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RESEARCH ARTICLE

A COMPARATIVE EVALUATION OF SHEAR BOND STRENGTH OF THE PORCELIAN LAMINATES USING DIFFERENT SURFACE TREATMENTS: AN IN-VITRO STUDY

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ABSTRACT

Background: Porcelain laminate veneers rely on the synergistic bonding achieved between the porcelain and resin cement in order to survive the rigours of the oral environment. The enhancement of bonding through modification of the internal porcelain surface is advocated in order to increase the intimacy of the bond and may be achieved by exposing the porcelain surface to various treatments.
Objectives: To compare and evaluate the effect of different ceramic surface pre-treatments on the shear bond strength of porcelain laminates with resin cements to human teeth.
Methods: Forty samples of porcelain laminates (IPS Empress 2) were fabricated and randomly assigned into three different surface pre treatment conditions: (i) Group A: etched with 1.23% of APF gel for 10 minutes, (ii) Group B: sandblasted with 50 µm Aluminium oxide, (iii) Group C: etched with 5% hydrofluoric acid for 20 seconds, (iv) Group D: with no surface treatment.All samples were silanated before cementation with Variolink N resin cement. The shear bond strength was measured in a universal testing machine with cross-head speed of 1.0 mm/min. Data was compared by one-way ANOVA at 5% significance level.
Results: Surface pre treatment with 5% HF exhibited the highest mean shear bond strength (22.451 ± 2.710 MPa) followed by sandblasting (15.659 ± 3.569), APF gel (13.025 ± 1.618) and control (10.60 ± 1.384) group. There was a significant difference between 5% HF ($p<0.001$) and other groups.
Conclusion: Within the limitations of this study, it may be concluded that etching with 5 % HF produced favorable micromechanical retention.

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INTRODUCTION

Beauty of smile and esthetics of anterior teeth are considered synonymous. Any change in morphology and shade in relation to anterior teeth invites careful intervention of Prosthodontic treatment (Anusavice, 2003).A porcelain laminate veneer is an extremely thin shell of porcelain applied directly to the tooth structure. Porcelain laminate veneers have become a popular method of closing diastemas, restoring fractured and malaligned or worn teeth. They were also widely used in cases of enamel hypoplasia, tooth discoloration and intrinsic stains (Borges, 2007; Shillingburg, 1997).Tooth preparation is minimal and thus cementation process is vital for the clinical success of ceramic restorations. Ceramic restorations may be cemented with zinc phosphate, glassionomer, which requires adequate retention form. When this is compromised, resin luting systems are recommended (Borges, 2007;Shillingburg, 1997). Resin luting materials are a mix of monofunctional monomers with a variable amount of fillers of varying sizes, forms and composition. The amount of filler is reduced in comparison to restorative resins, in order to decrease viscosity and allow better adaptation of a rigid restoration to a cavity surface (Bagheri,2010).Based on the concept of "Surface Strengthening", hydrofluoric acid or Stripit solution was introduced in 1983 for etching porcelain laminate veneer restoration. Since then, lot of studies with different approaches like sandblasting, silane treatment, combination of acid etching and silane treatment were advocated for better bonding between porcelain to resin cement (Ozcan, 2001; Lee, 2003; Nagayassu, 2006). Etching ceramics with hydrofluoric acid, ammonium bifluoride, or acidulated phosphate fluoride gel creates sufficient resin bond strength by producing micromechanical retention, and the use of silane provides a chemical interaction, which is attributed to its bifunctional characteristics. A high proportion of porcelain's allows reaction of the silane agent both to the crystal portion of the

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treated porcelain and to the organic portion of the luting agent, thus modifying the porcelain surface texture (Ozcan, 2001; Lee, 2003; Nagayassu, 2006). Etching and silanization increases the surface energy and the wettability of the ceramic substrate. Bonding is usually obtained by the micromechanical retention provided by acid-etching of the ceramic surface and chemical coupling by application of a silane coupling agent (Nagayassu, 2006; Proenca *et al.,* 2008).Clinicians are often confused regarding the most effective way to treat the intaglio surfaces of indirect porcelain restorations prior to placement with various adhesives and luting resins. They are often equally perplexed about the "ideal" surface treatment for the intraoral repair of preexisting porcelain restorations. This is not surprising, as there appears to be no clear consensus in the literature, among "opinion leaders" or from dental manufacturers on exactly what the optimal surface treatment should, in fact be. Dental laboratory technicians also appear to lack standardized protocols on how they should treat the surface of finished porcelain (Alex, 2011). So, hereby we are making an attempt to address some of these questions, provide some useful general guidelines regarding the management of surface treatment of porcelain restorations by comparing different types of surface treatments with resin cement to ultimately achieve a successful long standing clinical success.

