Comparison of shear bond strength of resin cements to IPS empress 2 ceramic under four surface conditioning treatments: An in vitro study

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Abstract
1. To evaluate and compare the shear bond strength of four resin cements to IPS Empress 2 ceramic under four surface treatment conditions.
2. To compare the shear bond strength of light cure and Dual cure resin cements to IPS Empress 2 ceramic under four surface treatment conditions.

Materials and Methods:
The resin cements used in the study were Rely X™ veneer cement - light cure, Variolink II light cure and dual cure and Calibra dual care. The ceramic surface treatments were 1. Abrasion with 600 grit silicone carbide paper 2. Etching with 9.5% hydrofluoric acid for four minutes 3. Treating with silane for one minute and 4. Combination of etching and silane application. The ceramic used in the study was IPS Empress 2.

Results:
One-way ANOVA of shear bond strength of resin cements showed hydrofluoric acid etching followed by silane application produced bond strength in the highest statistical group and there were no significant differences among the four cements when hydrofluoric acid etching was followed by silane application. One way ANOVA of shear bond strength of light cure and dual cure resin cements showed that the dual cure resin cements showed higher bond strength.

Conclusion: Within the limitation of the study hydrofluoric acid etching followed by silane produced the best bond with the four resin cements. The dual cure resin cements produced higher bonds strength to ceramic.

Keywords: shear bond strength, ceramic surface treatment, resin cements

Introduction
In today’s fixed prosthodontic and aesthetic dentistry procedures, bonding techniques are becoming increasingly important. It ensures the durability of aesthetically pleasing all-ceramic restorations like porcelain veneers, inlays and onlays. The popularity of these restorations are primarily attributed to their esthetic qualities, fracture resistance, tissue acceptance and zero incidences of caries. According to Christensen [9] they have several advantages over direct composites, such as control of the polymerization shrinkage, more resistance to wear and better marginal adaptation and anatomic form. Above all, the success of these restorations is also attributed to its low debonding rate [15].

For many years, retention of crowns, inlays and onlays could only be attained by the use of standardized tooth preparations and by micromechanical interlocking of the luting cement into the irregularities present on the restorative surface and the tooth. The introduction of resin luting agents revolutionized the adhesive dentistry by providing an efficient bond between teeth and ceramic, thereby increasing the resistance to fracture and reinforcing the remaining tooth structure. Composition of the resin luting agents and their polymerization forms may influence their properties. Resin cements are low-viscosity versions of restorative composites containing varying proportions of resin and fillers. Organosilanes are used to bond filler particles to the resin matrix and create a durable, wear-resistant material. Depending on the polymerization process, resin cements can be of three types- chemically cured, light cured, or dual cured.
Dual cure resin cements can be used for cementation of most of the indirect restorations except for cementation of veneers, in which case a light-cured cement would be preferred. A strong resin bond relies on micro mechanical interlocking and chemical bonding to the ceramic surface, which requires roughening and cleaning for adequate surface activation [3]. Common treatment options are grinding, abrasion with diamond cutting instruments, air borne particle abrasion with roughening and cleaning for adequate surface activation [8].

60% by volume of lithium disilicate glass crystals. Ten wax generation of pressable ceramic material made of more than Materials & Methods 

sprues of 10 mm in diameter and 30 mm in height were invested and heat pressed with the IPS empress system using acid etching of porcelain for effective bonding to resin cements. The ceramic discs were embedded in acrylic using a specially designed mold former cementation assembly designed by Sree Chithra Thirunal Institute of Medical Science and Technology, Trivandrum (Fig 2). It consist of two parts, lower part with a mould space in the center, which can be divided into two equal halves. The upper part consists of a rectangular block of metal (5x4x1 cm) with two halves that is used for application of resin cement. All specimens were first sanded with 600 grit silicone carbide papers on a water irrigated grinding wheel, dried and subsequently treated with 37% phosphoric acid for one minute to clean off abrasive particles. All specimens were again rinsed under running water and dried. The sanded specimens were randomly divided into four main groups (24 each) (Fig 3). The four surface treatments tested represented one control and three surface treatments, commonly recommended by various manufactures. The surface treatment performed on each group were as follows

