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EVALUATION OF MICRO SHEAR BOND STRENGTH OF ORMOCER AFTER VARIOUS CERAMIC MATERIALS SURFACE TREATMENTS

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ABSTRACT

Objective: The purpose of this study was to assess the impact of various surface treatments on the ormocer's micro-shear bond strength bonded to different CAD/CAM materials. **Materials and Methods:** Zirconia Reinforced Lithium Silicate Glass-ceramic and Flexible Nanoceramic materials CAD-CAM blocks were used. Different surface treatments were performed on sliced specimens where in Group1, no surface treatments; Group 2, Sandblasting using 50m aluminum oxide powder and silane, Group 3; 8% hydrofluoric acid etching and silane, and Group 4; dry grinding using a green coded diamond stone and silane. The repair material underwent thermocycling for 5000 cycles between 5^{O} C and 55 ^{O} C. A universal testing device assesed the micro shear bond strength at a crosshead speed of 0.5 mm/min until failure occurs. A stereomicroscope was used to test the failure modes. In addition to the Kruskal-Wallis and Mann-Whitney tests, 2-way ANOVA was used to statistically examine the data ($p < 0.05$). **Results:** The hydrofluoric acid etching had the highest (mean ±SD) value in the zirconia reinforced lithium silicate ceramic and there was no significant difference between all other groups. For the Flexible Nanoceramic, grinding had the highest (mean ±SD) value and the three surface treatments did not differ significantly from one another. **Conclusion:** The best surface treatment protocol for the repair of Zirconia Reinforced Lithium Silicate Glass-ceramic is the hydrofluoric acid etching followed by silane. The flexible nanoceramic repair could be done using either sandblasting, hydrofluoric acid etchant or dry grinding, followed by silane.

KEYWORDS: Ceramic, Micro-shear bond strength, Surface treatment, Ormocer

INTRODUCTION

Over the last few years, digital computer technology has been rapidly developing in dentistry[(1)]. CAD/CAM technology has been introduced as an alternative to traditional manufacturing processes which have led to advances in dental ceramic materials and adhesive technology [(2)]. While ceramic restorations fabricated from machined ceramic blocks may enhance structural reliability, the impact of the machining process on the long-term durability of these restorations must be considered [(3)].

The concept of adhesive dentistry made it possible to fix pre-existing restoration as opposed to replacing them entirely [(4)]. A lot of factors influence the bond strength between materials in case of performing a repair procedure. Factors influencing adhesion in repair attempts include material compositions, surface conditioning methods, the application of silane coupling agents, and the timing of repairs (immediate or delayed) [(5)].

Before the composite repair material is applied, hydrofluoric acid etching or tribochemical silica

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coating (Co-jet) can be used to address the intraoral surface treatment of damaged glass-ceramic restorations. A recognized conditioning technique for promoting the adhesion of resin-based materials to feldspathic, leucite, and lithium-disilicate glass-ceramic restorations involves etching with hydrofluoric acid gel and then silanization [(6)].

Silane coupling agents are synthetic, hybrid inorganic-organic chemicals that improve the resin composite's bonding to different types of ceramics; glass-based fillers, silica-coated metals, hydrofluoric-etchable ceramics and oxide ceramics[(7)].

The repair bond strength to zirconia reinforced lithium silicate ceramics and lithium disilicate glass ceramics may be strengthened by hydrofluoric acid etching and salinization, as well as by sandblasting ceramic surfaces with Co-jet and silanization subsequently [(8)].

Grinding can be used anywhere quickly and easily by the operator without the need of any sophisticated or expensive equipment and without taking the risk of handling a biologically unsafe material [(8)].

Ormocer are hybrid materials combining inorganic "ceramic-like" and organic "polymer-like" components, it has been adapted for dental use, especially in restorative and repair contexts. However, the effectiveness of the surface treatments with Ormocer repair material has not been thoroughly investigated. The purpose of this study was to assess how various surface treatments affected the micro-shear bond strength of resin composite repair material based (Ormocer) that was attached to various CAD/CAM components.

