

ASSESSMENT OF ER, CR: YSGG LASER SURFACE TREATMENT AND SELF-ADHESIVE RESIN CEMENTS FORMULAE ON MICROTENSILE BOND STRENGTH TO VARIOUS CAD/CAM CERAMIC ESTHETIC MATERIALS: AN IN VITRO STUDY

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ABSTRACT

Purpose: To assess the effect of Er,Cr:YSGG pulsed laser surface treatment versus hydrofluoric acid etching on microtensile bond strength (μ TBS) to three different types of CAD/CAM ceramic materials cemented with two self-adhesive resin cements.

Materials and Methods: Ceramic slices (n=24) were prepared from three different types of CAD/CAM blocks: Vita Enamic (group: VE), IPS e.max CAD (group: EM) and Obsidian (group: OB). Two surface treatments were applied followed by silane primer: Er,Cr:YSGG laser (Subgroup ER) and hydrofluoric acid (Subgroup HF as a control). ESEM evaluation was performed before and after surface treatments, accompanied with measurement of roughness (Ra). Two self-adhesive resin cements were used: BisCem (Division BC) & Panavia SA Cement plus (Division PA). After 24 hours storage in distilled water, the ceramic-resin blocks were sectioned into microbeams. A total of 480 microbeams were subjected to (μ TBS) testing. Data was tabulated and statistically analyzed.

Results: ESEM revealed variations in surface texture. Regarding VE group, HF recorded statistically higher significant μ TBS with PA. While, EM group, HF showed statistically higher significant μ TBS for both cements compared to ER. OB group HF showed statistically higher significant μ TBS with PA in contrast to ER which showed statistically higher significant μ TBS with BC.

Conclusions: The interaction of Er,Cr:YSGG pulsed laser with the surfaces of the tested CAD/CAM materials seemed to be different and dependent on the crystalline structure of these ceramics. Variation of the chemical formulae of self-adhesive resin cements played a great role in determining the μ TBS to the tested CAD/CAM materials.

KEY WORDS: Ceramic- Er,Cr:YSGG laser- Resin cements- Microtensile Bond Strength.

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INTRODUCTION

The growing development on the efficacy of CAD/CAM ceramic materials was designed with the objective of promoting highly esthetic restorations.³² Although the great improvement in their mechanical efficiency in addition to biocompatibility,^{24,42} these restorations endured low resistance to fracture and brittleness.²³ Many attempts were employed to overcome these problems as incorporation of different crystals such as leucite, fluormica or alumina in order to increase the resistance to crack propagation leading to their structure reinforcement.³⁴ Also, the incorporation of lithium dioxide to glass ceramics improved the mechanical properties enabling the ceramic material to be used in different indications without compromising their esthetic quality.¹⁹

Lithium Disilicate ceramic (IPS e.max) (Ivoclar Vivadent; Schaan, Liechtenstein) has become one of the most commonly used monolithic ceramics with high mechanical and esthetic characteristics for different indications as veneers, inlays, onlays, single crowns, three-unit anterior fixed partial dentures and crowns for implants with flexural strength up to 440 MPa.^{1,8}

Recently, a new lithium silicate ceramic has been introduced to the dental market known as (Obsidian Lithium Silicate Ceramic) (Glidewell Dental Laboratories, Newport Beach, CA, USA). The material is indicated for various esthetic restorations. In contrast to bilayered ceramics, Obsidian ceramic restorations are resistant to chipping, as a result of their monolithic composition and flexural strength (385MPa). This new ceramic material is claimed to combine the merits of esthetics; they exhibit translucency reflecting the vitality of natural teeth, and strength which exceeds the ISO requirements. They can be conventionally or adhesively cemented. According to the manufacturer, Obsidian is formed of a combination of over 20 elemental oxides including zirconia. Also, it contains high content of

ultrafine nano-meter size lithium silicate and lithium phosphate crystals.²⁰

A polymer-infiltrated ceramic network material (Vita Enamic hybrid ceramic material) (Vita Zahnfabrik, Bad Säckingen, Germany) has been earlier introduced, which contains a dominant ceramic network (86% by weight), reinforced with acrylic resin polymer network (14% by weight).⁴⁵ Therefore, the hybrid structure combines the positive merits of ceramics regarding the high strength and color stability with that of resin composites as reduced brittleness providing a cushion effect thus reducing the mastication forces, enhanced flexibility with fewer fracture rate and low abrasiveness, so less damage occurs to the opposing teeth.^{11,18,41}

Adhesive cementation of indirect restorations provides many benefits such as enhancement of marginal adaptation, reducing microleakage, improved retention and increased fracture resistance.^{6,22,30} The success of the indirect restorations is greatly dependent on the efficient adhesion at the restoration/cement / tooth interfaces.³⁵ In order to optimize the bonding efficacy between the indirect restorations and the resin cement, micromechanical as well as chemical surface treatments should be applied. Different surface roughening procedures and adhesion promoting agents were used.²⁵ Roughening may include grinding with diamond rotary instruments,⁴⁰ silica coating,^{7,39} air abrasion using aluminum oxide particles,¹⁶ sandblasting,^{26,39,44} hydrofluoric acid etching,^{21,36} laser,^{21,39,44} or combination of any of these procedures. In previous studies, treatment of the ceramic surface with hydrofluoric acid etching prior to silane coupling agent was reported with a proven success.^{6,35,36,44} Hydrofluoric acid is characterized by its tendency to disintegrate the glassy phase of the ceramics, thus exposing the crystals creating micro porosities into which the silane coupling agent is infiltrated providing micromechanical retention and chemical bonding ability with ceramic and the

overlying resin adhesive cement.^{10,13,46} However, this approach represents health hazards to both the patient and the operator, and cannot be used in repair cases, where a more biocompatible treatment is preferred.²⁸ Er:YAG, Nd:YAG, and Er,Cr:YSGG lasers have been proposed as an alternative surface treatment to condition the surfaces of dental materials.¹² 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.²¹ The wavelength of Er:YAG (2940 nm) and Er,Cr:YSGG (2780 nm) lasers are considered similar, so their results could be compared.²⁸

Resin cements can be categorized according to their adhesive protocol into total-etch, self-etch or self-adhesive cements.⁴³ Self-adhesive resin cements, are one-step cements which do not need any pretreatment for the dental hard tissues.^{31,43} These resin cements contain carboxylic acid groups or phosphoric acid groups attached to (meth)acrylate monomers,⁴⁷ where, these acidic groups bond chemically to hydroxyapatite crystals through hydrogen bonds after their hydrolysis in addition to metals and zirconia.¹⁷ Moreover, 10-MDP monomer (10-methacryloyloxydecyl dihydrogen phosphate) owns the ideal bonding agent property; that its polar phosphate group is initially hydrophilic, but becomes more hydrophobic once polymerized.²

The impact of Er,Cr:YSGG (erbium, chromium: yttrium, scandium, gallium, garnet) pulsed laser on the adhesion efficiency between the resin cements and ceramics was not extensively declared in the previous literature²⁸. Accordingly, the objectives of this research was to evaluate the influence of Er,Cr:YSGG radiation versus hydrofluoric acid surface treatment on the μ TBS to the newly introduced (Obsidian, Vita Enamic) and IPS e.max when cemented with two self-adhesive resin cements formulae.