1. Group I – After sanding, no further surface treatment was performed to the control group.
2. Group II – Ceramic specimens were etched with 9.5% hydrofluoric acid (Porcelain etch, Vivadent USA) for 4 minutes. After etching the specimens were submerged in 10% solution of baking soda and water until the acid is neutralized. Then the specimens were cleaned in detergent solution and rinsed under running tap water for 5 seconds and dried with oil free compressed air from an air syringe for 10 seconds.
3. Group III – A silane solution (Mono bond S) was applied on the ceramic specimens with a tip applicator for one minute and allowed to air dry.
4. Group IV – Porcelain specimens were etched with hydrofluoric acid as described for group III and Silane solution was then applied as in group II.

Ceramic – Resin Cement bonding 

Each of the four main groups were divided into four sub groups (6 each) for each of 4 cements. The ceramic surface was treated as described above and, ceramic embedded acrylic mould was placed in the mold space and ceramic surface was covered with adhesive with a 4 mm diameter punched hole (Leitz, Germany). The margin of the tape was burnished to ensure complete adaptation on the flat surface. This created a standardized bonding area. The upper part of the mould former-cementation assembly was positioned so that the smaller hole is in line with bonding area. Teflon tubing with 4 mm diameter was adapted inside the smaller hole for easy separation of the cement cylinders later. The upper and lower parts were clamped together with machine screws. Each cement was mixed according to the manufacturer’s directions and applied through the smaller hole. The probe of light cure unit was introduced through the larger hole and cured. (Astralis 5, Vivadent)

Resin cements used were 

Rely X™ veneer- light cure (3M, ESPE) 
Variolink II- light cure (Ivoclar, Vivadent) 
Variolink II dual cure (Ivoclar, Vivadent) 
Calibra dual cure (Dentsply Caulk)

After curing, the machine screws were removed and the upper part was disassembled and the ceramic with the resin cements attached were placed in distilled water for 24 hours in an incubator.

Testing procedure 

The mounted specimens were locked in position into the stainless steel Jig on the lower member of the universal testing machine in shearing mode. (Fig 4). A stainless steel wire loop was engaged into the test specimen at ceramic – cement interface. The shearing force was then applied at the crosshead speed of 1 mm/min to the ceramic surface, until the debonding occurred. Shearing force required to debond the cements was recorded in Newton and converted to stress in Mega Pascal.
Result

The dependent variable for the present study was bond strength measured in MPa. The two independent variables were surface treatments (Abrasion with Silicon Carbide Paper, Application of Silane, Application of Hydrofluoric Acid, Combination of Silane + Hydrofluoric Acid), and cements (Rely X TM Veneer, Variolink II light cure and Variolink II dual cure, Calibra dual cure). The data were analyzed with One way analysis of variance (ANOVA), Student’s t test and Duncan’s multiple range test.

When different ceramic surface treatment groups were compared, the samples etched with hydrofluoric acid and treated with silane shows the highest mean bond strength values. The values were statistically highly significant (p <0.001). Variolink II dual cure cement showed higher shear bond strength values compared to all other cements in all groups. The values were significantly different from other cements in all groups except hydrofluoric acid + silane treated groups. (Table 1 & 2)

When shear bond strength of light cure and dual cure resin cements were compared on different surface treatment groups, the dual cure cements showed the higher mean shear bond strength values but the values were statistically significant only for silane treated group (p <0.01) and hydrofluoric acid +silane treated group (P<0.05). (Table 3)

Discussion

The introduction of lithium-disilicate glass-ceramic core veneered with a sintered glass-ceramic (for example; Ivoclar, Vivadent) offers higher fracture strength that allows them for the fabrication of short –span fixed partial denture (FPDs). IPS Empress 2 system is indicated for inlays, onlays, crowns, laminate veneers as well as three-unit bridges. Another advantage of IPS Empress 2 is the wear compatibility and thus the ability to re-establish proper canine disclusion and anterior guidance without abrading the opposing dentition. This has been a concern when utilizing conventional porcelain.

The present study evaluated the shear bond strength of four resin cements to IPS Empress 2 ceramic surface under four different surface treatment conditions. The ceramic conditioning treatments were
1. Sanding with 600 grit silicone carbide paper,
2. Etching with 9.5% hydrofluoric acid for four minutes,
3. Treating with silane primer for one minute and
4. The combination of etching and silane application.