MATEREIALS AND METHODS

This invitro study was conducted to evaluate and compare the shear bond strength of porcelain laminates using different pre-surface treatments luted to the tooth structure using resin cement. Codes, batch numbers, and manufacturers of the materials tested are listed in Table 1.

A. Laminate fabrication

Forty recently extracted human permanent anterior teeth without any previous restoration were mounted in PMMA cold cure acrylic resin using custom made jig. The facial surfaces of the teeth were prepared to accommodate laminates using laminate preparation diamond bur. Facial reduction was 0.3 mm at the cervical third and 0.5 mm at the middle and incisal thirds and1 mm of incisal reduction with 1 mm height palatal chamfer was done for incisal and palatal surfaces with rotary instruments and a water coolant. All tooth preparations were completed entirely in enamel. Once the tooth was prepared Silicone separating media was applied on the surface. Then Wax pattern was fabricated with Press wax (DELTA) with uniform thickness of 0.6mm and then the wax pattern was kept in water for 10 minutes in order to relieve the residual stress. Then the Wax pattern was sprued and invested by phosphate bonded investment using auto mixer machine. The investment was allowed to set for 45 minutes before keeping in burn-out furnace (NEY, VULCAN 3-130) for wax elimination at 930^0C, then the ring was immediately placed in the pre heated (700^0C) pressable ceramic furnace with ceramic button and plunger. Once the pressing was over the ring was allowed for bench cooling. All procedures were performed with IPS Empress 2 materials and protocol. After divestment, the laminate fit was verified with green aerosol sprayed over the tooth surface. High spots on the laminates were removed with a diamond medium grit round bur. All ceramic veneers were then reduced to 0.3 mm at the cervical third and 0.5 mm at the incisal two thirds with green stones. Their dimensions were standardized again after measurement with an electronic Vernier caliper (DIGIMATIC) for the height and thickness. The laminates were then glazed in a ceramic oven.

B. Surface treatment

The samples were randomly divided into 4 groups (n=10 each) and the laminates were treated with different pre surface treatments as follows:

Group A: Ceramic veneers were treated with 1.23% APF gel for 10 minutes .Then the specimens were ultrasonically cleaned for 3 minutes in distilled water.
Group B: Ceramic veneers sandblasted with 50 µm aluminium oxide at 60 psi at 0.5 Mpa for 5 seconds at a distance of 10mm, then ultrasonically cleaned for 3 minutes in distilled water.
Group C: Ceramic veneers were acid etched with hydrofluoric acid of 5% for 20 seconds. Then the specimens were ultrasonically cleaned for 3 minutes in distilled water.
Group D: No pre-surface treatment (control group).

C. Cementation procedure

The surface treated ceramic veneers were cemented using variolink N resin cement as per the manufacturing instructions on the prepared teeth with light finger pressure and excess cement was removed with an explorer before an oxygen blocking gel (glycerin gel) was applied to the margin and the cement was further polymerized using a light cure unit (HIFLEX) for 40 seconds. The surface of the test samples was calculated using electronic Vernier caliper .In this manner, forty samples were cemented to the enamel with three different surface pre-treatment of porcelain laminates and the samples in the control group were cemented without any pre-surface treatment.

D. Shear bond strength test

In all the groups, luted teeth were stored in distilled water at 37^0 C for 24 hours and thermo cycled between 5 and 55°C for 5,000 cycles with 30-second dwell times. The shear bond values were measured in a Universal testing machine (AG-IS, SAHIMADZU) at a cross head speed of 1.0mm/min until fracture occurred and maximum load recorded for each specimen. The values were then divided by the surface area of the sample to obtain the shear bond strength values in MPa. Total of 40 test samples were tested in identical manner.The recorded shear bond strength values were analyzed statistically.

E. SEM analysis

All specimens were gold coated with a sputter coater (HITACHI, E-1010) for 180 seconds at 40 mA. They were then mounted on coded brass stubs and loaded using scanning electron microscopy (HITACHI, S-3400N) operated at 10Kv, and examined for the surface topography of the samples at 1000x magnifications by the same operator.

Statistical analysis

All the statistical tabulations were done using Microsoft Excel (Microsoft, U.S.A.). The SPSS (SPSS for Windows 10.05, SPSS Software Corporation, Munich, Germany) software package was used for statistical analysis. One-way ANOVA was used to compare the mean values of the four groups (A, B, C and D), with p value < 0.05 was considered statistically highly significant.

