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EVALUATION OF BOND STRENGTH OF A HEAT PRESSED CERAMIC TO RESIN CEMENT AFTER VARIOUS SURFACE TREATMENT: AN IN VITRO STUDY

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ABSTRACT
The aim of this study was to evaluate the bond strength of heat pressed ceramic to resin base cement after various surface treatments. Sixty six samples of 4 mm diameter and 3 mm thickness were fabricated from heat pressed ceramic ingots by lost wax technique. Samples were divided into three groups for three surface treatments. Group 1 was treated with 37% orthophosphoric acid for 20 seconds, Group 2 was sandblasted with 250 µm Alumina under 4.1 bar air pressure for 5 seconds and Group 3 was treated with 9% hydrofluoric acid for 20 seconds. After treatment all samples were cleaned, dried and silane coupling agent was applied for 60 seconds. Surface roughness was measured with scanning electron microscope and shear bond strength was measured with universal testing machine. There was highly significant difference in the mean value of shear bond strength among different groups $p<0.001$. ANOVA is highly significant also. Shear bond strength was found maximum in hydrofluoric acid etched samples followed by sandblasting samples and it was found to be least in ortho-phosphoric acid samples.

KEYWORDS: Press ceramic, surface roughness, shear bond strength, hydrofluoric acid, sand blasting, ortho-phosphoric acid.

INTRODUCTION

Ceramics are wildly used in Prosthodontics with the advent of new technologies such as heat press, injection mould, slip cast, glass infiltration and computer aided design or computer aided manufacturing. Ceramics are inert with good biocompatibility, mechanical properties and aesthetics. Ceramics have been successfully used as a core material in full ceramic dental restorations.[1]

Patient's demand for metal-free, tooth colored restorations has led to the introduction and development of all ceramic crown and fixed dental restorations. However, the indication for all ceramic restorations has been limited by numerous problems, such as low fracture strength of conventional dental ceramics and complex manufacturing techniques. The hardness of ceramic, similar to that of enamel, is desirable to minimize wear of the restoration as well as the enamel. The susceptibility of fracture is a drawback, particularly when flaws and tensile stress co-exist in the same region of a ceramic restoration. Chemical inertness is an important characteristic because it ensures that the surface does not react with oral fluids and food materials to form by-products. It also reduces the risk of surface roughening which increases susceptibility to bacterial adhesion over time.[2]

Long term retention of the restoration depends on the strength and durability of the bond of the luting agent to the tooth and porcelain surfaces. This bond may be created in two ways, either by micro mechanical preparation (sand blasting or chemical etching) or by application of silane coupling agent.[3] Various surface modification techniques have been proposed to improve the bonding strength between ceramic and tooth. Surface treatment of porcelain increases the surface area and creates micro porosities, which enhances the potential for mechanical retention of the luting agent. Mechanical procedure such as air borne particle abrasion with alumina particle and grinding with a mounted stone, have also been discussed in the literature.[4,5,6] Chemical surface treatment of dental ceramics can be accomplished by the use of orthophosphoric acid, sulphuric acid, nitric acid, hydrofluoric acid and ammonium hydrogen di-fluoride. In general, conditioning of dental material surfaces is the treatment by which the critical surface energy may be increased. The concept of critical surface energy tension is highly significant. When the surface tension of a liquid is less than the critical surface energy of a particular surface, spontaneous spreading of the liquid will occur, which improves the wetting of the surface. The critical surface energy is dependent on roughness and chemical composition of the substrate. Two approaches are possible in dentistry, pure chemical and physico-chemical conditioning. Often etchants, such as hydrofluoric acid that is washed off the surface, are considered as conditioners. The concept of a physical bond caused by etching of the porcelain surface remains strong among researchers and clinicians. Effective etching of the porcelain surface is considered an essential step for the success of bonded restorations.

Luting agents used with ceramic restorations are generally composite resins provided with more than one polymerization modes. Auto polymerized luting agents are indicated for cementing restorations whereas light polymerized agents are used for laminates veneers, whereas the light can penetrate through the restoration. Conversion of photoacivated material is affected by both the thickness and the transmission coefficient of ceramic material. Dual polymerized luting agents were developed to combine the properties of both autopolymerize and light polymerized agents to overcome insufficient conversion of photoactivated materials.[7,8]

The ceramic restorations are attributed to debonding. So it is necessary to evaluate the shear bond strength of surface treated press ceramic to dual cure cement.

