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Behaviour of Shear Bond Strength to Different Surface Treatments of Hybrid Ceramic Bonded with Adhesive Resin Cement (An In-vitro study)

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Keywords

Shear bond strength, Surface treatment, hybrid ceramic, Cerasmart

Abstract

Purpose: to evaluate effect of both different surface treatments on the shear bond strength between resin cement and Cerasmart.

Materials and Methods: Forty five cerasmart specimens were cut with uniform thickness 1.5 mm. They were divided into 3 equal groups (n=15) according to the surface treatment applied: Group 1, includes 15 specimens that would receive no surface treatment. Group 2, includes 15 specimens that would receive hydrofluoric acid etching. Group 3, includes 15 specimens that would receive sandblasting. Each specimen was embedded in pink-wax prior to surface treatment. After surface treatments Bisco silane was applied for all specimens. Each Cerasmart specimen received one resin cylinder Irise of polyethylene tube having 2.5 mm diameter and 1.5 mm height were positioned over the plate surface and filled with resin cement then all samples were individually and horizontally mounted on a computer controlled materials testing machine (Model 3345; Instron Industrial Products, Norwood, USA). After bond strength test, all the failed specimens were examined using USB digital microscope to determine mode of failure. Then Data were explored for normality by checking the data distribution and using Kolmogorov-Smirnov and Shapiro-Wilk tests. One-way ANOVA were used to compare mean values. The significance level was set at $P \leq 0.05$.

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Results: The highest mean ± SD values of shear bond strength were recorded for **Sandblasting** group (9.55±1.88 MPa) followed by **Hydrofluoric** group mean ± SD values (7.58±1.24 MPa) meanwhile the lowest mean ± SD value was recorded with **Control** group (4.18±0.84 MPa). The difference between groups was statistically **significant** as indicated by one way ANOVA. **Conclusion:** Both Sandblasting and Hydrofluoric acid etching treatment in combination with a universal adhesive increases the shear bond strength of Cerasmart.

Introduction

The Goal for achieving the ultimate esthetic requires the array of different factors; proper case selection, clinical experiences and selecting the appropriate material considered to be the corner stone. In the last few years it has been noticed the demand for metal-free restorations has increased as patient's esthetics demands increased. So, there is a markable increase in chair side dental computer-aided design/computer aided manufacturing(CAD/CAM) machinable materials. Ceramics have successful natural looking outcomes, high mechanical properties, optical properties, chemical stability and biocompatibility, so they gained popularity in dental applications. A strong & durable bond between ceramics and resin cements is the key for success and long term clinical survival of the restorations**.** One of these blocks is Cerasmart which is nano-hybrid composite with inorganic fillers. It contains 71% silica and barium glass filler by weight. In addition, it has high fracture resistance, high strength under compressive loading, and higher wear potential than commonly used CAD-CAM materials with respect to the mechanical performance. But for a long-term survival of adhesive esthetic restorations there is another critical factor which is successful bonding between the ceramics, bonding agent and dental structure. So attempts have been done to improve bonding of resin cements (the materials of choice for the adhesive cementation of composite resin blocks) to ceramics via different surface treatments. Nano resin ceramic blocks are new in the market and study on them are not enough. The objective of this study is to evaluate effect of both different surface treatments on the shear bond strength between resin cement & Cerasmart.

Materials and Methods

1.1. Resin nano-ceramic blocks: Specific type of ceramic blocks was used which is Resin nano-ceramic blocks (commercially known as Cerasmart). Cerasmart consists of UDMA,Bis-MEPP,SiO2,barium glass. [1]

1.2. Adhesive resin cement: A dual-curing adhesive composite-based luting system was used that is supplied in a form of a syringe containing a base paste and a catalyst paste. It is known commercially as Duo-Link.

1.3. Acid Etch: Porcelain acid Etch consists of 9.5% buffered hydrofluoric acid gel (Commercially known as Bisco porcelain etchant) was used in this study to increase surface area of porcelain available for bonding.

