations, particularly when a standard layering technique is to be used in order to simulate the esthetics of natural teeth. Multiple firing might affect the esthetics along with the mechanical properties of all ceramic restorative material.

Yet, there is still no sufficient reports regarding the assessment of the mechanical properties of lithium silicate based ceramics, and how they are affected by the change of temperatures during the different firing cycles. Therefore, this study will be conducted to evaluate differences that might occur in their bond strength, as well as their flexural strength in relation to alteration of temperatures.

Materials and methods

Specimens made of Lithium Silicate (obsidian) blocks were divided into 2 groups according to the measurements done.

We named the samples for the flexural strength testing **group F** and the samples for the microshear bond strength testing **group S.**

For Group F, the Obsidian Lithium silicate blocks were sectioned into 36 bar-shaped samples with the following dimensions 14mm *2mm* 2mm for three point bending test for measuring flexural strength, according to the ISO 6872 standard using using IsoMet 5000 microsaw with cutting speed 2500 rpm.

While for Group S the blocks were used to prepare 32 slices with the following dimensions: 14mm x 12mm x 1mm Using IsoMet 4000 micro saw with cooling water system, by a dimond disk 0.6 mm thickness with cutting speed 2500 rpm.

Each Obsidian cut specimen was manually polished by using silicon carbide abrasives mounted on low speed hand piece and then checked using digital calliper and any defective specimen was discarded.

All Obsidian samples were crystallized according to manufacture recommendations in ceramic furnace, manually polished and each group was divided into four subgroups of according to the number of the firing cycles which were 1, 2, 3 and 5 fire cycles respectively. First subgroups were crystalized at 820°C as shown in table (1), second subgroups were subjected to 2 different firing cycles simulating Crystallization (820°C), and Stain and glaze Temperature (800°C) using the same furnace, while the third subgroups received three firing cycles using the same furnace which are the crystallization cycle (820°C), a second cycle for stain and glaze (800°C) and a third add-on cycle (760°C). The fourth Subgroups received five firing cycles using the same furnace which are the crystallization cycle (820°C), stain and glaze cycle (800°C) and three add-on cycles (760°C).

Table 1: Obsidian crystallization parameters according to manufacturer manual

Crystallization Heating Cycle for Obsidian® Milling Blocks		
Ivoclar Programat® CS2, EP 5010, EP 5000, P300 or similar		
Vacuum Quality	72 mbar	N/A
Standby Temperature / Closing Time S	400°C	3 minutes
Heating Rate t_1 ↗	90°C/min	N/A
Holding Temperature T_1 / Holding Time H_1	780°C	10 seconds
Heating Rate t_2 ↗	40°C/min	N/A
Holding Temperature T_2 / Holding Time H_2	820°C	10 minutes
Cooling Rate ↘ t_L	50°C/min	N/A
Long-term Cooling L	680°C	N/A
Vacuum 1 Level $V1_1$ (on) / $V2_1$ (off)	400°C	780°C
Vacuum 2 Level $V1_2$ (on) / $V2_2$ (off)	780°C	819°C

For **group F**, three-point bending tests were done on the samples using ElectroPuls E3000, Instron, USA; a universal testing machine with a distance between the supporting rollers of 12 mm. It was then uniaxially loaded and the loading was elevated steadily with a 1 mm/min crosshead speed until fracture happened.

The study then calculated the flexural strength according to the following formula:

$\sigma = 3FL/ 2wt^2$

There "F" is load at fracture point; "L" is span length of supports; "w" is the width of specimen, "t" – thickness of specimen, "l" – length of specimen and "F" – load at fracture point.

For **group S**, For easier handling and fixation during the micro shear test, the whole group of 32 slices of Obsidian Lithium Silicate material were embedded in an acrylic blocks. Obsidian rectangular slices were placed inside the polyvinyl chloride (PVC) pipes and then resin was poured resulting in specimens embedded in acrylic blocks and each acrylic block was given a number according to the number of cycles done. They were etched using IPS ceramic hydrofluoric etching gel 5% according to the specified etching duration by the manufacturer for 10 seconds and then they were washed with an air water spray for 30 seconds and were air-dried with the air/water syringe. Silane coupling agent was then applied for 60 seconds and then they were air dried.

