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EFFECT OF DIFFERENT SURFACE TREATMENTS ON BOND STRENGTH OF RESIN CEMENT WITH LITHIUM SILICATE CERAMICS

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ABSTRACT

Objective: This study was designed to evaluate different surface treatments affecting bond strength of different lithium silicate ceramics, due to long-term survival of adhesive esthetic restorations greatly depends on achieving maximum adhesive bonding strength, however one of the most common failures is loss of retention &de-bonding of ceramic restorations. **Materials and methods:** Total number of 42 lithium silicate squared samples were fabricated with a dimension of 1 cm^2, categorized as two groups (N=21) according to the two different materials. (Obsidian and Vita Suprinity). Every group of them receives three different types of surface treatments: acid etch, sand blasting and laser then all samples were bonded using a resin cement (RC) to a composite substrate. After that, all samples were subjected to thermocycling. Shear bond strength was assessed using a universal test machine while the surface treatment effect on the ceramic surface was observed by scanning electron microscope (SEM). **Results:** This study revealed that acid etching gave superior bonding strength ($8.91\pm2.42^{A\,a}$ for Obsidian and $9.05\pm2.87^{A\,a}$ for Suprinity) in comparison to sandblasting ($6.94\pm1.1^{A\,a}$ for Obsidian and $6.81 \pm 1.14^{AB\,a}$ for Suprinity), yet Laser gave the least results ($4.16\pm0.83^{B\,a}$ for Obsidian and $3.94\pm1.1^{B\,a}$ for Suprinity). **Conclusion:** Acid etching is the best surface treatment to be used with Lithium silicate ceramics concerning bond strength, Sandblasting could be used for much less retention, while Laser at the used parameters shouldn't be used as a surface treatment for Lithium silicate ceramics.

KEYWORDS: Glass ceramics, Acid Etch, Sandblasting, Laser, Shear Bond strength .

INTRODUCTION

In the last few years, many research has been focusing on ceramic development with intentions to test the hardness and the strength of the restorative materials, which resulted into high antagonistic tooth wear. Researchers have tried to mimic the mechanical behavior of natural teeth, so they created a bio-mimetic materials through running some modifications on ceramics to simulate the phyiscal properties of dentin & enamel. And still, they have strengthThat surpass conventional ceramics [(1)].

As a result for that new materials were introduced in dental market, glass ceramic materials was introduced as heat pressed and also being introduced for CAD/CAM use [(1)]. Recently in the past few years a new glass ceramic made of lithium silicate was introduced under the name of Obsidian[(2)].

In another attempt to enhance the physical properties of Zirconia- reinforced Lithium Silicate (ZLS) has been recently introduced to the market, with high mechanical and physical properties, for

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manufacturing of crowns in anterior and posterior area, veneers, onlays, inlays and superstructure of implant [3].

The final strength of these materials and hence their survival rate is totally dependent on their good bonding to the tooth structure. The proper adhesive cementation can increase fracture resistance in the ceramic by up to 69% [2].

Trying to enhance bonding of ceramics to resin cement, different surface treatments can facilitate chemical and micromechanical retention such as hydrofluoric acid etching, sandblasting, tribochemical like CoJet and laser) have been used. Etching the ceramic surface with hydrofluoric acid, one of the most preferred surface treatment methods, which is the best way of roughening ceramic surfaces for bonding resin composite [4].

Silane coupling agent was recommended to increase the bond strength between ceramics and RC, by increasing the surface energy of ceramic substrates and improves adhesive and/or cement wettability [5].

Many studies were made to utilize new techniques for surface treatment of ceramic materials to establish reliable bond strength to RC. Sandblasting with aluminum oxide particles is a surface treatment option that produces irregularities in the ceramic surface. It relies on blasting the ceramic surface with different particle sizes ranging from 30-250 micron[6]. Moreover, Er:YAG, Nd:YAG, and Er,Cr:YSGG lasers have been proposed as an alternative surface treatment to condition the surfaces of dental materials [7]. This different surface treatment and their effect on the bonding of ceramics has not been thoroughly investigated in the literature, specially comparing these different modalities.