1.4. Silane coupling agent: Silane coupling agent based on metha cryloxy propyl was used to enhance bonding between resin cement & cerasmart.

1.5. Aluminum-oxide powder: having a size of 50 microns.

2) Methodology:

2.1 Specimen grouping and study design

According to sample size calculation a total of 45 specimens obtained in this study would be sufficient with a power 90% & a significance level of 5% were divided into 3 groups (n=15). Group 1: Control group: Includes 15 specimens that would receive no surface treatment. Group 2: Intervention 1 group: Includes 15 specimens that would receive hydrofluoric acid etching. Group 3: Intervention 2 group: Includes 15 specimens that would receive sandblasting.

2.2 Preparation of substrates A total no of (7) Cerasmart blocks were cut into specimens using ISOMET 4000 with blade speed & continuous water irrigation. The specimens are with the following dimension (14mm*12mm*1.5mm). Uniform thickness is confirmed by digital caliper.

2.3 Surface treatment:

2.3.1 Hydrofluoric acid etching: Specimens treated with Hydrofluoric acid etching were embedded in pink-wax prior to acid application, in order to facilitate handling. Specimens surfaces were treated with Bisco porcelain acid etch having 9.5% buffered acid concentration for 60 seconds, then washed for 180 seconds with air/water spray and finally dried with oil/water free compressed air. The acid etching was used in this study to increase surface area of porcelain available for bonding by creating micropores into which uncured flowable resin penetrates to provide durable micromechanical interlocking.

2.3.2 Sandblasting: A custom-made metal frame was fabricated to hold the specimens during sandblasting in order to standardize the distance between specimens' surface and sandblasting nozzle which was centralized. Sandblasting was made using 50 microns aluminum-oxide powder at an angle of 90, distance 1cm for 10 seconds and 2- bar pressure. After surface treatments Bisco silane was applied for all specimens. A single layer of silane coupling agent was applied using a microbrush, allowed to dry for 60 seconds and then further gentle dryness was done for 10 seconds using oil/water free compressed air.

2.4 Application of resin cement: Each Cerasmart specimen received one resin cylinder Irise of polyethylene tube having 2.5 mm diameter and 1.5 mm height were positioned over the plate surface and filled with resin cement, creating resin cylinder having the same dimensions. Resin cement was injected into the irise of polyethylene tube lumen using the supplied auto-mixing tips with intra-radicular tips. After filling the polyethylene tube irises, resin cement was light-cured for 20seconds as recommended by the manufacturer, using Woodpecker LED light –curing unit of $1100mW/cm^2$. Polyethylene irises were removed after 24 hours to ensure a complete polymerization of the dual- cured resin cement, in order not to subject the resin cylinders to shear stress, polyethylene irises were sectioned vertically using blade No. (12).

2.5 Shear bond strength test: A circular interface shear test was designed to evaluate the bond strength. All samples were individually and horizontally mounted on a computer controlled materials testing machine (Model 3345; Instron Industrial Products, Norwood, USA) with a loadcell of 5 kN and data were recorded using computer software (Bluehill Lite; Instron Instruments). Samples were fixed to specially designed sample holder [metal block with central hole for sample housing] secured to the lower fixed compartment of testing machine by tightening screws. Shearing test was done by tensile mode of load applied at ceramic-resin interface using metal loop prepared from an orthodontic wire (0.014" in diameter) wrapped around the bonded cylinder assembly as close as possible to the base of the cylinder and aligned with the loading axis of the upper movable compartment of the testing machine at cross-head speed of 0.5 mm/min. The load required to debonding was recorded in Newton.

Shear bond strength calculation;

The load at failure was divided by bonding area to express the bond strength in MPa :

$$\tau = P/ \pi r^2$$

where ; τ =shear bond strength (MPa, P =load at failure(N)

π =3.14 and r =radius of resin disc(mm)

2.6 Fractography & Failure mode:

After bond strength test, all the failed specimens were examined using USB digital microscope (Scope Capture Digital Microscope, Guangdong,China) at 40x magnification and photographed using image analysis software (Scope Capture 1.1.1.1. Ltd Co.) to determine the nature of their failure modes. Failure type was classified into:

1) Adhesive failure: (no resin cement remained on the CAD/CAM ceramic surface).

