INFLUENCE OF DIFFERENT DUAL CURED RESIN LUTING AGENTS ON FRACTURE RESISTANCE AND SHEAR BOND STRENGTH OF LITHIUM DISILICATE OCCLUSAL VENEERS

Hala M. Abdel Moaty 1*, MSc, Ahmed S. Elkadi 2 PhD, Amal E. Fahmy 3 PhD

ABSTRACT

INTRODUCTION: Occlusal veneer are considered promising solutions for coronal tooth structure loss. Pressed lithium disilicate is the first choice indirect restorative material. Resin cements are the weak point in this biomimetic solution.

OBJECTIVES: Investigate the fracture resistance and shear bond strength of lithium di-silicate occlusal veneer using different dual cured resin cements.

MATERIALS AND METHODS: 28 sound extracted human permanent molars were used. They were divided into 2 groups (n=14) according to 2 different tooth preparation. For group A the occlusal surface was anatomically reduced for fracture resistance test, while for group B the occlusal surface was flattened for shear bond strength test. Each group was divided into subgroups (n=7) according to resin cement system type used. For group A1 and B1, Panavia F 2.0 self-etch resin cement. While for group A2, B2 Variolink Ethetics total etch resin cement was used. Lithium disilicate veneers (1mm) and discs (2×4 mm) were cemented according to manufacturer's instructions. Bonded samples were tested for fracture resistance and shear bond strength after thermocycling. Failure mode was determined. The data were statistically analyzed (p≤0.05).

RESULTS: Type of resin cement had a statistically significant difference on the fracture resistance and shear bond strength with P ≤ 0.05. Group (A2) Total etch Variolink Ethetic resin cement provided higher fracture strength (2259.5 N). Also, Group (B2) provided higher bond strength (34.36 MPa).

CONCLUSION: The lithium disilicate occlusal veneers are preferred using total etch adhesive to get the best bond strength and fracture resistance sufficient to withstand loading in molar region.

KEYWORDS: Fracture resistance, Shear bond, Resin cement, Occlusal veneers.
of indirect restorations is cementation. Dual-cure resin cements cured by means of both chemicals and light. They include self-cure initiators that can cure the cement and curing light activate the photoinitiator (8).

Long term success of the occlusal veneer is influenced by different factors, including tooth surface substrate, preparation depth and design, type and thickness of the restoration and its surface treatment. The resin cement and dental adhesive type, tooth morphology, functional and abnormal behaviors are also important parameters affecting longevity of occlusal veneers (9).

Although clinical trials are the most appropriate tools to evaluate the effectiveness of the adhesive systems, long-term clinical trials are hard to perform because of the time and rapid evolutions and changes in the adhesive systems. Therefore, laboratory studies are still commonly used to estimate the clinical behavior of dental materials (10).

The null hypothesis for this study is that the type of different dual cure luting resin cements either self etch or total etch would not influence the fracture resistance and shear bond strength of the lithium disilicate occlusal veneers.

MATERIALS AND METHODS

Table 1 Shows the list of resin luting materials composition used in this study.

Tooth specimen preparation

Twenty eight sound extracted human upper molars from old diabetic patients approximately the same dimensions ±0.5mm buccolingually, mesiodistally and occlusocervically mounted in autopolymerizing acrylic resin (Acreston, Egypt) and stored in distilled water (11). After mechanical debridement of the teeth by using ultrasonic scaler, they were subsequently kept in 0.2% thymol solution for seven days which is effective to destroy all kinds of microorganisms (12).

Before starting preparation, accurate silicon indices of all teeth was taken to help in the check of the amount of reduction. The indexes were cut in buccolingual and mesio-distal directions to be used as an index or guideline to return the tooth to its normal anatomical configuration. The indexes extended minimally 3 mm beyond the crown of the tooth cervically to ensure accurate replacement of the mold each time (13, 14).

Grouping of the specimens

The twenty eight specimens were randomly divided according to different design of tooth surface preparations into two main groups of 14 specimens each (n=14).

Group A: Represented (Anatomical reduction) for fracture resistance test.

Group B: Represented (Flat reduction) for shear bond strength test.

