Influence of Different Luting Cements on the Shear Bond Strength of Pretreated Lithium Disilicate Materials

Mohammed Alkhurays¹, Fawaz Alqahtani²

ABSTRACT

Aims: The aim of the study was to examine the shear bond strength of different luting cements bonding to pre-treated lithium disilicate materials. **Materials and methods:** Sixty A2 shade lithium disilicate discs were subjected to either micro-etch with aluminum trioxide and etching by 10% hydrofluoric acid (micro-etch group; n = 30); or etching with 10% hydrofluoric acid (acid-etch group; n = 30) before cementation. Three dual-cure Variolink Esthetic (VDC), RelyX Ultimate (RUT), and RelyX Unicem (RUC) and three light-cure Variolink Veneer (VV), Variolink Esthetic (VLE), RelyX Veneer (RV) resin cements were used for cementation. The specimens from each group were tested for shear bond strength (SBS). The data were analyzed using two-way ANOVA; p < 0.05 is considered statistically significant.

Results: For all resin cements tested with different surface treatments, there was a statistically significant difference within resin cements per surface treatment (p < 0.05). The SBS in the micro-etch group was significantly higher across all the cements tested when compared to the acid-etch group (p < 0.05), thus suggesting that surface treatment affects the SBS largely irrespective of the resin cement. Their interaction between cement and the surface treatment was significantly different across groups (p < 0.001).

Conclusion: Under the limitations of the present study, it can be concluded that surface treatment influences the bond strength irrespective of the resin cement (light/dual-cure) used for indirect restorations' cementation. The shear bond strength in the sand blast/acid etch group was significantly higher across all the cements tested when compared to the acid-etch alone.

Clinical significance: The surface treatment of porcelain veneer hugely influence the SBS, which will directly affect the veneer clinical success rate. The micro-etching recorded a higher shear bond strength when compared to those with acid-etch only.

Keywords: Adhesion, Lithium disilicate, Luting, Microetching, Resin cements.

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INTRODUCTION

With the recent rapid development of dental materials, the expectations of the dental community and patients have risen markedly, owing to its higher standards for esthetic and functional performances. Porcelain laminate veneers (PLV) are widely used in modern dental practice owing to their high esthetic outcome and conservative tooth preparation. PLV showed a high clinical success rate of approximately 93% over 15 years.¹ Although clinically successful, various features such as cementation procedure, composition, the luting cement, and the ageing procedure can affect their durability, adhesion, and thereby clinical success.^{2–8}

The strength and durability of the adhesion complex formed between the tooth surface, resin cement, and porcelain surface are considered the most critical factors determining the longevity of the PLV.⁹ The failure modes most frequently associated with laminate veneers are debonding or fracture.¹⁰ Adhesive failure occurs at the porcelain-cement interface, leading to a complete debonding of the veneer.^{11,12} Although the failure is largely dependent on the magnitude of load; it may also depend on the types of shear stresses.^{13–16} Hence, bond strength to counter shear stresses is significant in determining the longevity of laminate veneers' postcementation.

Resin cements are the most used materials for the cementation of indirect restorations.¹⁷ The advantages of resin cements include improved marginal seal, reduced risk of postoperative sensitivity, low solubility, and superior mechanical properties, compared to zinc phosphate and glass-ionomer cements.^{18,19} The clinical outcome of indirect restorative procedures also depends largely on the cement used to bond onto the teeth.^{20,21} Therefore, diligent

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selection of resin cement in the restorative process is much needed for long-term success of indirect restorations.

Depending on the mode of activation, resin cements are often classified in three groups: chemically activated (self-cured), photo-activated, and dual-cured cements. Self-cured cements are designated for cementing metallic and non-metallic restorations. It has been suggested that light-curing cements may be restricted to laminate veneers owing to the ability of the veneers material to transmit the light that results in curing of the cement.²² Porcelain veneer adhesive luting can be achieved using both dual-cured and light-cured cements.²³ Dual-cured cements were developed to combine the desirable features of self-cured and photoactivated cements. Light-curing materials used as luting agents can easily be managed and are characterized by well-regulated hardening times

© The Author(s). 2019 Open Access This article is distributed under the terms of the Creative Commons Attribution 4.0 International License (https://creativecommons. org/licenses/by-nc/4.0/), which permits unrestricted use, distribution, and non-commercial reproduction in any medium, provided you give appropriate credit to the original author(s) and the source, provide a link to the Creative Commons license, and indicate if changes were made. The Creative Commons Public Domain Dedication waiver (http://creativecommons.org/publicdomain/zero/1.0/) applies to the data made available in this article, unless otherwise stated. and no time constraints. It is easier to attain a precise setting of the veneer and to eliminate all the excess cement, creating high-quality margins precisely.²⁴ However, with a wide variety of resin cements available, the selection of the most beneficial adhesives for long-term retention of restoration is desirous. In addition, restoration surface treatment is known to improve adhesion.²⁰ With options such as micro-etch and acid-etch techniques, the selection of surface treatment requires that the specialist makes a cognizant decision as to what the perfect surface treatment ought to be.