The resin cements used were Rely X™ veneer cement – light cure, Variolink II light cure and dual cure and Calibra dual cure.

A strong, durable resin bond provides high retention, improves marginal adaptation, prevents micro leakage, and increase fracture resistance of the restored tooth and the restoration. The bonding of composite to ceramic materials has played an important role in aesthetic dentistry. In order to achieve a strong resin bond between ceramic and tooth surface, surface preparation of the ceramic material is very important. The bond strengths achievable by grinding of the treated surface alone were not sufficient. Mechanical roughening of the porcelain surfaces with a coarse diamond improves repair strength [4, 30]. Common treatment options are grinding, abrasion with diamond, abrasion with aluminum oxide, acid etching, and combinations of any of these methods. Acid etching with solutions of hydrofluoric acid (HF) or ammonium bifluoride can achieve proper surface texture and roughness [8].

In the present study 9.5% hydrofluoric acid etching for four minutes followed by silane application (HF + Silane) proved to be the most effective and reliable surface treatment. For all four cements, the bond strengths resulting from HF+Silane treatment were statistically as high as any other treatment. HF + Silane bond strength ranged from 10.8 MPa to 17.04 MPa. Some of the other surface treatments also yielded bond strengths in this range but not for all four cements. This finding is in agreement with the work of other authors who found that HF + Silane was consistently the most effective ceramic surface treatment, although it was not always statistically better than other treatment that involved roughening and silane application.

Airborne particle abrasion alone will not provide sufficient bond strengths [4, 15]. Excessive airborne particle abrasion induced chipping or a high loss of ceramic material and is there for not recommended for cementation of silica based all ceramic restorations. Kato et al. [15] compared airborne particle abrasion with different acid etching agents and found that hydrofluoric acid and hydrofluoric acid-sulfuric acid provided the highest and most durable bond strengths. According to Della Bona and Van Noort [10], the application of hydrofluoric acid removes the surface morphology created by the treatments prior to etching due to its aggressive potential. Hydrofluoric acid reacts with glass content of porcelain producing micro porosities on the porcelain surface similar to the micro porosities produced on the tooth surface when etched with phosphoric acid. While etching, the glassy matrix is selectively removed and crystalline structures were exposed. The number, size, and distribution of glass crystals influence the formation of micro porosities created by acid etching. Resin cement is thus able to flow into these etched surfaces to form a strong bond with porcelain. This is further proved by the scanning electron microscopic analysis of hydrofluoric acid etched ceramic surface and the surface sanded with 600 grit silicone carbide paper. The SEM of ceramic surface treated with 600 grit sand paper showed scratches only while the surface etched with hydrofluoric acid showed numerous micro porosities. (Fig. 5&6)

For the leucite-reinforced feldspathic porcelain IPS Empress, solution of 9% HF applied for 60 seconds were most successful. The lithium disilicate glass-ceramic IPS Empress 2 has a high crystalline content and exhibits significantly higher bond strengths than IPS Empress independent from surface conditioning. It seems that the ceramic microstructure has a significant influence on the fracture resistance of the composite ceramic adhesion zone [19]. Earlier studies by J.R. Calamia [15] (1983) and Stangle et al. [30] (1987) used varying concentrations of hydrofluoric acids to treat the surface of porcelain and to study its surface characteristic and bond strength. High concentration of hydrofluoric acid (20%) was used in the laboratory for many years. But because of its caustic nature, the current generations of hydrofluoric acid gels are made to concentrations of about 10%.

