

Bond Strength of Hybrid Ceramic to Different Foundation Materials

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ABSRACT

Objective: This study was carried out to evaluate the effect of different surface treatments on the shear bond strength of hybrid ceramic material.

Materials and methods: A total of forty two machinable hybrid ceramic discs of Vita Enamic (VE) (8×3 mm in dimensions) were fabricated using CAD/CAM system. All discs were divided into 2 main groups (n=21) according to the type of the surface conditioning that was used: group (S): sandblasting with aluminum oxide particles (Al_2O_3) , group (SP): sandblasting with Al_2O_3 particles and ceramic primer. Each main group was subdivided into 3 subgroups (n=7) according to the type of the foundation material that was used: subgroup (C): composite resin, subgroup (G): glass ionomer, and subgroup (D): dentin. Each foundation material disc was cemented to its corresponding VE disc using adhesive resin cement (Multilink® N). One hour after cementation, bonded specimens were stored in water bath at 37°C for 6 months followed by thermo cycling for 10000 thermal cycles. Shear bond strength was recorded for all specimens using universal testing machine, all data were tabulated and analyzed statistically. Scanning Electron Microscope (SEM) was used for failure mode examination.

Results: There was statistically significant difference of shear bond strength (SBS) between tested groups treated with sandblasting/primer indicated that foundation material had statistically significant effect on SBS. The highest mean SBS values at maximum load were among CSP group (19.90 ± 3.23) , GSP (16.92 \pm 2.83) and GS (8.87 \pm 2.62), then CS (8.42 \pm 1.61), DSP (8.06 \pm 1.91), and the least was DS (6.49 ± 1.87) .

Conclusion: The results of this in vitro study showed that the ceramic material treated with sandblasting followed by primer application,

increased SBS more than sandblasting only. Regarding the foundation material, composite showed the highest bond strength among other foundation materials, so composite was recommended to be the foundation material of choice.

KEYWORDS: Hybrid ceramics, Surface treatment, Ceramic primer, Resin cements, Bond strength.

I. **INTRODUCTION**

Digital dentistry has been recently introduced and has become a new challenge for
dental practitioners. Computer-aided Computer-aided design/computer-aided manufacturing (CAD/CAM) technology is broadly used in daily dental practice due to its advantages such as its speed, ease of use, and quality of therapy. **(1)** This technology can be used in both the dental laboratory and the dental office with multiple applications which include the fabrication of indirect restorations (inlays, onlays, veneers and crowns), fixed partial dentures, implant abutments, full-mouth reconstruction and orthodontics. $(1, 2)$

CAD/CAM technology was developed in order to ensure the sufficient strength of tooth restorations, to improve esthetic restorations with a natural appearance and to make the techniques easier, faster and more accurate. ⁽³⁾ More specifically, digital scans of the denture provide faster and easier treatment in comparison with the conventional impressions because casts, wax-ups, investing, casting and firing are eliminated. Moreover, having a milling machine on-site means that patients can receive their permanent restorations at the first appointment without the need to have provisional restorations, which take time to fabricate and fit. The quality of CAD/CAM restorations is high because measurements and

fabrication are precise due to the applied digital technology. **(4)**

On the other hand, there are also some disadvantages of CAD/CAM technology, most important among these, are the initial cost of the equipment and software and the need to spend time and money on training. **(2)** Indirect tooth restorations are a very common indication for dental CAD/CAM technology. **(5)** The CAD/CAM materials of choice for different types of restorations are either ceramic or composite. Recently, there has been an evolution of CAD/CAM composite materials due to their improved physical and mechanical properties in comparison to their ceramic counterparts, which were achieved by changes in their manufacturing methods (high pressure, high temperature) and structure (glass ceramic networks).**(6,7)**

CAD/CAM composite materials present less hardness and stiffness compared to ceramics, and as a result, the opposing tooth tissues are subjected to less wear clinically and they are easily fabricated by the milling machine. Furthermore, composites are easily fabricated, repaired and they are less brittle than ceramic, **(8)** leading to less chipping and crack formation during manufacturing, **(9)** and to improved marginal quality (potential thickness up to 0.2 mm). $(10, 11)$