The tested null hypotheses were, first: there was no difference between Er,Cr:YSGG laser and hydrofluoric acid surface treatment on μ TBS to the tested CAD/CAM ceramics. Second: there was no effect of the type of self-adhesive resin cement formula on μ TBS to the tested CAD/CAM ceramics.

MATERIALS AND METHODS

Three groups of CAD/CAM materials were selected for the current study: Vita Enamic (VE), IPS e.max CAD (EM) and Obsidian (OB), with two surface treatments: Er,Cr:YSGG laser (Subgroup ER) and Hydrofluoric acid etchant (Subgroup HF as a control) followed by Silane primer. Two self-adhesive resin cements were used: Panavia SA Cement plus (Division PA) and BisCem (Division BC). The manufacturers and composition of the materials are listed in Table 1.

TABLE (1): Material type, product name, manufacturer and composition:

Material Type	Product Name	Manufacturer	Composition*
Lithium disilicate ceramic	IPS e.max CAD blocks	IvoclarVivadent; Schaan, Liechtenstein	SiO ₂ , Li ₂ O, K ₂ O, P ₂ O ₅ , ZrO ₂ , ZnO, Al ₂ O ₃ , MgO and colouring oxides.
Lithium silicate ceramic	Obsidian glass ceramic blocks	Glidewell Dental Laboratories, Newport Beach, CA, USA	More than 20 metal oxides and zirconia
polymer infiltrated ceramic network	Vita Enamic blocks	Vita Zahnfabrik, Bad Säckingen, Germany	SiO ₂ , Al ₂ O ₃ , Na ₂ O, K ₂ O, B ₂ O ₃ , CaO, TiO ₂ , TEG-DMA, UDMA

Material Type	Product Name	Manufacturer	Composition*
Dual-cured self-adhesive resin cement (1)	Panavia SA Cement Plus	Kuraray Noritake; Okayama, Japan	Paste A: 10-MDP, Bis-GMA, TEGDMA, HEMA, hydrophobic aromatic dimethacrylate, silanated barium glass filler, silanated colloidal silica, dl camphorquinone, peroxide, catalysts and pigments. Paste B: hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, silanated barium glass filler, silanated colloidal silica, surface treated sodium fluoride, accelerators and pigments. Approximately 40 vol% inorganic fillers. Particle size (0.02-20 μ m)
Dual-cured self-adhesive resin cement (2)	BisCem	BISCO, Schaumburg, IL, USA	Base: Bis-GMA, uncured dimethacrylate monomer, Di-HEMA glass filler (36 vol% with particle size 1 μ m) Catalyst: phosphate acid monomer, glass filler (40 vol% with particle size 3.5 μ m)
Acid Etchant	Porcelain Etchant	BISCO, Schaumburg, IL, USA	9.5% Buffered hydrofluoric acid gel
Single component Pre-Hydrolyzed silane primer	Porcelain Primer	BISCO, Schaumburg, IL, USA	Silane Coupling Agent in an alcohol and acetone base

* According to the manufacturers' data. TEG-DMA: triethyleneglycoldimethacrylate; UDMA: urethane dimethacrylate; 10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate; Bis-GMA: bisphenol-A-glycidylmethacrylate; HEMA: hydroxyethyl methacrylate.

Preparation of Ceramic slices:

A total of 24 slices were cut off CAD/CAM ceramic blocks. The blocks were cut using water cooled diamond blade with a low speed cutting saw (Isomet 4000, BUEHLER, Lakebluff, USA) to obtain slices with dimensions of (10 x 10 x 3 mm) (n=8 slices for each ceramic material). The slices were then polished in a circular motion using silicon carbide papers of grits 600 and 1200 under continuous water irrigation for 20 seconds each. The slices were then cleaned ultrasonically in distilled water for 30 seconds and air dried.

Surface Treatments:

The slices of each CAD/CAM materials were randomly divided into two subgroups (n=4 for each subgroup) according to the surface treatments applied. Er,Cr:YSGG (ER) Treatment: The ceramic slices were subjected to laser irradiation followed by the application of silane primer. In this group Er,Cr:YSGG laser (Water lase i Plus; Biolase Technology Inc., Irvine, CA, USA) 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 were 400 μ m in diameter and 4mm in length, was

arranged perpendicular over each ceramic slice and moved manually in a sweeping manner to cover all the surface area during the adjusted exposure period. For EM, OB groups, the laser parameters were adjusted so that, the power was 5 W,^{28,38} while it was 2 W for VE⁴ group. The repetition rate was 20 Hz for 20 seconds at 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.

Hydrofluoric (HF) acid etching (control): The ceramic slices were treated with hydrofluoric acid etching prior to silane primer application. In this group, Porcelain Etchant was applied to the slices followed by application of Porcelain Primer. According to manufacturer's instructions for ceramic etching; 20 seconds for EM, 10 seconds for OB, and 60 seconds for VE. This was followed by rinsing for one minute and air drying for all the slices. After that, silane primer was added and left for 60 seconds, then air dried for another 60 seconds.

Environmental scanning electron microscopic (ESEM) analysis of ceramic material surfaces:

Two slices from each ceramic material were evaluated at base line (non- treated) and after treatment with either ER or HF acid etching (control) for assessment of the surface texture using ESEM analysis. Ceramic material surface topography was evaluated using ESEM Model Quanta 250 FEG (Field Emission Gun) attached with EDX Unit (Energy Dispersive X-ray Analyses), with accelerating voltage 30 K.V, magnification 14x up to 1000000 and resolution for Gun.1n) in The Egyptian Mineral Resources Authority ,Central Laboratories Sector, Giza, Egypt). Electron micrographs were obtained at 30 kilovoltage (KV) using secondary electron live fiber detection (LFD) detector under the magnification of (2000x) with a spot size (4.7 - 5.3 nm) for each CAD/CAM material slice. Additionally, roughness parameter (Ra) in μm unit for each previously ESEM evaluated ceramic slice

was measured by an image analysis attached to the ESEM unit at (500x).