The null hypothesis, which was investigated, was that the micro shear bond strength of the Ormocer repair material was unaffected by different surface treatments.

MATERIALS AND METHODS

1. Sample preparation

Discs with dimensions of 14 mm x 12 mm x 1 mm were made using blocks of two different CAD/CAM restorative materials: VITA Suprinity (VITA Zahnfabrik, Bad Säckingen, Germany) and CERASMART (GC Corporation, Tokyo, Japan).

A total of 8 discs from each material were cut using a micro-tome sectioning device (IsoMet 4000 micro- saw, Buehler, USA) under cooling water system, using a diamond disk 0.6 mm thickness and a cutting speed 2500 rpm. Programat P310 oven was used for crystallization of VITA Suprinity CAD slices, according to its firing program and manufacturer's instructions. All discs were checked for surface integrity and the defective discs were discarded. The discs of both the VITA Suprinity and the CERASMART were kept at 37°C for 48 hours in distilled water as the initial aging procedure to replicate the environment of the oral cavity.

All ceramics discs of both groups were embedded in acrylic blocks (Acrostone Dental & Medical supplies, Cairo, Egypt) in order to facilitate handling and fixing throughout the micro shear test. The VITA SUPRINITY discs were embedded in pink acrylic blocks, while the CERASMART blocks were embedded in green acrylic blocks, (Fig.1)

2. Sample grouping

The samples were randomly divided into 2 main groups (Group **"S"** representing VITA SUPRINITY & Group **"C"** representing CERASMART ceramics, according to the ceramic type used. Each group was sub-divided into 4 groups according to the surface treatment used.

Group 1 – Control (S1 &C1): The Samples received no treatment.

Group 2 – Sandblasting (S2 & C2): Discs of the ceramics were subjected to sandblasting using 50 microns aluminum oxide powder.

FIG (1) Acrylic blocks with embedded CAD/CAM discs

Group 3 - Hydrofluoric Acid (S3 & C3): The ceramic discs were etched with HF acid 8% (Itena, Paris, France).

Group 4 - Grinding with Green Coded Diamond Bur (S4 & C4): Dry grinding using a green coded tapered with round end diamond bur (VladMiVa, Russia).

3. Methodology of surface treatment:

Group 1 – Control (S1 &C1): universal adhesive Futura bond M+ (VOCO, Cuxhaven, Germany) was applied in compliance with the manufacturer's guidelines.

Group 2 – Sandblasting (S2 & C2): Sandblasting using 50 microns aluminum oxide powder, at an angle of 90^{o}, distance 10mm, for 20 seconds and 2.8 bar pressure. (Fig. 2) For standardization a stand was fabricated to hold the hand piece and the acrylic blocks (holding the ceramic discs). After applying 97% alcohol to the discs, they were air-dried. After 60 seconds of application, the silane coupling agent was air dried.

Group 3 - Hydrofluoric Acid (S3 & C3): The ceramic discs were etched with HF acid 8% (Itena, France). For the ceramic discs embedded in the pink blocks (VITA SUPRINITY) the acid was applied for 20 seconds and for the discs embedded in the green blocks (CERASMART) the hydrofluoric acid was applied for 60 seconds; according to manufacturer instructions. Then, they were washed and air-dried. Afterwards silane coupling agent was applied using a micro brush for 60 seconds for both ceramic groups, then air-dried.

Group 4 - Grinding with Green Coded Diamond Bur (S4 & C4): Dry grinding using a green coded tapered with round end diamond bur (VladMiVa, Russia) for 30 seconds with 25000 RPM speed. Discs were then etched by phosphoric acid 37% to clean the surface from the grinding debris. It was applied for 1 min followed by air water spray for 1 min. Silane coupling agent was applied for 60 seconds then air dried.

