

# The effect of surface treatment and thermocycling on the shear bond strength of porcelain laminate veneering material cemented with different luting cements

Fawaz Alqahtani<sup>1,\*</sup>, Mohammed Alkhourays<sup>2</sup>

<sup>1</sup> Associate Professor Department of Prosthodontics, College of Dentistry, Prince Sattam Bin Abdulaziz University, Al-Kharj 11942, Saudi-Arabia.

<sup>2</sup> Specialist, Department of Prosthetic Dental Sciences, ministry of Health - Asser region, Saudi Arabia.

**Corresponding author:**

Fawaz Alqahtani, 3603 Imam Abdullah bin Saud Road, unit # 646, 8356-13225 Riyadh.  
E-mail: [implantologist@yahoo.com](mailto:implantologist@yahoo.com)

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**Aim:** The study aimed to evaluate and compare the effect of different surface treatment and thermocycling on the shear bond strength (SBS) of different dual-/light-cure cements bonding porcelain laminate veneers (PLV). **Methods:** One hundred and twenty A2 shade lithium disilicate discs were divided into three groups based on the resin cement used and on the pretreatment received and then divided into two subgroups: thermocycling and control. The surface treatment were either micro-etched with aluminium trioxide and 10% hydrofluoric acid or etched with 10% hydrofluoric acid only before cementation. Three dual-cure (Variolink Esthetic (I), RelyX Ultimate (II), and RelyX Unicem (III)) and three light-cure (Variolink Veneer (IV), Variolink Esthetic (V), RelyX Veneer (VI)) resin cements were used for cementation. The SBS of the samples was evaluated and analysed using three -way ANOVA with statistical significant set at  $\alpha=0.05$ . **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 shear bond strength in the micro-etch group was significant higher than the acid-etch group ( $p<0.05$ ) There was statistically significant interaction observed between the surface treatment and thermocycling ( $p<0.05$ ) as well as the cement and thermocycling ( $p<0.05$ ). It was observed that the reduction in shear bond strength after thermocycling was more pronounced in the acid etch subgroup as compared to the microetch subgroup. However, the interaction between the three factors: surface treatments, thermocycling and



resin cements did not demonstrate statistically significant differences between and within groups ( $p=0.087$ ). **Conclusions:** Within the limitations of the present study, it can be concluded that Dual cure resin cements showed a higher Shear bond strength as compared to light cure resin cements. Thermal cycling significantly decreased the shear bond strength for both ceramic surface treatments. After thermocycling, the specimens with 10% HF surface treatment showed lower shear bond strength values when compared to those treated by sandblasting with  $Al_2O_3$  particles.

**Keywords:** Resin cements. Dental cements. Dental porcelain. Shear strength.

## Introduction

The most desirable characteristics of a dental restoration are good esthetics, strength and chemical stability, and ceramics inarguably possess these qualities<sup>1-3</sup>. The ceramic restorations is the first choice to be used for indirect restoration<sup>1,3</sup>. The SBS is getting more promising due to the newly developed resin cements with new composition of ceramic materials<sup>3</sup>. Resin cements are the most used materials for the cementation of indirect restorations. 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<sup>4,6</sup>. However, the success of ceramic restorations depends on factors, like the composition of the ceramic material and the cementation procedure<sup>7-9</sup>. Therefore, for the long-term success of ceramics, selection of the appropriate resin cement as well as the bonding procedure is imperative.

The efforts to improve resin bonding to ceramic include the application of different ceramic surface treatments. Lithium disilicate glass ceramic (IPS e.max Press, Ivoclar Vivadent, Schaan, Liechtenstein) may be adhesively cemented, but the retention may be inadequate when the retentive area is small. Etching with hydrofluoric acid roughens the surface on the bonding area of the ceramic material to enhance bonding by micromechanical interlocking between the ceramic and resin cement. It also creates irregularities within the lithium disilicate crystals by removing the glass matrix and the second crystalline phase<sup>3,9-11</sup>. Airborne particle abrasion with 50- $\mu$ m aluminum oxide ( $Al_2O_3$ ) particles is another surface treatment recommended for ceramic surfaces to aid in mechanical retention<sup>10-14</sup>. It leads to the coating of the ceramic surface must with a suitable silane, thereby resulting in the formation of chemical bonds between the inorganic phase of the ceramic and the organic phase of the resin cement<sup>11,15-17</sup>.