Then each group subdivided according to type of resin cement systems used into subgroups (n = 7):

Fracture resistance test


Shear bond test


Tooth preparation

After complete polymerization of the self-curing acrylic resin blocks tooth preparation of the specimens was performed:

For anatomical preparation reduction (Groups A1 & A2)

In total, 14 teeth were included for anatomical preparation reduction.

Evenly reduce the anatomical shape occlusally. The average occlusal clearance was 1 mm. A standardized preparation was done on occlusal surface using a conical diamond bur (646 KR 314 016 ,Komet ) in high speed with a water coolant following the depth orientation grooves, The axial walls were prepared creating 90 degree shoulder finish line at the junction between occlusal and middle third with the round end cylindrical diamond bur (836KR 314018, Komet). Fine diamond bur (888 36 KR 314018, Komet) were used to finish the preparation to make it round and smooth. Finally, polishing was performed using abrasive rubber points (9608 314030, Komet) (13).

For the flat preparation reduction (Groups B1 & B2)

In total, 14 teeth were included for the flat preparation.

The entire coronal structure was ground perpendicular to the long axis of each tooth using a diamond disc (Jota AG Rüthi/SG Switzerland) under continuous water cooling then surfaces were polished using a smooth sand paper disc (600- grit. Sic) under running water for 1 minute leaving a flat area of enamel (6 mm occlusal to the CEJ). For luting the ceramic discs to the middle third of the occlusal surface.

Ceramic specimen fabrication:

A total of 14 wax up with the one mm thickness occlusal veneers were constructed from IPS e.max press ceramic material to be bonded to the prepared teeth surfaces for fracture resistance test.
A total of 14 ceramic discs (4mm in diameter and 2 mm in height) were constructed from IPS e.max press ceramic material to be bonded to the prepared teeth surfaces for shear bond strength test. 

Surface treatment and bonding procedure 
Table 2 shows the enamel and ceramic surfaces treatment, priming and luting according to manufacturer instructions for each cement after sub-grouping. 

The cementation was done according to manufacturer's recommendations under a constant load of 2.0 kgs for five minutes using a special static loading device. 

Aging of the bonded specimens 
The bonded specimens were stored in distilled water at 37 °C for 24h and were then thermocycled 2500 cycles between 5 °C and 55 °C with a dwell time of 15 seconds at each temperature to mimic approximately 6 months of intraoral use. 

Fracture resistance testing 
The samples were attached to the lower arm of the universal testing machine (Instron 3345), while the 8 mm stainless steel ball was fixed on the upper arm of the machine. 

Compressive force was adjusted (at cross-head speed of 0.5 mm/min) down the long axis of each specimen along the central fossa of the occlusal veneer specimens, So that the forces were shared by the triangular ridges of the cusps. Additionally a 0.6mm rubber dam was placed between ball end and specimen in order to distribute the load homogenously. (Fig 1) 

Each specimen was loaded till fracture occurred and the forces in Newton was recorded. 

Shear bond strength testing 
Shear bond test was done to debond the ceramic discs from the prepared occlusal surface using the universal testing machine. 
The specimens were oriented so that the stainless steel chisel blade perpendicular to the junction between the ceramic disc and tooth surface at a cross head speed of 0.5mm/min (Fig 2). 

The load at which the de-bonding occurred was recorded in Newtons for each specimen in both groups then the shear bond strength was calculated in MPa. 

Failure mode investigation 
After fracture resistance testing. All the specimens were classified based on structures involved in the fracture according to Egbert in 2015 into: (Mode I: failure of restoration, mode II: failure of restoration and enamel, mode III: failure of restoration, enamel and dentin) (17). 

After shear bond testing failure mode was investigated according to Scherrer et al. into three types (18) using operating microscope under 1.8 × magnification. The results were categorized according to the amount of resin cement still present on the tooth surface as follows: Adhesive failure (if the resin still present is equal or less than 25 % in the bonded area). Cohesive failure (if the resin still present was equal or more than 75% in the bonded area). Mixed failure (if the resin still present was between 25-75 % of the total adhesion area). 

Statistical Analysis 
The used tests were: 
Chi-square test 
For categorical variables, to compare between different groups. 
Monte Carlo correction 
Correction for chi-square when more than 20% of the cells have expected count less than 5 
Student t-test 
For normally distributed quantitative variables, to compare between two studied groups. 

