

Effect of different surface treatments on bond strength of resin cement to two recent ceramic materials

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ABSTRACT

Background: The adhesion of indirect restorations to the tooth structure is a very critical step that a lot of operators underestimate it & don't understand its importance which will influence durability and success of restorations. By overcoming failures that occur due to improper bonding we can reach the best durability of the restoration helping us reach our goal which is fabricating a restoration that mimics natural teeth. So Bonding between indirect restorations and tooth, will fulfill high retention, preventing microleakage, and creating good marginal adaptation.

Aim of the Work: This research was performed to study the effect of different surface treatments which are: Acid etching & silanation (9.5 % HF acid etching), Al₂O₃ sandblasting & silanation, 2W, Er,Cr:YSGG laser etching with repetition rate 20 HZ for 20 secs & silanation, to two recent ceramic materials: resin nano ceramic (Crystal Ultra) & True hybrid ceramic polymer infiltrated ceramic network material (Vita Enamic).

Materials and Methods: Blocks of two CAD CAM esthetic restorative materials (VITA Enamic and Crystal Ultra) were used to prepare slices with the following dimensions: 14mm x 12mm x 1mm for Vita Enamic ceramic material & 19mm x 15mm x 1mm for Crystal Ultra ceramic material. Using IsoMet 4000 micro saw with cooling water system, by a diamond disk 0.6 mm thickness with cutting speed 2500 rpm. For easier handling and fixation during the micro shear test, a number of 9 slices of crystal Ultra Material & 12 slices of Vita Enamic material were embedded in an acrylic blocks, before any surface treatments.

Results: For VITA Enamic, hydrofluoric acid etching & silanation as Surface treatment had the highest (mean±SD) value, followed by sandblasting & silanation,

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while Laser surface etching followed by silanation had the lowest (mean±SD) values. For Crystal Ultra, sandblasting & silanation had the highest (mean±SD) value, followed by hydrofluoric acid etching & silanation,, while the laser surface etching followed by silanation had the lowest (mean±SD) value.

Conclusion: Crystal Ultra showed the highest bond strength after sandblasting & silanation. In Vita Enamic HF acid etching & silanation had the highest bond strength than other surface treatments. Laser surface etching & silanation had significantly lower values for both materials used.

Keywords: Surface treatments, hybrid ceramics, bond strength, PICN materials

INTRODUCTION

The development of ceramics during the last couple of years was huge. However, the focus was on the hardness and the strength of the restorative materials, which resulted into high antagonistic tooth wear. There was a new approach for the material tested, to modify ceramics to create a bio-mimetic material simulating dentin and enamel's physical properties, as the long-term aim is to imitate the mechanical behavior of a natural tooth. And still they have strength similar to or near conventional ceramics. As a result for that new materials in dental market were introduced, Polymer infiltrated ceramic network (PICN) materials Also known as hybrid ceramics are currently being introduced for CAD/CAM use. (PICN) CAD/CAM materials have reduced brittleness, rigidity and hardness with improved flexibility, fracture toughness and better machinability compared to ceramics and due to the presence of two connected phases within (PICN) crack propagation is generally limited because of interfacial crack deflection. The phase with the higher strain to failure enhances the fracture resistance by bridging the cracks introduced to the other phase.

Computer Aided design computer aided manufacturing (CAD/CAM) has been known widely and gained popularity in dental

applications especially in the last decade as it minimizes time and effort made by dentists, technicians and patients, restores and maintains patient oral function and aesthetic, while providing high quality outcome. This explains the appearance of new CAD/CAM ceramic materials keeping pace with the new technology & fulfilling the concept of fabrication of tooth like restorations.