2) Mixed failure: (some resin cement remained on the CAD/CAM ceramic surface and cracks formed within the CAD/CAM ceramic surface).

3) Cohesive failure: (failure occurred within the resin cement and cracks formed within the CAD/CAM ceramic or fracturing occurred within the CAD/ CAM ceramic surface).

2.6 Statistical Analysis: Data were collected and analyzed as mean, standard deviation (SD), range (Minimum – Maximum) for values. Data were explored for normality by checking the data distribution and using Kolmogorov-Smirnov and Shapiro-Wilk tests. One-way ANOVA were used to compare mean values. Tukey's post-hoc test was used for pair-wise comparisons when ANOVA test is significant. The significance level was set at $P \leq 0.05$ and 95% Confidence interval. Statistical analysis was performed using Graph Pad Instat (Graph Pad, Inc.) software for windows.

Results

Shear bond strength Descriptive statistics showing mean values and standard deviation of **shear bond strength** test results measured in mega-Pascal (MPa). The highest mean ± SD values of shear bond strength were recorded for **Sandblasting** group (9.55±1.88 MPa) followed by **Hydrofluoric** group mean ± SD values (7.58±1.24 MPa) meanwhile the lowest mean ± SD value was recorded with **Control** group (4.18±0.84 MPa). The difference between groups was statistically **significant** as indicated by one way ANOVA (F=57.42, P=<0.0001<0.05) followed by Tukey's pair-wise post-hoc test.

Failure mode analysis The observed modes of failure were predominantly adhesive or mixed failure in control group with no cohesive failure. For Sandblasting and Hydrofluoric groups all samples showed mixed failure with no record for adhesive or cohesive ones. Chi square test showed significant difference in failure mode distribution between groups (p=<0.0001 <0.5).

Discussion:

Recently, the revolution in dental ceramics in respect to optical properties, microstructure and wide range of indications, moreover the increase in demand and interest in achieving the ultimate esthetic concluded that ceramic restorations are used widely.[2] The main concern of bonding ceramic restorations to tooth structure is the bond strength at the two interfaces: tooth/resin interface and ceramic/ resin interface, as the weak bond at any interface will significantly affects the final bond strength so affecting the clinical success of the ceramic restoration. Cerasmart have a new different microstructure composed of resinous polymer and nanoceramics, and being one of the first materials manufactured using nanotechnologies, the previously mentioned points were considered to be the driving reasons for using Cerasmart in this study. A critical aspect in the durability of resin nano-ceramics is the optimum uniform thickness of the material, so according to many studies Isomet 4000 was used to perform cutting of specimens with even thickness. Isomet 4000 simulate the actual fabrication technique used for indirect restorations.[1,3] A major aspect in adhesion and enhancement the bond between indirect restorations and resin cements is the surface treatment. Adhesion between indirect restorations and resin cements depends mainly on micro-mechanical retention and chemical bonding, where surface treatment provides the micro-mechanical retention needed. According to ceramic composition and micro-structure different surface treatment protocols were applied.[4-6] Creating a microscopically porous, high energy and micro-retentive bonding surface in glass ceramics requires the application of hydrofluoric acid, while indirect composites and acid resistant ceramics should be treated by sandblasting. By classifying Cerasmart as a hybrid ceramic (Resin Nano Ceramics) both types of surface treatments were applied to evaluate the effect of each type of surface treatment on bond strength especially that sandblasting would affect the resin phase while etching would have some effect on the ceramic phase. As a conclusion the more effective surface treatment for Cerasmart was assessed in this study.