Recent ceramics provide wide range of indications for restorative dentistry, however one of the most common failures is loss of retention &de-bonding of ceramic restorations, that's why achieving maximum adhesive bonding strength is a major goal. Therefore, durability of adhesive esthetic restorations is a big challenge and the strength of ceramic bonding, the luting agents and the dental substrates are the three factors impacting this challenge. In trying to enhance bonding of ceramics to RC, different surface treatments have been recommended to facilitate chemical and micromechanical retention, yet choosing the right protocol for each particular material is the key for success of different adhesive restorations [8].

The laboratory bond strength tests can be static or dynamic tests [9].In static tests, load is applied when the test specimen is stationery on the other hand in dynamic tests the specimen is in dynamic state. Static tests are categorized into macro tests where the bond area is >3 mm^2 and micro-tests with <3 mm^2 bond area [10]. Shear bond strength (SBS) testing with bonded cross-sectional areas of 3 mm^2 or less is referred to as "micro" SBS. It permits efficient screening of adhesive systems, regional and depth profiling of a variety of substrates, and conservation of teeth. A significant advantage over micro-tensile strength (TBS) methods is that the SBS specimen is pre-stressed prior to testing only by mold removal. However, the use of the mold for composite placement can lead to the introduction of flaws and different stress concentrations upon shear loading [11].

Therefore, this research was performed to study the effect of acid etching, sandblasting and laser etching over ZLS material (Vita Suprinity) and Lithium Silicate Ceramic All ceramic version (Obsidian) CAD/CAM Blocks on the shear bond strength of resin cement.

MATERIALS AND METHODS

Materials:

TABLE (1) Brand name, material description, composition, manufacturer and lot number of materials & equipment used.

Brand Name/ description	**Composition**	**Manufacturer**
RelyX™ Ultimate / Self-Adhesive dual cured luting RC	Base: methacrylate monomers; Radiopaque; salinized fillings; initiator and stabilizer components; rheological additives. Catalyst: methacrylate monomers; alkaline radiopaque; initiator and stabilizer components; pigments; fluorescent dyes; rheological additives, cure in the dark; activator of the Scotchbond Universal adhesive.	3M ESPE
Obsidian®/ Lithium silicate ceramic	More than 20 unique elemental oxides, SiO_2, Li_2O, K_2O-Al_2O_3, ZrO_2, P_2O_5, including zirconia (4-6%).	Glidewell
Vita Suprinity®/ ZLS	SiO_2 (58 – 64%), Li_2O (15-21%), K_2O (1-4%), P_2O_5 (3-8%), AL_2O_3 (1-4%), ZrO_2 (8-12%), CeO_2 (0- 1%), La_2O_3 (0.1%), Pigments (0-6%)	Vita Zahnfabrik
IPS ceramic/ etching gel	5% HF	IvoclarVivadentSchaan, Liechtenstein, Germany
Bisco Silane Primer/silane coupling agent	Pre Hydrolized porcelain silane	Bisco, Inc. Schaumburg, USA
Cobra/ sandblasting abrasive	99.7% Al2O3 of 50-micron in particle size	RenfertHilzingen, Germany
Water lase i plus	Er,Cr:YSGG laser of 2780 nm wave length	Biolase technology Inc, Irvine, CA, USA

Samples grouping:

A total sample size of 42 specimens (7 for each group) was determined according to a one-way ANOVA study, it was calculated with a power of 95% and a significance level of 95%.) [12]. Sample size was calculated using G power version 3.1.9.7 (Heinrich-Heine-Universität Düsseldorf, Düsseldorf, Germany).

Twenty-one specimens of either Vita Suprinity (group 1) and Obsidian (group 2) are categorized in two groups. Each group were subdivided in to three different subgroups to received three different types of surface treatment.

Preparation of the specimens:

Blocks of the two CAD CAM glass ceramic materials (VITA suprinity and Obsidian) were used to prepare slices with the following dimensions: 10mm x 10mm x 1mm for Vita Suprinity ceramic material & the same for Obsidian ceramic material. Using IsoMet 4000 microsaw (Buehler USA) with cooling water system, by a diamond disk 0.6 mm thickness with cutting speed 2500 rpm. Then each ceramic disc was examined with a digital Caliber to make sure they all had the same thickness 1mm each.

