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Effect of Different Surface Treatment Methods on Micro-Shear Bond Strength of CAD-CAM Restorative Materials to Resin Cement.

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ABSTRACT

This study aimed to investigate the micro-shear bond strength (μSBS) of surface treated CAD-CAM materials to resin cement. The specimens obtained from IPS e.max CAD, Lava Ultimate, Cerasmart and Vita Enamic were divided according to the surface treatment method applied as: no treatment, 3W and 2W Er, Cr:YSGG laser irradiation, sandblasting and 5% hydrofluoric acid (HF) application. Then, μSBS and field emission-scanning electron microscope analysis were performed. Data were analyzed using the Mann Whitney U and the Kruskal Wallis tests. For all materials, the highest μSBS values were demonstrated in HF acid applied groups. Regarding the μSBS values of IPS e.max CAD, no significant differences were found among control, 2W Er, Cr:YSGG laser and sandblasting groups ($p > 0.05$). For Cerasmart and Lava Ultimate; 2W Er, Cr:YSGG laser treated group showed significantly lowest μSBS values while there was no significant difference among control, 3W Er, Cr:YSGG and sandblasting groups. HF applied Lava Ultimate and IPS e.max CAD groups exhibited the highest μSBS values among all the groups. For Vita Enamic; significantly lowest μSBS values were obtained in sandblasting group, whereas there was no significant difference among control, 3W Er, Cr:YSGG and 2W Er, Cr:YSGG groups ($p > 0.05$). The FE-SEM images of all CAD-CAM materials submitted to surface treatment revealed an increase in surface alterations compared to control groups. It can be concluded that prior to bonding 5% HF acid treatment is the best surface treatment method regarding the bond strength for all CAD-CAM restorative materials. Er, Cr:YSGG laser application with energy level of 3W can be recommended for IPS e.max CAD.

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Introduction

Computer aided design-computer aided manufacturing (CAD-CAM) techniques in dentistry have become progressively popular in the last few years with the advent of adhesive techniques and development of new materials [1,2]. CAD-CAM systems have many advantages such as allowing better standardization of manufacturing process of the restorations with lower cost of production [3,4]. The materials for the fabrication of indirect restorations with CAD-CAM technologies have been reinforced to improve mechanical strength against occlusal load and bond strength to tooth structure [5]. Ceramics reinforced with lithium di-silicate have been suggested as the best option for CAD-CAM restorations due to the properties such as high mechanical strength, excellent adhesion properties to tooth structures, and excellent esthetics [6,7].

The latest developments of CAD-CAM restorative materials are especially associated with novel microstructures containing dispersed fillers which is called 'resin nano-ceramic' or polymer-infiltrated ceramic network (PICN) [8]. The first marketed resin nano-ceramic material is Lava Ultimate which has approximately 80 wt% of zirconia-silica nanofillers (in the form of non-agglomerated/non-aggregated particles) bound in the 20 wt% of resin matrix composed of urethane di-methacrylate (UDMA) and is totally heated rather than photopolymerized [9]. Similar to Lava Ultimate, Cerasmart is a heated and polymerized nano-ceramic material with 71 wt% of filled nano-composite based on UDMA matrix [9,10]. More recently, Vita Enamic is developed as a polymer-infiltrated ceramic network material. In the structure of this material, unlike resin nano-ceramic materials; ceramic particles are partially sintered and then infiltrated with a low-viscosity polymer composed of UDMA and triethylene glycol dimethacrylate (TEGDMA) mixture [4,10].

The quality and durability of the bond between indirect CAD-CAM restorations and the tooth is of clinical significance in terms of the longevity and success of the restoration [4,11,13]. Various surface treatment techniques as sandblasting [3], grinding with bur [14], tribo-chemical silica coating (CoJet) [15] and acid etching [13] have been suggested to provide micromechanical and chemical retention which results better bond strength of CAD-CAM restorative materials to resin cement. As a recent method; the use of erbium, chromium: yttrium, scandium, gallium, and garnet (Er, Cr:YSGG) laser for roughening the surface of a PICN material was recommended in a previous study [4]. However, the data regarding the micro-shear bond strength of Er, Cr:YSGG laser treated CAD-CAM restorative materials to resin cement is limited.

Therefore, the aim of the present study was to investigate the micro-shear bond strength of a dual-cured resin cement to a lithium di-silicate ceramic, 2 resin nano-ceramics and a PICN material treated with sandblasting, 5% hydrofluoric acid (HF) and Er, Cr:YSGG laser with the energy levels of 2W and 3W. The study also aimed to analyze the surface topography of the CAD-CAM materials after surface treatment methods by using field emission scanning electron microscope (FE-SEM).

Materials and Methods

The compositions and manufacturers of the materials tested in the study are presented in Table 1. Fifteen sections with the dimensions of 6 mm × 7 mm × 1 mm

Table 1. Materials and compositions used in this study.