Hybrid ceramic material have been divided in further two subgroups: Interpenetrating glass ceramic network infiltrated by resin network (polymer infiltrated ceramic) which is called true hybrid ceramic because it consist mainly of a dominant ceramic network (86 wt.%) strengthened by an acrylate polymer network (14 wt.%) example of this Material is (VITA Enamic, VITA Zahnfabrik, Bad Sackingen, Germany) it has aluminum-oxide enriched, fine-structure feldspar matrix infused by polymer including urethane dimethacrylate (UDMA) and others. Resin matrix or network infiltrated with glass ceramic fillers refered to as CAD/CAM Resin Nano ceramics. Example of this subgroup: Paradigm MZ100, Lava Ultimate, Cerasmart and Crystal Ultra, which is rescently applied in market by Dental Laboratory Milling Supplies, LLC (DLMS). Crystal Ultra is Sailinated glass ceramic polymer hybrid restorative with glass fillers of 70.02% (wt). The adhesion of indirect restorations to the tooth structure is a very critical step that a lot of operators underestimate it & don't understand its importance which will influence durability and success of restorations.

AIM OF THE WORK

This research is performed to study the effect of different surface treatments which are: Acid etching & silanation (9.5 % HF acid etching). Al₂O₃ sandblasting & silanation. 2W, Er,Cr:YSGG laser etching with repition rate 20 HZ for 20 secs& silanation.

To two recent ceramic materials: Resin nano ceramic (Crystal Ultra). True hybrid ceramic polymer infiltrated ceramic network material. (Vita Enamic)

MATERIALS AND METHODS:

I. Materials:

Brand name, material description, manufacturer and lotnumber are listed in table (1).

Table (1): List of materials& instruments used.

	Brand name	Material description	Manufacturer	Lot #
1	Breeze self adhesive resin cement	Self adhesive dual cured luting resin cement	Pentron	7027824
2	Crystal ultra	Resin nano ceramic	Dental Laboratory Milling Supplies, LLC (DLMS)	P2.61C
3	Vita enamic	Hybrid ceramic	Vita Zahnfabrik, Bad Sackingen, Germany	41720
4	Bisco porcelain etchant	Hydro fluoric acid	Bisco, Inc. Schaumburg,IL60193	1700003266
5	Bisco silane primer	Pre hydrolyzed Porcelain silane	Bisco, Inc. Schaumburg,IL60193	1700003403
6	Cobra	Aluminum oxide	Renfert Hilzingen, Germany	15941205
7	Water lase i plus	Er,Cr:YSGG laser of 2780 n.m wave length	Biolase technology Inc,Irvine,CA,USA	

1. Breeze self adhesive resin cement:

Dual cured Self-Adhesive Resin Cement with auto-mix delivery system upon application. Figure(1)



Figure (1): Breeze self adhesive resin cement

2. Crystal Ultra CAD/CAM ceramic material:

The Crystal Ultra CAD/CAM formed materials consist of interpenetrating networks of glass ceramic and polymer material to form a solid block of material. A perfect 70/30 blends of silanated glass and advanced polymers. The 30% polymer matrix gives the material its elasticity, and silanizing causes the ceramic to bond chemically to the polymers giving the material its strength.



Figure (2): Crystal Ultra CAD block

Table (2): Physical Properties of Crystal Ultra:

Physical Property	Measurement	Standard Value
Vickers Hardness (200 p)	710 MPa	None specified
Modulus of Elasticity (Young Modulus)	10,000 MPa	None specified
Flexural Strength	175 MPa	ISO 10477 \geq 50 ISO 6872 \geq 100
Compressive Strength	480-500 MPa	ISO 4049 ADA 27
Density	1.89 g/cm ³	None specified
Chemical Solubility	1.29 μ g/cm ³	ISO 6872 \leq 100
Water Absorption	16.94 μ g/mm ³	ISO 10477 \leq 40
Water Solubility	0.04 μ g/mm ³	ISO 10477 \leq 7.5
Biocompatibility	Confirmed	ISO 10993
Ceramic Filler	70.02% (wt)	None Specified
Color Stability	Confirmed	ISO 10477
Shade Consistency	Confirmed	ISO 10477
Surface Finish	Smooth	ISO 10477
Bond Strength	27-37 MPa	ISO 10477 \geq 5

3. Vita enamic:

Vita enamic is a true hybridceramic because it consist mainly of a dominant ceramic network (86 wt.%) strengthened by an acrylate polymer network (14 wt.%) it has aluminum-oxide enriched, fine-structure feldspar matrix infused by polymer including urethane di-methacrylate (UDMA) andothers.