Surface Treatments:

Before surface treatment, all the specimen were crystallized according to manufacturer instructions in Vita Vacumat 6000MP ceramic furnace [2] then embedded in an acrylic block to ease the handling and fixation during the shear test. 70% ethyl alcohol was used for cleaning the surface from any debris and drying these surfaces very well.

1) HF Etching + Silane:

Seven of the Obsidian, and of the Vita Suprinity ceramic were etched using HF 5% for 30 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 [13].

2) Sandblasting:

Sandblasting was performed using 30 microns aluminum oxide powder at an angle of 90, distance ≈10 mm for better standardization for sandblasting done in all specimens a cylindrical metal holding device which allowed sandblaster tip movement right, left, up & down directions without changing the distance which gave us better standardization. This was done for 20 seconds and 2.8 bar pressure for each ceramic disc in this subgroup. 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 [13].

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 lased-powered hydrokinetics, was used. Vapor and air were adjusted to 50% of the laser unit. The optical fiber of the laser unit was 400µm in diameter and 4mm in length, arranged perpendicular at distance≈ 1mm 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 with a repetition rate 20 Hz for 20 seconds at surface of the slices (**Ghallab**, et al, 2018) [14]. The slices were then rinsed with distilled water and airdried. Silane primer was then applied to the irradiated surfaces for 60 seconds and then air dried for 60 seconds.

Application of resin cement (RC) material and Composite substrate construction:

A specially constructed cylindrical split Teflon mold was fabricated. The mold has a circular central hole 2 mm in diameter and 2 mm in thicknesses, with an outer cupper ring that served for the assembling of the two halves of the Teflon mold. Forty-two composite resin discs (2mm diameter and 2mm thickness) A3 shade were fabricated according to manufacturer’s instructions.

A thin layer of separating medium was applied on the Teflon mold that was seated on a dry clean glass slab. Composite resin was applied incrementally, each increment was 2 mm in thickness using a non-metallic plastic instrument. After application of the increment, a mylar strip was pressed on the glass plate in order to provide optimum smoothness. A LED light curing unit (Miraj, LED.D curing light, Korea) with a mean light intensity of 1400 mW/cm^2 and optical wavelength of 420-480 nm was used for composite resin activation for 40 seconds.

Cementation of specimens after surface treatment:

The etched surfaces were thoroughly rinsed using water spray for 60 seconds, followed by ultrasonic cleansing in distilled water for another 60 seconds, then dried by oil-free compressed air for 30 seconds. A layer of silane coupling agent was applied to the etched surface for 60 seconds followed by air thinning.

The cemented side of composite resin discs were manually finished using wet silicon carbide paper 320 and 600 grit (Norton S.A., São Paulo, Brazil) then washed with tap water for 1 minute, and ultrasonically cleaned in distilled water for10 minutes[15].

In order to stabilize the applied load, a unique cementation machine was fabricated from stainless steel and formed from several parts: A cementation mold to tie all samples in the base of the machine. The mold can be classified into 4 sections: Teflon section that consists of 2 Teflon halves engraved from the middle to form a hole with 2mm diameter and 2 mm thickness to keep the composite diameter and substrate fixed.

Aside from Teflon section, there are 2 metal sections (A& B)both are circular in shape and have a squared hole in the center and 10 mm inner diameter, 0.5 mm thickness and 2 mm thickness respectively. Part A is creating a way out for extra cement while Part B is designed to fixate the ceramic disc in the center.

Now moving to the Teflon Ring: its outer & inner diameter is 30 mm and 25 mm respectively. Teflon section was created for the purpose of holding the other parts during loading.

Cementation device (fig 1) that consists of 4 parts: a) Two horizontal metal plates rectangular in shape (upper and lower) with sample fixation screws connected together with two vertical metal arms attached to both plates. b) Two supporting vertical metal arms attached to the upper and lower horizontal metal plates. c) A T shaped metallic rod is attached to the upper metal plates and can move freely vertically, it also carries at its upper end a disc shaped plate over which the required load will be placed, while at its lower end a Teflon rod with 10 mm diameter tip was attached. d) A fixed load was placed on the disc shaped plate of part (c) [16].