CAD/CAM composite materials can be classified based on their microstructural geometry into two main categories: a) resin with dispersed fillers and b) polymer infiltrated ceramic networks (PICN). **(12)** The first category includes composite blocks containing a basic monomer type [bisphenol A diglycidylmethacrylate (BisGMA), urethane dimethacrylate (UDMA), triethylene glycol dimethacrylate (TEGDMA), etc.] as an organic matrix with dispersed filler particles (silica, zirconia, barium glass, etc.). **(5)** The second category**:** PICN (Vita Enamic) materials consist of a three dimensional ceramic network which is

infiltrated with a monomer mixture, offering a higher Weibull modulus and making the material less brittle than glass ceramics . **(13,14)**

Indirect restorations made by CAD/CAM composite materials are bonded to the tooth surfaces using resin cements. The increase of the bond strength between indirect CAD/CAM restorations and resin cement is essential to improve fracture resistance and to preserve the marginal integrity of restorations. (**15, 16)**

To achieve an adequate bond to CAD/CAM composite restorative materials, mechanical or chemical pre-treatments to the bonded surfaces are necessary . **(17,18)** Chemical bonds between resin cement and resin-based restorative material, as well as the application of primers in order to wet polymeric resin surfaces, significantly enhance the adhesive bonding. **(17,19,20)** In addition, micromechanical pretreatments by sandblasting with aluminum oxide particles (A_1, O_3) can also improve the bonding of the surfaces. **(21)**

Surface modification achieved by sandblasting with Al_2O_3 particles creates a microretentive surface that enables the mechanical interlocking of the resin cement. **(22)** This micromechanical interlocking of the interface between the two bonding surfaces is strongly dependent on their surface roughness and surface morphology.⁽²³⁾ In view of the limited research on the surface treatments of the CAD/CAM composite blocks and the need to evaluate the ideal surface characteristics of the material to achieve the best adhesion to tooth tissues. Thus, the aim of this invitro study was to evaluate the effect of surface treatments on the shear bond strength of hybrid ceramic to different foundation materials.

The null hypothesis of this in-vitro study was that different foundation materials and surface treatment methods have no effect on shear bond strength of hybrid ceramic.

II. MATERIALS & METHODS

Materials used in this study and their basic compositions are shown in **(Table 1)**.

TABLE (1): Description of materials utilized in the study.

Machinable Hybrid Ceramic Discs Fabrication

Forty- two Vita-Enamic (VE) discs with the dimensions of 8 mm diameter and 3 mm thickness were fabricated using CAD/CAM technology as follow: composite resin discs (8×3 mm in dimensions) were fabricated using specially designed teflon mold then scanned using Ceramill Map 400+ (Amann Girbach, Germany) for obtaining standardized ceramic discs.

VE discs were wet milled from VE block (Amann Girrbach, Austria) by using ceramill®

Motion 2 CAD/CAM machine (Amann Girrbach, Austria). The discs were wet ground on only one surface using 600 grit silicon carbide (SiC) paper. All discs were carefully checked using magnifying lens and examined for any surface defects. Thickness and diameter of all discs were checked at different points of each disc and at the margin using a digital caliper.

All discs were finished and polished by using VE polishing set technical as manufacturer's

instructions. The pink polishers of the VE polishing set was used with water at (7000-10000 rpm). The high gloss polishing was done with the grey diamond-coated polishers of the VE polishing set at (5000-8000rpm).The untreated surface was marked by red color water proof pen to be easily identified from the treated surfaces. All discs were cleaned with alcoholic swab, and ultrasonically cleaned after milling then carefully held with straight tweezer to keep the surfaces of the discs untouched. All discs were divided into two main groups with 21 discs in each one according to their surface treatments then each group was wrapped in closed sterilization bags to be ready for surface roughening and cementation procedure.

Composite Resin Discs Fabrication

A total number of 14 composite resin discs were fabricated using custom made resin pattern with numerous holes (4 mm internal diameter \times 3 mm thickness). The hole was incrementally filled in 2 increments with composite resin (SDI LUNA) that was light polymerized with light curing unit (COXO, China) for 20 sec for each increment to fabricate the composite resin discs. Composite discs were inspected for any defects after removal from resin pattern.