Application of Self Adhesive Resin Cements:

Two self-adhesive resin cements were selected for this research: Panavia SA Cement Plus Automix (Division PA) and BisCem (Division BC). Twelve subdivisions of three CAD/CAM materials (two slices each) were obtained. The two self-adhesive resin cements were directly applied to the silanated HF acid etched and laser irradiated surfaces in a single increment of 3 mm height within a special silicon mold.²⁸ A transparent glass slide was then applied to the surface of the cement in order to obtain a flat uniform surface. The resin cement was then light cured using LED light curing unit (Elipar S 10, 3M, St Paul, USA) with an output 1200 mW/cm² for 60 seconds. The mold was then removed and each side of the resin cement was light cured for additional 20 seconds. The ceramic slices with bonded resin cement blocks were stored in distilled water for 24 hours before subjecting to microtensile bond strength testing.

Micro Tensile Bond Strength (μTBS) Testing:

A total of 24 blocks composed of ceramic slices with overlying self-adhesive resin cement were vertically sectioned using water cooled diamond blade with a low speed cutting saw (Isomet 4000, BUEHLER, Lakebluff, USA) into serial slabs followed by rotating the block 90° to make additional vertical cuts in order to obtain thin long microbeams with the following dimensions (1 mm x 1 mm x 6 mm). The peripheral microbeams were discarded to avoid excess or deficient resin cement at the periphery which might affect the results. Twenty microbeams were obtained from each block (40 microbeams for each division) (with a total of 480 microbeams). Geraldini's jig was used to mount the microbeams onto the universal testing machine (Instron 3345, Instron, Norwood, Massachusetts, USA). Each microbeam was aligned in the central groove of the jig, glued in place by its ends using cyanoacrylate based glue. The jig was then placed

onto the universal testing machine with a load cell of 500 N. At a cross-head speed of 0.5 mm/min, tensile loading was kept until debonding failure of each microbeam was obtained. Microtensile bond strength was calculated in Mega Pascal (MPa) by dividing the load of debonding failure (Newton) over the whole bonded surface area (mm^2).

Statistical Analysis:

The mean and standard deviation values were calculated for each study variable. Data was explored for normality using Kolmogorov-Smirnov and Shapiro-Wilk tests and data showed parametric (normal) distribution. One-way ANOVA followed by Tukey post hoc test was used to compare between more than two non-related samples. Independent sample t-test was used to compare between two non-related samples. The significance level was set at $P \leq 0.05$. Statistical analysis was performed with IBM® SPSS® Statistics Version 20 for Windows. Three-way ANOVA was used to show the interaction between the study variables.

RESULTS:

ESEM Analysis:

The ESEM photomicrographic evaluation of the three tested materials revealed variations in surface texture and surface roughness (Ra) measurements as shown in figures (1- 9).

VE group with ER treatment (Fig. 2) revealed a mild effect on the surface roughness as the irradiated surface showed traces of ablation and melting of the surface, while for the HF acid etched surface (control), a high degree of surface roughness was revealed (Fig. 3).

EM group with ER treatment (Fig. 5) showed a relative ablated smooth surface, while for the HF acid etched surface (control) showed distinct open pits distributed all over the material surface (Fig. 6).

OB group with ER treatment (Fig. 8) revealed discrete areas of irregularities, while for the HF acid etched surface(control), the surface irregularities obviously increased with visible Lithium silicate crystals (Fig. 9).

Microtensile bond strength (μ TBS) results for the tested divisions:

The mean in (MPa), standard deviations (SD) and P-values for all tested study variables are presented in table (2).

Regarding (group VE), the subgroups treated with (ER) showed no statistically significant difference between the μ TBS mean values for both resin cements. While for the subgroup treated with (HF) acid etching (control), (PA) recorded higher statistically significant mean value of μ TBS than (BC).

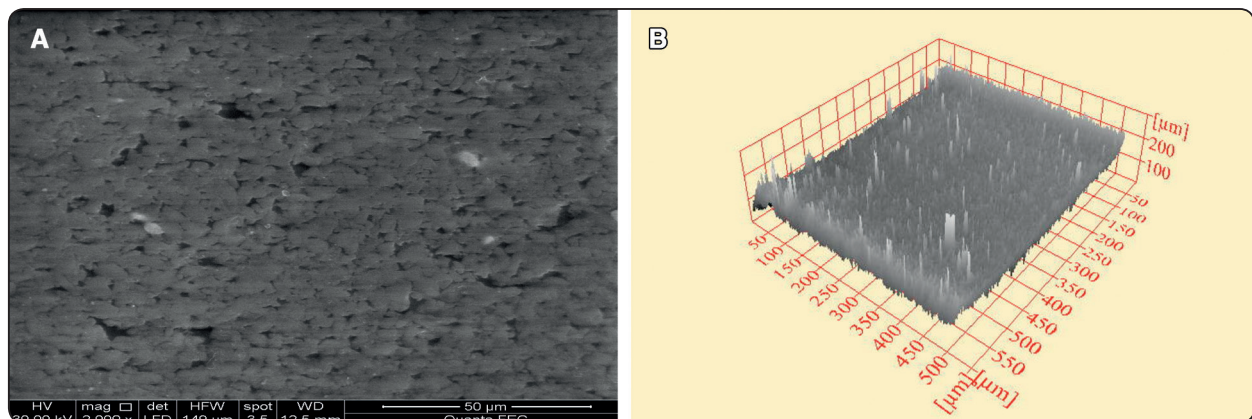


Fig. (1): (A): ESEM Photomicrograph (2000x) of Vita Enamic material (VE) non treated surface showing relative degree of smoothness. (B): image analysis of roughness (500x) with $R_a=10.15 \mu\text{m}$.

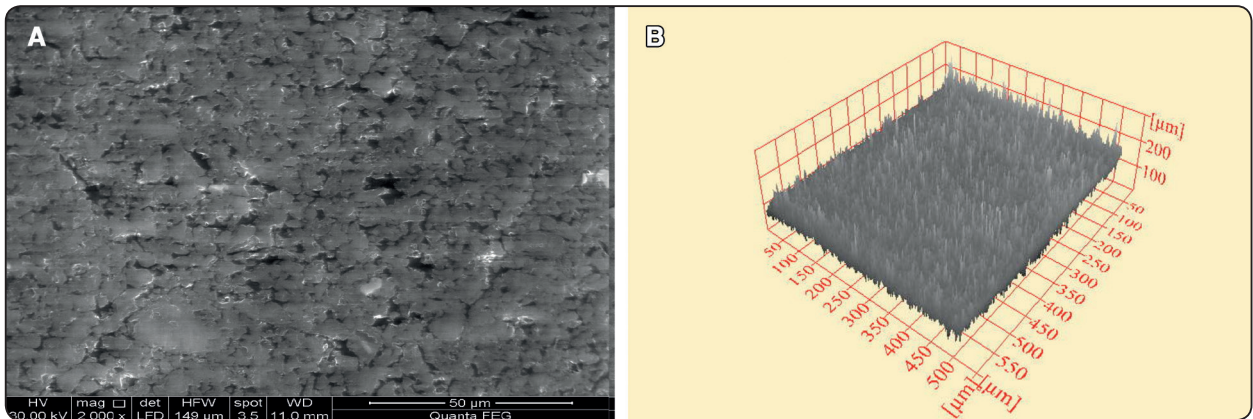


Fig. (2): **(A)**: ESEM Photomicrograph (2000x) of Vita Enamic material (VE) surface treated with ER showing mild degree of surface irregularities similar to non-treated (VE). **(B)**: image analysis of roughness (500x) with $R_a=11.44 \mu\text{m}$.