Clinically, when ceramic restorations are cemented and exposed to the oral environment, factors that could result in fatigue may influence their physical and mechanical properties. Fatigue fracture is a form of failure that occurs in structures with microscopic cracks subjected to dynamic and fluctuating stresses<sup>9</sup>. Thermal variations and the evaluation of fatigue resistance of dental ceramics could provide a more detailed understanding of clinical failures<sup>18</sup>. Long-term water storage and thermocycling of bonded specimens are accepted methods to simulate aging and to stress the bond-

ing interface<sup>19</sup>. Most studies that apply these methods reveal significant differences between early and late bond strength values<sup>20</sup>. Microtensile, shear or tensile testing methods with and without simulated aging and/or thermocycling have been used and conflicting results are reported regarding the effect on bond strength after aging in water and thermocycling<sup>21-25</sup>.

Hence, there is a need to examine the effect of thermocycling and restoration surface treatment on the longevity of restorations estimated using shear bond strength. The aim of this study was to evaluate and compare the effect of different surface treatment and thermocycling on the SBS of different dual-/light-cure cements bonding PLV. The null hypothesis was that there is no difference in the bond strength of differently pre-treated and thermocycled PLV cemented using different light-/dual-cure resin cements.

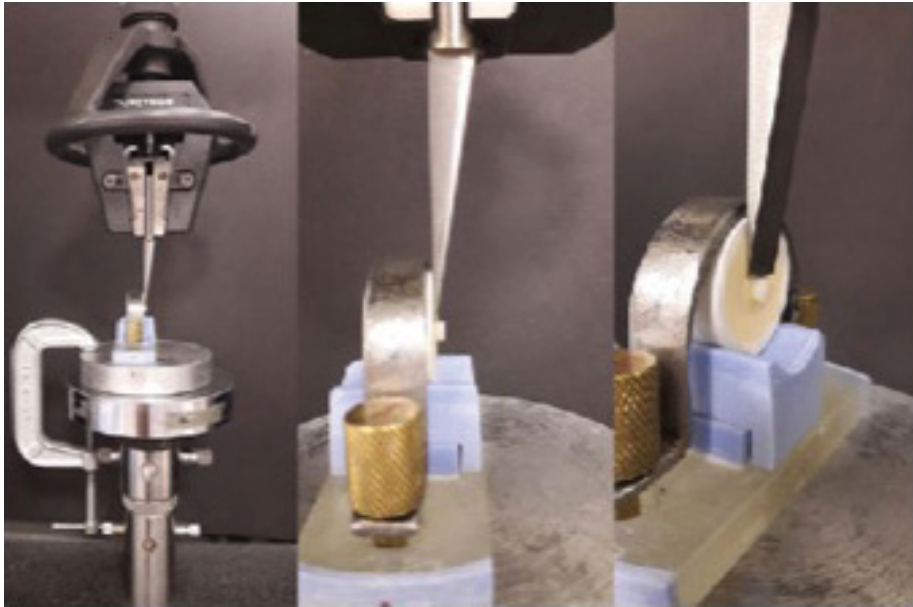
## Material and Methods

An in vitro experimental study was conducted to evaluate the effect of thermocycling and two different surface treatments on the shear bond strength of PLV cemented with three light cure and three dual cure cements.

Lithium disilicate Computer Aided Design/Computer Aided Manufacturing (CAD/CAM) blocks (Ivoclar Vivadent, Schaan, Liechtenstein) were used to prepare one hundred and twenty A2 shade digitally calibrated discs (3mm × 10 mm) 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)<sup>21,26</sup>. To ensure surface standardisation, the ceramic surfaces were finished and polished using the manufacturers' recommended kit (LUS80, Meisinger, USA). The firing of the specimens was done at 850°C followed by embedding in the autopolymerising acrylic resin. The discs were sanded with 400-grit followed by 600-grit wet silicon carbide paper until the ceramic discs were perfectly flush with the acrylic resin. To clean off the abrasive particles, the specimens were rinsed, dried, and subsequently treated with 37% phosphoric acid for 1 minute. All specimens were again rinsed under running water and dried.