Glass Inomer Discs Fabrication

A total number of 14 glass ionomer discs were fabricated using custom made resin pattern with numerous holes (4 mm internal diameter \times 3 mm thickness). Glass ionomer discs were prepared by mixing powder with liquid according to manufacturer's instructions, and then pattern holes were filled with the material. After 2 min. of material setting, glass ionomer discs were inspected for any defects after removal from resin pattern.

Dentin Discs Fabrication

A total number of 14 freshly extracted sound human molars extracted for periodontal and orthodontic reasons, were gathered from healthy patients with the age range from $30 - 45$ years following approval of the ethics committee of Faculty of Dentistry, Mansoura University. The patients were informed about the use of their extracted teeth in this study, after cleaning, scaling and infection control standards, teeth were stored in distilled water that was changed daily till fabrication of dentin discs. Dentin discs were fabricated by Computer Numerical Control machine that's commonly called CNC. Dentin discs were fabricated (4 mm diameter \times 3 mm thicness) and inspected for any defects.

Surface Conditioning of Ceramic Discs

 According to the manufacturer's instructions, the discs were cleaned in ultrasonic bath (CD-4820, Codyson,China) for 5 min using 95% ethyl alcohol placed in plastic cup in order to remove any remaining acid then dried with oil-free air stream for 3 sec.

Group 1 Sandblasting with Al_2O_3 (50 μ m).

The discs were sandblasted (Ney; Blastmate II, Yucaipa, CA) with 50 μ m Al₂O₃ for 20 sec; 2 bar pressure was maintained for air abrasion. Discs were mounted in a special holder forming right angles where the distance between the nozzle and the surface of disc was 10 mm. The discs were cleaned in distilled water, and then were air dried.

Group 2 Sandblasting with 50 μ m Al₂O₃ and **priming**

The discs were sandblasted as mentioned before for group 1, then discs surfaces were treated with porcelain primer applied by micro brush for 3 min on the bonding surface, then fully removed with a powerful jet of air/water spray according to manufacturer's instructions.

Bonding procedures

Foundation material discs were cemented to previously treated surfaces of VE discs using resin cement (Multilink®N, ivoclar vivadent, Liechtenstein) for both groups according to the manufacturer's instructions. Multistep adhesive resin cement (Multilink N) base and catalyst pastes were dispensed into equal mounts, automixed through the disposable automix tip and applied on the treated surface of ceramic discs. Ceramic discs were secured to a specially designed device to deliver a constant load of 2 Kg for 5 min on the foundation material discs during cementation. Each foundation material disc was then placed onto its ceramic disc and the constant load 2 Kg was applied on the foundation material disc using loading device, excess resin cement was removed with a brush then curing was done using light cure unit (COXO, China) from four directions for 40 sec. from each surface for a total of 160 sec. and the constant load was left for 5min.**(24)**

Artificial aging (Thermocycling)

One hour after adhesive bonding of the specimens, artificial aging procedure was performed for 6 months for all tested groups that were held in a water bath at 37°C. Subsequently, the specimens were subjected to 10,000 cycles of

thermocycling in distilled water using a thermocycler system (Julabo GmbH, FT200, Seelbach, Germany) between 5°C and 55°C with 30 sec dwells times and 6 sec transfer time . **(25)**

Shear bond strength Test

Specimens were placed inside the testing device, which was fixed in a universal testing machine (Instron 3350, Instron industrial products, Grove City, US). Shear loading was applied at the interface between the cement and ceramic surface at a cross-speed of 1 mm/min. The maximum debonding force (N) for each specimen was recorded and used in calculating the SBS value (in MPa), according to the equation: $SBS = N/A$, where A is the cross-sectional area (in mm). **(24)**

Failure pattern analysis

The bonding surface of the deboned specimens were evaluated with optical reflection microscope (JEOL.JSM.6510LV, Japan) at 10x magnification to determine failure pattern. The failure patterns were classified into 3 types . **(26)**

1. Adhesive pattern of failure: failures between resin cement and ceramics or between resin cements and foundation materials (at interface).

2. Cohesive pattern of failure: failure took place in the foundation material discs or in cement layer.

3. Mixed pattern of failure: involving cohesive failure of the cement and adhesive failure between ceramic and resin cement. Further analysis of representative specimens of each failure pattern was exmined utilizing Scanning Electron Microscope (SEM) (Quanta 250-FEG,FEI, Netherland) with a voltage of acceleration and working distance of 10 mm at different magnifications (100x,500x,1000x,2000x,3000x) .

