EVALUATION OF ZIRCONIA-REINFORCED LITHIUM SILICATE CERAMIC SURFACE TREATMENT ON THEIR SHEAR BOND STRENGTH TO DENTINE FOLLOWING IMMEDIATE DENTIN SEALING

Mahmoud Abdel Salam Shakal*

ABSTRACT

Objectives: The purpose of this study was to evaluate zirconia-reinforced lithium silicate ceramic surface treatment on their shear bond strength to dentine following immediate dentin sealing.

Methods: One hundred sixty extracted molars were initially divided into two main groups each counting eighty molars: based on dentine sealing protocols (without immediate dentin sealing S- and with immediate dentin sealing S+), those groups were further subdivided into four subgroups each counting twenty based on the ceramic surface treatment protocol. Each were further subdivided into two subdivisions to evaluate the bonding durability after thermodynamic aging process each counting ten specimens. All restorations were adhesively bonded using a light curing resin cement. The thermo-mechanical group were subjected to 10,000 thermal cycles between (5-55ºC) with a 30-s dwell time, 20 seconds transfer time. specimens were then subjected to maximum vertical load of 10 kg with cyclic frequency of 1.7 Hz for 240,000 cycles, which corresponds to one year of clinical service. Specimens were tested for shear bond strength using Instron testing machine. Data were statistically analyzed using two-way ANOVA and Tukey’s post-hoc tests.

Result: A significantly higher shear bond strength of restorations (p ≤ 0.001) was obtained when immediate dentin sealing was followed regardless of the surface treatments of ceramics with a maximum value of 10.50±0.412 MPa. Both ceramic surface treatment and artificial aging had a statistically significant effect on the shear bond strength.

Conclusion: Immediate dentin sealing protocol is recommended and HF surface treatment is recommended for ceramic restorations adhesion to achieve better durability.

KEYWORDS: Immediate dentine sealing, Hydrofluoric acid, Cojet system, Thermodynamic stressing

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INTRODUCTION

Over years, glass ceramics have been evolved in their compositions and processing techniques. Crystal phase was the main structure of Leucite or lithium disilicate of the first generation of these materials and presented as injectable ingots. Lithium disilicate–based glass ceramics are now available in computer-aided design and computer-aided manufacturing (CAD-CAM) blocks and satisfactorily served as monolithic restorations. Zirconia-reinforced lithium silicate ceramic (ZLS), was launched recently under the argument that zirconia could act as a crystal phase that can reinforce the material to avoid crack propagation. Ceramic surface treatment is performed to improve the adhesion procedure through, creating surface irregularities via mechanical or chemical treatments, such as roughening with diamond burs, etching with hydrofluoric acid and sandblasting are the surface treatments recommended for ceramics. Acid etching increases the surface area and the bonding potential of ceramic to resin through increasing ceramic wettability via changing their surface energy. Ceramic surface abrasion with aluminum oxide particles produces irregularities in acid-resistant ceramics. However, In-Ceram ceramics air abrasion has presented effective initial but unstable bonding, since it presented failure subjecting the specimens to storage and thermocycling for longer periods in water. This may be due to the fact that this treatment creates surface irregularities without micromechanical retention.

Ceramic surface treatment with silica as reported by some authors may jeopardize the material mechanical strength as a result of crack propagation, while others has indicated no deleterious effect on long-term mechanical behavior. There are no data in the literature on the effects of silica coating on lithium silicate reinforced by zirconia ceramic. Silica coating can be particularly important if the zirconia content present in in ZLS makes it less sensitive to acid conditioning.

During the standard dentin bonding procedure, dentin bonding agent is applied when laminate is seated on the prepared tooth during cementation. Incomplete seating of the restoration can be avoided by maintaining the adhesive resin unpolymerized before laminate veneer is seated. This is attributed to the fact that the polymerized dentin adhesive thickness varies from 60 - 80 μm to 200 - 300 μm depending on the structure of tooth surfaces, although less than 40 μm thickness is recommended before setting of restoration. Additionally, the oxygen inhibition layer which plays an important role in bonding with resin reaches up to 40 μm in thickness, making dentin adhesive excessively thin that might weaken the bond strength between bonding agent and resin. This thick film could interfere with the proper seating of the restoration if the dentine bonding agent is light-cured before seating of laminate veneer. However, it is also reported that curing dentin adhesive and resin cement individually resulted in greater bonding strength values than curing both in one step. This comes from the fact that unpolymerized dentin-resin hybrid layer collapses during the placement of restoration.