Figure (1): Fracture resistance test (A 0.6mm rubber dam placed between specimen and 8 mm stainless steel ball of Universal Testing Machine during load application)
Figure (2): Shear bond test (A chisel blade of Universal Testing Machine aligned perpendicular to ceramic disc and teeth during load application).

Table (1): List of resin luting materials composition used in this study.

<table>
<thead>
<tr>
<th>Brand name</th>
<th>Composition</th>
<th>Manufacture</th>
</tr>
</thead>
<tbody>
<tr>
<td>Variolink Esthetic Dual cure</td>
<td>The monomer matrix: Urethane dimethacrylate. The inorganic fillers: Ytterbium trifluoride. Spheroid mixed oxide. Total volume of inorganic fillers is: Approx. 38%. The particle size: 0.04-0.2 μm Other ingredients: Initiators: Both Photo-initiator (Camphor-Quinone) and Self cure initiator (Benzoyl peroxide) with Co-initiator (Tertiary amine). Stabilizers and pigments. Tetric N-Bond: Contain phosphoric acid acid acrylate, HEMA, Bis-GMA, Urethan dimethacrylate, ethanol, film forming agent, Catalysts and stabilizer. Monobond® Plus: Ethanol, 3-(trimethoxysilyl)propylmethacrylate, phosphoric acid methacrylate, silane methacrylate and sulfide methacrylate.</td>
<td>Ivoclar Vivadent, Schaan, Liechtenstein REF #66619WW</td>
</tr>
</tbody>
</table>

Table (2): The enamel and ceramic surfaces treatment, priming and luting according to manufacturer instructions for each resin luting materials after subgrouping.

<table>
<thead>
<tr>
<th>Subgroups</th>
<th>Luting agent</th>
<th>Tooth bonding agent</th>
<th>Ceramic bonding agent</th>
</tr>
</thead>
<tbody>
<tr>
<td>A1 &amp; B1</td>
<td>Panavia F 2.0</td>
<td>Panavia F 2.0 Primer</td>
<td>Clearfil Tri S Bond Universal</td>
</tr>
<tr>
<td>A2 &amp; B2</td>
<td>Variolink Ethetics</td>
<td>Tetric N bond Universal</td>
<td>Monobond Plus</td>
</tr>
</tbody>
</table>

Table (3): Comparison between the studied resin luting materials on the fracture resistance (N) and on the shear bond strength (MPa).

<table>
<thead>
<tr>
<th>Fracture resistance (N)</th>
<th>Panavia F2.0 (n = 7)</th>
<th>Variolink (n = 7)</th>
<th>t</th>
<th>p</th>
</tr>
</thead>
<tbody>
<tr>
<td>Min. – Max.</td>
<td>576.5 – 2480.6</td>
<td>1830.0 – 2691.8</td>
<td>2.738</td>
<td>&lt;0.018</td>
</tr>
<tr>
<td>Mean ± SD.</td>
<td>1330.1 ± 812.2</td>
<td>2259.5 ± 382.9</td>
<td>6.930</td>
<td>&lt;0.001</td>
</tr>
<tr>
<td>Median (IQR)</td>
<td>1009.9(707.4 –1914.5)</td>
<td>2297.9(1898.5 –2599.8)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Shear (MPA)</th>
<th>Panavia F2.0 (n = 7)</th>
<th>Variolink (n = 7)</th>
<th>t</th>
<th>p</th>
</tr>
</thead>
<tbody>
<tr>
<td>Min. – Max.</td>
<td>23.89 – 26.01</td>
<td>30.70 – 38.85</td>
<td>6.930</td>
<td>&lt;0.001</td>
</tr>
<tr>
<td>Mean ± SD.</td>
<td>25.18 ±0.91</td>
<td>34.68 ±3.51</td>
<td>6.930</td>
<td>&lt;0.001</td>
</tr>
<tr>
<td>Median (IQR)</td>
<td>25.60 (24.59 – 25.81)</td>
<td>34.36 (31.62 – 37.82)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