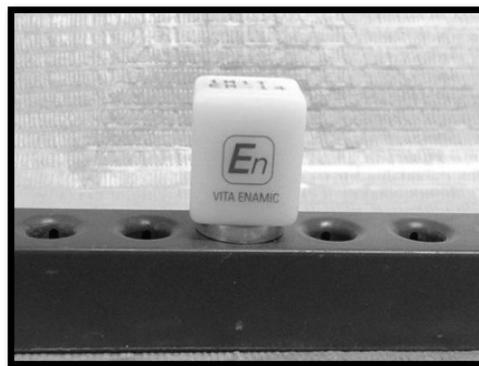


Figure (3): Vita Enamic CAD block

Composition of the polymer part (14wt% / 25 vol%)

- **UDMA** (urethane dimethacrylate)
- **TEGDMA** (triethylene glycol dimethacrylate)

4. Porcelain etchant:

Buffered viscous 9.5% hydrofluoric acid that will etch ceramics of all types to make a micro-porous surface which gives a strong mechanical interlock with composite resin materials.

5. Silane primer:

A single component silane coupling agent in an alcohol and acetone base that is used to improve the adhesive bond between porcelain and resin cements. Silane is a dual function monomer consisting of a silanol group that reacts with the porcelain surface, and a methacrylate group that co-polymerizes with the resin matrix of the composite.

6. Aluminum Oxide:

Cobra is the abrasive used in the sandblasting, approximately 99.7% Al₂O₃ of 50 micron in particle size.

II. Levels of investigation and factorial design:

Samples grouping:

Table (3): Samples grouping & factorial design.

Surface Treatment \ Type of Hybrid Ceramic	I. HF acid etching + silane (E)	II. Sand blasting + silane (S)	III. Laser etching + silane (L)	Total Number Of Samples
Vita Enamic Group V	VE N=20	VS N=20	VL N=20	N=60
Crystal Ultra Group C	CE N=20	CS N=20	CL N=20	N=60
Total Number Of Samples	N=40	N=40	N=40	N=120

III. Preparation of the substrate:

Blocks of two CAD CAM esthetic restorative materials (VITA Enamic and Crystal Ultra) were used to prepare slices with the following dimensions: 14mm x 12mm x 1mm for Vita Enamic ceramic material & 19mm x 15mm x 1mm for Crystal Ultra ceramic material.

Using IsoMet 4000 micro saw *** with cooling water system, by a diamond disk 0.6 mm thickness with cutting speed 2500 rpm. (Fig.4)

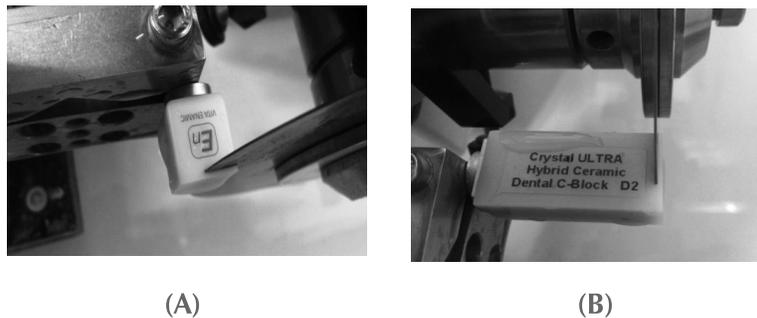


Figure (4): Vita Enamic block (A) Crystal Ultra block (B) being cut into slices using isoMet 4000.