Cementation was accomplished according to the manufacturer instructions. Dual cure Rely X Ultimate (A self-adhesive resin luting agent) was used. The cement was injected from the double- push syringe and mixed within the spatula according to the manufacturer recommendation to the prepared surface of each composite substrate.

The cementation mold was placed on the rectangular shaped metallic base of the cementation device, and then secured in centralized position by fixation screw to ensure its placement in the same location each time during cementation. Each composite disc was positioned inside the Teflon mold cavity part (a) with its prepared surface facing the luting cement. The metal part (b) was then placed over part (a), and then part (c) was placed over them to secure the ceramic disc in place. Finally, Teflon ring accommodated the three parts (a, b and c) together during loading.

A 1 Kg constant load was applied on the disk-shaped plate at the upper end of the T-shaped metallic part of the cementation device and was left for 3 minutes. The excess cement was then removed with a sharp lancet from the corner of the square hole of metal part (b), the resin material was polymerized from three directions for 40 seconds with light cure device at a power of 3.200 mW/cm2. After completion of curing, the outer Teflon ring was removed to disassemble the cementation mold parts, the cemented ceramic discs were removed, then Finishing was made using finishing bur and according to manufacturer recommendations by the same operator for standardization.

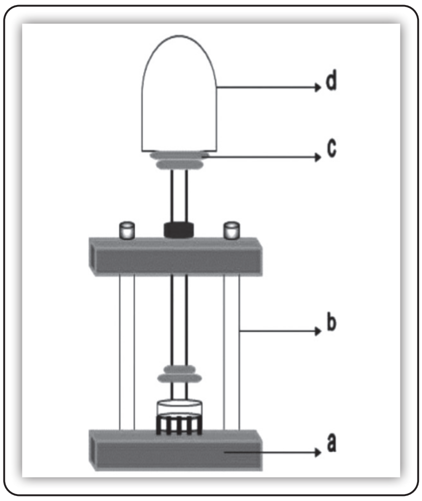

FIG (1) Schematic diagram for the cementation device

Thermocycling:

All cemented specimens where subjected to aging factor through a custom-made thermocycling machine where the thermocycling unit contains a hot water path (55ºC±1) and a cold-water path (5ºC±1), the samples where immersed in each path for 15 seconds. 5000 cycle where adjusted using the control board of the machine which is equivalent to 6 months of restorations serving in oral cavity [17].

Shear Bond Strength Test:

Each block with its own bonded specimen 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 shearing load with tensile mode of force was applied via materials testing machine at a crosshead speed of 1mm/min. Debonding load was measured in Newton.

Bond strength was calculated by dividing the load at failure over area of bonding:

$\tau = P/ \pi r2$

Where; τ= 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 specimen of Vita Suprinity & one other disc from Obsidian was examined under the scanning electron microscope before and after any surface treatment done to be able to compare it afterwards with the different surface treatments done in our study. Each specimen was mounted on aluminum stud & examined with scanning electron microscope using low vacuum mode with magnification of 2000 x (JSM-5400LV, Jeol, Jeol Ltd. Massachusetts, USA).

Statistical analysis

Results were presented as mean and standard deviation (SD) values. Data were explored for normality using Kolmogorov-Smirnov test and for homogeneity using Levene test. The data showed a normal and homogenous distribution (parametric data). Therefore, one way analysis of variance (ANOVA) test was used to compare the results of shear bond strength test between different surface treatment methods. This was followed by Tukey's post-hoc test when the difference was found to be significant. Independent T-test was used to compare the results of shear bond strength test between the two tested materials. The significance level was set at $p < 0.05$. For analysis of mode of failure, Chi square test was used. Statistical analysis was performed with SPSS 25 for Windows (Statistical Package for Scientific Studies, SPSS, Inc., Chicago, IL, USA).

RESULTS

TABLE (2) Mean (MPa) and standard deviation for shear bond strength (MPa) of different groups.