4. Application of Repair Material:

Each disc of both material VITA SUPRINITY (S) and CERASMART (C) received five ORMOCER micro-cylinders. After applying universal bond, iris-shaped polyethylene tubes measuring 1 mm in diameter and 1 mm in height were placed over the disc surface and filled with Ormocer material using the provided tip.

Then, as recommended by the manufacturer, Ormocer was light cured for 20 seconds, using LED light curing unite (3M ESPE Elipar) of 1200 mW/cm2. The irises of the polyethylene tube were left in place to avoid shear stress at the interface and to rule out any pretest failures. (Fig. 3)

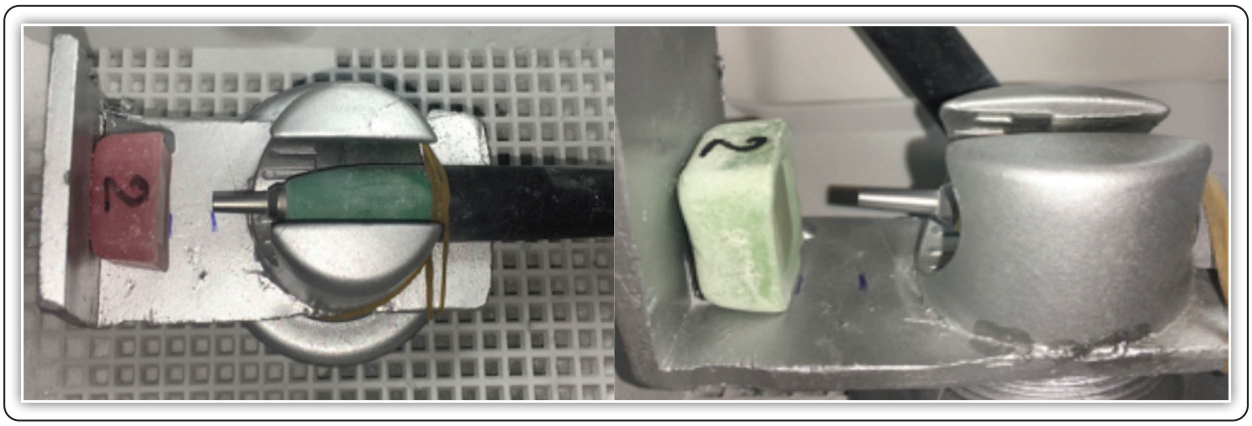

FIG (2) Sandblasting of VITA Suprinity, & Cerasmart discs

FIG (3) Irises of polyethylene tube positioned over CAD/CAM slices

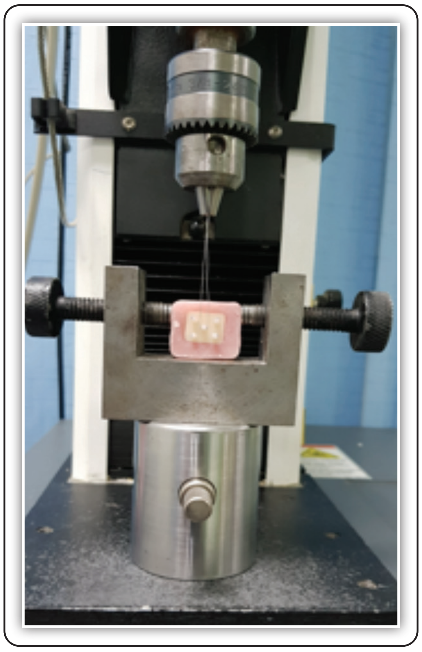

FIG (4) Testing machine, Model 3345; Instron Industrial Products, Norwood, MA, USA

5. Ageing process:

Using the THE-1100 SD Mechatronics thermocycler (SD-Mechatronik, Westerham, Germany), specimens were thermo-cycled to replicate the media found in the oral cavity, 5000 cycles between 5 and 55 degrees Celsius with a dwell time of 20s and a transfer time of 5 seconds, which corresponds to approximately 6 months of clinical use [9,23].