Material	Product	Composition
Lithium disilicate glass-ceramic	IPS e.max CAD, Ivoclar Vivadent, Schaan, Liechtenstein	Lithium disilicate reinforced glass-ceramic of the Li2O–K2O–P2O5–MgO-material system
Resin nano-ceramic	Lava Ultimate, 3M ESPE, St Paul, MN, USA	20wt% composite resin material (BisGMA, UDMA, BisEMA, TEGDMA) with 80wt% silica and zirconia nanoparticles and zirconia/silica nanoclusters
Resin nano-ceramic	Cerasmart, GC Dental Products, Leuven, Belgium	Composite resin material (BisMEPP, UDMA, DMA) with 71wt% silica and barium glass nanoparticles
Polymer-infiltrated ceramic network (PICN)	VITA Enamic, VITA Zahnfabrik, Bad Säckingen, Germany	Polymer-infiltrated-feldspatic ceramic-network material (UDMA, TEGDMA) with 86wt% ceramic
Dual-cured resin luting cement	Variolink N, Ivoclar Vivadent, Schaan, Liechtenstein	Bis-GMA, TEGDMA, UDMA, Inorganic fillers (barium glass, ytterbium trifluoride, Ba-Al-fluorosilicate glass, and spheroid mixed oxide), initiators, stabilizers, pigments, benzoyl peroxide

from each CAD-CAM restorative materials (IPS e.max CAD, Lava Ultimate, Cerasmart, Vita Enamic) were constructed by using a CAD-CAM system (CEREC inLAb MC X5, Sirona Dental Systems, Bensheim, Germany). IPS e.max CAD specimens were crystallized with Programat EP5000 (Ivoclar Vivadent, Schaan, Liechtenstein) furnace according to the manufacturer's instructions. Only one surfaces of the sections were wet polished with 400-grit followed by 600-grit wet silicon carbide paper under water rinsing for surface standardization and then all the sections were stored in distilled water for 24 hours. After storage period; all the specimens were randomly divided into 5 groups each containing 3 sections according to the surface treatment method applied:

Group 1: no surface treatment (control group)

Group 2: The specimens were irradiated with Er, Cr:YSGG laser on hard tissue mode with a MG6 sapphire tip using a non-contact mode at an energy level of 3W, a repetition rate of 10 Hz, and 140 μs pulse duration with 55% water and 65% air for 20 sec.

Group 3: The specimens were irradiated with Er, Cr:YSGG laser (Waterlase MD, Biolase, Irvine, CA, USA) on hard tissue mode with a MG6 sapphire tip using a non-contact mode at an energy level of 2W, a repetition rate of 10 Hz, and 140 μs pulse duration with 55% water and 65% air for 20 sec.

Group 4: The specimens were sandblasted with 50 μm Al_2O_3 particles (Korox 50, Bego, Bremen, Germany) for 30 seconds at a distance of 10 mm.

Group 5: The IPS e.max CAD specimens were etched with 5% HF acid gel for 20 s, Vita Enamic, Cerasmart and Lava Ultimate specimens were etched with 5% HF acid gel for 60 s. All specimens were rinsed with distilled water for 2 min and air-dried after etching.

Following surface treatment procedures, all of the specimens were rinsed with distilled water and air dried. Prior to the placement of tygon tubes for micro-shear bond strength test, a thin coat of Monobond Plus (Ivoclar Vivadent, Schaan, Liechtenstein) was applied to all surfaces with a micro-brush, allowed to react for 60 s and any remaining excess was dispersed with a strong stream of air according to the manufacturer's instructions. Three tygon tubes with a thickness of 1 mm and diameter of 0.75 mm were placed on each specimen. After Variolink N Base and Catalyst was

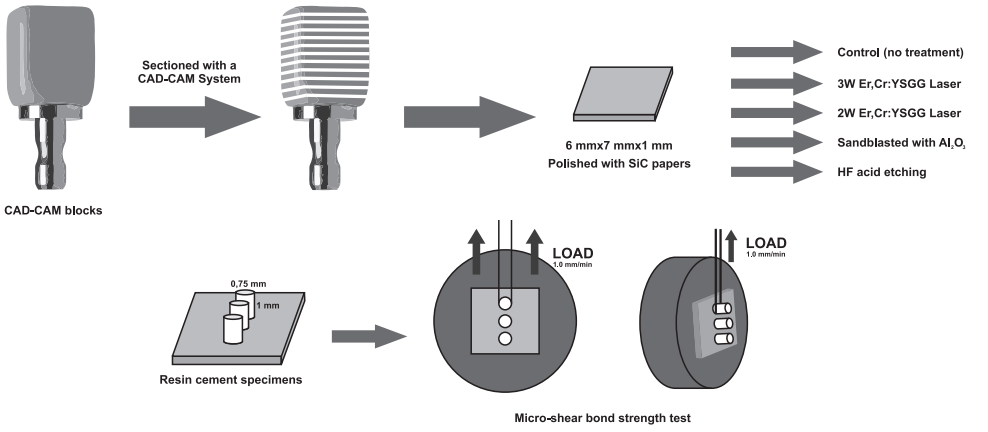

Figure 1. Schematic representation of micro-shear bond strength test.

mixed in a ratio 1:1 on a mixing pad for 10 s, resin cement was filled into the tubes. The resin cement in the tubes was light cured with a quartz-tungsten halogen light-curing unit (Blue Luxcer M-835, Monitex, New Taipei City, Taiwan) in standard mode with an intensity setting of 800 mW/cm^2 for 20 s. Nine cylindrical specimens were obtained for each surface treatment group of each CAD-CAM restorative material (n = 9). The specimens were stored in distilled water at 37 °C for 24 h. After the storage period, the tygon tubes were carefully removed with a sharp scalpel. Specimens that were failed before testing were not included in the analyses; new specimens were fabricated instead.