Statistical analysis

 Statistical analysis of data was performed with SPSS 20 in several steps.

III. RESULTS

Shear Bond Strength (SBS) results

Two- way ANOVA and Serial One-way ANOVA tests were utilized to statically analyze the data. When ANOVAs tests showed significance, the Post-Hoc Tukey (HSD) test was used for comparing the mean of each two tested groups. The mean difference was significant with ($P \le 0.05$). Two-way ANOVA test showed that, type of

foundation materials and surface treatments had statistically significant effect on SBS ($p<0.0001$ & p<0.0001, respectively). Also the interaction between the studied independent factors had a statistically significant effect on bond strength (p<0.0001). **(Table 2)**

One-way ANOVA test showed no statistically significant differences in SBS between tested groups at the level of foundation material (p=0.1). However, it showed statistically significant difference in the SBS between tested groups at the level of surface treatment (p=0.001). **(Table 3)**

Quantitative data were described mean and standard deviation. Significance of the obtained results was judged at the 5% level. The used tests were F-test (ANOVA) For normally distributed quantitative variables, to compare between more than two groups, and Post Hoc test (Turkey) for pairwise comparisons. The highest mean Bond strength at maximum load was among CSP group (19.90 \pm 3.23), GSP (16.92 \pm 2.83) and GS (8.87 \pm 2.62), then CS (8.42 \pm 1.61), DSP (8.06 \pm 1.91), and the least was DS (6.49 \pm 1.87). (**Table 4)**

Post Hoc Tukey tests showed statistically significant difference of SBS between tested groups GS and GSP, CS and CSP (P=0.000). However there was no statistically significant difference between tested groups DS and DSP (P= 0.83).). There was no statistically significant difference between mean SBS of tested groups GS and CS $(P=1)$, GS and DS $(P=0.4)$, CS and DS $(P=0.7)$, GSP and CSP $(P=0.2)$. However there was statistically significant difference between tested groups GSP and DSP, CSP and DSP (P=0.000). **(Table 5)**

Scanning Electron Microscopy (SEM)

In this in-vitro study, failure pattern of all groups was evaluated using SEM at different magnifications. Failure pattern of all deboned specimens showed mainly mixed failure in which adhesive failure occurred between hybrid ceramic and cement with remnants of resin cement adherent to the ceramic discs. **(Fig. 1)** Some of specimens showed adhesive failure between ceramic and resin cement as the specimen surface was free from any remnants of resin cement. **(Fig. 2)** On the other hand, cohesive failure pattern was minimum. **(Fig. 3) (Table 6)**

TABLE (2): Two-way ANOVA test used to compare between foundation materials and types of surface treatments on shear bond strength

a. R Squared=.836 (Adjusted R Squared=.814) *Indicate statistically significant difference.

TABLE (3): Serial one-way ANOVA results of foundation materials and surface treatments

*Indicate statistically significant difference.

TABLE (4): Descriptive analysis of the studied cases according to Bond strength

	Foundation materials		
	Mean \pm SD.	Mean \pm SD.	Mean \pm SD.
Level of treatments			
	8.87 ± 2.62	8.42 ± 1.61	6.49 ± 1.87
SP	16.92 ± 2.83	19.90 ± 3.23	8.06 ± 1.91
Total	12.90 ± 4.93	14.16 ± 6.44	7.27 ± 1.99

Data was expressed using Mean \pm SD for 7 replica each SD: Standard deviation

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*Indicate statistically significant difference.

Fig. (1) Representative SEM micrograph from group GSP, (A) and group CSP, (B) demonstrating mixed failure pattern between hybrid ceramic and luting cement.

cement/ceramic interface. Luting cement.

IV. DISCUSSION

This in vitro study aimed to evaluate the effect of surface treatments on the shear bond strength of hybrid ceramic to different foundation materials. There was difference in the shear bond strength based on type of foundation material and type of surface treatment, so the null hypothesis of this study was rejected.