6. Micro-Shear Bond Strength Test (µSBS):

Using universal testing machine (Model 3345; Instron Industrial Products, Norwood, MA, USA) with a 5 KN loadcell had its own bonded micro-cylinders attached to the lowest fixed compartment horizontally with tightening screws.

The bonded micro-cylinder assembly was wrapped with a loop of orthodontic wire (0.014” in diameter), which was then securely placed at the junction of the ceramic disc and micro-cylinder, to align the loop with the loading axis of the testing machine's upper movable compartment. A material testing device was used to apply a shearing load with a tensile mode of force at a crosshead speed of 0.5 mm/min. Computer software was used to record the results, and the load needed to de-bond was measured in Newton units. By dividing the load at failure by the bonding area, the bond strength was calculated and given in MPa.

$$\textbf{Bond Strength} = \frac{\textbf{\textit{Load at Fracture}}}{\textbf{\textit{Bonding Area}}}$$

This calculation results in a value, expressed in Megapascals (MPa), representing the stress at the interface when the bond fails.

7. Digital Microscope Analysis:

Digital Microscope Assessment: Following a micro shear bond strength test, the cracked surfaces of the CAD/CAM restorative materials were inspected at a magnification of 55x using a digital microscope (Dino-Lite). Failure mechanisms were divided into three categories: cohesive (inside the resin composite or CAD/CAM restorative material), adhesive (at the interface between the resin composite and CAD/CAM restorative material), and mixed.

IBM® (IBM Corporation, NY, USA) SPSS® (®SPSS, Inc., an IBM Company) Statistics Version 2.0 for Windows was used to conduct the statistical analysis. Numerical data were checked for normalcy by looking at the distribution of the data, calculating the mean and median, and using the Shapiro-Wilk and Kolmogorov-Smirnov tests. The data's non-normal distribution was demonstrated using the mean, median, and standard deviation (SD) values. For every test, the significance level was set at $P \leq 0.05$. Using two-way ANOVA, the effects of several measured variables and their interactions on μ-Shear Bond Strength (MPa) were investigated. The Kruskal-Wallis and Bonferroni corrected Mann-Whitney tests were used to compare the impact of surface treatment within each type of material. The Mann-Whitney test was used to compare the effects of each surface treatment.

RESULTS

a. Statistical analysis

Table (1) displays the mean, standard deviation, median, and P-value for the impact of material types and surface treatments on μ-Shear Bond Strength.

For VITA Suprinity (S) Group, Surface treatment (S3 "HF group") had the significantly highest value (10.51±7.81), while surface treatment (S1 "no treatment group") had the significantly lowest value (0). The groups did not significantly differ from one another. (C1"no treatment group"), (C2 "Sandblasting group") and (C4 "Grinding with stone group").

For CERASMART (C) Group, surface treatment (C4 "Grinding with stone group") had the significantly highest value (16.46±12.13), while surface treatment (C1 "no treatment group") had the significantly lowest value (4.90±6.82). There was no significant difference among the four surface treatment groups. Regarding surface treatment (Group 1"no treatment group"), (Group 2 "Sandblasting group") and (Group 4 "Grinding with stone group"), CERASMART (C) had a significantly higher value than VITA Suprinity (S).

Whereas for surface treatment (Group 3 "HF group"), showed no significant difference between two different types of ceramics.

b. Fracture Analysis:

Fracture analysis of VITA SUPRINITY (S) groups revealed that In the majority of groups, the adhesive type was the most common failure mode; except for Group (3) surface treatment, the dominant mode was cohesive in composite. For CERASMART (C) groups, With surface treatment groups (Groups 1 and 3), the most frequent failure mode was adhesive; with groups 2 and 4, the most frequent failure mode was cohesive in the composite. Table (2) shows the percentage distribution of each group's various failure modes.