I. Surface Treatments:

For easier handling and fixation during the micro shear test, a number of 9 slices of crystal Ultra Material & 12 slices of Vita Enamic material were embedded in acrylic blocks, before any surface treatments.

To easily differentiate between the two materials during the rest of the procedures, two different color coded acrylic resin blocks were selected. Pink color was used for Crystal Ultra slices, and Green color for Vita Enamic slices.

Then each acrylic block was given the initial letter related to the surface treatment applied, as mentioned in fig (5)

*** (Buehler USA)



(A)



(B)

Figure (5): Vita Enamic slices (A) & Crystal Ultra slices (B) Embedded in acrylic blocks each given initial related to surface treatment that will be done.

Before any surface treatments done, 70% ethyl alcohol was used on each of 21 slices for cleaning the surface from any debris, and drying these surfaces very well.

1) Hydrofluoric Acid Etching + Silane:

Acrylic blocks carrying CE & VE initials were etched using Hydrofluoric acid 9.5% for 60 seconds, afterwards washed and air-dried with oil-free air/water syringe. Silane coupling agent was applied for 60 seconds for both types of ceramics. Then air drying was done for the specimens using oil free air way syringe.

2) Sandblasting:

Sandblasting was performed on Vita Enamic & Crystal Ultra ceramic slices embedded in acrylic blocks holding VS & CS initials, using 50 microns aluminium oxide powder at an angle of 90, distance ≈ 10 mm for 20 seconds and 2.8 bar pressure.

Then alcohol 97% was applied on the slices and air dried with oil-free air/water syringe until the surface became matt.

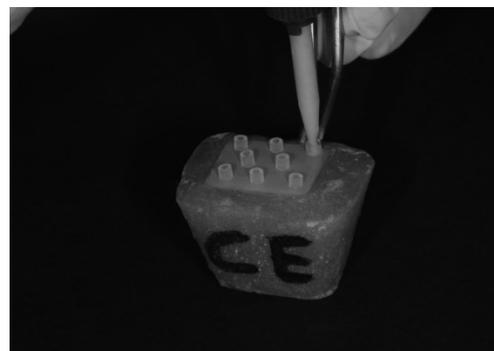
Silane coupling agent was applied for 60 seconds then dried with oil free air way syringe.

3) Laser etching + silanation:

The ceramic slices were subjected to laser irradiation followed by the application of silane primer. In this group Er,Cr:YSGG laser with wave length 2780nm, pulsed laser-powered hydrokinetics, was used. Vapor and air were adjusted to 50% of the laser unit. The optical fiber of the laser unit were 400 μ m in diameter and 4mm in length, arranged perpendicular over each ceramic slice and moved manually in a sweeping manner to cover all the surface area during the adjusted exposure period. The laser parameters were adjusted so that, the power was 2 W for VL & CL acrylic blocks carrying Vita Enamic & Crystal ultra ceramic slices. The repetition rate was 20 Hz for 20 seconds at surface of the slices. The slices were then rinsed with distilled water and air dried. Silane primer was then applied to the irradiated surfaces for 60 seconds and then air dried for 60 seconds.

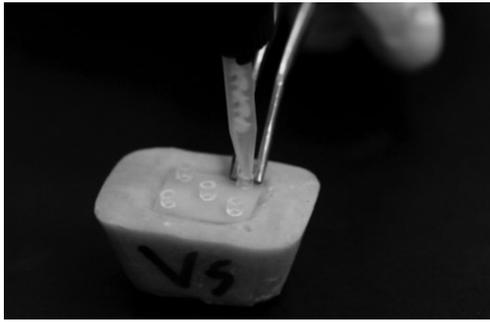
II. Application of resin cement material:

VITA Enamic slice Received 5 resin micro cylinders and Crystal Ultra slice Received 8-6 resin micro Cylinders. Irises of polyethylene tube having 1mm diameter and 1mm height were positioned over the disc surface, then cement was injected into the tubes through the mixing tip. Light curing was done through the tube for 20 seconds, with a LED light-curing unit*** with an irradiance of 1200 mW/cm² according to the manufacturer's instructions. Polyethylene tube irises were not removed in order not to subject the resin micro cylinders to shear stress at the interface, and to eliminate any pretest failures.