Albasheer et al (1990) found that Acidulated Phospho Fluoride (APF) gel produced superficial etched pattern on porcelain surface [2]. Lacey et al evaluated the bond strength of Inceram porcelain etched with APF for ten minutes and 9.5% hydrofluoric acid for four minutes [4]. They found no significant differences in the bond strengths between APF and hydrofluoric acid etched surface. They concluded that 1.23% APF gel can be a substitute for 9.5% Hydrofluoric acid gel to etched porcelain.
Suliman et al. found that surface roughness treatment has no significant effect on bond strength \([11]\). However, these authors treated all specimens with silane after each surface treatment. Roulet et al. found that acid etching with 10% hydrofluoric acid gel or 10% ammonium bifluoride was much more effective than air-particle abrasion or grinding \([23]\). Ozden et al. found application of silane on mechanically roughened ceramic surface is most effective in increasing bond strength \([26]\). When used in conjunction with a diamond bur, silane treatment resulted in bond strengths twice as high as those obtained with hydrofluoric acid etching. Thurmond et al. found that mechanical alteration of the porcelain surface with aluminium oxide air-abrasion and hydrofluoric acid etching followed by silane application produced the highest bond strength at 3 months \([30]\).

Kupiec and Wuertz reported that best bonds were obtained immediately after bonding and at 3 months when silane was used \([19]\). Kamada et al. found that a silane-coupling agent with or without phosphoric acid etching improved the shear bond strength between ceramic and each of the 4 luting agents that they studied \([17]\). Madani et al. found that when Panavia 31 was used with Clearfil silane on air particle abraded In-Ceram specimens, higher mean shear bond strength values were observed than those etched with 9.5% hydrofluoric acid. An increased concentration of hydrofluoric acid (from 5% to 9.5%) resulted in increased shear bond strength values \([20]\).

Silane coupling agents are used to improve the bond strength between composite and porcelain. Chemical bonding produced by a silane coupling agent on the porcelain surface is an important factor in improving the bond strength between resin cement and porcelain \([6]\). Silanes are bifunctional molecules, that bond silicone dioxide with the hydroxyl groups on the ceramic surface. They also have a degradable functional group that copolymerizes with the organic matrix of the resin. Silane coupling agents usually contain an alkoxyl silane group and a weak acid, which enhances the formation of siloxane bonds. Application of silane also increases the interfacial adhesion and thereby the wettability of the ceramic surface. In a study by Lacy et al. \([4]\) showed that airborne particle abraded silica based ceramic was not retentive unless silane coupling agent was applied. Some silane conditioners that contained carboxylic acid provided sufficient bond strengths even without HF acid etching. Sorensen et al. showed that ceramic etching and silanization significantly decreased the micro leakage, which was not achieved by exclusive silane treatment \([24]\). Silanes can be presented as single-phase pre-activated solutions or two component systems that has to be mixed in order to initiate the hydrolysis reaction. The silane used in the present study, Monobond-S, is a prehydrolyzed monofunctional g-methacryloxy propyl trimethoxy silane (MPS) diluted (1 wt. %) in a water-ethanol solution.

It follows that the application of silane after ceramic surface etching is a crucial step. Specimens that were sanded or etched with hydrofluoric acid, but had no silane treatment were associated with low bond strength. Even though the values of bond strength of resin cements to ceramic surface with silanization alone was not statistically significant but silanization after etching with hydrofluoric acid resulted in a bond strength of 10-17 MPa which was statistically significant in the present study. The hydrofluoric acid treatment alone, not followed by silane application provided a high bond strength of 11.4 MPa for Variolink dual cure cement samples only. This finding, which is in agreement with the reports of Kupiec and Wuertz \([19]\) and Kamada et al., \([17]\) is an indication of the importance of silane for long term bonding.

The results of the present study and others published previously, suggest that the efficiently etched ceramic surface in combination with a silane treatment usually provides the highest bond strength. The different result from study to study probably are due to the use of different porcelains, different hydrofluoric acid concentrations, etching times and different micro etching pressures and particles.

In the present study shear bond strength of light cure and dual cure cements were compared on different ceramic surface treatment groups. The mean shear bond strength of dual cure cements to ceramic were higher than the light cure cements but statistical significance was observed only for silane treated and hydrofluoric acid +silane treated groups. In this group dual cure cements showed Shear bond strength value of 15.39 MPa and of light cure cements were 10.99 MPa

Resin based composites also known as resin cements are the material of choice for the adhesive luting of all ceramic restorations. Composite cements have compositions similar to conventional restorative composites and contain inorganic fillers embedded in an organic matrix. Composite cements can be classified according to their initiation mode as auto polymerizing (chemically activated), light activated, or dual-activated materials. The differences in the polymerization mechanism are based on the chemical type of initiator. Self-cure resin cements use a peroxide-amine initiator, dual-cure resin cements use a combination of amine and photo initiator; and light-cure resin cements use a photo initiator only. Light curing and dual curing resin cements have been advocated for luting all-ceramic restorations. When comparing these cements, light polymerized materials offer the advantages of extended working time, polymerization on demand, and improved color stability \([12]\). However, for cementing all-ceramic restorations, adequate polymerization of a resin luting agent is a crucial factor to ensure optimal bond strength at the ceramic-resin luting agent-dentin interface, as well as optimal physical properties \([13]\).