Table 1. The materials used for this study are as follows:

Sl. No	Material	Manufacturer	Type	Batch no	Chemical composition
1.	Variolink N	Ivoclar Vivadent, Schaan, Liechtenstein	Dual Cure resin cement	Catalyst N01584 Base N01552	BisGMA*, UDMA**, TEGDMA$^{+,}$ Barium glass, and silica fillers, YbF3^{++}
2.	Variolink N (Excite DSC)	Ivoclar Vivadent, Schaan, Liechtenstein	Bonding agent	M04952 Two-step/etch and rinse	HEMA$^{\#}$,DMA$^{\#\#}$, phosphoric acid acrylate, highly dispersed silicon dioxide/ethanol
3.	Monobond-S	Ivoclar Vivadent, Schaan, Liechtenstein	Silane coupling agent	N01595	Alcoholic solution of silane methacrylate
4.	IPS Ceramic etching gel	Ivoclar Vivadent, Schaan, Liechtenstein	Etchant	N39215	5% Hydrofluoric Acid
5.	Professional APF gel	Pascal	Etchant	091114	Acidulated Phosphate Fluoride

Table 2. Comparison between mean shear bond strength values of Group A (APF gel), Group B (sandblasting) ,Group C (HF acid) and Group D (control), test samples using One-way ANOVA

Group	Number of Samples	Mean Shear Bond Strength (MPa)	Standard Deviation	P–value
A	10	13.025	1.618	<0.001 HS
B	10	15.659	3.569	
C	10	22.451	2.710	
D	10	10.60	1.384	