TABLE (1) Mean ± Standard deviation (SD), Median and P-value for the effect of types of material and surface treatments on μ-Shear Bond Strength (MPa).Different superscript letters within the same row indicates a statistically significant difference

Type of material	Type of surface treatment								P-value
	No Surface treatment (1)		Sandblasting & Silane (2)		Hydrofluoric Acid & Silane (3)		Diamond Grinding & Silane (4)		
	Mean±SD	Median	Mean±SD	Median	Mean±SD	Median	Mean±SD	Median	
VITA Suprinity (S)	0^{A}	0.00	0.24 ± 0.64^{AB}	0.00	10.51 ± 7.81^{C}	13.70	1.41 ± 1.54^{B}	0.91	<0.001*
CERASMART (C)	4.90 ± 6.82^{A}	0.00	11.33 ± 9.43^{AB}	12.44	9.27 ± 8.67^{AB}	8.41	16.46 ± 12.13^{B}	20.26	0.092NS
P-value	<0.001*		0.014*		0.515NS		<0.001*		

**: significant (p ≤ 0.05); NS: non-significant (p>0.05)*

TABLE (2) Percentage distribution of different failure modes in each group.

Type of Material	Type of surface treatment	Mode of Failure	
		Adhesive	Cohesive in composite
VITA Suprinity (S)	**No Surface treatment (1)**	100%	0%
	Sandblasting & Silane (2)	100%	0%
	Hydrofluoric Acid & Silane (3)	40%	60%
	Diamond Grinding & Silane (4)	100%	0%
CERASMART (C)	**No Surface treatment (1)**	100%	0%
	Sandblasting & Silane (2)	40%	60%
	Hydrofluoric Acid & Silane (3)	70%	30%
	Diamond Grinding & Silane (4)	20%	80%

DISCUSSION

The two materials used in this study; VITA SUPRINITY and CERASMART are newly introduced CAD/CAM materials, each of a different type of ceramics. The VITA Suprinity is classified as Synthetic glass-matrix ceramics and the CERASMART as resin-matrix ceramics.

The most crucial factor in determining the repair strength of restorative materials is mechanical interlocking. Increasing surface roughness improves the mechanical interlocking of the bonding surface[10].

In order to assess and test the surface of the CAD/CAM materials, various surface treatments were used in this investigation. These surface treatments included acid etching with 8% buffered hydrofluoric acid, grinding with a green banded diamond bur (125 µm grain size), and air-particle abrasion with 50 µm Al_2O_3. These techniques are frequently applied as surface treatments when ceramic restorations are repaired intraorally.

Hydrofluoric acid “HF” etching was the second surface treatment used. The method most frequently used to increase bonding strength is acid etching.

By forming micro-pores into which uncured flowable resin can enter, etching increases the surface area and produces long-lasting micro-mechanical interlocking [(11)]. A 20-second etching process causes the glassy phase to dissolve primarily around the crystals, forming tiny, isolated pores and fissures [(12)]. Restorations usually fail after being aged in a humid, thermally active oral environment [(13)].

Thermo-cycling, a method for artificially aging dental materials, was employed to simulate the circumstances of the oral cavity both before and after repair. The failure of some specimens during thermo-cycling may indicate the stress and alterations caused by thermal aging on the recovered surfaces of restorative materials. Failure analysis revealed that most failures occurred in the control groups that did not get surface treatment.

Thermocycling showed an unfavorable effect on bond strength in this study, which is translated through the failures that occurred during thermocycling especially in both control groups of the VITA Suprinity and CERASMART. There have been prior reports of composite resin's decreased bonding strength to zirconia-reinforced lithium silicate and CAD/CAM resin-ceramic hybrid restorations. [(14,15)].

Compared to VITA Suprinity, CERASMART showed a noticeably greater bonding strength value. It was explained by Ustun et al in 2018 [(16)] that the zirconia content of the VITA Suprinity material could be adversely affected by silanization.