Previous studies demonstrated incomplete polymerization of light-activated resin luting agents due to attenuation of the light energy by the restorative material \([6]\). The degree of this light attenuation is primarily dependent on the type and thickness of the restorative material \([3, 16]\). Black men et al. found that polymerization beneath ceramic inlays is to be safe up to 3 mm distance from the tip of a standard curing light \([5]\). Dual-activated composites offer extended working time and controlled polymerization, although chemical activators ensure a high degree of polymerization \([16]\). Most dual-activated resin cements still require photo polymerization and demonstrated inferior hardness when light polymerization was omitted. Auto polymerizing resin cements have fixed setting times and are generally indicated for resin bonding metal-based or opaque, high strength ceramic restorations.

Linden \([23]\) and peumans \([27]\) believed that a chemical catalyst might be desirable for porcelain veneer bonding especially in thicker (>0.7mm) porcelain veneers. Herrin have also asserted that the thickness of the veneer has a substantial effect on the light energy passing through the veneers. Dual-cure agents should not be used on thinner veneer because they do not polymerize as effectively as visible light activated resins and may be susceptible to discoloration with time. Peumans et al. \([27]\) agreed that in case of dual cure resin cement the catalyst is problematic because it causes discoloration of anterior porcelain veneers. They believed that in case of thicker porcelain veneers, an increase in exposure
time is sufficient to overcome the problem of unsatisfactory polymerization. In addition, Cardash concluded that dual cure cement should be preferred only for porcelain restorations that are 2 mm thick or greater [7]. In case of Porcelain Laminate Veneer (PLV) with 0.5 mm to 1 mm thickness requires only light cure material which can be used for reliable and safe bonding.

As stated earlier, when bonding to tooth structure is considered two different interfaces need to be optimized; the tooth-cement interface and the cement-ceramic interface. The debonding of restorations will occur at the weaker interface.

The present study evaluated the bond strengths at the ceramic cement interface. The mean shear bond strength values of resin cements to ceramic surface ranged from 2 MPa to 15 MPa. Among the four cements tested the Variolink II dual cure cements showed higher shear bond strength to IPS Empress 2 ceramic at different surface treatment conditions. Calibra dual cure showed high bond strength value at combination of surface treatment group (Hydrofluoric acid + silane). The bond strength of resin cements to enamel and dentine were studied separately.

Table 1: One-way ANOVA of shear bond strength of ceramic specimens under different surface treatment condition

| Group              | No. of samples | Mean  | SD    | F    | p value |
|--------------------|----------------|-------|-------|------|---------|
| Control            | 24             | 2.3995 | 0.9025 | 48.190 | <0.001 |
| Hydrofluoric acid  | 24             | 7.6951 | 2.7978 |      |         |
| Silane             | 24             | 6.3253 | 1.9737 |      |         |
| HF + Silane        | 24             | 13.1921| 5.2123 |      |         |

Table 2: One way ANOVA & Post Hoc Tests of shear bond strength of resin cements to ceramic under different surface treatment