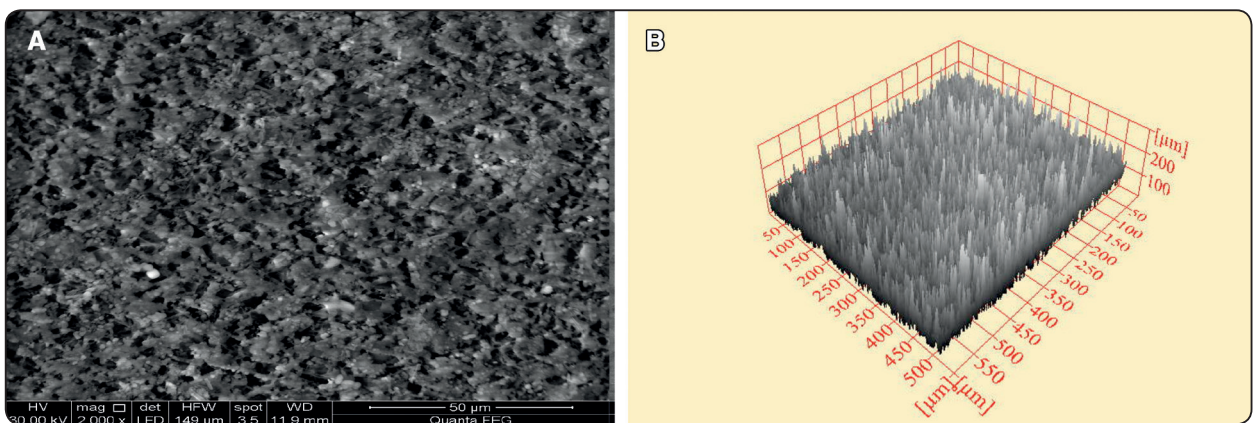


Fig. (3): **(A)**: ESEM Photomicrograph (2000x) of Vita Enamic material (VE) surface treated with HF acid etching (control) showing exposure of surface crystals with high degree of surface roughness. **(B)**: image analysis of roughness (500x) with $R_a=27.44 \mu\text{m}$.

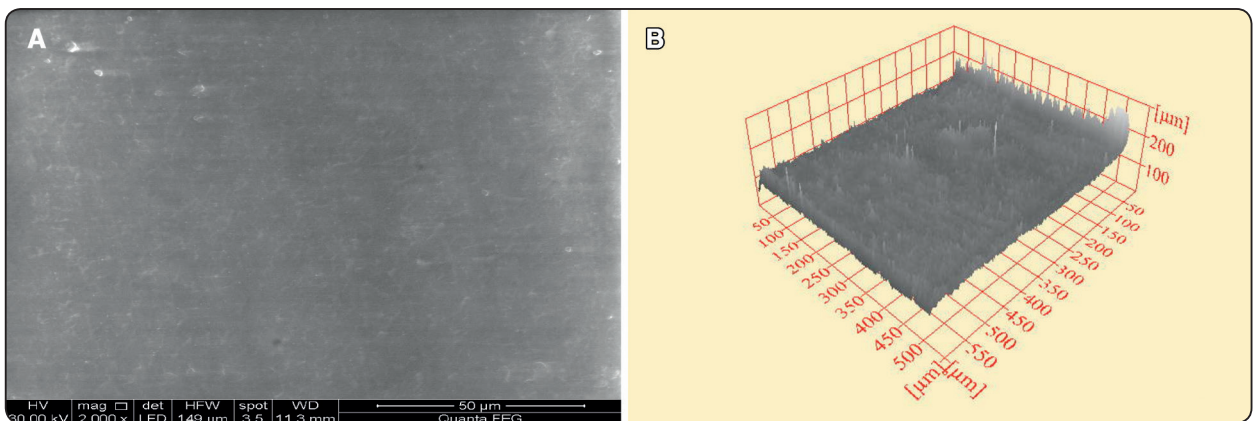


Fig. (4): **(A)**: ESEM Photomicrograph (2000x) of e.max material (EM) non- treated surface showing an apparent smooth surface. **(B)**: image analysis of roughness (500x) with $R_a=4.21 \mu\text{m}$.

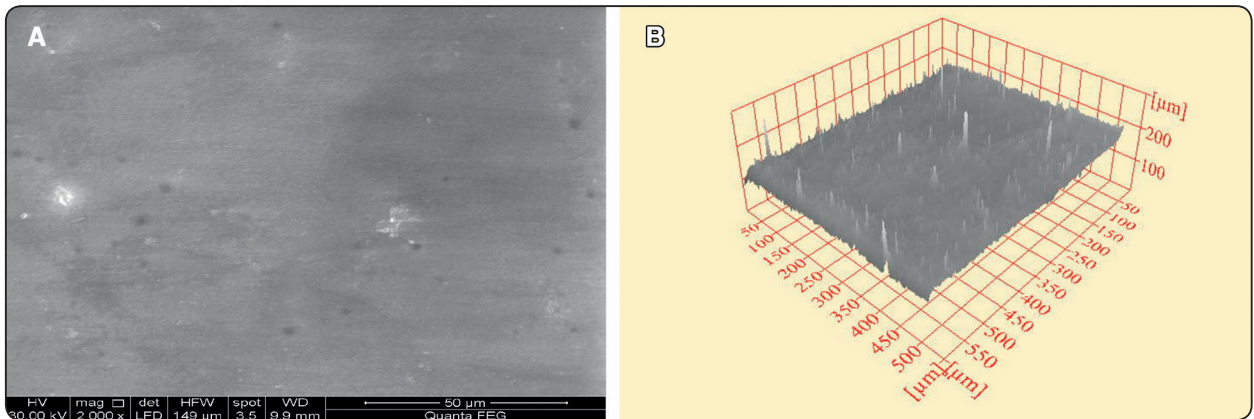


Figure (5): (A): ESEM Photomicrograph (2000x) of e.max material (EM) surface treated with ER showing a relative smooth surface. (B): image analysis of roughness (500x) with $R_a=3.19 \mu\text{m}$.