Micro-shear bond strength test was performed by a universal testing machine (EZtest-500 N Shimadzu; Kyoto, Japan). The specimens were attached to the machine with a cyanoacrylate adhesive (Zapit, Dental Ventures of America; Corona, CA, USA). A 0.2-mm-diameter wire was looped around the cylinder on the specimens, making contact with half of the cylinder base and held flush against the bonded area [16]. A shear force was applied to each specimen at a crosshead speed of 1.0 mm/min until failure occurred. The wire loop and the center of the load cell were positioned as straight as possible to ensure that the requested orientation of shear stress application was maintained (Figure 1). The μSBS values at failure were displayed in Newton's and converted to MPa by dividing the load at failure by the surface area of the cylinder.

To determine the mode of failure, specimens were examined by a single operator under a stereomicroscope (Olympus SZ61, Olympus Optical; Tokyo, Japan) at 40X magnification and failure modes were categorized as follows: adhesive failure between the resin cement and the CAD-CAM restorative materials; cohesive failure within the resin cement, cohesive failure within the CAD-CAM restorative materials; mixed failure composed of adhesive and cohesive failure of resin cement or CAD-CAM restorative materials.

For FE-SEM analysis of the specimens treated with different procedures, additional specimens of CAD-CAM restorative materials were prepared for each group. All samples were coated with platinum/palladium before mounting on the stub of FE-SEM (SUPRA, Carl Zeiss, Oberkochen, Germany). Surface topography of the samples was

recorded at 5000X magnification. All measurements on digital FE-SEM images were performed by one blind calibrated examiner.

Kruskal-Wallis analysis was used to determine any significant change in μSBS values in all groups. When significant change was detected, Mann–Whitney U test was used to make intragroup comparisons. Values of $p < 0.05$ were considered as statistically significant.

Results

The mean μSBS values and standard deviations of CAD-CAM restorative materials treated with different methods to resin cement are presented in Table 2. For all materials, the highest μSBS values were demonstrated in HF acid applied groups. Regarding the μSBS values of IPS e.max CAD, no significant differences were found among control, 2W Er, Cr:YSGG laser and sandblasting groups ($p > 0.05$). For Cerasmart and Lava Ultimate; 2W Er, Cr:YSGG laser treated group showed significantly lowest μSBS values while there was no significant difference among control, 3W Er, Cr:YSGG and sandblasting groups. HF applied Lava Ultimate and IPS e.max CAD groups exhibited the highest μSBS values among all the groups. For Vita Enamic; significantly lowest μSBS values were obtained in sandblasting group, whereas there was no significant difference among control, 3W Er, Cr:YSGG and 2W Er, Cr:YSGG groups ($p > 0.05$).

Representative FE-SEM images are shown in Figures 2-5. The FE-SEM images of all CAD-CAM materials submitted to surface treatment revealed an increase in surface alterations compared to control groups. For Er, Cr:YSGG laser treated Cerasmart, Lava Ultimate and Vita Enamic specimens; FE-SEM images displayed irregular morphologic changes like shallow pits, whereas smoother surfaces were observed in Er, Cr:YSGG laser treated IPS e.max CAD specimens especially with 2W irradiation (Figure 2C). In HF treated specimens of IPS e.max CAD (Figure 2E), a honeycomb-like topography was observed. For the other materials, HF created blister-like globules on the surfaces.

The number of failure modes of the tested materials are presented in Table 3. According to the results of the fracture mode analysis, the most observed fracture pattern was adhesive failure for all groups. Cohesive failures were only observed within resin cement and the number of this type of failure was higher in HF acid applied groups compared to other groups. No mixed failure was displayed.

Table 2. The mean μSBS values (MPa) and standard deviations of CAD-CAM restorative materials treated with different methods to resin cement.