Concerning ceramic etching protocols it is clear that each material should be treated properly regarding to its composition, micro-structure and surface topography. Etching concentration and duration are considered to be an important aspect in respect to their effects on creating a retentive surface so influence the bond strength. Etching Glass ceramics having a valuable amount of glass/ silica compositions using 4-9.5% hydrofluoric acid was widely applied and proved to be a very effective etching protocol in creating the micro-mechanical retention required.[7-9] Another concern in etching ceramic surface is the duration of acid application. Several studies claimed that as etching duration increase, the surface roughness of the ceramic surface increase so providing a more retentive surface. in contrast to that **Canay et al.,2001**[10] assumed that increasing the duration of etching may lead to stress concentration in the adhesive interface, in addition to weaken the ceramic surface, which was supported by **Nagayassu et al., in 2006** . Manufactures of Vita Enamic (hybrid ceramic) and Cerasmart (flexible nano-ceramic) recommended acid etching as a surface treatment for 60 second. That was the reason of applying the etchant for one minute in our study**.**[11]The second applied surface treatment in this study was sandblasting. As mentioned previously that ceramic composition and microstructure determine the suitable surface treatment that should be applied. Surface treatments applied to provide the mechanical retention which is an important aspect in adhesion promotion, additionally a chemical bonding can be achieved by using a silane coupling agents. Silane considered to be another important aspect in ceramic/resin bonding, that promotes adhesion by increasing the critical surface energy of substrate so secure an even spreading of liquids, and reducing the ceramic surface tension allowing a convenient penetration of resin composite cements into the micro-retentive pores.[12,13]

Duo-Link dual-curing adhesive resin cement was also selected in this study. As claimed by its manufacturer a significant bond strength values where obtained after a pure chemical curing was applied, which considered being a very important aspect in resin/cement bonding as light-curing is not secured in many cases (opaque materials). Moreover being dual-curing resin cement provides intimate bond strength at the beginning of luting producers, where self-curing resin cements have poor initial bond strength.[14,15]

Shear bond strength test was selected to evaluate the bond strength in this study, due to its relative simplicity in application especially when compared to tensile bond strength test. In order to create resin cylinders over the substrate surface a polyethylene tubes were used. [16,17] This method was mainly chosen over using starch-based template method that was developed by **Tedesco et al., in 2013** [18]due it simplicity and ease of fabrication especially that a large number of specimens were fabricated in this study. After debonding of Cerasmart specimens in this study, Failure mode was evaluated which is classified into the following types: adhesive failure, mixed failure and cohesive failure. Cohesive failure of the luting resin exhibits the perfect bonding status that can be obtained, as the principal source of failure arises from flaws within the resin and not at the interface.

Taking a closer look on the results seeking to analyze and discuss the out findings of this study, it turns out that, sandblasting showed a higher significant difference than HF- acid etching. These outcomes were in agreement with the results of a study by **zamzam et al in 2014** [19] were they also reported a higher but non-significant difference of sandblasting results than those obtained by HF-acid etching on the micro-shear bond strength of the resin ceramic block.[20] The results of this study was in disagreement with **H.Ozdemir AND L.Aladag, 2017** [21] who reported that HF acid etching showed higher BS values than sandblasting and this may be attributed to the difference in the structure of the ceramic blocks used as mentioned before the composition of the ceramic should be considered to determine the surface treatment method as the ceramic structure directly affected the bonding. While both researches agreed that the control group showed the least bong strength. With respect to that, Cerasmart is considered to be a composite block after all, and recommended to treat its surface by sandblasting rather than acid-etching. As mentioned before, according to materials composition and micro-structure a suitable surface treatment should be applied, in order to provide a micro-retentive surface so enhancing the bond strength of the indirect materials.[22] Cerasmart described as a resin nano-ceramic blocks. In this study results of both surface treatments; sandblasting and HF-acid etching were comparable as mentioned, which could be explained that, due to the hybrid composition of Cerasmart each of the surface treatments applied would have an effect on either the resin part or the ceramic part. While analyzing fractography and failure mode, it was found that the only group that had adhesive failure was the control group which its specimens had no surface treatment While the two other groups which had the intervention either Hydrofluoric acid or Sandblasting had mixed failure only. So Failure mode analysis supported the results of shear bond strength test.

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