Each ceramic specimen received 5 resin micro cylinders. Irises of polyethylene tube having 1mm diameter and 1mm height were positioned over the disc surface, then cement was injected into the tubes through the mixing tip, light curing was done through the tube for 40 seconds.

Polyethylene tube irises were not removed in order not to subject the resin micro cylinders to shear stress at the interface and to eliminate any cause of failures.

At the end Micro-shear bond strength test was executed on all 160 micro-tubes.

For measuring the Micro-shear bond strength, the specimen containing the bonded micro-cylinders was secured horizontally and using the tightening screws it was fixed to the lower fixed compartment of the universal testing machine. A loadcell of 5 kN was used and using computer software the data was recorded.

An orthodontic wire was used to make a loop that was enfolded around the bonded micro-cylinder assembly and it was allied with the loading axis of the upper movable compartment of the testing machine.

Shear load with tensile mode of force at a crosshead speed of 0.5 mm/min was applied by the machine. The load required for debonding was documented in Newtons.

Micro-Shear Bond Strength Calculation;

Bond strength was obtained by dividing the load at failure by bonding area in MPa:

$\tau = P/ \pi r2$

Where; τ = µ-shear bond strength (in MPa), P =load at failure (in N), π =3.14 and r = radius of micro-cylinder (in mm).

The numerical data were checked for normality by checking the data distribution using Shapiro-Wilk tests. The data presented parametric distribution, so they were represented by mean and standard deviation (SD) values. Intergroup comparisons were done using One-way ANOVA followed by Tukey's post hoc test. Correlation between shear and flexural bond strength was analysed using Spearman rank order correlation coefficient. Within all the tests the significance level was fixed at $p \leq 0.05$. Statistical analysis was achieved with IBM® SPSS® Statistics Version 26 for Windows.

Results

No significant difference was found between the values of different groups (p=0.166). Control samples showed the highest flexural strength (MPa) value (213.38±15.19), followed by samples subjected to 5 cycles (193.94±33.64), then samples subjected to 2 cycles (190.83±16.21), while the 3 cycles samples showed the lowest value(189.53±15.86).

For group S, No significant difference was found between vales of different groups (p=0.363). Samples subjected to 3 cycles showed the highest micro shear bond strength (MPa) value (33.13±5.96), followed by control samples (31.29±4.16), then samples subjected to 5 cycles (30.95±4.47), while the 2 cycles samples had the lowest value (29.49±3.03).

Table 2 : Mean ± standard deviation (SD) of flexural strength (MPa) for different groups

Flexural bond strength (MPa) (mean±SD)				p-value
Control	2 cycles	3 cycles	5 cycles	
213.38±15.19	190.83±16.21	189.53±15.86	193.94±33.64	**0.166ns**

Means with different superscript letters are statistically significantly different *; significant ($p \leq 0.05$) ns; non-significant ($p>0.05$)

Table 3 : Mean ± standard deviation (SD) of shear bond strength (MPa) for different groups

Shear bond strength (MPa) (mean±SD)				p-value
Control	2 cycles	3 cycles	5 cycles	
31.29 ± 4.16^{A}	29.49 ± 3.03^{A}	33.13 ± 5.96^{A}	30.95 ± 4.47^{A}	**0.363ns**

Means with different superscript letters are statistically significantly different *; significant ($p \leq 0.05$) ns; non-significant ($p>0.05$)

Discussion

Lithium silicate glass-ceramic Obsidian Milling Blocks (Glidewell Laboratories, Newport Beach, USA) were introduced for posterior and anterior uses. It is characterized by a composition of an exceptional mixture of elemental oxides and a great content of ultrafine nanometre-size lithium silicate crystals. It is promoted to have a high survival rate against chipping and fracture after three years in use, which is comparable to other glass-ceramics.[4] They also offer a combination of adequate translucency and improved mechanical properties.[5]