P-value<0.05 denotes significance at the 5% level, HS – Highly Significant

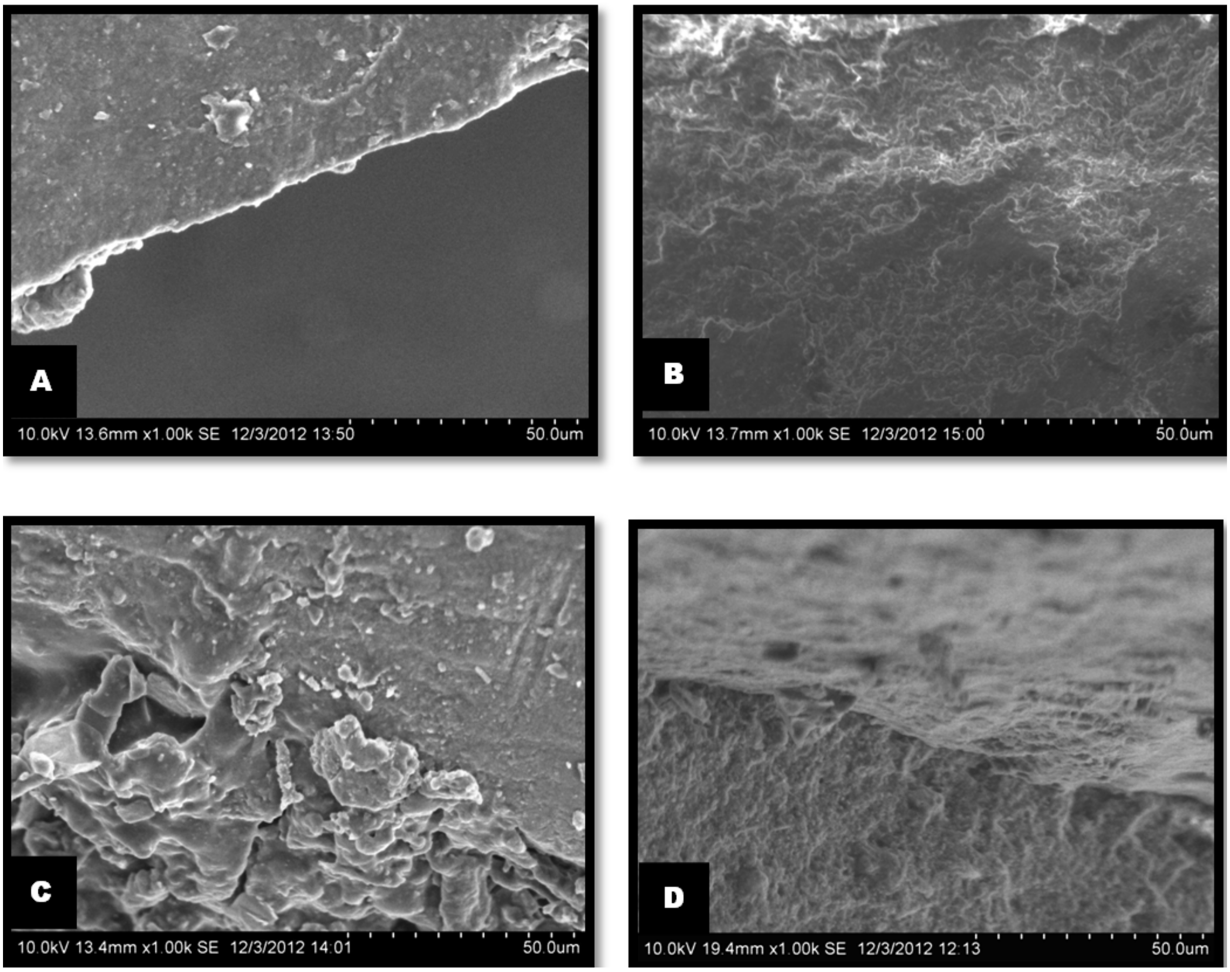

Figure 1. SEM of IPS Empress 2. A, Control. B, sandblasted with 50 µm aluminium oxide for 5 seconds.C, Etching with 5% hydrofluoric acid for 20 seconds. D, Etching with 1.23% APF gel for 10 minutes. (Original magnification x1)

RESULTS

On comparison between the mean shear bond strengths of Group A, Group B, Group C and Group D (Table II), using One-way ANOVA it was found that there was a statistically significant difference between the mean shear bond strength of the four groups. Group C (HF etching) had the highest mean shear bond strength followed by Group B (sandblasting) and the lowest shear bond strength value was observed in Group D (control).

The SEM photomicrograph of the Group A samples, surface pre-treated with 1.23% APF gel showed a slightly roughened surface with many irregularities and ill-defined micro porosities in some areas (Fig.1.D).Group B samples, surface pre-treated with 50 µm aluminium oxide showed an etched pattern with well defined small grains (Fig.1.B).Group C samples, surface pre-treated with 5% hydrofluoric acid showed an etched relief pattern with cotton wool appearance which presented as a porous and dendritic appearance, sufficient for creating micromechanical retention (Fig.1.C).Group D samples with no pre-surface treatment showed a uniform, smooth, flat appearance of the adhesive junction (Fig.1.A).

DISCUSSION

The porcelain veneer technique includes the bonding of porcelain laminate to the tooth surface using adhesive techniques. The success of the porcelain veneer is greatly determined by the strength and durability of the formed bond between the three different components of the bonded veneer complex, as there are the tooth surface, the luting agent and the porcelain veneer (Peumans, 2000). The aim of pre-cementation surface modification of the laminate is to modify its structure, to increase the surface area available for bonding and to create undercuts that increase the micromechanical retention and strength of the bond to the resin luting cement (Nagayassu, 2006; Spohr, 2003). IPS Empress 2 a pressable mutliphasic popular glass ceramic with a high degree of crystallinity was developed in 1998 with the same laboratory procedure and equipment used for the initial version of IPS Empress. It is composed of approximately 70% leucite, which confers improved mechanical properties. This material has generated considerable interest because of its ease of fabrication based on the lost wax technique and excellent esthetic feature. So this material was used in our study and veneering material consisted of an appetite glass ceramic (Proenca *et al.,* 2006; Addison, 2007). HF is an inorganic acid capable of etching glass surfaces (Alex, 2011). Horn in 1983 proposed etching porcelain laminate veneer restoration with either hydrofluoric acid or Stripit solution, and it is a standard protocol to bond etchable porcelains to teeth (Lee, 2003; Spohr, 2003). The HF acid attacks the glass phase of ceramics, partially dissolving it and creating microporous retention by exposing areas of crystals which make up the crystalline phase of the material (Filho, 2004). Silanes are a class of organic molecules that contain one or more silicon atoms. Rochette first advocated the use of silane as a coupling agent, and Lacy et al showed that silane treatment increased almost 5 times higher bond strength than that of acid etched porcelain surface (Lee, 2003). The bond with ceramics occurs via condensation reaction between the silanol group (Si–OH) of the ceramic surface and the silanol group of the hydrolyzed silane molecule, creating a siloxane bond (Si–O–Si) and producing a water molecule as byproduct (Filho, 2004). Moreover, silanization increases the wettability of ceramic surface, facilitating the spreading of resin cement that fulfils the superficial irregularities (Blatz, 2003). Resin luting cements are a mix of mono functional monomers with a variable amount of (55-70%, v/w) of filler of varying sizes, forms and composition. The amount of filler is reduced in comparison to restorative resins, in order to decrease viscosity and allow better adaptation of a rigid restoration to a cavity surface (Bagheri, 2010). They are classified according to their initiation mode as autopolymerising (chemically activated), photo activated, or dual activated materials (Borges, 2003). In this study, we had used dual-activated resin cements as they offer extended working time and controlled polymerization, although chemical activators ensure a high degree of polymerization (Borges, 2003). For a reliable and satisfactory union between ceramic and resin cement, a combination of chemical and mechanical retention must occur. Porcelain surface treatments modify its texture, increasing the micromechanical retention of the resin cement. Chemical retention is achieved with the use of silane agents that reacts with the vitreous compounds of the ceramic and with the composite organic matrix. An alternative for creation of micromechanical retention is aluminum oxide sandblasting. This technique was included in the present study for being a commonly employed procedure in prosthodontic laboratories and due to its adaptation to dental offices by means of the use of miniaturized devices, which facilitates the use of this technique. The mechanical retention provided by surface treatment is of paramount importance for proper adhesion. However, the association with a chemical procedure (silanization) is required for better results (Blatz, 2003). For this reason, a silane agent was used in all specimens before bonding in the present study.