Vita Enamic material was chosen as it is new type of material consists of a three dimensional ceramic network which is infiltrated with a monomer mixture, offering a higher Weibull modulus and making the material less brittlle.**(13, 14)** Vita Enamic materials present less hardness and stiffness compared to other ceramics, and as a result, the opposing tooth tissues are subjected to less wear clinically and they are easily fabricated by the milling machine. Furthermore, composites are easily repaired and they are less brittle than ceramic, **(8)** leading to less chipping and crack formation during manufacturing. **(9)**

To achieve an adequate bond to vita enamic materials, mechanical or chemical pretreatments to the bonded surfaces are necessary. **(17, 18)** Chemical bonds between resin cement and resinbased restorative material, as well as the application of primers that contain phosphoric acid monomers in order to wet polymeric resin surfaces, significantly enhance the adhesive bonding. **(17,19,20)** In addition, micromechanical pretreatments by sandblasting with aluminum oxide (A_1,Q_3) particles can also improve the bonding of the surfaces due to increasing the surface area and chemical activation of the bonding surface by removing organic contaminants. **(21)**Surface modification achieved by sandblasting with $Al₂O₃$ particles created a microretentive surface that enables the mechanical

Fig.(2) Representative SEM micrograph from Fig.(3) Representative SEM micrograph from group GS demonstrating adhesive failure at group CS demonstrating cohesive failure within lui group CS demonstrating cohesive failure within luting

interlocking of the resin cement. **(22)** This micromechanical interlocking of the interface between the two bonding surfaces is strongly dependent on their surface roughness and surface morphology. **(23)**

Thermocycling was used at 10000 cycles to simulate thermal changes occurring in oral cavity during eating, drinking, or breathing which may result in stressing the adhesive interfaces and to allow for water saturation of the luting cements to simulate the oral environment for one year. **(25)**

 In this study, VE discs were cemented to three different foundation materials, composite resin, glass ionomer and dentin. Composite resin was selected as foundation material due to many of the desirable properties that were combined into one material. They have adequate strength, ease of handling and they can be bonded to the tooth structure. Regardless the esthetics, resin composite foundations have a number of advantages over amalgam, due to the immediate polymerization that enables teeth to be prepared for a crown restoration at the same appointment. Resin composites can also be bonded to dowels and crowns whenever appropriate bonding techniques are used . **(27)**

Glass-ionomer materials are clinically attractive dental materials that have certain unique properties which make them useful as restorative and luting materials. Glass-ionomer (GI) was introduced by Wilson and Kent in 1972 as a "new translucent dental filling material" recommended for the restoration of cervical lesions. The positive characteristics of the glass-ionomer materials include chemical adhesion to enamel and dentin in the presence of moisture, resistance to micro leakage, good marginal integrity, and dimensional stability at high humidity, coefficient of thermal

expansion similar to tooth structure,
biocompatibility, fluoride release, and less biocompatibility, fluoride shrinkage than resins upon setting with no free monomer being released. For these numerical advantages, it was selected as foundation material in this study. **(28)**

In the present study, dentin was selected as foundation material as freshly extracted teeth simulate clinical conditions. However, to obtain sufficient number of teeth it was collected over time and stored in saline throughout the study to prevent dehydration until they were further used. **(29)**

The shear bond strength test was chosen to test the bond strength between hybrid ceramics and foundation materials because it was the most frequently used bond testing method and it had the advantage of being fast and easy test. **(30)**

The cuirrent study revealed that significantly different bond strength values were recorded when a hybrid ceramic was bonded to three different foundation materials including composite resin, glass ionomer and dentin. There was statistically a significant difference of bond strength between tested groups treated with sandblasting and treated with sandblasting/primer indicating that surface treatment had statistically significant effect on bond strength. There was statistically a significant difference of bond strength between tested groups treated with sandblasting/primer indicated that foundation material had statistically significant effect on bond strength. The highest mean bond strength at maximum load was among CSP group (19.90 ± 3.23), GSP (16.92 \pm 2.83) and GS (8.87 \pm 2.62), then CS (8.42 \pm 1.61), DSP (8.06 \pm 1.91), and the least was DS (6.49 ± 1.87) .