A

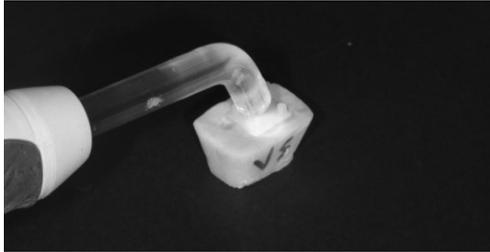
*** *Elipar S10, 3M Espe, St. Paul, MN



B



C



D



E



F

Figure (6): Resin cement injection into the grooves on the surfaces of ceramic discs in (A) for crystal ultra & in (B) for vita enamic, then light curing was done for (c) resin tubes on crystal ultra & for (D) resin tubes on vita enamic surfaces for 60 seconds for each specimen.

Fully cured resin tubes (E) on the crystal ultra ceramic surface & (F) on the vita enamic ceramic surface

VI. Thermocycling:

In order to simulate the oral cavity media, specimens were

Thermo-cycled using THE-1100 SD Mechatronics thermo-cycler between 5 °c and 55 °c for 5000 cycles with a 20 seconds dwell time and 5 seconds transfer time.

VII. Micro-Shear Bond Strength Test:

Each block with its own bonded micro-cylinders was secured horizontally with tightening screws to the lower fixed compartment of a universal testing machine***** with a loadcell of 5 kN and data were recorded using computer software*****. A loop prepared from an orthodontic wire (0.014" in diameter) was wrapped around the bonded micro-cylinder assembly as close as possible to the base of the micro-cylinder and aligned with the loading axis of the upper movable compartment of the testing machine (Fig.7). A shearing load with tensile mode of force was applied via materials testing machine at a crosshead speed of 0.5 mm/min. The load required to debonding was recorded in Newton.

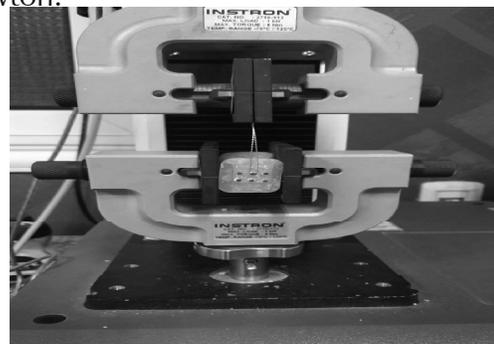


Figure (7): A loop wrapped around bonded micro cylinder.

Micro-Shear Bond Strength Calculation;

The load at failure was divided by bonding area to express the bond strength in MPa: $\tau = P / \pi r^2$

**** * (Model 3345; Instron Industrial Products, Norwood, MA, USA)

***** ** (Instron® Bluehill Lite Software).

Where; τ = μ -shear bond strength (in MPa), P =load at failure (in N), π =3.14 and r = radius of micro-cylinder (in mm)

Scanning Electron microscope (SEM) Analysis:

One disc of Vita Enamic & one other disc from Crystal Ultra was examined under The Electron microscope before any surface treatment done, to be able to compare it afterwards with the different surface treatments done in our study. Each disc was mounted on aluminum stud & examined with scanning electron microscope using low vacuum mode with magnification of 2000 x.

Then after micro shear test was done, each acrylic block carrying ceramic slice been mounted on Aluminum studs, examined under low vacuum mode with magnification 100 x of scanning electron microscope (SEM)^{*****} to ensure excellent resolution to a non conductive surface similar to the specimens we are working on it.