Aim of the work

The purpose of this study was to evaluate zirconia-reinforced lithium silicate ceramic surface treatment on their shear bond strength to dentine following immediate dentin sealing.
MATERIALS AND METHODS

Material

TABLE (1) Material used in this study:

<table>
<thead>
<tr>
<th>Materials</th>
<th>Type</th>
<th>Composition</th>
<th>Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>CAD/CAM Restorative Block Materials</td>
<td>Vita Suprinity</td>
<td>ZrO₂ , SiO₂ , Li₂O</td>
<td>Vita, Germany</td>
</tr>
<tr>
<td>Total Etch</td>
<td>Etching gel</td>
<td>37% phosphoric acid</td>
<td>Ardent, Arlandastad</td>
</tr>
<tr>
<td>Adhesive</td>
<td>Light cure all-bond universal</td>
<td>MDP, bis-GMA, HEMA, ethanol, water, initiators</td>
<td>BISCO,USA</td>
</tr>
<tr>
<td>Ceramic Etching Gel</td>
<td>Etching gel for dental ceramic</td>
<td>9% Hydrofluoric acid</td>
<td>Dentopond, France</td>
</tr>
<tr>
<td>Ceramic bonding agent</td>
<td>ESPE SIl</td>
<td>Pre-hydrolyed silane coupling agent, alcohol, water</td>
<td>3M ESPE,USA</td>
</tr>
<tr>
<td>silica coating</td>
<td>Cojet system</td>
<td>30-µmsilica-modified Al₂O₃ particles</td>
<td>3M ESPE,USA</td>
</tr>
<tr>
<td>Resin cement</td>
<td>Choice 2 veneer cement</td>
<td>BisGMA, Strontium glass, Amorphous Silica</td>
<td>BISCO</td>
</tr>
</tbody>
</table>

Methods

A total number of one hundred sixty sound extracted molars were collected and stored in thymol solution at room temperature for specimen’s preparation for two weeks. Teeth were then fixed along their long axis in custom made acrylic resin blocks. Flat mid coronal dentin surfaces (Fig.1) were created after removal of the occlusal half of the crown using low speed diamond discs under copious water cooling. Specimens were randomly divided according to their initial treatment procedure into two main groups each counting 80 (N=80). In the first main group (S-) neither the immediate dentin sealing protocol was applied nor the dentin was treated after preparation, while in the second main group (S+) an immediate dentin sealing protocol was applied using total etching protocol where the freshly cut and uncontaminated prepared enamel and dentine surface were treated with phosphoric acid 37%. Enamel was etched for 30 s while dentine was etched for 15 s. Then etchant was rinsed thoroughly with vigorous water spray for at least 15 s and then dried with air spray until the surfaces appeared chalky white. Bonding agent (All-bond universal, BISCO, Schaumburg, USA) was applied to the surfaces with gentle brushing motion by micro brush for at least 20 s and left to diffuse and then cured at first for 20 s.

The ZLS ceramic blocks (Vita Suprinity, Vita Zahnfabrik, Germany) were cut into disc (6×2mm) and crystallized according to the manufacturer’s instructions. The hundred and sixty discs were divided in to 4 groups each counting (40 discs)
according to surface treatment method as mentioned in (Table. 2), where group (N) did not received surface treatment, group (SB) was treated by airborne-particle abrasion using 50 micron Al₂O₃ particles at 4 bar pressure for 10 seconds from a distance of 10 mm perpendicular to the surface of the specimen, while (CS) group were coated with silica using CoJet system (Co Jet Sand, 3M ESPE) for 25 seconds at 2.5 bar and 15-mm distance at right angle to the treated surface. The (HF) group was treated using Hydrofluoric acid surface conditioning with 9% hydrofluoric acid for 60 seconds.

After following the previously mentioned protocol for ZLS ceramic discs surface treatment. The treated ceramic discs testing surface were salinized using (ESPE SIl, 3M ESPE, USA) then left 30 seconds to dry and cemented with light cured resin cement (Choice 2 veneer cement BISCO) (Fig. 2).

All hundred and sixty specimens were stored in distilled water for 24 hours at 37°C, and only eighty specimens were subjected to thermocycling for 10,000 cycles (5-55°C) with a 30-s dwell time, 20 seconds transfer time. This is corresponding to one year of clinical service. The thermocycled specimens were subjected to maximum vertical load of 10 kg with cyclic frequency of 1.7 Hz for 240,000 cycles, which corresponds to one year of clinical service (11). Load was applied occlusally with a custom-made load applicator [steel rod with flat tip (20x25mm) attached to the upper movable compartment of the machine.

### Measurement of shear bonding strength

Shear bond strength of the ZLS ceramic to dentine was measured using universal testing machine by applying load vertically at the adhesive bonding joint between ceramic and dentine at cross-head speed of 0.5 mm/min (Fig. 3). Failure load for the specimen were recorded and were transferred into stress unit (MPa). Data were analyzed using two-way analysis of variance (ANOVA) with Tukey’s post hoc test to determine statistical significance between each group.