To reach the final crystallization, glass-ceramic restorations must be fired before or after CAD/CAM milling. The composition and microstructure of the final product are determined by the nucleation and crystallization process that happens during this treatment which consecutively define their mechanical and their optical properties.[4] There is not any data on the exact number of firing cycles to attain the perfect ceramic restoration, yet, multiple firing procedures are necessary to attain better colour, contour and esthetics. Thus, an essential point is understanding the effect of these multiple firings on the ceramic material and obtaining the minimum number of firing cycles necessary to get restorations with proper performance.[6] The extra firings are mostly done for layering and staining of the restoration or if the dentist needed correction for shape and color.[7]

This in vitro study aimed to evaluate differences in the bond strength, as well as the flexural strength in relation to the change of temperatures after multiple corrections and firings were done to lithium silicate (Obsidian) samples.

Ceramics are characteristically known as brittle materials, which are likely to break under unavoidable bending chewing forces. In the intraoral conditions, restorations should have strength enough to endure regular masticatory force. Flexural strength generally denotes the capacity to bear the chewing forces.[8]

The three-point-bend test is a relatively simple method of producing consistent and repeatable relative strength values for dental ceramics.[9]

CAD/CAM materials are commonly provided in blocks, and the preparation of the rectangular-section bars necessary for 3PBT and 4PBT is simplified in comparison to the one required for BFT, where a disc has to be produced. [10]

Therefore in this study ; Obsidian milling blocks were sectioned into bar shaped samples of group F from blocks (lithium silicate obsidian milling blocks, Glidewell Dental, USA) were set for the three point bending test, according to the ISO 6872 (2015)[11] standards for ceramic testing.

Results for the flexural strength in this study stated that there was no significant difference between vales of different groups. The highest flexural strength (MPa) value was found in control samples, followed by samples subjected to 5 cycles, then samples subjected to 2 cycles, while the lowest value was found in 3 cycles' samples. Hence, we fail to reject the second null hypothesis stating that multiple firing cycles would not affect the flexural strength of lithium silicate glass ceramic. These results were in coinciding with Alper Ozdogan et al (2021)[12] who studied the frequent firing effects on the mechanical properties of lithium disilicate glass-ceramics. The study stated that both the hardness and the fracture toughness of the materials were affected by the several firings. However, their flexural strength wasn't affected and the difference in the results was not statistically significant (P>.05).

According also to Miranda et al (2020)[13] their study was consistent with the present study. They stated that the numerous firings did not affect the flexural strength of IPS Emax CAD ceramics. The flexural strength values for Lithium disilicate glass ceramics have been informed to be between 300 and 500 MPa. This range was attributed to diverse material thicknesses and the testing methods that were used. The number of firing cycles did not impact the chemical configuration of the used specimens for any of the groups that were involved.

Also Oh et al (2000)[14] experimented the changes that happen after several firing conditions in the flexural strength of lithium disilicate reinforced Empress 2 all-ceramic material. They concluded that after the seventh firing, the values of the flexural strength declined slightly. Yet, this decline was still not statistically significant.

In co-ordinance with results found in our study, R. Gozneli et al (2014)[7] noted the changes that happens in the biaxial flexural strength of some pressable all-ceramic materials after being exposed to repeated firings. The biaxial flexural strength wasn't affected. However, at the seventh firing, the strength values were dropped, yet the difference still wasn't statistically significant.

In addition, a study by Tang el al (2012)[15] studied how numerous firings affect the mechanical properties and the microstructure of veneering ceramics that are used with zirconia frameworks. The study showed that for all materials that were used, they noted that there was a significant difference in density and porosity between 2 and 10 firings. However, they found no significant differences shown on the flexural strength.