RESULTS
Table 3 Shows the comparison between the studied resin luting materials on the fracture resistance (N) and on the shear bond strength (MPa).
Fracture resistance
On comparing between 2 Types of resin cements used in each groups in enamel by using t_
test, the difference in the mean values of fracture resistance showed a statistically significant difference with \( p \leq 0.05 \). Group A2 (Variolink Esthetic) showed the highest mean value (2259.5±382.9 N) followed by group A1 (Panavia F2.0) mean value (1330.1±812.2 N). (Table 3)

Shear bond strength

There was statistically significant difference of the shear bond strength values between groups B1 and B2 \( (p \leq 0.05) \). The highest mean values of shear bond strength was recorded in group B2 (Variolink Esthetic) mean value (34.36±3.51 MPa) followed by group B1 (Panavia F2.0) mean value (25.18±0.91 MPa). (Table 3)

Failure mode of occlusal veneers after fracture

The relation between fracture load and mode of failure was tested using Chi square test there was no statistically significant difference between the two groups.

In group A1, (PanaviaF2.0 resin cement), All seven specimens (100 %) showed fractured in veneer restoration (Mode I). (Fig 3) While In group A2, (Variolink Esthetic resin cement), six specimens (85.7 %) showed fractured in veneer restoration (Mode I), while only one specimen (14.3 %) showed fractured in veneer restoration, enamel and dentin (mode III). (Fig 4)

Failure mode of de-bonded ceramic disks after shear test

There was no statistically significant difference in Failure mode of de-bonded ceramic disks after shear test. In group B1 (PanaviaF2.0 resin cement) three specimens showed cohesive pattern of failure representing (42.9%) while other two showed adhesive pattern (28.6%). The last two specimens showed mixed pattern of failure (28.6%).(Fig 5) While In group B2 (Variolink Esthetic resin cement) three specimen showed cohesive pattern of failure (42.9%) while one specimen showed adhesive pattern of failure (14.3%), and the final three specimens showed mixed pattern of failure (42.9%). (Fig 6)

**DISCUSSION**

Restorative Dentistry's main objective is preservation of the tooth structure. Wear, caries, aging process, bulimia nervosa or malposition may lead to loss the normal occlusal teeth contact. Treatment of this problem is very challenging because sometimes we need to remove the sound tooth structure to accept conventional material of restoration (19). Management of dental wear starting from knowing the main causes and saving teeth from additional damages , the restorative phase depends on the grade of destruction and needs cautious methods. Initial lesions require simply a clinical follow up, an interceptive minimalistic treatment approach using a non-prep sealing with composite resin restorations (20).

Re-enameling process using lithium disilicate non retentive occlusal veneers are becoming a current treatment solution to restore posterior teeth affected by wear. Occlusal veneers are monolithic indirect extra coronal overlay. The luting technique has an influence in bonded all ceramic
restorations are showed a higher fracture resistance than traditionally cemented restorations (13).

The present study was performed in vitro to evaluate the fracture resistance and shear bond strength of lithium disilicate occlusal veneers to enamel luted with two different dual cured brands of resin cements differing according to the etching technique either total etch or self-etch. Occlusal surface preparation was restricted to enamel thickness and any tooth that their preparation expose dentin surface was discarded.

The Adhesive system selected for this study was Panavia F2 and Variolink Esthetic® both were dual cure resin cements. One of the factors that play an essential role in the longevity of lithium disilicate occlusal veneers is the adhesive system. Appropriate adhesion to tooth structure proves successful function of ceramic restorations. Bond strengths are affected by many factors such as surface treatments and more importantly, luting cement type (21). Clinically, maximum bite forces in the posterior region can range from 200 to 540 N and up to 800 N in patients with bruxism (22). Thus the failures affecting whole (restoration-cement-tooth) complex as a result of high forces can be suspected in clinical situation due to the wide range of masticatory forces.

In the present study using both types of resin luting cements resulted in high fracture resistance (ranging between 1330.1 N in Panavia F2 and 2259.5 N in Variolink Esthetic), which is higher than the normal masticatory loads encountered in the posterior molars regions, indicating that occlusal veneer restorations constructed from IPS emax could be predictable long life restoration in these areas. The fracture resistance of all-ceramic materials increased by using adhesive luting resin cement that can prevent cracks propagation by penetrating into the irregularities of the restoration’s inner surface then all spaces could be filled and closed (33).