**TABLE (2) Specimens grouping and distribution**

<table>
<thead>
<tr>
<th>Without immediate dentin sealing S- (N=80)</th>
<th>Immediate dentin sealing/ Total etching S+ (N=80)</th>
</tr>
</thead>
<tbody>
<tr>
<td>N (n=20)</td>
<td>SB (n=20)</td>
</tr>
<tr>
<td>With artificial aging</td>
<td>No artificial aging</td>
</tr>
<tr>
<td>(n = 10)</td>
<td>(n = 10)</td>
</tr>
</tbody>
</table>

Fig. (2)  
Fig. (3)
RESULTS

The mean values of shear bond strength and the statistical groupings are shown in Table 3 and graphically represented in Fig 4. The shear bonding values recorded with the S+ group were significantly greater than those recorded for the S- group. The highest significant mean shear bond strength within the S- group was recorded for CS group with those specimens without artificial aging at the level of 9.90±0.671MPa, while the highest significant mean shear bonding strength was recorded for (HF) group of specimens in S- group with artificial aging was at the level of 8.00±0.795 MPa. While in S+ group of specimens, the highest significant mean shear bonding with the non-artificial aging group was also recorded for the (CS) group at the level of 10.50±0.412MPa, whereas the highest significant mean shear bonding strength recorded in S+ with artificial aging was recorded for the (HF) group at the level of 8.82±0.389MPa.

Based on Tukey’s post hoc significance analysis test, significance differences were observed between the S+ group compared to the S- group (p <0.001). Significance differences were found between data obtained from different surface treatments of N, HF, SB and CS regardless aging, whereas After aging CS group revealed significantly damaged in adhesion, while the HF group should no significant drop of bonding values even after artificial aging.

TABLE (3) Shear bond strength mean values in MPa

<table>
<thead>
<tr>
<th>Groups</th>
<th>S- With artificial aging</th>
<th>No artificial aging</th>
<th>S+ With artificial aging</th>
<th>No artificial aging</th>
</tr>
</thead>
<tbody>
<tr>
<td>N</td>
<td>4.44±0.527</td>
<td>6.00±0.791</td>
<td>4.88±0.544</td>
<td>7.52±0.37</td>
</tr>
<tr>
<td>HF</td>
<td>8.00±0.795</td>
<td>8.140±0.444</td>
<td>8.82±0.389</td>
<td>8.86±0.384</td>
</tr>
<tr>
<td>SB</td>
<td>7.50±0.786</td>
<td>9.42±0.563</td>
<td>8.00±0.316</td>
<td>10.06±0.44</td>
</tr>
<tr>
<td>CS</td>
<td>7.620±0.492</td>
<td>9.90±0.671</td>
<td>8.10±0.223</td>
<td>10.50±0.412</td>
</tr>
<tr>
<td>F</td>
<td></td>
<td></td>
<td>51.565</td>
<td></td>
</tr>
<tr>
<td>Sig</td>
<td></td>
<td></td>
<td>&lt;0.001**</td>
<td></td>
</tr>
</tbody>
</table>

Fig. (4) Shear bond strength mean values in MPa
DISCUSSION

The current study has evaluated the impact of ZLS different ceramic surface treatments and thermomechanical loading on its shear bond strength to resin cement on dentin surface following immediate dentine sealing protocol. Bonding values achieved with the three surface treatments tested (HF, SB, and CJ) were of similar baseline values.

The results of this study showed that the immediate dentin sealing exhibited significantly higher shear bond strength than non-sealing protocol, with significant difference between the shear bond strength recorded for all surface treatment protocols with or without thermodynamic, that goes in line of the findings of Choi Y-S etal who reported the improved the marginal adaptation between the restoration and the dentin following immediate dentin sealing (12), that could be interpreted by increasing the amount of fillers penetrating collagen fibers to stabilize the dentin-resin hybrid layer that has enhancing impact factor on bonding strength of dentin bonding agent(13-16).

The results of the current study showed high initial bonding values for most of all groups regardless the surface treatment technique and recording the highest values for the CS (Silica-coating group) compared to Sandblasting and hydrofluoric acid etching, that goes in line with the findings of both with Özcan, Vallittu(17) who concluded that mean shear bond strength values for the IZ ceramic treated with hydrofluoric acid, which was lower than the mean values for the groups blasted with aluminum oxide particles and the group treated with the Rocatec system (silica-coating). The higher bond strength values attained with silica coating (CS) is attributed to the dual (chemical and mechanical) adhesion mechanism that improve the bonding to composite resin, through surface roughness created by air abrasion providing a larger surface area for micromechanical retention, and improved chemical bond since the silica coated surface promotes better bonding with silane and resin adhesives (18,19).

Shear bonding values for most of all different ZLS surface treatment has been deteriorated significantly after thermo-mechanical loading the highest drop in the bonding values attained regardless following the immediate dentine sealing protocol was recorded for no surface treatment, (CS) group (silica coating) and sandblasting group while no apparent significant effect was observed with HF group, that could be explained by inadequacy of surface micro retention created by sandblasting (20). While the durability and stability of (HF) group could be related to the uniformity of surface activation as reported by Menees et al(21).

The small irregularities caused by silica coating as reported Della bone et al (22) and dimited ceramic roughness as stated by Kern and Wegner(23) and the induction of excessive gaps in the material surface as reported by Sato T et al (24) goes in line with the findings of our current study and explains the drop in the bonding values recorded after aging through thermodynamic stressing.

CONCLUSION

Within the limitation of this in vitro study
1. Immediate dentin sealing protocol is recommended
2. Ceramic surface prior bonding is essential for bonding initial and long-term stability
3. Ceramic surface treatment using Hydrofluoric acid has produced a stable and durable bonding in combination with immediate dentine protocol.

REFERENCES