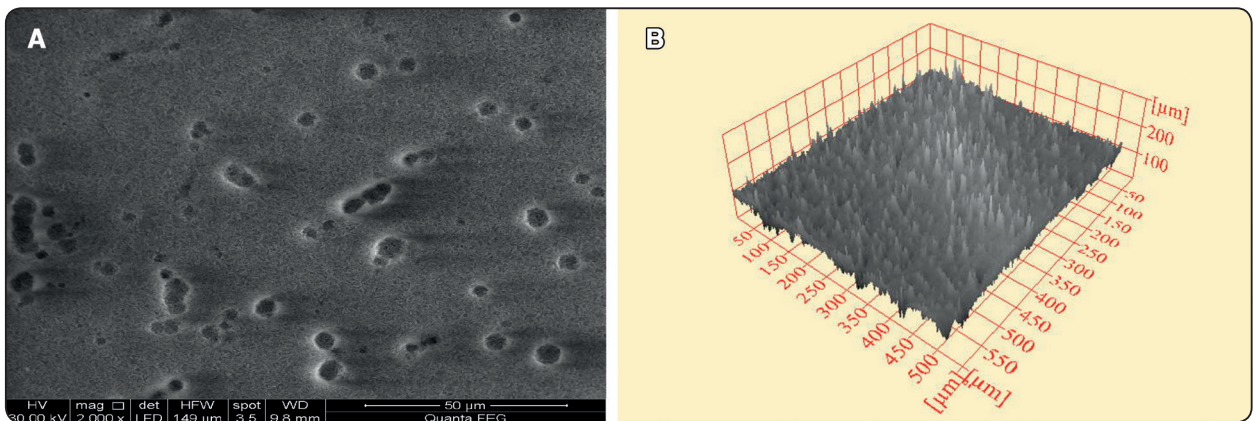


Figure (6): (A): ESEM Photomicrograph (2000x) of e.max material (EM) surface treated with HF acid etching (control) showing highly pitted surface. (B): image analysis of roughness (500x) with $R_a=10.28 \mu\text{m}$.

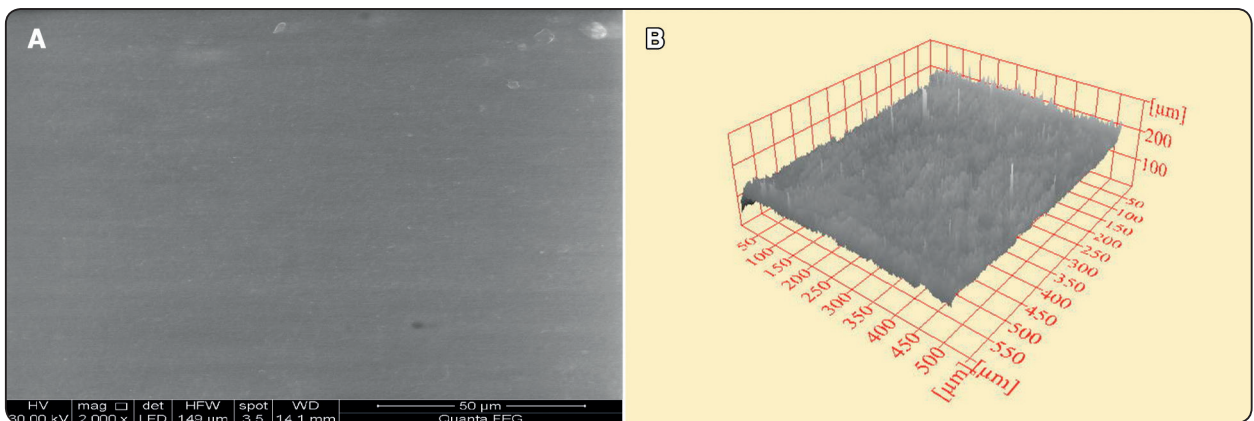


Figure (7): (A): ESEM Photomicrograph (2000x) of Obsidian (OB) non treated surface showing relative smooth surface. (B): image analysis of roughness (500x) with $R_a=5.4 \mu\text{m}$.

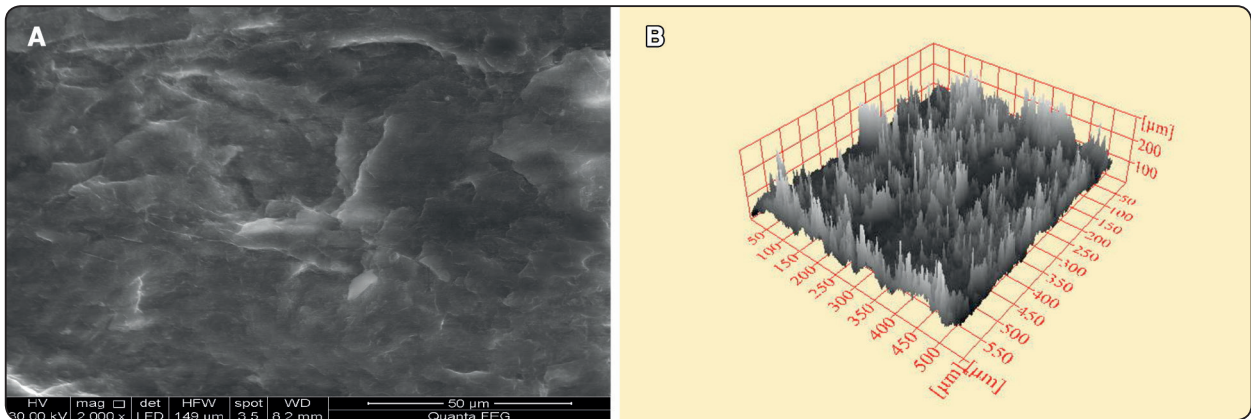


Fig. (8): (A): ESEM Photomicrograph (2000x) of Obsidien (OB) surface treated with ER showing discrete areas of irregularities distributed over the surface. (B): image analysis of roughness (500x) with $R_a=9.8 \mu\text{m}$.

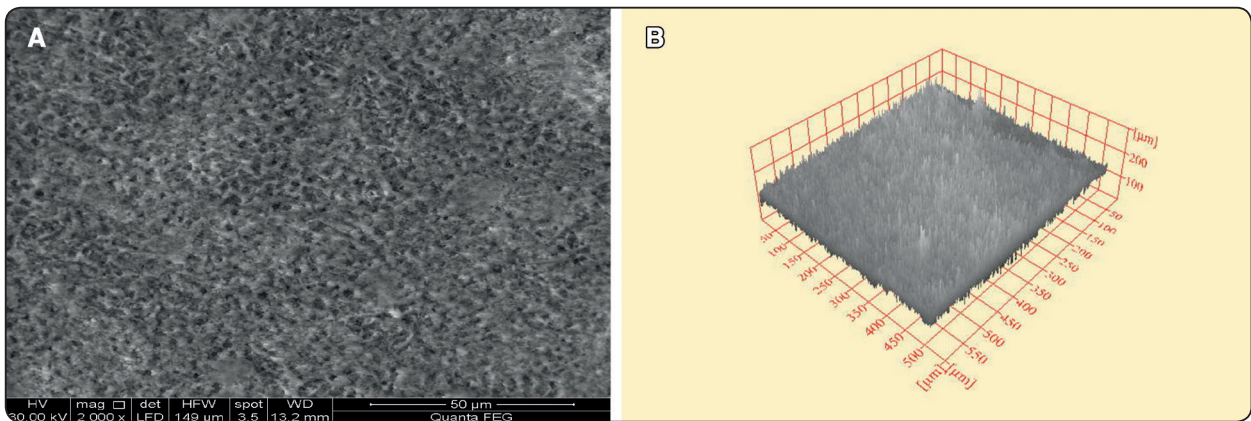


Fig. (9): (A): ESEM Photomicrograph (2000x) of Obsidien (OB) surface treated with HF acid etching (control) showing high surface roughness. (B): image analysis of roughness (500x) with $R_a=16.24 \mu\text{m}$.