	Surface treatment methods				
Material	Control	3W Er, Cr:YSGG	2W Er, Cr:YSGG	Sandblasting	HF acid
IPS e.max CAD	6.36 (1.27)^{a,A}	9.13 (2.80)^{c,B}	5.93 (1.17)^{f,A}	6.21 (0.98)^{h,A}	12.36 (2.81)^{k,C}
Lava Ultimate	7.73 (1.34)^{a,F}	4.37 (1.35)^{d,G}	6.66 (1.31)^{f,F}	8.91 (2.20)^{i,F}	12.56 (2.00)^{k,I}
Cerasmart	8.11 (1.57)^{b,D}	4.69 (1.17)^{d,E}	6.27 (1.32)^{f,D}	6.73 (2.35)^{h,D}	8.69 (1.96)^{l,D}
Vita Enamic	6.52 (1.93)^{a,J}	7.10 (1.95)^{e,J}	7.40 (1.79)^{g,J}	4.43 (1.22)^{j,K}	10.3 (1.99)^{m,L}

*Different superscript capital letters in columns and lower case letters in rows indicate statistically significant differences ($p < 0.05$).

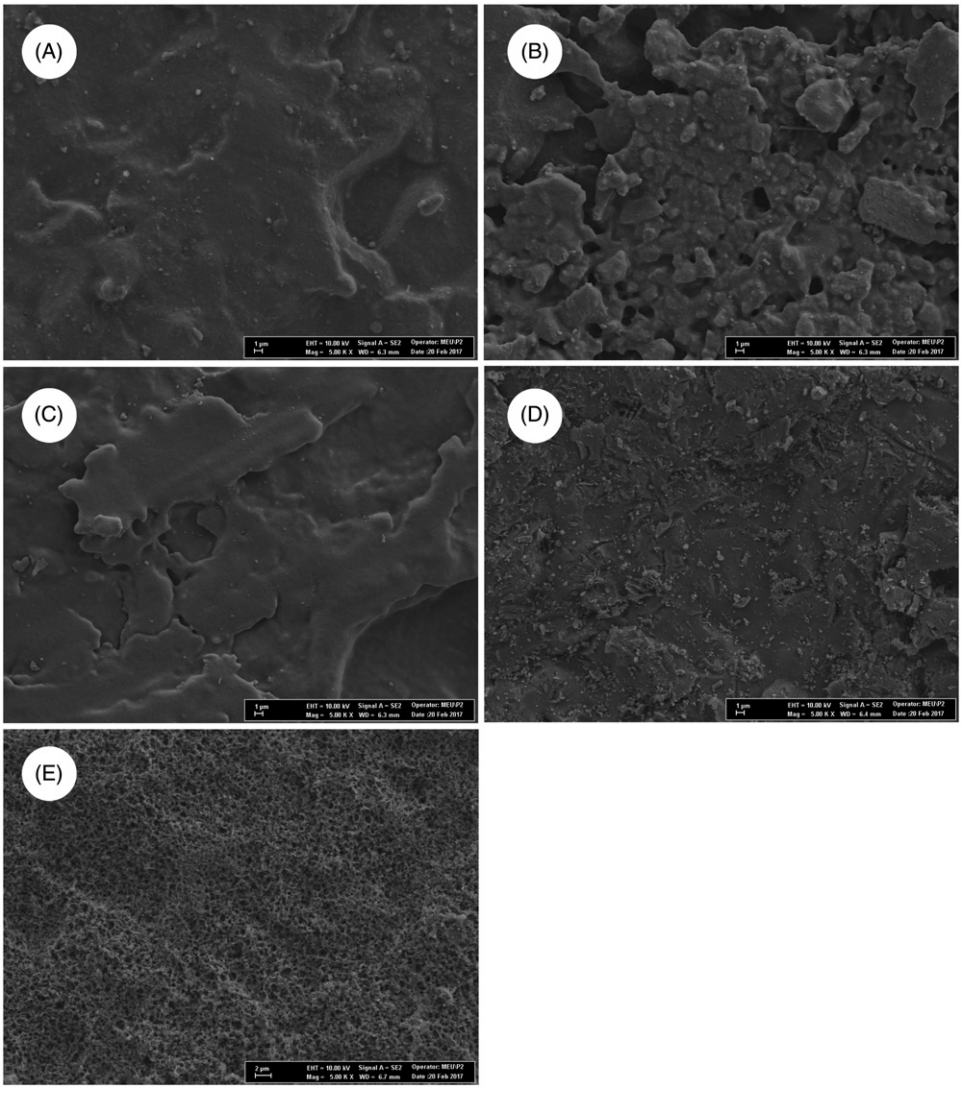

Figure 2. FE-SEM micrographs of IPS e.max CAD specimens (5000x) **A:** Control group **B**: 3W Er, Cr:YSGG laser treated **C**: 2W Er, Cr:YSGG laser treated **D:** Sandblasted **E:** HF acid etched.