The specimens were randomly divided into three light cure and three dual cure groups according to the cements used. Three dual-cure - Variolink Esthetic (I), RelyX Ultimate (II), and RelyX Unicem (III) and three light-cure - Variolink Veneer (IV), Variolink Esthetic (V), RelyX Veneer (VI) resin cements were used. Each group was further divided into two subgroups according to the surface treatment – micro-etch and acid-etch. The specimens were further divided into control and thermocycled subgroups. (Fig. 1)

The two surface treatments were micro-etching with Al<sub>2</sub>O<sub>3</sub> with particles size of 40 µm followed by etching with 10 % hydrofluoric acid (micro-etch) for two minutes and only etching with 10 % hydrofluoric acid (acid-etch) for two minutes. The debris was rinsed off and a special mould to provide a uniform area for cementation was placed at the center of each specimen. All resin cements were applied directly from a syringe on to the treated surface of the specimens. A 1-kg weight was placed on the top to form a uniform cemented layer. The specimens were then light cured for 40 seconds.



**Figure 1.** Flowchart showing the distribution of the study groups

Sixty specimens (5 from each subgroup) were subjected to thermocycling, 3500 times between 5°C and 55°C, with a dwell time of 30 seconds at each temperature and a transfer time of 15 seconds. The other 60 specimens that were not subjected to thermocycling served as the control group.

The specimens were tested for shear bond strength using a universal testing machine (Instron Corp, Canton, Mass., USA). 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/min. A knife-edge stainless steel chisel with a thickness 0.34 mm and diameter of 10 mm was used for loading. The shear load at failure was recorded by the software and the values were converted to stress in MPa.

### *Statistical Analysis*

The data was analyzed using software IBM SPSS v. 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. Three-way ANOVA with Bonferroni's correction for multiple group comparisons was used to test the interaction between factors: resin cement, surface treatment and thermocycling and its effect on the shear bond strength (MPa). P value less than 0.05 was considered statistically significant.

## **Results**

The mean and standard deviation for the shear bond strength at maximum load in MPa were compared using three-way ANOVA (Table 1). There was a statistically significant difference observed in the shear bond strength between the cements ( $p < 0.05$ ). There was a statistically significant difference seen in the shear bond strength of

**Table 1.** Shear bond strength of the tested cements per surface treatment and thermocycling

Surface treatment/ Cement	Shear bond strength (MPa) (mean ± SD)					
	Light-cure cements			Dual-cure cements		
	V	IV	VI	I	III	II
<b>Thermocycling</b>						
Acid	11.36 ± 0.06 <sup>a</sup>	6.95 ± 0.10 <sup>b</sup>	12.00 ± 0.12 <sup>a</sup>	9.42 ± 0.11 <sup>c</sup>	13.21 ± 0.16 <sup>d</sup>	12.31 ± 0.19 <sup>ad</sup>
Acid + Microetch	15.11 ± 0.35 <sup>A</sup>	8.50 ± 0.35 <sup>B</sup>	15.50 ± 0.96 <sup>A</sup>	11.15 ± 0.32 <sup>C</sup>	14.30 ± 0.74 <sup>AE</sup>	13.53 ± 0.44 <sup>DE</sup>
<b>No Thermocycling</b>						
Acid	12.13 ± 0.04 <sup>I</sup>	8.26 ± 0.06 <sup>II</sup>	14.08 ± 0.79 <sup>III</sup>	11.65 ± 0.26 <sup>I</sup>	13.37 ± 0.10 <sup>III</sup>	12.58 ± 0.53 <sup>I, III</sup>
Acid + Microetch	13.85 ± 0.56 <sup>ab</sup>	9.12 ± 0.19 <sup>B</sup>	15.96 ± 0.67 <sup>x</sup>	12.94 ± 0.23 <sup>a</sup>	14.30 ± 0.89 <sup>b</sup>	13.67 ± 0.26 <sup>ab</sup>

Three-way ANOVA;  $p < 0.05$  is significant.

Factor 1: Cement;  $p < 0.001$

Factor 2: Surface treatment;  $p < 0.001$

Factor 3: Thermocycling;  $p < 0.001$

Factor 1\*2;  $p < 0.001$

Factor 1\*3;  $p < 0.001$

Factor 2\*3;  $p < 0.001$

Factor 1\*2\*3;  $p = 0.087$

Different superscripts indicate significant differences across significant groups.

cements treated by different surface treatment ( $p < 0.05$ ). Within the acid etch group, the highest shear bond strength was observed by the dual cure cements III whereas the lowest shear bond strength was for light cure cement V followed by I which were significantly different from the other resin cements ( $p < 0.05$ ). Within the micro-etch group, the highest shear bond strength was observed for VI, whereas the lowest shear bond strength was for the V followed by I 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 as compared to the acid-etch group ( $p < 0.05$ ). Also, there was a statistically significant difference observed between the thermocycled and non thermocycled subgroups ( $p < 0.05$ ). Across all the resin cement groups, thermocycling significantly reduced the shear bond strength of the resin cements for both the surface treatments. However, the interaction between the three factors: surface treatments, thermocycling and resin cements did not demonstrate statistically significant differences between and within groups ( $p = 0.087$ ).