In this study, 5% hydrofluoric acid–etching followed by silane application (HF _ Sil) proved to be the most effective and reliable surface treatment. For all pre-surface treatments, the bond strength resulting from HF _ Sil treatment were statistically highly significant. The possible explanation could be as follows, IPS Empress 2 glass ceramic is formed by elongated crystals of lithium disilicate. A second phase is composed of lithium orthophosphate. A glass matrix surrounds both crystalline phases. Hydrofluoric acid attacks the glass phase of ceramics, partially dissolving it to the depth of a few microns and, as a result, the lithium disilicate crystals protrude from the glassy matrix. This treatment significantly changes the surface morphology, increasing the surface area and irregularities within the lithium disilicate ceramic. This will favour the infiltration and retention of adhesive materials and made the ceramic surface more retentive (Borges, 2003; Filho, 2004; Proenca *et al.,* 2006; Alex, 2011).This is in accordance with the results of previous studies. This finding is in agreement with the work of other authors who found that HF _Sil was consistently the most effective ceramic surface treatment (Blatz, 2003). The different results from study to study probably are due to the use of different porcelains, different hydrofluoric acid concentrations and etching times, and different micro etching pressures and particles. APF gel, widely used for in-office fluoride application, consists of sodium fluoride, phosphoric acid, and hydrofluoric acid. It is safe for oral tissue, unlike hydrofluoric acid, which can produce tissue crash and burn. Consequently, APF gel has been proposed as an alternative for ceramic surface etching before bonding with composite resin (Boonlert Kukiattrakoon, 2007). Kewalin *et al,* showed that a seven to 10-minute application of 1.23% APF gel on a leucite containing porcelain produced a shear bond strength to composite similar to a four-minute etch with 9.6% HF resin (Boonlert Kukiattrakoon, 2007). Other studies have also shown that etching with APF, even with prolonged application times, results in very shallow etching patterns when compared to HF etching for much shorter time periods (Alex, 2011). In addition,10-minute etching with APF gel is relatively time consuming compared to hydrofluoric acid etching.

Conclusion

This study was done to Compare and Evaluate the Shear bond strength of four different pre-surface treatments of porcelain laminates to human dental hard tissue. Within the limitation of this study following conclusions was made:

1. There is significance differences exist in long-term durability to human teeth between with and without pre-surface treatment of porcelain veneers to human dental hard tissue.
2. There was significant difference between APF gel/sandblasting/acid etching with HF.

3. Although HF is considered to be a hazardous substance in causing various disorders and in spite of difficulty in storing HF, it is still considered best among various pre-surface treatments.

Abbreviations

APF- Acidulated Phosphate Fluoride
BisGMA*:Bisphenol-A diglycidyl ether dimethacrylate
DMA##: aliphatic dimethacrylate.
HEMA#: 2-hydroxyethylmethacrylate;
HF-Hydroflouric acid
HF _ Sil - hydrofluoric acid–etching followed by silane application
SEM- Scanning Electron Microscope
TEGDMA+: Triethylene glycol dimethacrylate
UDMA**:7,7,9-trimethyl-4,13-dioxo-3,14-dioxa-5,12-diazahexadecane-1,16-dimethacrylate;
YbF3++: Ytterbium trifluoride

Conflict of interest – No conflict of interest

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