Discussion

Diverse methodologies have been suggested to determine the effectiveness of interfacial bonding between restorative materials and adhesive agents [17]. The micro-shear bond strength test which is a relatively simple test compared to other methods, allows efficient screening of adhesive systems, regional and depth profiling of a variety of substrates [17], and elimination of pre-stressing factors such as sectioning specimens [17,18]. Considering these advantages, micro-shear bond strength test was preferred for the present study. For the micro-shear test, the specimen tested is pre-stressed prior to testing only by the removal of the polyethylene tubes with a scalpel blade which was recommended by previous studies [16,19]. However, the pressure

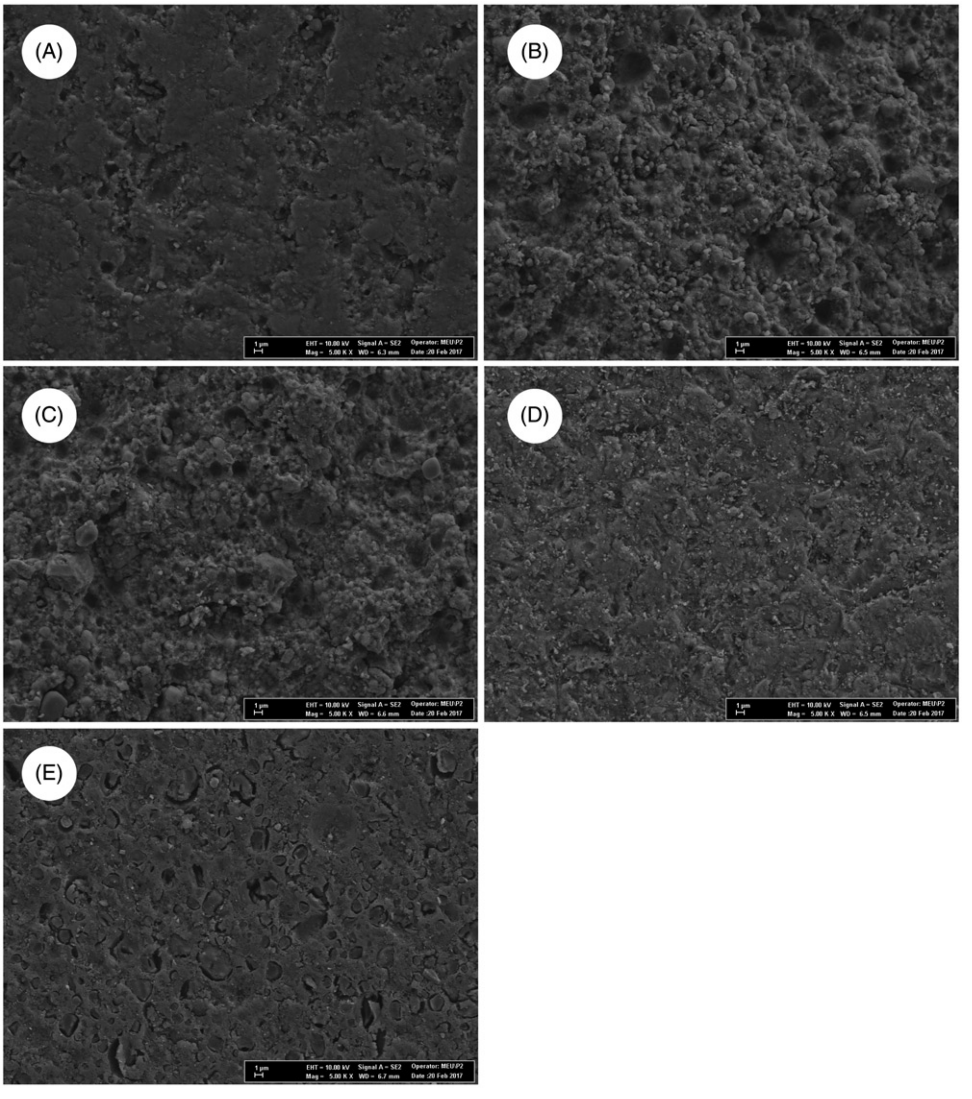

Figure 3. FE-SEM micrographs of Lava Ultimate specimens (5000x) **A:** Control group **B**: 3W Er, Cr:YSGG laser treated **C**: 2W Er, Cr:YSGG laser treated **D:** Sandblasted **E:** HF acid etched.

applied to the blade to remove the tubes may be transmitted to the cylinder, which may lead to stress accumulation on the adhesive interface and result in pre-test failures. In the present study, the number of pre-test failures was not high enough to misrepresent the performance of the materials; therefore, all pre-test failures were excluded from the analysis.

In order to promote bonding of CAD-CAM restorative materials to resin cement, the application of silane coupling agent was suggested by several studies [3,20,21]. Yoshida et al. [20] reported that silane coupling agent application improved the bond strength of resin cement to CAD-CAM restorative material. In agreement with this result, Higashi et al. [3] suggested that the improvement of bond strength after