Concerning the effect of surface treatment, the results of this current study revealed that hydrofluoric acid etching plus silane (S3 & C3) showed higher significant micro-shear bond strength value regardless of the material.

These findings are explained by several studies Neis et al [(10)] reported that the most effective method for infiltration and elimination of the vitreous stage, which exposes the crystalline structure and causes irregularities on the IPS ceramic surface, is HF etching. According to Cláudia et al [(17)] who confirmed similar findings, sandblasting of the IPS surface yielded lower micro-tensile bond strength (µTBS) values than silanization and HF etching. Additionally, Sato et al [(18)] evaluated how surface treatments affected the strength of the link between the polymerized composite blocks and the VITA SUPRINITY specimens. Additionally, they discovered that HF surface treatment produced positive µTBS outcomes. The study's findings for each material indicated that etching with hydrofluoric acid would be the most effective surface treatment for restoring VITA SUPRINITY.

These results supported the findings of Strasser et al [(19)] stated that hydrofluoric acid etching followed by silane application is the most widely used technique for establishing reliable resin bonding to silicate ceramics, including the novel zirconia-reinforced lithium-silicate ceramics VITA Suprinity. All glass-ceramics have a low tolerance to additional mechanical pre-treatments. After coarse grit grinding and sandblasting, microchipping and fracture development occurred.

The similar conclusion was reached by Menees et al [(20)] who demonstrated that sandblasting zirconia-reinforced lithium-silicate ceramic considerably reduced its flexural strength. Which is consistent with the current study's findings, which indicate that grinding and sandblasting produced the lowest values.

For the resin bond strength to ceramic polymer or composites, Spitznagel et al [(21)] stated that before applying silane, surface roughening is essential. The ceramic components were superficially dissolved by hydrofluoric acid. Low roughness was achieved in spite of the high secondary electrons induced by the hydrofluoric acid etching processes. Grinding is a promising technique for intraoral repairs, but it is clinically less common than sandblasting.

These statements explain the results of the different surface treatments of CERASMART after thermocycling, where diamond grinding surface treatment and sand blasting gave better values in comparison to acid etching and no treatment groups, but there was no significance between all groups which indicates that all of them can be used when repairing a CERASMART restoration or any similar CAD/CAM composite material.

The majority of them were adhesive failures, particularly in the VITA Suprinity groups, according to the failure mode analysis conducted for this trial. It suggests that CAD/CAM ceramics' bonding strength to resin composite can be assessed using the micro-shear test (22).

The composite's cohesive failures were more common in the groups with high bond strength values, such as the VITA Suprinity group's HF acid etching followed by silane and the CERASMART group's diamond grinding followed by silane. The CERASMART group also experienced sandblasting followed by silane, but in a smaller percentage.

CONCLUSIONS

Within the limitation of this study it was concluded that**:**

- Various surface treatment techniques had an impact on the flexible nano-ceramic (CERASMART) and CAD/CAM zirconia reinforced lithium silicate (VITA Suprinity) repair bond strength.
- For VITA Suprinity repair, hydrofluoric acid etching followed by silane is the most effective surface treatment.
- Different surface treatments, such as hydrofluoric acid etching followed by silane, sandblasting with 50-micron particles and silane, or grinding with green-coded diamond stone and silane, could be used for the CERASMART repair.

Limitations of the Study

A better evaluation of the surface topography changes following the various surface treatments would have been possible with the use of an electron microscope in conjunction with a digital microscope. This would have confirmed the findings of the current in vitro investigation.

RECOMMENDATIONS

This in vitro investigation indicates that hydrofluoric acid etching followed by silane application is the optimal surface treatment protocol for performing an intraoral repair on fractured zirconia reinforced glass-ceramic CAD/CAM blocks. Any of the three surface treatment methods; hydrofluoric acid etching followed by silane, sandblasting, or grinding with green-coded diamond stone, could be the most effective for the hybrid resin ceramic CAD/CAM blocks.

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