SEM Aanalysis was important to examine the fractured surface of each bondedassembly to determine the mode of failure that occurred.

Statistical analysis:

Categorical data were presented as frequencies and percentages and were analyzed using chi square test followed by multiple pairwise comparisons utilizing z-test with benferroni correction. Numerical data were explored for normality by checking the data distribution, calculating the mean and median values and using Kolmogorov-Smirnov and Shapiro-Wilk tests. Data showed parametric distribution so; it was represented by mean and standard deviation (SD) values. Two-way ANOVA was used to study the effect of different tested variables and their interaction. Comparison of main and simple effects were done utilizing benferroni correction. The significance level was set at $P \leq 0.05$ within all tests. Statistical analysis was performed with IBM^{*****}® SPSS^{*****}® Statistics Version 26 for Windows.

RESULTS

Table (4): Descriptive statistics for microshear bond strength (Mpa) of different groups.

Material	Surface treatment	Mean	Std. Deviation	Median	Range
Crystal Ultra	Etching & silanation	11.29	3.39	10.49	13.35
	Sandblasting&silanation	11.71	3.62	11.71	14.75
	Laser&silanation	6.34	2.20	7.25	7.47
Vita Enamic	Etching & silanation	12.35	3.56	12.27	12.52
	Sandblasting&silanation	9.39	3.31	8.44	11.58
	Laser&silanation	5.04	2.47	4.88	9.06

Table (5): Effect of different variables and their interactions on microshear bond strength (Mpa)

Source	Type III Sum of Squares	df	Mean Square	F	p-value
Material	21.44	1	21.44	2.18	0.143ns
Surface treatment	824.31	2	412.15	41.89	<0.001*
Material *Surface treatment	158.11	2	129.05	6.95	0.043*

df=degree of freedom*; significant ($p \leq 0.05$) ns; non-significant ($p > 0.05$)

***** thermo fisher scientific, Quattro S Environmental SEM

*****® IBM Corporation, NY, USA.

*****®SPSS, Inc., an IBM Company.

Table (6): Mean \pm standard deviation (SD) of microshear bond strength (Mpa) for different materials and surface treatments

Material	Surface treatment(mean \pm SD)			p-value
	Etching & silanation	Sandblasting&silanation	Laser&silanation	
Crystal Ultra	11.29 \pm 3.39 ^A	11.71 \pm 3.62 ^A	6.34 \pm 2.20 ^B	<0.001*
Vita Enamic	12.35 \pm 3.56 ^A	9.39 \pm 3.31 ^B	5.04 \pm 2.47 ^C	<0.001*
p-value	0.302ns	0.021*	0.195ns	

Different superscript letters indicate a statistically significant difference within the same horizontal row*; significant ($p \leq 0.05$) ns; non-significant ($p > 0.05$)

Table (7): Frequencies (n) and Percentages (%) of mode of failure in different surface treatments

Failure mode		Etching & silanation (n=40)	Sandblasting&silanation (n=40)	Laser&silanation (n=40)	p-value
Adhesive	n	3	10	25	
	%	7.5% ^B	25.0% ^B	62.5% ^A	
Cohesive	n	23	18	1	
	%	57.5% ^A	45.0% ^A	2.5% ^B	
Mixed	n	14	12	14	
	%	35.0% ^A	30.0% ^A	35.0% ^A	

Percentages with different superscript letters within the same horizontal row are significantly different*; significant ($p \leq 0.05$) ns; non-significant ($p > 0.05$)

DISCUSSION

In general, non-metal CAD/CAM restoratives are currently divided into two main categories: ceramics and composites.⁽¹⁾ Comparing composites to ceramics, composite indirect restorations are more compliant and soft, have easy finishing and polishing properties are less abrasive for opposing dentition, and are conducive to making add-on adjustments, although they experience high wear.⁽²⁾ However, esthetic properties of ceramic restorations are superior to those of composite materials.⁽³⁾ Furthermore, ceramics are more wear-resistant, more biocompatible, and more discoloration-resistant; however, they are more brittle & they cause excessive wear to opposing dentition. Combining these materials' positive properties, which consequently enhances the properties and longevity of indirect restorations, a novel material named polymer infiltrated ceramic network material has been developed (PICN)⁽²⁾.