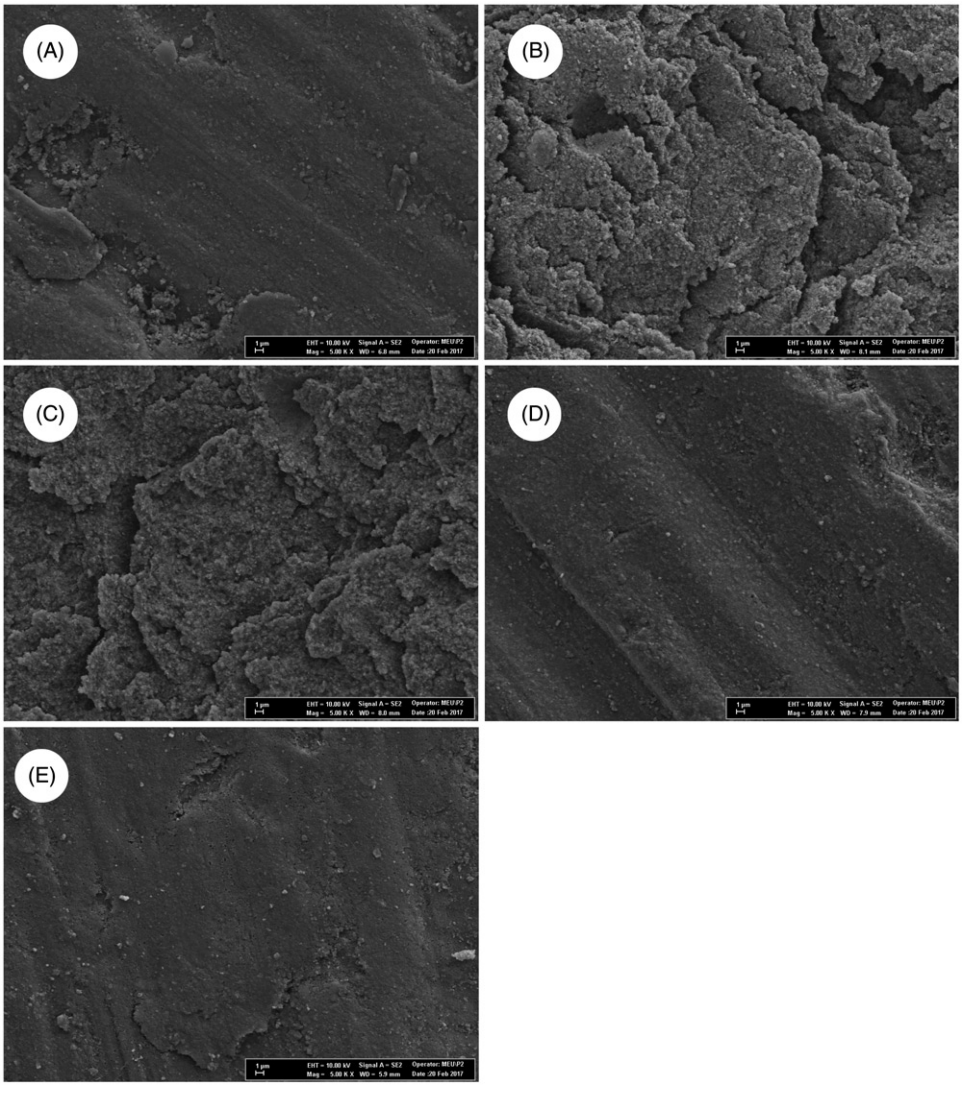

Figure 4. FE-SEM micrographs of Cerasmart specimens (5000x) **A:** Control group **B**: 3W Er, Cr:YSGG laser treated **C**: 2W Er, Cr:YSGG laser treated **D:** Sandblasted **E:** HF acid etched.

silanization may be due to the chemical bond between resin cement and restorative material created by silane coupling agent. It is also noteworthy that silanization improves filler-matrix bonding by covalently coating silica with methacrylate double-carbon bonds that can co-polymerize with the resin matrix [22]. Taking these results and recommendation of the manufacturer into consideration, a silane coupling agent (Monobond Plus) was applied to all specimens after surface treatment procedures.

For lithium di-silicate reinforced ceramic materials, HF acid etching was reported as the most reliable surface pretreatment method [11,23]. The results of the present study verified the effect of HF etching on IPS e.max CAD and also for all materials tested in the current study, HF applied specimens presented highest μSBS values compared to other surface treatment methods. Consistent with this result, previous