Comparing our results on the fracture resistance of lithium disilicate restorations, the results of specimens treated with total etch technique showed a significantly higher fracture resistance than self etch. Phosphoric acid etching improve bond strength because the smear layer was eliminated and allow the sufficient penetration of resin, self-etch resin had the high level of fillers and high viscosity of cement lead to insufficient penetration into demineralized enamel. This comes in agreement with Yildiz et al in 2013 (23) who evaluated the influence of the bonding technique on the fracture resistance of lithium disilicate partial crowns with occlusal thickness of 1.5 mm using total etch (Variolink II ) and self-etch (Multilink).

Our results were also in agreement with Angerame et al in 2019 and Ioannidis et al in 2019 (13, 24) studying load to fracture with lithium disilicate occlusal veneers 1mm thickness bonded to enamel using Variolink II in maxillary molars they found mean value were 2395 N ,1245 N respectively which are in the same range of our study result ( 2259.5 N)

Abo-Madina and Abdelaziz in 2009 (25) in their fracture resistance study using Panavia F2.0 showed fracture strength of 921 N after thermocycling which is in the same range of our study (1330 +/- 812 N). Moreover Al-Akhali et al in 2019 (26), when bonding to enamel using self-etch the average final fracture strength was 932 N and they reported that when bonding to enamel self-etch technique should be avoided in non-retentive occlusal veneers.

However, in contradiction to our findings Guess et al in 2013 evaluated the resistance to fracture of lithium disilicate overlays when bonded using Variolink II total etch technique median value were in a lower range (1300 N) than our study (2297 N). The lower values may be attributed to use of premolars instead of molars and differences in preparation design (27).

Using self-etch after pre-treatment of enamel with 37% phosphoric acid increase the fracture resistance significantly. Al Akhali et al in 2017 found the fracture resistance of aged occlusal veneers bonded to the premolars enamel (19), to be in the range of 1’545 N for the lithium disilicate ceramic in comparison to 1330 N in our study.

Also, the present study was performed in vitro to evaluate also the shear bond strength of lithium disilicate occlusal veneers to enamel luted with two different dual cured brands of resin cements differing according to the etching technique either total etch or self-etch. The Adhesive systems selected for this study were Panavia F2 and Variolink Esthetic® both were dual cure resin cements. Bond strengths are affected by the luting cement type (22).

Variolink Esthetic total-etch resin cement had a statistically significant higher bond strength (34.68MPa) than Panavia F2.0 self-etch resin cement (25.18 MPa), This may be clarified by, first, the high filler load and viscosity of the Panavia F2.0, which may decrease infiltration depth of the adhesive into the primed enamel. Second , the residual acids of ED primer may retard the chemical curing of the luting cement while the Variolink Esthetic used small-particle fillers that necessitated the incorporation of low-viscosity, highly reactive monomers to allow a high filler content. The hydrophilic/hydropobic features of the monomers were modified to the fillers to ensure optimal wetting of the fillers, (28)

This comes in agreement with Berke Bulut et al. in 2018 (28) and Abo Hamar SE in 2005 (29) Furthermore, Holiel et al in 2015, and Alqahtani in 2017 (30, 31) they also showed higher enamel bond
Optimizing the ceramic surface treatment when conditioned with HF and silane, the combination of micromechanical interlocking (physical bonding) and (chemical bonding) enhance bond strength. Tooth surface treatment using phosphoric acid prepares the topography of enamel, changing it to high surface energy which is more susceptible to adhesion instead of low reactive surface. Also, an enlargement of the surface area of enamel after acid etching allow bond penetration through capillary attraction achieving micro mechanical retention that provided best bonding quality. Besides, bond strength not only depends on etching depth but also chemical composition and mechanical properties of the brand used. Elmarakby AM in 2019 found that nature of adhesive system, resin cement and their chemical composition play a significant role in making a long lasting bond between the tooth substrates and the indirect restoration (33). These previous reasons proposed by Elmarakby AM could explain the results of our study which includes tooth structure. Also, Oztruk study used buccal surface substrate rather than occlusal one.