MATERIALS AND METHOD

Sixty six samples were fabricated from heat pressed ceramic ingots (IPS e.max press, Ivoclar vivadent). Wax pattern with a diameter of 4.0 mm and a length of 12 mm cylinder was invested in a phosphate boned investment materials in an all ceramic ring. After setting of investment, ring was kept in burnout furnace at 700^{0}c for one hour to burnout wax. The ring was transferred to ceramic furnace for press cycle at 960^{0}c. After cooling sprue was cut and ceramic cylinder was retrieved. Three samples of 3mm were cut from each cylinder. Sixty six samples were fabricated using this method. All samples were smooth with 2500 grit silica carbide paper.

Surface Treatment of Ceramic Samples: Sixty ceramic samples were divided into three groups. Group 1 was treated with 37% orthophosphoric acid (3M ESPE, Germany) for 20 seconds, Group 2 was sand blasted with 250 µm Alumina (Karox. Bego, Germany) under 4.1 bar air pressure for 5 seconds and Group 3 was treated with 9% hydrofluoric acid (Ultradent Products, USA) for 20 seconds. After treatment all samples were cleaned with distilled water and dry.

Preparations of Extracted Molars: Sixty extracted permanent human mandibular molar teeth without any defect were chosen. The teeth were cleaned and sectioned horizontally with a disc to cut root of the teeth, occlusal surface was made flat and finished with 600 grit silicon carbide paper. All the teeth were embedded in autopolymerized acrylic blocks (Dentsply, India) and used for bonding with dual cure cement.

Silane coupling agent (Monobond S, Ivoclar vivadent) was applied to all samples for 60 seconds. All the samples were bonded to the tooth surface with dual cure resin cement (VariolinkII, Ivoclar vivadent) in accordance with the manufacturer's instructions. During cementation period, static load of 5 kilogram is applied to the samples. Excess cements were removed using "flicking- off" with a hand instrument. Light polymerization was applied for 40 second with light cure unit (Blue phase C5, Ivoclar Vivadend).

Shear bond strength between ceramic samples and the resin cement were conducted on an Instron testing machine. The machine applied an increasing shear load to the specimen at 1mm/min cross head speed till debonding occurred. Surface roughness was measure with the help of scanning electron microscope (Hitachi, S – 530, Japan). Six samples were analyzed under a SEM at 5000x magnification. Two samples from each group were electroplated with pure gold using an electronic ion coater and introduced into the SEM chamber three most prominent defects were identified. The length of these defects was measured and averaged using analyzing digital images software.

RESULTS

All the data recorded and analyzed with the help of the statistical package for social scientist (SPSS) computer software version 19. Group wise comparison showed shear bond strength values for all groups (table1). Hydrofluoric acid group 3 showed maximum shear bond strength 6.0127 MPa, orthophosphoric acid group 1 showed minimum shear bond strength 1.99462 MPa while sand blast group 2 showed shear bond strength of 3.08278 MPa.

Fig. 1: wax pattern and ring.

Fig. 2: Ceramic samples.

Fig. 3: Press furnace.

Fig. 4: SEM of orthophosphoric acid samples.

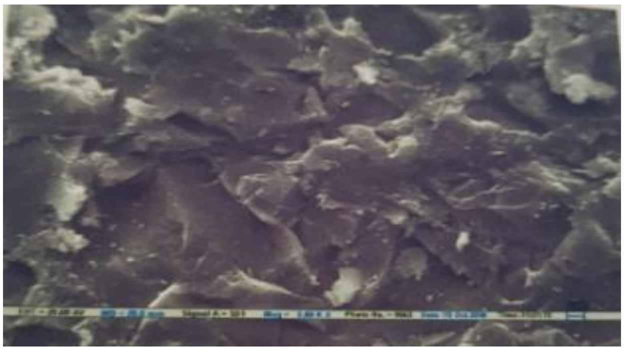

Fig. 5: SEM of sand blasted samples.

Fig. 6: SEM of hydrofluoric acid samples.

Table 1: Shear bond strength among various groups.