The SBS of all tested groups after six months of water storage and 10000 cycles of thermocycling in this study could be decreased significantly because of resin cement hydrolytic degradation, water uptake, reduction of luting cement's mechanical properties, and hydrothermal aging. Moreover, it might absorb water and permit internal stress relaxation caused by polymerization shrinkage. **(31)**

The results of present study was in accordance with other published researches investigating the SBS to variable foundation materials using adhesive resin cement. **(32,33)** Additionally, the differences in the coefficient of thermal expansion at ceramic-resin cementfoundation material interfaces, especially after long-term water storage, raise the undesirable effect of water storage. The durability of resin

cement depends on its resistance to any temperature-induced stress. The mismatching in the coefficient of thermal expansion between hybrid ceramic, luting cements, and different foundation materials could result in high-stress concentration, partial resin cement degradation or imbibition, and undesirable effect on their micromechanical bonding. However, the matched coefficient of thermal expansion, water absorption ability, and mechanical properties between the resin cements and composite resin foundation material were the reason for their reaction to thermal stress nearly identical. This might justify that the highest SBS reported in the current study, was in the composite resin test group, and the lowest was dentin group. **(34)** These results were in agreement with Al-Manei et al, (2020) **(35)** and Hewlett et al, (2010) **(36)** who reported that the highest SBS was for the composite group. Tavakolizadeh et al, (2021) **(37)** , studied the SBS of ceramics to composite, nonprecious gold alloy (NPG), ceramics, and human dentin. They found that the composite resin group was the highest while the dentin group was the lowest.

Bonding of hybrid ceramic to glass ionomer foundation material is based on the micromechanical and chemical bond, which is significantly decreased after the aging protocol due to the behaving of HEMA in glass ionomer as hydrogels, water absorbing, and stresses produced from the polymerization reaction. Also, many studies reported that glass ionomer is sensitive to water storage, and its mechanical properties are affected after long-term storage in water due to water imbibition. **(38)** That explained the lower SBS of glass ionomer compared to composite resin.

The high organic contents and the heterogeneous microstructure of dentin might result in interpreting errors in the bond strength and that might explain the cause of their mostly adhesive failure mode. Furthermore, the polymerization reaction could pull the resin cement from the weakest interface (dentin) toward the strongest interface (hybrid ceramic surface). Moreover, the fragile bond strength of these tested groups were due to hydrolytic effect of water, thermocycling effect on cement degradation, or a weak micromechanical bond. **(39,40)**

Results of this study were in agreement with our research work that showed by Tanış et al. (2014) **(41)** who reported that the use of a primer containing adhesive phosphate monomer MDP (Z-Prime Plus primer) on a sandblasted ceramic surface increased the bond strength between ceramic and resin cement.

In the current study, the bond strength value was higher after sandblasting and primer application, this was due to a synergistic effect produced by the increased contact area on the chemical interactions between the Prime and the hybrid ceramic surface. The recorded data of the present study was in agreement with Kobayashi et al. (2009) ^{(42)} who reported that the use of priming agents enhanced bond strength of an indirect composite veneering material to ceramic.

The outcome of the present study was in agreement with Fushiki et al. (2012) **(43)** who revealed that the use of ceramic primers increased shear bond strength between an indirect composite layering material and ceramics. The findings of present in-vitro study were in agreement with Yi YA et al. (2015) ^{(44)} who noted that use of sandblasting and ceramic primers raised shear bond strength between ceramic and a self-adhesive resin cement.

This can be explained on the basis that air abrasion with coarser alumina particles resulted in increasing surface irregularities, which in turn increases the surface area allowed for bonding with the luting material and hence improving the micromechanical retention with subsequent increase in the bond strength values. **(45, 46)**

This was in accordance with several studies that evaluated the effectiveness of this method of using small particles with a diameter between 30 and 50 µm.**(47)**

The increase in roughness and surface energy that resulted from the air-abrasion method fascilated the resin cement flow into the microretention, thereby increased the micromechanical interlocking between the resin cements and ceramic surfaces. Moreover, an increased surface area would have been generated by air abrasion, increasing the chemical reaction and improving the micromechanical interlocking. **(48)**

V. CONCLUSIONS

Within the limitations of this in vitro study, the following conclusions were drawn:

1- Ceramic material treated with sandblasting followed by primer application increased SBS more than sandblasting only

2- Regarding the foundation material, composite showed the highest bond strength among other foundation materials, so composite was recommended to be the foundation material of choice.

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