There was no statistically significant difference of mean values of μTBS between (ER) or (HF) subgroups using (BC). On the other hand, the use of (HF) acid etching (control) recorded higher statistically significant mean value of μTBS compared to (ER) with (PA).

For (group EM), the subgroups treated with (ER) or (HF) acid etching (control), there was no statistically significant difference between the μTBS mean values of both resin cements (BC) and (PA).

There was a statistically significant difference of mean values of μTBS between (ER) and (HF) acid regarding both resin cements.

In (group OB), the subgroups treated with (ER), (BC) division recorded statistically higher mean μTBS value compared to (PA). While for (HF) acid

etching (control), (PA) division revealed statistically significant higher mean μTBS value compared to (BC).

Three-way ANOVA for the effect of the different study variables on mean microtensile bond strength showed that, the different CAD/CAM ceramic materials had a statistically significant effect on mean microtensile bond strength at F-value 30.547 and P-value <0.001. Surface treatments have a statistically significant effect at F-value 278.904 and P-value <0.001. Also; Resin cement formula had a statistically significant effect on mean microtensile bond strength at F-value 12.190 and P-value 0.001. The interaction between the three variables had a statistically significant effect on mean microtensile bond strength at F-value 68.333 and P-value <0.001.

TABLE (2): The mean (MPa), standard deviation (SD) and p-values for the all tested variables of the study:

Variables	Microtensile bond strength (MPa)												p-value
	Vita Enamic (VE)				IPS e.max CAD (EM)				Obsidian (OB)				
	Laser (ER)		Hydrofluoric acid (HF)		Laser (ER)		Hydrofluoric acid (HF)		Laser (ER)		Hydrofluoric acid (HF)		
	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	
BisCem (BC)	14.20 ^{aB}	2.18	13.46 ^{bB}	2.24	3.45 ^{aD}	0.93	19.15 ^{aA}	0.96	15.49 ^{aB}	2.02	9.97 ^{bC}	1.07	<0.001*
Panavia (PA)	15.59 ^{aB}	0.79	19.49 ^{aA}	2.95	4.40 ^{aD}	0.17	18.54 ^{aA}	2.35	4.70 ^{bD}	0.17	22.46 ^{aA}	1.53	<0.001*
p-value	0.217ns		0.007*		0.196ns		0.606ns		<0.001*		<0.001*		

Mean with different lower-case letters in the same column and different upper-case letters in the same row indicate statistically significant difference *; significant ($p \leq 0.05$) ns; non-significant ($p > 0.05$).

DISCUSSION

Achieving of a strong bond between the resin cements and ceramic restorations is one of the important factors influencing the clinical success of these restorations¹. Experimental initial high μ TBS could be seductive parameter for selection the type of surface treatment as well as the resin cement¹. The clinical durability of the bond strength could be the evidence of the optimum selection. In this study, dual cured self-adhesive resin cements (PA) and (BC) were selected as dual cured resin was postulated to have an excellent self-curing capability, as the visible light could not reach to achieve maximum polymerization.²⁷ Furthermore, the acidic monomers undergo an acid-base setting reaction with the basic inorganic fillers of the materials far from the polymerization reaction of the material.³³ The assessment of the bond strength between resin cement and CAD/CAM materials is important, as it evaluates the weak link during the cementation procedure.^{18,37} Microtensile bond strength testing was used in this study as it was found to be more sensitive in evaluation of bond strength when compared to shear bond strength

testing. As conventional shear and tensile tests were reported to suffer from non-uniform stresses created at the resin-ceramic interface.^{9,15} Vita Enamic was used in this study as it represents one of the recent developments in the CAD/CAM materials industry which combines the advantages of ceramics with those of resin composite. Obsidian glass ceramic was chosen also as it is the most recent novel ceramic material derived from the group of Lithium Silicate Ceramics. Laser surface treatment was also assessed as it represents a safe alternative to conventional hydrofluoric acid etching of CAD/CAM materials.²⁸

In the (VE group), no significant difference in the mean of μ TBS values between (HF) acid etching (control) and (ER) sub groups when (BC) self adhesive resin cement was used in spite of the variations in the surface roughness created by both surface treatments as shown in fig.(2, 3). The degree of roughness was more pronounced in case of (HF) acid etching than (ER) surface treatment, however, (BC) showed higher non statistically significant μ TBS value. This could be attributed to the fact that, the degree of roughness created by (ER) was

homogenous allowing better wettability of (BC) on the surface of (VE) ceramic. Regarding (PA) resin cement, which recorded a higher statistically significant μ TBS mean value compared to (BC) in (HF) acid etching subgroup, this might be explained by the difference in the chemical compositions of the two self adhesive resin cements, where (PA) resin cement contains 10-MDP (its vinyl group reacted with monomers in the cement during polymerization)^{5,14} and other functional monomers which may be postulated that, they enhanced the bonding to the resin portion found in VE. In addition to the presence of variant particle size of silanated silica and glass fillers which might have been deeply embedded in the more roughened (HF) acid etching treated (VE) ceramic surface and bonded chemically with the silane layer, in a way that formed a resilient layer which can further withstand the fracture forces.

In the (EM group), (HF) acid etching (control) surface treatment subgroup recorded statistically significant higher mean μ TBS values compared to (ER) for both self-adhesive resin cements (PA and BC). This finding was in accordance with the findings of Kursuglo et al.,2013²⁸ and other studies^{10,13,46}, who reported that, (HF) acid etching surface treatment recorded higher bond strength when compared with laser of different power settings (1.5, 2.5 and 6W). This can be attributed to the effect of (HF) acid etching on (EM) ceramic surface, where the glass matrix (silica part) reacted with (HF) acid etchant forming hexa fluorosilicates, which had been selectively removed and dissolved exposing the lithium disilicate crystals fig.(6), thus forming a favorable topography; allowing micromechanical interlocking and higher surface energy before receiving the silane primer, that helped in improvement of resin wettability and chemical bonding with both the ceramic surface and resin cements. Also Aboushelib and Sleem, 2014¹ suggested that, the use of high energy level of pulsed Er,Cr:YSGG failed to increase the bond

strength to lithium disilicate ceramics, as it may destruct the surface material. So a great attention should be taken in selecting the suitable surface treatment of glass ceramics.