Hence, there is a need to examine the effect of cement type (light/dual-cure) and restoration surface treatment on longevity of restorations estimated using shear bond strength. The null hypothesis tested in the study was that there is no difference in the bond strength of differently pretreated PLV cemented using different light-/dual-cure resin cements.

MATERIALS AND METHODS

Study Design

This experiment was designed and approved by all authors and were conducted at the dental school, Riyadh Elm University. It is an *in vitro* experiment of PLVs that evaluated the effect of three light curing and three dual curing luting cements on two different surface treatments by means of a shear bond strength.

Specimen Preparation

Sixty A2 shade digitally calibrated discs (3 mm \times 10 mm) using a digital caliper were prepared from lithium disilicate computer aided design/computer aided manufacturing (CAD/CAM) blocks (Ivoclar Vivadent, Schaan, Liechtenstein) according to the manufacturer's instructions. The specimens were designed using the 3D builder software and saved as stereolithography (STL) file. Subsequently, milling was done with CAM 5-s1 (VHF, Ammerbuch, Germany).^{2,25} The ceramic surfaces were finished and polished using the manufacturers' recommended kit (LUS80, Meisinger, USA) to ensure surface standardization. The specimens were fired at 850°C. The ceramic discs were subsequently embedded in the autopolymerizing acrylic resin (Fig. 1). The discs were sanded with a 400-grit followed by a 600-grit wet silicon carbide paper until the ceramic discs were perfectly flush with the acrylic resin. All specimens were rinsed under running water, dried, and subsequently treated with 37% phosphoric acid for 1 minute to

clean off the abrasive particles. All specimens were again rinsed under running water and dried.

Study Groups

The sanded specimens were randomly divided into three light cure and three dual cure groups according to the cements used as per Flowchart 1. Three dual-cure—Variolink Esthetic (VDC), RelyX Ultimate (RUT), and RelyX Unicem (RUC) as well as three light-cure— Variolink Veneer (VV), Variolink Esthetic (VLE), and RelyX Veneer (RV) resin cements were used for disc cementation. Each group was further divided into two subgroups according to the surface treatment: micro-etch and acid-etch (Table 1).

Specimen Cementation

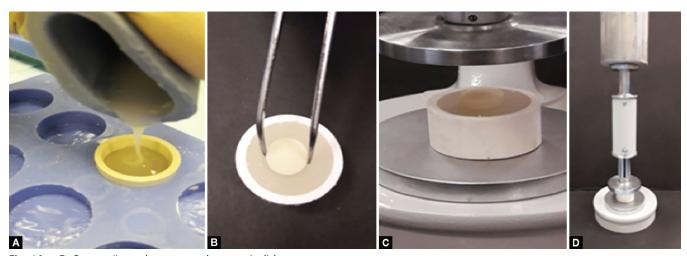
Before cementation, the ceramic surfaces were treated as per groups; micro-etching with 30 µm alumina from 10 mm at 55 KPa for 10 seconds followed by 20 seconds etching with 10% hydrofluoric acid (micro-etch) and only etching with 10% hydrofluoric acid (acidetch) for 20 seconds. All specimens were rinsed under running tap water to remove the debris. A mould of 4 mm diameter and 2 mm thickness was fabricated to provide a uniform area for cementation. Subsequently, it was placed at the center of each specimen. All resin cements were applied directly from an auto-mix syringe onto the treated surface of the specimens after syringe bleed to not use the first cement layer. A 1 kg weight was placed on the top to form a uniform cemented layer. Subsequently, the top surfaces of all specimens were light-cured in direct contact for 40 seconds to simulate clinical conditions.

Shear Bond Strength Assessment

The specimens from each group were tested for shear bond strength. For testing, a universal testing machine (Instron Corp., Canton, MA, USA) was used. The specimens were fixed by using a jig, and the interface between the specimens and resin was loaded at a crosshead speed of 1 mm/minute.² A knife-edge stainless steel chisel with a thickness 0.34 mm and diameter of 10 mm was used for loading (Fig. 2). The shear load at failure was recorded by the software and the values were converted to stress in MPa.