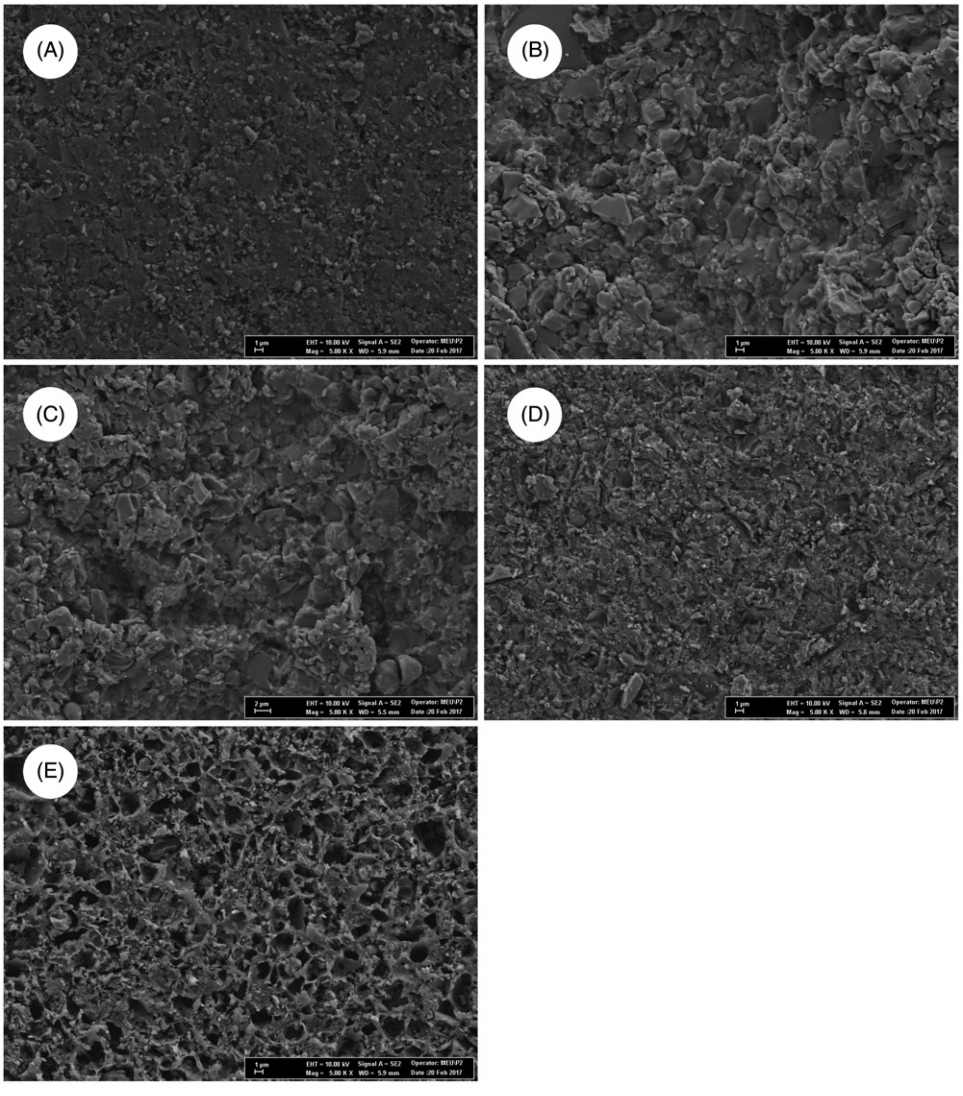

Figure 5. FE-SEM micrographs of Vita Enamic specimens (5000x) **A:** Control group **B**: 3W Er, Cr:YSGG laser treated **C**: 2W Er, Cr:YSGG laser treated **D:** Sandblasted **E:** HF acid etched.

studies [15,23,24] concluded that HF etching prior to bonding enhance the bond strength of CAD-CAM restorative materials. In a previous study [25] which examined the morphological changes in the surface of lithium di-silicate reinforced ceramic materials after HF application, it was reported that the glassy matrix of ceramics was dissolved by 10% HF and lithium di-silicate crystals were observed as unaffected. As a result of dissolution, micro-porosities and honey-comb like topography were formed as observed in the FE-SEM images (Figure 2E) which increase the surface area leading to micromechanical interlocking and high bond strength values. Although HF acid pretreatment is not recommended for Lava Ultimate by the manufacturer, the highest bond strength values of Lava Ultimate were obtained in HF group. In agreement with this result, previous studies [15,26] reported that HF acid

Table 3. The number of failure modes of specimens after micro-shear bond strength test.

Test group	CAD-CAM material	The number of failure modes			
		Adhesive	Cohesive in resin cement	Cohesive in CAD-CAM material	Mixed
Control	IPS e.max CAD	9	–	–	–
	Lava Ultimate	8	1	–	–
	Cerasmart	7	2	–	–
	Vita Enamic	9	–	–	–
Sandblasting	IPS e.max CAD	9	–	–	–
	Lava Ultimate	7	2	–	–
	Cerasmart	8	1	–	–
	Vita Enamic	9	–	–	–
HF acid etching	IPS e.max CAD	6	3	–	–
	Lava Ultimate	7	2	–	–
	Cerasmart	8	1	–	–
	Vita Enamic	7	2	–	–
2W Er, Cr: YSGG laser	IPS e.max CAD	9	–	–	–
	Lava Ultimate	9	–	–	–
	Cerasmart	9	–	–	–
	Vita Enamic	9	–	–	–
3W Er, Cr: YSGG laser	IPS e.max CAD	7	2	–	–
	Lava Ultimate	9	–	–	–
	Cerasmart	9	–	–	–
	Vita Enamic	9	–	–	–

etching improved the bond strength of Lava Ultimate to resin cement. However, Frankenberger et al. [24] demonstrated a detrimental effect of HF acid on the bond strength of Lava Ultimate and suggested to follow manufacturer's instructions. The discrepancy between the results may be explained by different methodology like thermocycling they used. In the structure of Lava Ultimate, the inorganic filler particles are embedded in a polymer matrix without interconnections [11] therefore; as a result of thermocycling, water penetration into the resin matrix of the restorative materials may be the reason of lower bond strength values.