	Acid Etching Mean (MPa) ± S.D	Sandblasting Mean (MPa) ± S.D	Laser Mean (MPa) ± S.D	P value
Obsidian	$8.91 \pm 2.42^{A\,a}$	$6.94 \pm 1.1^{A\,a}$	$4.16 \pm 0.83^{B\,a}$	0.002 *
Vita Suprinity	$9.05 \pm 2.87^{A\,a}$	$6.81 \pm 1.14^{AB\,a}$	$3.94 \pm 1.1^{B\,a}$	0.005 *
P value	0.937	0.875	0.729	

** Indicates the mean difference is statistically significant at the 0.05 level.*

Different capital litter indicates statistically significant difference in the same rows. (p-value ≤ 0.05).

Different small litter indicates statistically significant difference in the same column. (p-value ≤ 0.05).

The highest shear bond strength was recorded after treating the surface of Vita Suprinity with acid etching method (9.05 MPa ±2.87), followed by samples of Obsidian treated by sandblasting (8.91 MPa ±2.42), followed by Obsidian samples treated by acid etching (6.94 MPa ±1.1), followed by samples of sandblasted Vita Suprinity samples (6.81 MPa ±1.14), followed by Obsidian samples treated by Laser (4.16 MPa ±0.83). However, the lowest shear bond strength value was recorded in Vita Suprinity samples treated by Laser (3.94 MPa ±1.1). (Table 2).

SEM Analysis for the ceramic surface before and after surface treatment:

The alterations in surfaces after surface treatment methods were evaluated with SEM at a magnification of 2000×, SEM images of The Al2O3 sandblasted surfaces of Vita Suprinity showed clearly elevated and depressed micro-size areas with crevices and pits caused by sandblast particles. While for sandblasted Obsidian surface revealed an irregular surface and microcracks. The SEM images of HF acid treated groups showed the surface alterations on the treated surface with several randomly distributed and irregular micropores and gaps rendering a honey comb appearance, partially dissolved and glassy phases of glass ceramic can be seen. While SEM images of HF acid etching for Obsidian group showed tiny micro-pores and pits were appeared on the Obsidian surface leaving a smooth surface without extensive dissolution after HF acid application.