Group	N	Mean	Std. deviatiion	Std. Error	95%confidence Interval for mean		Minimum	Maximum
					Lower bound	Upper bound		
Orthophosphoric acid	20	1.99462	1.132279	0.253185	1.46647	2.52454	0.312	4.309
Sand blast	20	3.08278	1.387959	0.310357	2.43319	3.73236	0.778	5.906
Hydrofluoric acid	20	6.0127	2.766885	0.618694	4.71775	7.30764	0.98	10.05
Total	60	3.6967	2.535092	0.327279	3.04181	4.35158	0.312	10.05

Analysis of variance revealed statistically significant differences amongst all groups ($p<0.001$) table 2. Inter group comparison also showed statistically significant differences between groups ($p<0.001$).

Table 2: Analysis of variance. (ANOVA)

	Sum of Squares	DF	Mean Square	F	Significance
Between groups	172.756	2	86.378	23.852	<0.001
Within groups	206.419	57	3.621		
Total	379.175	59			

DISCUSSION

Lithium disilicate IPS e.max press was selected to its acceptable physical properties and durability with a relatively low refractive index and similarity to the natural teeth.[9] The durability of ceramic restoration depends on strong and stable bond between resin cement and the ceramic. Micromechanical attachment is one of the common methods to give an adequate bond between resin cement.[10-12] Different ceramic surface treatments have been introduced to improve the bond to the resin luting cement, roughening of the inner surface of the ceramic restorations increases the area available for the penetration of the resin based materials resulting in enhancement of the mechanical bond. Current available roughening techniques are: (1) grinding, (2) abrasion with diamond (or other) rotary instruments, (3) air abrasion with alumina (or other) particles, (4) acid etching (typically HF), and (5) combination of any of these techniques.[13,14]

Air borne abrasion with alumina was chosen because of its capacity to change the surface morphology of the ceramic and increase surface zone to gain micromechanical bonding, additionally it is considered a delicate procedure as significantly less material is removed from the surface and lower temperature was created.

37% ortho-phosphoric acid and 9% Hydrofluoric acid were also used in the present study as other surface treatment materials. It was assumed in other studies that they showed morphological changes such as pores and grooves which are considered to be important for the interlocking of the composite resin luting cement to the ceramic. Hydrofluoric acid treated samples on SEM showed pores and grooves of variable sizes. This observation supports the result of the previously published studies.[15-19] All these studies have compared the effect of hydrofluoric acid against various conditioning agents like ammonium hydrogen bifluoride, ortho-phosphoric acid and acidulated phosphate fluoride among others. The application of such acidic compounds, though advocated, was not found to be as effective for ceramic materials. The application time of 20 seconds used in this study makes it more clinically acceptable. The result of this study indicates that hydrofluoric acid produce higher shear bond strength than ortho-phosphoric acid or air borne particle abrasion methods. This result of present study was in accordance with the result of Ismael B Alashal et.al.[14]

Retentive characteristics of the sand blasted and the etched surfaces were evaluated using dual cure resin cement because it represent better properties in comparison to self etch self adhesive resin cement.[20] Silane was used in all the three groups. Bond strength is provided by combination of physical, mechanical and chemical factors. The physical component is dependent on the surface state of the substrate, which can be characterized by its surface energy. Etching or silanation of this surface increases or decreases the surface energy and the adhesion. Thus, etching the dental enamel by acids increases its surface energy. Energy characterization determines the surface energy of a substrate such as dental tissue or biomaterial, enabling adhesion potential to be evaluated. This is rarer for dental surfaces and biomaterials such as ceramics, which exhibit the topographic effects associated with highly polarized, high surface energy.

The present study confirmed that insufficient surface modification was produced by etching with orthophosphoric acid in a concentration of 37% and this appeared to affect the retention. This result was in accordance with the results of Lacy et. al. and Barkmeier et. al.[17,18]

There are some limitations of the present study also. Although, surface roughness was measured, volume loss from the ceramic surface, which could possibly jeopardize the fit of the restoration, was not estimated. Another limitation of the study was that the combined effects of higher acid concentrations and/or longer application periods were not observed. It should be considered that a suitable combination of acid concentration and etching time for the acids used might have some importance. Only one type of ceramic material and luting agent was used, further studies using different ceramic materials and other luting cements with different concentrations of etchant can be done.

CONCLUSION

Within the limitation of this study, it was concluded that The shear bond strength of lithium di silicate press ceramic was increased after surface treatment.

1) 9% Hydrofluoric acid showed maximum shear bond strength and create surface roughness more distinct than others.
2) 9% Hydrofluoric acid can be used as a regular etchant during bonding of lithium di silicate ceramic.
3) 37% orthophosphoric acid can't be used as an etchant for of lithium di silicate press ceramic.

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