There was statistically significant interaction observed between the surface treatment and thermocycling ( $p < 0.05$ ). It was observed that the reduction in shear bond strength after thermocycling was more pronounced in the acid etch subgroup as compared to the microetch subgroup. For all resin cements tested with different surface treatments, there was a statistically significant difference within resin cements per surface treatment ( $p < 0.05$ ).

## DISCUSSION

The results of this study show that there are significant differences in bond strength between the thermocycled and non-treated specimens. Aging in water significantly affected the bond strength of the pre-treated specimens. This effect was more pro-

nounced for the specimens that received additional air abrasion with acid etching. Therefore, the null hypothesis tested in the study was that there is no difference in the bond strength of differently pre-treated and aged PLV cemented using different light-/dual-cure resin cements has been rejected.

The adhesive porcelain veneer complex has been proven to be very strong in vitro and in vivo. An optimal bonded restoration can be obtained especially if the preparation is done properly, correct adhesive treatment procedures are performed, and a suitable resin cement is chosen. The medium- to long-term esthetic maintenance of porcelain veneers is excellent, resulting in high patient satisfaction. Also, there are no adverse effects on the gingival health in patients with an optimum oral hygiene. However, the performance of the adhesive resin cements the natural oral habitat has been a topic of debate amongst clinicians<sup>27</sup>.

The accepted methods to simulate aging and to stress the bonding interface are long-term water storage and thermocycling of the bonded specimens<sup>28-30</sup>. This is typically performed with temperatures between 5°C and 55°C<sup>28-32</sup>. In several studies, thermocycling was combined with a second treatment, such as dynamic loading, which was termed artificial aging<sup>31,33</sup>. Most studies that used these methods revealed significant differences between early and late bond strength values<sup>34</sup>.

White et al.<sup>35</sup> showed that immersion of ceramics in water decreased their static strength and increased the crack velocity. Subramanian et al.<sup>36</sup> reported a decrease in flexural strength of aluminous and feldspathic porcelains when tested in water. They observed that the failure of the restorations and postoperative cracking can arise because of thermal variations. Moreover, the resin cement used for luting the laminate veneer may impose surface changes on the veneer when it is subjected to thermocycling.

Stacey<sup>37</sup> (1993) investigated the relative bond strength surface-treated porcelain and the effectiveness of silane treatment of the etched porcelain. He found that after thermocycling, etching the porcelain surface with acid did not create a sufficiently reliable bond with enamel for PLVs. Thermocycling did not significantly reduce the strength of etched enamel/composite resin/etched porcelain bonding when the porcelain was treated with silane. Silane treatment of the etched porcelain surface can be considered a practical and a necessary procedure. In the present study, it was observed that though thermocycling reduced the SBS of the resin cements, the reduction was significantly higher in the specimens which underwent only acid etching. Hence, it can be inferred that microetching is an advantageous procedure which helps in improvement of the durability of PLVs.

Understanding the mechanism behind the effects of water on the mechanical properties of polymers is of utmost importance. The sensitivity of resin-based materials to water depends on a multitude of factors such as the degree of monomer conversion, degree of polymer crosslinking, volume fraction of intrinsic nanometer-sized pores, and the quantity and presence of fillers<sup>9,38,39</sup>. One study found that an increase in water sorption was observed by increasing the ratio of triethyleneglycol dimethacrylate (TEGDMA) and urethane dimethacrylate to bisphenol-A-glycidyl dimethacrylate<sup>40-41</sup>. This possible effect of TEGDMA in the resin cement on water sorption and thereby

its effects on the mechanical properties of the resin cement after fatigue testing and thermocycling need to be investigated in future studies. This could provide a basis for future research for improving the stability of resin cements in the oral environment, thereby improving the longevity of PLVs.

Within the limitations of the present study, the following conclusions can be drawn:

1. Dual cure resin cements showed a higher Shear bond strength as compared to light cure resin cements.
2. Thermal cycling significantly decreased the shear bond strength for both ceramic surface treatments.
3. After thermocycling, the specimens with 10% HF surface treatment showed lower shear bond strength values when compared to those treated by sandblasting with  $Al_2O_3$  particles

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