However, Gökçe et al., 2007²¹ found that, laser irradiation using ER: YAG recorded higher yet not statistically significant bond strength than (HF) acid etching. This could be attributed to the different type of laser used, the testing method used, as shear bond strength was used instead of microtensile bond strength testing that was used in the present study. Moreover, the higher laser power setting of 6W was used instead of 5W applied in the current study.

On the other hand, surface treatment by (ER) might cause ablation and inappropriate micro depths which resulted by the 5W irradiation power, rendering (EM) ceramic surface smoother than non-treated one, which was not favorable for bonding. Taking into consideration, heat damage of the ceramic surface could not have occurred due to the hydrokinetic system of Er,Cr:YSGG laser type, as the laser energy absorbs water microdroplets and is supposed to be responsible for the cutting potency of hard tissue.²⁹

In the (OB group), (BC) recorded statistically significant higher μ TBS mean value than (PA) in the (ER) subgroup, while in (HF) acid etching subgroup (control), (PA) recorded statistically significant higher μ TBS than (BC). These findings could be due to the difference in the rheological properties of the tested self-adhesive resin cements combined with very high non homogenous peaks on the (OB) ceramic laser treated surface as shown in fig. (8), which affected the penetration of (PA) which had higher viscosity and variant larger particle sizes that might prevent the cement penetration and interaction with the ceramic surface. Moreover, the interaction of (ER) with (OB) ceramic surface might remove the contaminant without phase transformation; not exposing the crystalline structure, depriving 10-MDP in (PA) from the chemical bonding with

the zirconium and metal oxides. In contrast, the lower viscosity of (BC) and smaller particle sizes might allow more adaptation increasing the bond strength. Other factors among the cements such as, the different degree of conversion rates, variable portions of the resin monomer, the initiators and the activators, the surface energy, pH, the duration of self-curing polymerization process and adhesive property which related to the methacrylate monomers and their acidity might also affect the results.^{3,14} (HF) acid etching caused higher surface roughness than (ER) with more homogenous distribution of peaks fig.(9) which might have played a role in the penetration of (PA) and its chemical interaction with the (OB) ceramic substrate surface.

Based on the findings of this study, the two hypotheses were totally rejected. Within the limitations of this research, the following conclusions could be drawn:

- 1- The interaction of Er,Cr:YSGG pulsed laser with the surfaces of the tested CAD/CAM ceramic materials seemed to be different and dependent on the crystalline structure of these materials.
- 2- Variation of the chemical formulae of self-adhesive resin cements played a great role in determining the μ TBS to the tested CAD/CAM ceramic materials.

Clinical Relevance:

For promoting a reliable bond during cementation or repair of resin cemented ceramic restorations, the use of Er,Cr:YSGG pulsed laser as an intraoral safe surface treatment can be clinically applied, putting in consideration the microstructure and composition of CAD/CAM materials combined with the chemical formulae of resin cements.

Conflicts of interest:

Each author has no conflicts of interest.

Financial support and sponsorship:

There was no a financial support and sponsorship from any person or organization.

ACKNOWLEDGEMENT

The authors acknowledge the valuable inspiration and continuous emotional support of late Dr. Mokhtar Nagi, Professor of Operative Dentistry, Faculty of Dentistry, Ain-Shams University. His memory will always be with us.

REFERENCES

1. Aboushelib MN and Sleem D. Microtensile Bond Strength of Lithium Disilicate Ceramics to Resin Adhesives. *J Adhes Dent* 2014; 16: 547-552.
2. Alex G. Universal adhesives: the next evolution in adhesive dentistry? *Compend Contin Educ Dent* 2015;36:15-26.
3. Alkurt M, Duymus ZY, Gundogdu M, Karadas M. Comparison of temperature change among different adhesive resin cement during polymerization process. *The Journal of Indian Prosthodontic Society* 2017;17:183-188.
4. Barutcigil K, Barutcigil C, Kul E, Özarslan MM, Buyukkaplan US. Effect of Different Surface Treatments on Bond Strength of Resin Cement to a CAD/CAM Restorative Material. *J Prosthet Dent* 2009;102:306-312 .
5. Berijani N, Mahshid M, Tabatabaian F, Sadr SJ. Dipping Impact on the Bond Strength between Zirconia Ceramic and a Resin Cement. *Regeneration, Reconstruction, Restoration* 2017;2:20-25.
6. Blatz MB, Sadan A, Kern M. Resin-ceramic bonding: a review of the literature. *J Prosthet Dent* 2003; 89:268-274.
7. Boscato N, Della Bona A, Del Bel Cury AA. Influence of ceramic pre-treatments on tensile bond strength and mode of failure of resin bonded to ceramics. *Am J Dent* 2007; 20:103-108.
8. Bunek SS, Swift EJ Jr. Contemporary ceramics and cements. *J Esthet Restor Dent* 2014; 26: 297-301.
9. Carvalho AA, Stefani A, de Sa Barbosa WF, Lopes LG, Giannini M. Influence of curing mode of resin luting cements on bond strength to dentin. *Braz J Oral Sci* 2016;15:258-263.
10. Colares RC, Neri JR, de Souza AM, Pontes KM, Mendonca JS, Santiago SL. Effect of Surface Pretreatments on the