While in contradiction to what was found in our study low shear bond strength value were reported by Secilmis in 2015 (34) and Oztruk in 2016 (35). Panavia F2.0 with lithium disilicate ceramic were 7.50 MPa and 3.62 MPa respectively. This inconsistent results might be due to the use of different parameters. Secilmis luted cement directly to the restorative material surface differed as it involved only two substrates, ceramic and cement, as opposed to the current study which includes tooth structure. Also, Oztruk study used buccal surface substrate rather than occlusal one.

Optimizing the ceramic surface treatment when conditioned with HF and silane, the combination of micromechanical interlocking (physical bonding) and (chemical bonding) enhance bond strength. Tooth surface treatment using phosphoric acid prepares the topography of enamel, changing it to high surface energy which is more susceptible to adhesion instead of low reactive surface. Also, an enlargement of the surface area of enamel after acid etching allow bond penetration through capillary attraction achieving micro mechanical retention that provided best bonding quality. Besides, bond strength not only depends on etching depth but also chemical composition and mechanical properties of the brand used. Elmarakby AM in 2019 found that nature of adhesive system, resin cement and their chemical composition play a significant role in making a long lasting bond between the tooth substrates and the indirect restoration (33). These previous reasons proposed by Elmarakby AM could explain the results of our study because Variolink Esthetic contain Ivocerin rather than conventional initiator systems.

Alkhudhairy et al in 2018 reported that degree of conversion and optimal cure is essential as it commands the physical and mechanical properties of resin cement. A new initiator instead of the camphor quinine amine initiator systems is Ivocerin. Ivocerin is a Norrish type I photoinitiator. A higher photo curing activity than camphor quinine amine because of its higher absorption in the wavelength region between 400 and 450 nm. This allow light to penetrate deeply to perform a chemical bond within the initiator molecule and form two radicals. These radicals eventually react with the monomer to produce a polymer network and therefore achieves the best polymerization results due to higher degree of conversion and decreased polymerization stresses, thus improving the bond strength and mechanical properties of indirect restorations (36).

Concerning fracture resistance test; modes of failures were found to be limited to the occlusal veneers and don’t include tooth structure. This improve long term outcome of restored tooth because the occlusal veneers can be easily exchanged. If tooth structure was involved the endodontic treatment or extraction might be necessitated.

However, in the shear bond strength results of the present study most of the failures in Variolink Esthetic group were cohesive in nature at the cement/tooth but in Panavia F 0.2 group most of failures were adhesive failures in nature which means higher bond strength in Variolink Esthetic group. However, in the SBS results of the current study, most of the failures in Variolink Esthetic group and Panavia F2.0 were cohesive in nature at the cement/tooth interface. Secilmis et al in 2016 in a similar study stated that the greater shear bond strength value showed greatest liability for cohesive fractures in the luting resin (35). The value shown by Variolink Esthetic and Panavia F2.0 (42.9%) are in agreement with this conclusion. Higher adhesive failures (28.6%) were found in Panavia F2.0 than in Variolink Esthetic (14.3%). However, those differences were insignificant (28).

The results of the present study approve the refusal of the null hypothesis that there is no difference in the fracture resistance and shear bond strength of lithium disilicate occlusal veneers cemented with 2 different resin cements. Panavia F 2.0 self etch groups exhibited lower fracture resistance and shear bond strength values than the Variolink Esthetic total etch groups.

**CONCLUSION**

Within the limitations of this study, it was concluded that:

Lithium disilicate ceramic occlusal veneers material are the biomimetic conservative treatment option for occlusal wear and should replace conventional crown restorations.

Fracture resistance for both resin cements with lithium disilicate occlusal veneers showed sufficient strength to withstand occlusal load to be used in molar region.

The glass lithium disilicate ceramics are better luted with a total etch resin cement in order to obtain the best bond strength from enamel.

**CONFLICT OF INTEREST**

The authors declare that they have no conflict of interest.
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REFERENCES
24. Ioannidis A, Mühlemann S, Özcan M, Hüsler J, Hämmerle CHF, Benic GI. Ultra-thin occlusal veneers bonded to enamel and made of ceramic or hybrid materials exhibit load-bearing capacities not different from conventional...
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