Microscopic Examination

Specimens was scanned under a digital stereo zoom microscope (Hirox, Tokyo, Japan) at 50× magnification to determine the mode



Figs 1A to D: Custom Jig made to mount the ceramic disk



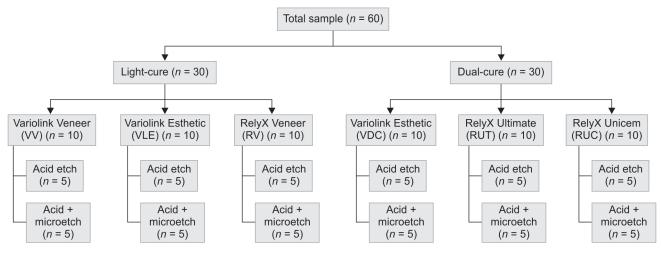
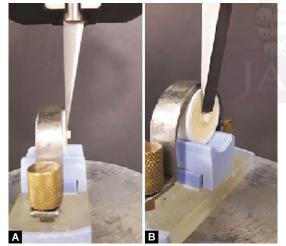


Table 1: Materials used in the study

Material	Types	Manufactures		
Ceramic	Lithium-disilicate based	Ips E.Max Press, Variolink Veneer, Ivoclar Vivadent, Schaan, Liechtenstein		
Resin cement	Light-cure	Rely X Veneer, 3m Espe, St. Paul, Minneapolis, USA		
		Variolink II, Ivoclar Vivadent, Schaan, Liechtenstein		
		Variolink Esthetic LC, Ivoclar Vivadent, Schaan, Liechtenstein		
	Dual-cure	Rely X Ultimate, 3M ESPE, St. Paul, Minneapolis, USA		
		Rely X Unicem, 3M ESPE, St. Paul, Minneapolis, USA		
		Variolink Esthetic DC, Ivoclar Vivadent, Schaan, Liechtenstein		
Ceramic primer	Variolink ceramic prime and etch	Monobond, Ivoclar Vivadent, Schaan, Liechtenstein		
Etching gel	Hydrofluoric acid	Hydrofluoric acid, Ivoclar Vivadent, Schaan Liechtenstein		
Bonding agent	Ceramic bonding	Single Bond Universal, 3m Espe, St. Paul, Minneapolis, USA		



Figs 2A and B: Custom knife-edge stainless steel chisel at shear loading

of failure. Failure mode was classified into three types: adhesive failure at the interface between ceramic/cement, cohesive failure in ceramic or cement, and mixed failure.

Statistical Analysis

The data were entered in Microsoft Office Excel worksheets and analyzed using IBM SPSS software, version 20.0 (IBM Statistics, SPSS,

Chicago, USA). The normality of the data was assessed using the Shapiro–Wilk test, while Levene's test for equality of error variances was used to analyze the homogeneity of error variances. Two-way ANOVA with Bonferroni's correction for multiple group comparisons was used to analyze the data with factors: resin cement and surface treatment for dependent variable shear bond strength (MPa). Statistical significance was determined at $\alpha = 0.05$.

RESULTS

The mean \pm standard deviation for the shear bond strength at maximum load in MPa were recorded, tabulated, and compared using two-way ANOVA (Table 2). There was a statistically significant difference observed in the shear bond strength between the two surface treatment groups (p = 0.007). Within the resin cement groups, there was statistically significant difference observed in the shear bond strength (p = 0.004). The interaction between the two factors: surface treatments and resin cements demonstrated statistically significant differences between and within groups (p < 0.001).

For all resin cements tested with different surface treatments, there was a statistically significant difference within resin cements per surface treatment (p < 0.05). Within the acid etch group, the highest shear bond strength was observed by the dual cure cements RUC, whereas the lowest shear bond strength was for light cure cement VLE followed by VDC, which were significantly



Tabl	e 2: Shear	bond	strength	of the	tested	cements	per sui	face treatment
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Shear bond strength (MPa) (mean \pm SD)									
Surface treatment/	Light-cure cements			Dual-cure cements					
cement	VV	VLE	RV	VDC	RUC	RUT			
Acid	11.36 ± 0.12 ^{d,l}	6.95 ± 0.22 ^{c,l}	$12.00 \pm 0.26^{\text{ad,I}}$	9.42 ± 0.24 ^{b,l}	13.21 ± 0.37 ^{a,I}	$12.31 \pm 0.43^{\text{ad,I}}$			
Acid + microetch	15.11 ± 0.79 ^{A,II}	$8.50\pm0.78^{\text{D,II}}$	15.50 ± 0.96 ^{A,II}	11.15 <u>+</u> 0.72 ^{C,II}	$14.30 \pm 0.74^{\text{AB,II}}$	$13.53 \pm 0.98^{\text{B,II}}$			