According to the results of the present study, there is no statistical difference between the bond strength values of HF applied Lava Ultimate and IPS e.max CAD specimens although FE-SEM images of these specimens exhibited different characterizations. Similar to FE-SEM results of the present study, Peumans et al. [15] reported that tiny micro-pores and pits were appeared on the Lava Ultimate surface without extensive dissolution after HF acid application unlike IPS e.max CAD specimens. This result showed that the mechanical retention due to the surface irregularities was not the only factor effecting bonding effectiveness. The higher bond strength values of HF acid applied Lava Ultimate specimens may be attributed to the structure of the resin nano-ceramic material which contains 20% UDMA with zirconia-silica nanofillers.

To increase surface area and improve mechanical interlocking of CAD-CAM restorative materials, also sandblasting is suggested by previous studies [3,12,26,27]. However, according to the results of the present study, for lithium di-silicate ceramic and resin nano-ceramic materials, sandblasting had no positive effect on the bond strength to resin cement; for PICN material, bond strength values decreased after sandblasting. Consistent with the results of the present study, previous studies [4,15] stated that the shear bond strength of the sandblasting surface treatment group was

lower than HF acid treatment groups for Vita Enamic and IPS e.max CAD. FE-SEM images of the sandblasted block surfaces revealed an irregular surface and microcracks for all CAD–CAM materials investigated in this study. Peumans et al. [15] reported that microcracks observed after sandblasting may lead to premature failures result in lack of internal and marginal adaptation which may be the result of lower bond strength values in sandblasting groups. As Elsaka [26] mentioned before, the results of the study showed that higher surface roughness will not ensure a higher bond strength.

Laser irradiation is a more recent surface treatment method to enhance the bond strength of restorative materials to resin cement [4,28–31]. The majority of the previous studies have evaluated the effect of erbium:yttrium-aluminum-garnet (Er:YAG) and neodmium:yttrium-aluminum-garnet (Nd:YAG) lasers on zirconia ceramics [31–34] and have demonstrated controversial results. According to Turp et al. [31], Er:YAG laser etching may be an alternative to air-particle abrasion for zirconia ceramics however, the effects of laser treatment on zirconia ceramics are very different than those on lithium-based ceramics because of the presence of a glass matrix in the composition of lithium-based ceramics. Er, Cr:YSGG laser irradiation shows its effect on hard and soft tissues through the interaction of laser energy with atomized water droplets on the tissue interface, resulting in micro-explosions and ablation of the tissue [35]. Therefore, the effect of Er, Cr:YSGG laser on different restorative materials might vary due to the water content of the restorative materials. In the present study; for IPS e.max CAD, 2W Er, Cr:YSGG laser treatment had no positive effect on bond strength however, Er, Cr:YSGG laser irradiation at an energy level of 3W increased the bond strength values in comparison to control group. This result showed that the modifications on the surface of restorative materials after laser etching depend on the energy level of the laser radiation as well as on the type of irradiated material as mentioned in a previous study [36]. This result was also supported by FE-SEM images of 3W laser group which demonstrates micro-pores and irregularities that was not observed in 2W group. The findings of the current study do not agree with a study [37] which reported that as the laser power setting increased, the bond strength values of lithium di-silicate ceramics decreased. The high energy levels (4W-10W) applied in that study may be the reason of different results.

In contrast; for resin nano-ceramic materials evaluated in this study, 3W Er, Cr:YSGG laser applied groups showed lower bond strength values compared to control group and 2W laser groups. According to Gokce et al. [37], low bond strengths after higher laser power settings may be explained by a formation of heat damaged layer due to higher laser power settings. This layer might be poorly attached to the infra layers of the restorative material, while the outer layer of the material still strongly bonded to the silane and resin cement [37]. For PICN material, 2W and 3W laser irradiations show similar effect on bond strength and surface roughness. Laser application increased the bond strength values compared to control group however, this difference is not significant. Barutcigil et al. [4] concluded that 2W Er, Cr:YSGG irradiation is as effective as sandblasting and HF acid application on bond strength of Vita Enamic to resin cement which was not in accordance with the results of the current study.

Regarding the failure modes of tested materials, the higher number of adhesive failures observed in all groups of the present study may be attributed to the small bonded cross-sectional areas (1mm^2 or less) required for the micro-shear bond strength test [17,18]. Cohesive failures observed in resin cement indicate that the bond between the restorative materials and resin cement seemed to exceed the strength of the resin cement.

Conclusion

Within the limitations of the study, it can be concluded that prior to bonding 5% HF acid treatment is the best surface treatment method regarding the bond strength for all CAD-CAM restorative materials. Er, Cr:YSGG laser application with energy level of 3W can be recommended for IPS e.max CAD. However further studies that evaluate mechanical and optical properties of surface treated CAD-CAM materials are needed to prove the effectiveness of surface treatment procedures

Disclosure statement

The authors declare that they have no conflict of interests. This study was not supported by any foundation.

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