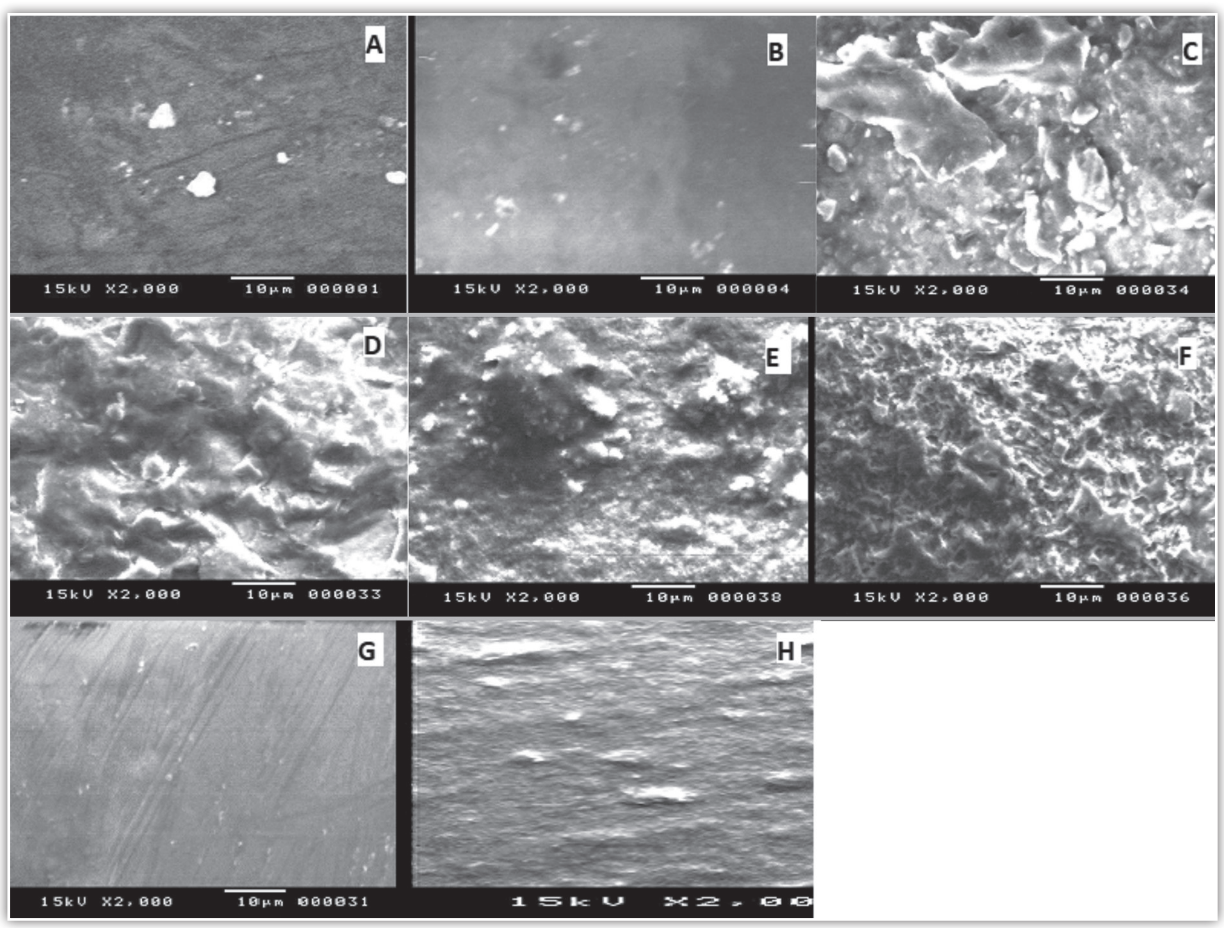

FIG (2) SEM photomicrograph of tested materials. A: untreated Vita Suprinity, B: untreated Obsidian, C: acid etched Vita Suprinity, D: acid etched Obsidian, E: sandblasted Vita Suprinity, F: sandblasted Obsidian, G: laser treated Vita Suprinity and H: laser treated Obsidian.

The SEM analyses verified the surface roughness results, except for Er,Cr:YSGG laser-treated group. Because in the analysis of the SEM images of the laser group, a deep laser craters and traces of ablation, combustion, and melting of the Er,Cr:YSGG irradiation on the hybrid ceramic block surfaces can be observed.

DISCUSSION

Recently, the implication of CAD/CAM technology in the led to a revolution in dental ceramics development regarding optical properties, microstructure and created many new products being introduced in very short intervals [18]. The two materials used in this study; VITA Suprinity and Obsidian are of the newly introduced CAD/CAM materials, they both lie under the same category of ceramics. The Obsidian as Lithium silicate ceramic and VITA Suprinity as zirconia reinforced ceramic[19].

The clinical success of a ceramic restoration is mainly dependent on the quality and durability of the bond between the RC and the restoration [20]. Hence, it is recommended to apply various surface treatments to improve bonding of RC to ceramics[21].

Different surface treatments were applied on the CAD/CAM materials surface to be evaluated and tested, these surface treatments include air-particle abrasion (50 um AL_2O_3), acid etching (5% buffered HF and ErCr:YSGG laser treatment, were applied. These methods are commonly used surface treatments for bonding of ceramic restorations [22].

The first applied surface treatment was sandblasting, which exposes a new surface layer high in purity and activity beneath the top-bonding surface; the surface energy of the new layer decreases when combined and attracted to other chemicals compounds [23].

The second applied surface treatment was HF etching. Which is the most commonly employed technique to improve the bond strength. Etching increases the surface area by creating micro-pores into which uncured flowable resin penetrates to provide durable micro-mechanical interlocking [21]. Etching for a short duration of 60 seconds results in dissolution of the glassy phase creating small isolated pores and fissures [25].

The third applied surface treatment is Err: YSGG laser treatment. Irradiation the surface with laser is a recent surface method to promote the bond strength of restorative materials to RC [26,27]. Barutcigil et al. concluded that 2W Er, Cr:YSGG irradiation has the same efficiency as sandblasting and HF application on bond strength of Vita Suprinity to RC [28].