Two-way ANOVA; p < 0.05 is significant

Factor 1: cement; p = 0.004Factor 2: surface treatment; p = 0.007

Factor 1 × 2; *p* < 0.001

Capital (A, B, C, D)/small letter (a, b, c, d) alphabets demonstrate significant differences between resin cement groups per surface treatment Roman numbers (I, II) indicate significant differences between surface treatment per resin cement

different from the other resin cements (p < 0.05). Within the microetch group, the highest shear bond strength was observed for RV, whereas the lowest shear bond strength was for the VLE followed by VDC, which were significantly different from the other resin cements (p < 0.05). The shear bond strength in the micro-etch group was significantly higher across all the cements tested when compared to the acid-etch group (p < 0.05), thus suggesting that surface treatment affects the bond strength largely irrespective of the resin cement. The microscopic examination of the failures demonstrated that most of the failure among all the group was mixed failure (69% from the scanned specimens represent this mode of failure); followed by cohesive failure occurred in 28% and it mainly occurs in the cement; only 3% represented the adhesive failure, which was in ceramic cement interface.

DISCUSSION

Three dual-cured (RelyX Ultimate, RelyX Unicem, Variolink Esthetic) and three light-cured (RelyX Veneer, Variolink Veneer, Variolink Esthetic) resin cement systems from different manufacturers were chosen in this study to evaluate their effect on adhesion to PLV treated with two different methods. The results of this study showed that there was a significant interaction between cement and surface treatment. The additional micro-etching leads to a significant increase in the shear bond strength of both light cure and dual cure cements. Hence the null hypothesis was partially rejected as the curing process did not influence the bond strength.

The stresses at the interfaces of restorations are complex. However, they can be identified as primarily tensile or shear type, created by forces working either perpendicular or parallel to the tooth surface.^{14–16} The difference in the shear bond strength can be interpreted as the difference in fracture of the resistance of the luting agents, to which shearing load was applied during the test. The shear strength is the degree to which a material or bond can resist shear before fracture. Shear and tensile tests are used usually to measure the bond strength of dental materials because they are easy to achieve, and requires minimum equipment and specimen preparation.²⁵ The shear bonding effectiveness and cementation process play a fundamental role in the clinical success of all-ceramic restorations.^{26,27} Therefore, in this study, shear bond tests were performed to assess the adhesive bonding of ceramic material with resin luting agents.

The results of this study showed that the values of shear bond strength vary with different resin cements. This result is in accordance with other studies that concluded that the properties and bond strengths of resin cements might be influenced by their composition.^{2–5} Dual-cured resin cements offer extended working times and controlled polymerization when compared to light cure cements.^{28,29} Our results were partially similar with results from a study conducted by Braga et al., who studied the early SBS of porcelain to dentin of certain resin cements.³ They found that no differences in the mechanical properties of different dual cured resin cements compared to the mechanically cured cement. Mechanically cured cements showed the lowest SBS compared with other dual cured cement.

In this study two different surface treatments were used: acidetch, and micro-etching followed by acid etch. The result showed a significant difference in the mean of SBS between the HF acid etching and Al₂O₃ micro-etching. The samples with additional micro-etching recorded a higher shear bond strength when compared to those with acid-etch only. The result of the present study in accordance with the previous research that demonstrated differences between the type of surface treatment.²⁹⁻³¹ Roulet et al.³⁰ studied the effect of the surface treatment on the bond strength of ceramic to the resin cement. Three surface treatments (etching, sandblasting, grinding) have been tested. They found that acid-etching with 10% hydrofluoric acid gel or 10% ammonium bifluoride was much more effective than air-particle abrasion or grinding. In a similar study by Ozden et al.³¹ (wherein they compared acid etching with hydrofluoric acid), the porcelain was roughened with a diamond bur and the silane coupling agent is used alone and in combinations with these surface treatments. They concluded that silane application on mechanically roughened ceramic surfaces to be most effective on SBS. When used in conjunction with a diamond bur, silane treatment resulted in bond strengths twice as high as those obtained with hydrofluoric acid-etching alone. In another study by Thurmond et al.³² where they tested ten combinations of different surface treatments on the bond strength of composite resin to porcelain. The mechanical alteration of the porcelain surface with aluminum oxide air-abrasion and hydrofluoric acid-etching followed by silane application produced the highest bond strengths at 3 months compared with other nine porcelain surface-treatment techniques.

An inherent limitation of the study is its nature of being *in vitro* as the methodology does not completely replicate the oral environment. However, a clinical study might reveal different insights into the perspective of the present study. Hence, a clinical trial examining the effect of resin cements with micro-etching is suggested to analyze the influence of curing process on the longevity of restoration.

CONCLUSION

Under the limitations of the present study, it can be concluded that surface treatment influences the bond strength irrespective of the resin cement (light/dual-cure) used for indirect restorations' cementation. The shear bond strength in the sand blast/acid etch group was significant higher across all the cements tested when compared to the acid-etch alone.

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