- Microtensile Bond Strength of Lithium-Disilicate Ceramic Repaired with Composite Resin. *Brazilian Dental Journal* 2013;24:349-352.
11. Coldea A, Swain MV, Thiel N. Mechanical properties of polymer-infiltrated-ceramic-network materials. *Dent Mater* 2013;29:419-426.
 12. da Silva Ferreira S, Hanashiro FS, de Souza-Zaroni WC, Turbino ML, Youssef MN. Influence of aluminum oxide sandblasting associated with Nd:YAG or Er:YAG lasers on shear bond strength of a feldspathic ceramic to resin cements. *Photomed Laser Surg* 2010;28:471-475.
 13. Duzyol M, Sagsoz O, Sagsoz NP, Akgul N, YildizM. The effect of surface treatments on the bond strength between CAD/CAM blocks and composite resin. *J Prosth* 2015;24:1-6.
 14. Dziejczak DSM, Prohny JPS, Picharski GL, Furuse AY. Influence of curing protocols on water sorption and solubility of a self-adhesive resin-cement. *Braz J Oral Sci* 2016;15:144-150.
 15. Elaska SE. Bond strength of novel CAD/CAM restorative materials to self-adhesive resin cement: the effect of surface treatments. *J Adhes Dent* 2014;16:531-540.
 16. Fabianelli A, Pollington S, Papacchini F, Goracci C, Cantoro A, Ferrari M, van Noort R. The effect of different surface treatments on bond strength between leucite reinforced feldspathic ceramic and composite resin. *J Dent* 2010; 38:39-43.
 17. Ferracane JL, Stansbury JW, Burke FJ. Self-adhesive resin cements—chemistry, properties and clinical considerations. *J Oral Rehabil* 2011; 38:295-314.
 18. Frankenberger R, Hartmann VE, Krech M, Krämer N, Reich S, Braun A, Roggendorf M. Adhesive luting of new CAD/CAM materials. *Inter J Comput Dent* 2015;18: 9–20.
 19. Gehrt M, Wolfart S, Rafai N, Reich S, Edelhoff D. Clinical results of lithium-disilicate crowns after up to 9 years of service. *Clin Oral Investig*; 17:275-284.
 20. GlidewellDental.com/services/all-ceramics/obsidian-ceramic-restorations.
 21. Gökçe B, Ozpınar B, Dündar M, Cömlekoglu E, Sen BH, Güngör MA. Bond strengths of all-ceramics: acid vs laser etching. *Oper Dent* 2007; 32:173-178.
 22. Guarda GB, Correr AB, Goncalves LS, Costa AR, Borges GA, Sinhoretto MA, Correr-Sobrinho L. Effects of surface treatments, thermocycling, and cyclic loading on the bond strength of a resin cement bonded to a lithium disilicate glass ceramic. *Oper Dent* 2013; 38:208-217.
 23. Kara HB, Dilber E, Koc O, Ozturk AN, Bulbul M. Effect of different surface treatments on roughness of IPS Empress 2 ceramic. *Lasers Med Sci* 2012;27:267-272.
 24. Kassem AS, Atta O, El-Mowafy O. Fatigue resistance and microleakage of CAD/CAM ceramic and composite molar crowns. *J Prosth* 2012;21:28-32.
 25. Kim BK, Bae HE, Shim JS, Lee KW. The influence of ceramic surface treatments on the tensile bond strength of composite resin to all-ceramic coping materials. *J Prosthet Dent* 2005; 94: 357-362.
 26. Kiyani VH, Saraceni CH, da Silveira BL, Aranha AC, Eduardo Cda P. The influence of internal surface treatments on tensile bond strength for two ceramic systems. *Oper Dent* 2007; 32:457-465.
 27. Kumbuloglu O, Lassila LV, User A, Vallittu PK. Bonding of resin composite luting cements to zirconium oxide by two air-particle abrasion methods. *Oper Dent* 2006;31: 248-255.
 28. Kursoglu P, Motro PFK, Yurdagüven H. Shear bond strength of resin cement to an acid etched and a laser irradiated ceramic surface. *J Adv Prosthodont* 2013;5:98-103
 29. Kursoglu P, Yurdagüven H, Kazazoglu E, Çalikkocaoglu S, Gursoy T. Effect of Er,Cr:YSGG laser on ceramic surface. *Balk J Stomatol* 2006;10:103-109.
 30. Le M, Papia E, Larsson C. The clinical success of tooth- and implant-supported zirconia-based fixed dental prostheses. A systematic review. *J Oral Rehabil* 2015; 42:467-480.
 31. Manso AP, Silva NR, Bonfante EA, et al. Cements and adhesives for all-ceramic restorations. *Dent Clin North Am.* 2011; 55:311-332.
 32. Marocho SM, Özcan M, Amaral R, Bottino MA, Valandro LF. Effect of resin cement type on the microtensile bond strength to lithium disilicate ceramic and dentin using different test assemblies. *J Adhes Dent* 2013; 15: 361-368.
 33. Moraes RR, Boscato N, Jardim PS, Schneider LF. Dual and Self-curing Potential of Self- adhesive Resin Cements as Thin Films. *Oper Dent* 2011;36:635-642.
 34. Oh SC, Dong JK, Lüthy H, Schärer P. Strength and microstructure of IPS Empress 2 glass-ceramic after different treatments. *Int J Prosth* 2000;13:468-472.

35. Özcan M, Vallittu PK. Effect of surface conditioning methods on the bond strength of luting cement to ceramics. *Dent Mater* 2003; 19:725-731.
36. Panah FG, Rezai SM, Ahmadian L. The influence of ceramic surface treatments on the micro-shear bond strength of composite resin to IPS Empress 2. *J Prosthodont* 2008; 17:409-414.
37. Peumans M, Valjakova EB, De Munck J, Mishevska CB, Van Meerbeek B. Bonding effectiveness of luting composites to different CAD/CAM materials. *J Adhes Dent* 2016;18:289-302.
38. Sadeghi M, Davari A, Mahani AA, and Hakimi H. Influence of Different Power Outputs of Er:YAG Laser on Shear Bond Strength of a Resin Composite to Feldspathic Porcelain. *J Dent*;2015;16:30–36.
39. Saraç D, Saraç YS, Külünk S, Erkoçak A. Effect of various surface treatments on the bond strength of porcelain repair. *Int J PerioRestor Dent* 2013; 33:120-126.
40. Saraçoğlu A, Cura C, Cötert HS. Effect of various surface treatment methods on the bond strength of the heat-pressed ceramic samples. *J Oral Rehabil* 2004; 31:790-797.
41. Schlichting LH, Maia HP, Baratieri LN, Magne P. Novel design ultra-thin CAD/CAM composite resin and ceramic occlusal veneers for the treatment of severe dental erosion. *J Prosthet Dent* 2011; 105:217-226.
42. Siervo S, Pampalone A, Siervo P, Siervo R. Where is the gap? Machinable ceramic systems and conventional laboratory restorations at a glance. *Quint Int* 1994;25:773-779.
43. Stamatacos C, Simon JF. Cementation of indirect restorations: an overview of resin cements. *Compend Contin Educ Dent*. 2013; 34:42- 46.
44. Yavuz T, Dilber E, Kara HB, Tuncdemir AR, Ozturk AN. Effects of different surface treatments on shear bond strength in two different ceramic systems. *Lasers Med Sci* 2013; 28:1233-1239.
45. Zimmermann M, Mehl A, Reich S. New CAD/CAM materials and blocks for chair side procedures. *Int J Comput Dent* 2013; 16:173-181.
46. Zogheib LV, Della Bona A, Kimpara ET, McCabe JF. Effect of hydrofluoric acid etching duration on the roughness and flexural strength of a lithium disilicate-based glass ceramic. *Braz Dent J* 2011;22:45-50.
47. Zorzini J, Belli R, Wagner A, Petschelt A, Lohbauer U. Self-adhesive resin cements: adhesive performance to indirect restorative ceramics. *J Adhes Dent*. 2014; 16:541-546.