Therefore we used two of newly introduced CAD/CAM polymer infiltrated ceramic materials of different sub families:

Vita Enamic classified as **Glass ceramic in a resin interpenetrating matrix** while Crystal Ultra classified as **Resin nano ceramic**.

Vita Enamic:.. was manufactured by introducing a lower elastic modulus polymeric second phase into ceramic networks. Therefore, the hybrid ceramic combines the positive characteristics of ceramics and composites.⁽⁴⁾

Crystal ultra is a hybrid nano ceramic/resin milling material that is a highly esthetic and easy to use alternative to all ceramic restorations. The higher compressive strength of Crystal Ultra is the result of special manufacturing processes used to bond the glass and polymer materials, including the use of a special silanated glass ceramic which chemically bonds to the polymers.⁽⁵⁾

An important aspect required for the success of such hybrid ceramic restorations is the establishment of proper adhesion between substrate and adherent upon the light of this our study was applied In an attempt to find the best surface treatment to improve bonding of resin cements to hybrid ceramics, that facilitate chemical and micromechanical retention.

Therefore we aimed at our study to test for bond strength, using micro shear bonding strength test due to the fact of micro-shear test is a relatively simple “micro” test that permits efficient screening of adhesive systems, Moreover, micro-shear method was shown to have the advantage of producing fewer cohesive failures and presenting lower data dispersion⁽⁶⁾.

Our study aims at evaluation of the effect of material & surface treatments on the bond strength of ceramic-glass polymer CAD-CAM material to the resin cement.

Therefore different surface treatments were applied in this study on the CAD/CAM materials' surface to be evaluated and tested, these surface treatments include: acid etching (9.5% buffered hydrofluoric acid) followed by silane primer, air-particle abrasion (50 μm Al₂O₃) followed by silane primer & laser etching using Er,Cr:YSGG pulsed laser followed by silane primer.

The first applied surface treatment was hydrofluoric acid etching. Acid etching is the most commonly employed technique to improve the bond strength. The HF surface treatment modifies the microstructures of CAD/CAM hybrid ceramic surface by partial dissolution of the polymer and glassy phase of the feldspar ceramic, forming microporosity on the ceramic surface.⁽⁷⁾ It increases the surface area by creating micro-pores into which uncured flowable resin penetrates to provide durable micro-mechanical interlocking.⁽⁸⁾

The ceramic slices were treated with hydrofluoric acid etching prior to silane primer application, Etching was done for 60 seconds, results in the dissolution of the glassy phase predominantly and creating small isolated pores and fissures, & subsequent silanization was performed for 60 seconds this protocol coincide with **Güngör et al. in 2016**⁽⁹⁾.

The second applied surface treatment was sandblasting, which causes a new surface layer underlying the top bonding surface to be exposed. This new surface layer is highly pure and active. In view of its high surface energy, it has a high tendency to attract and combine with other chemical compounds, that causes

its surface energy to decrease.⁽¹⁰⁾ In this study, aluminum oxide powder of 50 microns was used at 90° angulation to the specimens' surface, at distance of 10 mm for 20 seconds and pressure of 2.8 bar. This protocol coincides with **Wahsh et al. in 2015**.⁽¹¹⁾

Silane primer was then applied afterwards for 60 seconds then gently air dried which coincide with Elsaka et al⁽¹²⁾: surface treatment of hybrid ceramic specimens either with HF acid or sandblasting, followed by chemical treatment with silane, is a more effective way to obtain higher bond strength values as silanization is inevitable to assure a durable bond following surface conditioning application, which was mentioned above. Thus, a better mechanical interlocking between disposed surface and substrate may be achieved⁽¹³⁾.