Although laboratory evaluation and in-vitro studies cannot exactly simulate conditions in the oral cavity, such as the clinical environment, moisture and stresses inflicted on teeth and restorations, they can to some extent, simulate the oral cavity environment through aging procedures of teeth and/or restorations In the field of laboratory research, out of the currently available systems able to reproduce dynamic stresses, thermal cycling is one of the most widely used procedures that is also widely accepted in international literature [17].

Bond strengths to RC was found to be significantly higher in VITA Suprinity ceramic compared to Obsedian ceramics. Elsaka et al, supported that Vita Suprinity has higher mechanical properties [21]. So, it was found that the higher filler content of zirconia (10% by mass) in Vita suprinity was the reason behind its better bond strength when compared with Obsidian ceramics.

Those findings were revealed by Era Cengiz-Yabardag et al in 2018, they have determined that the best surface treatment is to use 5% HF acid as a treatment before bonding for all CAD-CAM restorative materials regarding the bond strength. They have stated that highest SBS values were achieved using HF in compared to other treatments[29].

Puppin-Rontani et al. mentioned that regardless of etching times, the higher the HF concentrations, the higher the SBS values, the greater the dissolution of the vitreous phase and exposure of lithium disilicate crystals. [30].

Results within each material in this study stated that the best surface treatment for Glass ceramics in general would be Hydrofluoric acid when compared to Sandblasting and Laser groups, Although Obsidian showed higher results regarding Sandblasting. Yet it was statistically insignificant.

Yoshihara et al in 2017, explained that sandblasting achieved similar surface energy values and strong roughness. High pressure sandblasting as demanded by the manufacturers achieved an average increase in roughness values. Thus, sandblasting composite CAD/CAM materials with reduced pressure is recommended [31].

Strasser et al in concluded that the sandblasted specimens showed better results than HF etching, which was in agreement with this study concerning Obsidian, stating that sandblasting was practiced more clinically than the other procedures [32].

On the other hand, Laser application on both Vita suprinity and Obsidian showed the least results when compared to HF etching and Sandblasting. Those results were verified by using SEM images for laser group which posses micro-pores and irregularities.

According to Gokce et al. In 2007 , lower values of bond strength following higher setting of laser power were justified by heat damaged layer formation because of the higher power of laser . This layer may have been poorly attached to the restorative material infra layers , despite that the outer layer which is strongly bonded to the silane and RC [33].

Cengiz-Yabardag et al in 2018 , got the conclusion of having modifications on the restorative meterials surface were due to both energy level and irradiated material type of the laser radiation, stating that glass ceramic can be etched by ER,CR:YSGG laser with 3W energy level, which was in agreement with this study [29].

Concerning the best surface treatment that goes with VITA Suprinity, HF etching showed significantly higher values among Sandblasting and Laser groups.

According to Elsaka et al in 2014 HF forms micro-porosities on the restorative material surface, which increases the surface, area and enhances the establishment of mechanical interlocking with luting resin, and that roughening the indirect esthetic restorative material surface followed by silanization was anticipated as the preferred technique to achieve a high bond strength between indirect restorations and resin based luting agents which came in agreement with this study [21].

This also came in agreement with Campus et al in 2016, who stated that the ceramic content of the PICN would “guide” the surface treatment, consequently, HF etching was the most reliable treatment where the glass content of this kind of ceramic suffers a selective dissolution when exposed to HF, increasing the surface roughness and promoting a better micromechanical interlocking with the RC [34].

Limitations of this study was excluding other factors such as cyclic loading, multiple firing, different acidities intra orally and other factors that might affects the final results, also it’s a vitro study where the intra oral environment is a multi-factorial incident that could occur all at once.

CONCLUSION

Keeping the limitations of this study into consideration, the conclusion can be stated as follows:

- The best surface treatment for high bond strength for both Vita Suprinity and Obsidian is HF etching followed by silane.
- Laser etching treatment of glass ceramic is not sufficient to obtain a high bond strength.

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