The present study reflects the limitations associated with in-vitro studies. The 3-point bending test used still does not represent the real flexural strength in the clinical condition owing to various environmental and loading circumstances. However, in-vitro studies are important to throw the light before conducting in-vivo clinical studies. In addition, the study was accomplished under dry and static environment, so it is still not strictly simulating the overall wet and cyclic nature of the oral environment.

The three point bending performed in the present study has the limitation of a monotonic test, in which the load is applied until specimen failure. This is not completely representative of the clinical situation in which the restoration is subjected to cyclical load and thermal variations. The test method is a very essential parameter for brittle materials and it has been established that a change in the test method can result in significantly different flexural strength values. Since no systematic reviews on flexural strength are currently available, in the present study the standard ISO 6872:2008 [11]was strictly followed, for specimen preparation and storage, for test apparatus set-up, and for expression of the results.

This study also assessed the Shear Bond strength of resin bonding of lithium silicate glass-ceramics that was subjected to several numbers of firings when HF etching and silanization were implemented.

In this study to test for bond strength using micro shear bonding strength test due to the fact that Micro-shear bond strength test is a reasonably simple test that allows proficient studying of the adhesive protocols, regional and depth profiling of a diversity of substrates.[16]

In this study; etching was done with 5% HF acid concentration according to manufacturer instructions. Silane primer was then applied afterwards for 60 seconds then gently air dried which coincide with SE Elsaka (2014).[17]

Most micro-shear studies use polyethylene tubes as a mould, which are later filled with a resin composite. [18] In the current study, the polyethylene tubes irises were not detached by a blade in order not to subject the self-adhesive resin cement micro cylinders to shear stresses at the interface and to eradicate any pre-test failures according to Andrade et al. in 2012.[16]

Results for the microshear bond strength in this study stated that there was no significant difference between values of different groups. The highest shear bond strength (MPa) value was found in samples subjected to 3 cycles, followed by control samples, then samples subjected to 5 cycles, while the lowest value was found in 2 cycles' samples. The results allow the acceptance of the tested null hypothesis since the multiple firings had no significant impact on the bonding with the luting agent.

In co ordinance with H. Meng et al (2018) [19] whom their study was evaluating the micro shear bond strength. They evaluated how different firing cycles affect the bonding strength to resin. They also studied how it affects the surface hardness, the fracture toughness as well as the roughness of lithium disilicate glass-ceramic. Samples were subjected to 0ne to four firing cycles. No significant effect on the shear bond strength was noted after repeated firings and 3-mo water storage. However, it affected partially the mechanical properties. Based on the surface roughness test results in this study, they concluded that the repeated firings had no significant effect on the surface roughness, resulting in related micromechanical retention between the ceramic and resin.

This finding is also agrees with a study that Carla kassis et al (2021)[20] made. It concluded that the intrinsic staining did not affect the shear bond strength between the lithium disilicate ceramics and resin cement. The study noted that the several heating temperatures and the acid etching altered the topography of the ceramic material. The results were influenced as Oxygen and silicon were the most existing chemical components in the intrinsically stained surface, thus proposing abundance of Silicon dioxide (SiO_2) that favours shear bond strength. Thus, no significant impact was found on the shear bond strength after a second firing cycle was done after the lithium disilicate‘s crystallization.

Conclusions

Within the limitation of this in Vitro study, it was concluded that:

1. Multiple firings did not affect the flexural strength of lithium silicate glass ceramics.
2. Multiple firings did not affect microshear bond strength of lithium silicate based glass ceramics.

Recommendation

Obsidian can be fired up to 5 times safely while more investigations should be conducted as the effect of repeated firing beyond 5 cycles is not known. Further research should be proposed especially in vivo studies are further needed to reach a decisive conclusion concerning the strength of the materials that were examined; also as this study was executed under dry and static circumstances, therefore it was not meticulously simulating the overall wet and cyclic nature of the oral cavity. Forthcoming studies should also assess the effects of the thermal procedures on the surface roughness, the microstructure, and the thermal alterations of the constituents.

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