The third applied surface treatment was laser etching, the ceramic slices were subjected to laser irradiation followed by the application of silane primer. In this group Er,Cr:YSGG laser (Water laser Plus; Biolase Technology Inc., Irvine, CA, USA) with wave length 2780nm, pulsed laser-powered hydrokinetics, was used. as ER:YAG (erbium: yttrium, aluminum, garnet) laser was reported to remove the glass phase of the ceramic creating rough surface suitable for bonding to the resin cement.⁽¹⁴⁾ so Vapor and air were adjusted to 50% of the laser unit. The optical fiber of the laser unit were 400 μm in diameter and 4mm in length, arranged perpendicular over each ceramic slice and moved manually in a sweeping manner to cover all the surface area during the adjusted exposure period.

Power used was 2 W, with the repetition rate 20 Hz for 20 seconds at approximately one mm distance from the surface of the slices. The slices were then rinsed with distilled water and air dried. Silane primer was then applied to the irradiated surfaces for 60 seconds and then air dried for 60 seconds. This protocol coincides with **Omaira H. Ghallab, et al, in 2018**⁽¹⁵⁾.

Restorations normally fail after being aged in a humid and thermally dynamic oral environment.⁽¹⁶⁾

Thermal cycling is an artificial aging method of dental materials, and thermal strain is simulated on the bonding surface by influence

of liquids and thermal change⁽¹⁷⁾

In the present study aging protocol was applied on all 6 groups it was done through thermo-cycling after surface treatments were done and self adhesive resin was applied. it was done to simulate the oral cavity environment after cementing the restoration using self adhesive cement.

Under thermal aging, the bond strength is affected by several factors including temperature settings, dwell time, and the number of cycles, in which the latest is the most influential factor.⁽¹¹⁾

Mean while in our study the aging protocol was done after application of surface treatments and resin micro-tubes positioned. A total of 5000 thermal cycles were done, which simulates 6 month of *in vivo* function. Temperature between 50°C and 55°C with 20 seconds dwell time and 5 seconds transfer time. This aging protocol was also used by **Al-Thagafi in 2016**⁽¹⁸⁾

After finishing the aging step through thermo-cycling, specimens were ready for testing its bond strength with CAD/CAM hybrid ceramic material, using microshear bond strength test (μ -SBS test), Which is considered a relatively simple test that permits efficient screening of adhesive protocols, regional and depth profiling of a variety of substrates⁽¹⁹⁾

Most micro-shear studies use polyethylene tubes as molds, which are then filled with a resin composite. After water storage for 24 h, the operator uses a scalpel blade to remove these tubes manually, resulting in cylindrical composite specimens. The pressure exerted on the blade by the operator in order to cut and remove the polyethylene tubes may be transferred to the resin cylinder and consequently form cracks along the specimen.

Therefore, it is fair to hypothesize that micro shear specimens may fail under relatively low loading levels or fail prematurely due to propagation of these cracks.⁽²⁰⁾

For this reason in the present study, the polyethylene tubes irises were not removed in order not to subject theOrmocer micro cylinders to shear stresses at the interface and

to eliminate any pretest failures according to **Andrade et al. in 2012**⁽⁶⁾

CONCLUSION

Crystal Ultra showed the highest bond strength after sandblasting & silanation. In Vita Enamic HF acid etching & silanation had the highest bond strength than other surface treatments. Laser surface etching & silanation had significantly lower values for both materials used.

RECOMMENDATIONS

In Crystal Ultra, Sandblasting followed by silane application is recommended. In Vita Enamic, HF acid etching followed by silane application is recommended. Laser surface etching followed by silane application gives lower bond strength values but this may be due to the used intensity or duration so various investigations are furtherly required.

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