| Ceramic surface treatment | Resin cements                      | No. of samples | Mean ± SD  | F    | p value |
|--------------------------|------------------------------------|----------------|------------|------|---------|
| Treated with 600 grit Silicon Carbide paper(control) | RelyXM veneer                      | 6              | 2.4708 ± 0.7594 | 3.843 | <0.05   |
|                          | Variolink II light cure            | 6              | 2.6380 ± 0.9500 |      |         |
|                          | Variolink II dual cure             | 6              | 2.9628 ± 0.8571 |      |         |
|                          | Calibra dual cure                  | 6              | 1.9283 ± 0.5035 |      |         |
| Treated with 9.5% hydrofluoric acid | RelyXM veneer                      | 6              | 7.1005 ± 1.4057 | 28.575 | <0.001 |
|                          | Variolink II light cure            | 6              | 7.5680 ± 0.9920 |      |         |
|                          | Variolink II dual cure             | 6              | 11.4983 ± 0.9957 |      |         |
|                          | Calibra dual cure                  | 6              | 6.6135 ± 1.6912 |      |         |
| Treated with silane      | RelyXM veneer                      | 6              | 3.9403 ± 0.5957 | 27.296 | <0.001 |
|                          | Variolink II light cure            | 6              | 6.5068 ± 1.2429 |      |         |
|                          | Variolink II dual cure             | 6              | 8.8150 ± 1.1361 |      |         |
|                          | Calibra dual cure                  | 6              | 6.0390 ± 0.5719 |      |         |
| Treated with 9.5% hydrofluoric acid + silane | RelyXM veneer                      | 6              | 10.8050 ± 4.3436 | 2.091  | >0.05   |
|                          | Variolink II light cure            | 6              | 11.1850 ± 2.9746 |      |         |
|                          | Variolink II dual cure             | 6              | 17.0450 ± 7.0270 |      |         |
|                          | Calibra dual cure                  | 6              | 13.7333 ± 4.2477 |      |         |

Table 3: Student’s t-test comparing shear bond strength of the light cure and dual cure resin cements under different ceramic surface treatments

| Surface treatment          | Resin cements | No. | Mean ± SD | t value | p value |
|----------------------------|---------------|-----|-----------|---------|---------|
| Control                    | Light cure    | 12  | 2.0822 ± 0.8137 | 1.805  | >0.05   |
|                           | Dual cure     | 12  | 2.7168 ± 0.9062 | -0.623 | >0.05   |
| Treated with 9.5% hydrofluoric acid | Light cure    | 12  | 7.3343 ± 1.854 | -3.256 | <0.01   |
|                           | Dual cure     | 12  | 8.0559 ± 3.8312 | -2.238 | <0.05   |
| Treated with silane        | Light cure    | 12  | 10.9950 ± 3.5549 | 1.6844 | <0.05   |
|                           | Dual cure     | 12  | 15.3892 ± 5.7998 | -2.238 | <0.05   |

Fig 1: Ceramic specimens (Disks of IPS Empress 2)
**Fig 2(a):** Mould former cum cementation assembly, (b) parts separated

**Fig 3:** Ceramic specimens embedded in acrylic with bonded cement

**Fig 4:** Shear stress testing in progress in Instron machine

**Fig 5:** Photomicrograph of ceramic surface treated with 600 grit silicone carbide paper. (a) 300x, (b) 3000x
Fig 6: Photomicrograph of ceramic surface etched with 9.5% Hydrofluoric acid for 4 minutes. (a).300x, (b). 3000x

Conclusions

The resin bond to silica based ceramics is well documented through numerous in vitro investigations. Preferred surface treatment methods are acid etching with Hydrofluoric acid solutions (2.5% to 10%) for 3-4 minutes and subsequent application of a silane coupling agent. The present study evaluated the shear bond strength of four different resin cements to IPS Empress-2 ceramic under four different surface treatment conditions.

The surface treatment used were
1. Sanding with 600 grit silicone carbide paper,
2. Etching with 9.5% hydrofluoric acid for four minutes,
3. Treating with silane primer for one minute.
4. The combination of etching and silane application.

The resin luting cements used were RelyX™ Veneers Light Cure Cement, Variolink II Light cure and Dual cure and Calibra Dual Cure.

Within the limitations of the present study and in regard to all four cements tested, bond strengths were highly dependent on surface conditioning. The following conclusions were drawn from the study.

1. Hydrofluoric acid-etching followed by silane application was the most effective and reliable surface treatment to bond IPS Empress 2 ceramic with the four cements tested.
2. Dual polymerizing cements yielded higher bond strength than light polymerizing cements. Scanning electron Microscopic analysis of hydrofluoric acid (9.5%) etched ceramic surface showed more porosities than the ceramic treated with 600 grit silicon carbide paper.

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