Effect of surface treatments and universal adhesive application on the microshear bond strength of CAD/CAM materials

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PURPOSE. The aim of this study was to evaluate the microshear bond strength (μSBS) of four computer-aided design/computer-aided manufacturing (CAD/CAM) blocks repaired with composite resin using three different surface treatment protocols. **MATERIALS AND METHODS.** Four different CAD/CAM blocks were used in this study: (1) flexible hybrid ceramic (FHC), (2) resin nanoceramic (RNC), (c) polymer infiltrated ceramic network (PICN) and (4) feldspar ceramic (FC). All groups were further divided into four subgroups according to surface treatment: control, hydrofluoric acid etching (HF), air-borne particle abrasion with aluminum oxide (AlO), and tribochemical silica coating (TSC). After surface treatments, silane was applied to half of the specimens. Then, a silane-containing universal adhesive was applied, and specimens were repaired with a composite, Next, μSBS test was performed. Additional specimens were examined with a contact profilometer and scanning electron microscopy. The data were analyzed with ANOVA and Tukey tests. **RESULTS.** The findings revealed that silane application yielded higher μSBS values ($P<.05$). All surface treatments were showed a significant increase in μSBS values compared to the control ($P<.05$). For FHC and RNC, the most influential treatments were AlO and TSC ($P<.05$). **CONCLUSION.** Surface treatment is mandatory when the silane is not preferred, but the best bond strength values were obtained with the combination of surface treatment and silane application. HF provides improved bond strength when the ceramic content of material increases, whereas AlO and TSC gives improved bond strength when the composite content of material increases. ***[J Adv Prosthodont 2020;12:22-32]***

KEYWORDS: Dental restoration repair; Ceramics; Shear strength; Dental bonding

INTRODUCTION

Computer-aided design/computer-aided manufacturing (CAD/CAM) has become a part of common practice in today's dentistry. With the help of CAD/CAM systems, restorations can be made in a single visit. CAD/CAM indirect restorative materials are mainly divided into two categories as ceramics and composites.[1,2] Although the most preferred indirect restoration material in clinical practice is ceramics, they have low fracture toughness and high brittleness.[3-5] To overcome this disadvantage, polymer-infiltrated ceramic network (PICN, hybrid ceramic) and resin nanoceramic materials were introduced. Hybrid ceramic is an interpenetrating phase composite material, which is formed by infiltration of 14% resin into 86% ceramic network.[6,7] Thus, the material has a hybrid surface that can be treated to both indirect composite or ceramic materials. On the other hand, the so-called resin nanoceramic material is a composite-ceramic restorative material, which combines the advantages of a

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highly cross-linked resin matrix (79% urethane dimethacrylate) and ceramic (zirconia-silica nano-fillers).[8,9] Due to their improved fracture toughness and reduced brittleness, hybrid ceramics and resin nanoceramics are in use today as an alternative to ceramics.[1,6]

One of the most common failures in CAD/CAM indirect restorations is fractures, whether they are made of ceramic or composite.[10,11] In such cases, the dentist must choose between total replacement or repair of the failed restoration. In most of the clinical cases, total replacement of the failed restoration may not be an appropriate choice since it may cause trauma to healthy dental tissues.[12] Time consumption and trauma risk could be minimized by repair of the restoration.[13] Therefore, it may be preferable to repair the failed indirect restorations rather than to replace them. However, achieving a durable and reliable bond between failed restoration and composite resin may be difficult.[14,15] Restoration repair is done by the preparation of the surface of the failed restoration and completion of the missing part with composite resin material. The clinical success of the repair depends on the bond strength between the failed restoration and the composite resin. Several methods are suggested to provide an adequate bond strength between the failed restoration and the repair composite as follows; coarse diamond bur abrasion,[16-18] hydrofluoric acid etching,[17,19-21] phosphoric acid etching,[19] air-borne particle abrasion with aluminum oxide,[18-23] tribochemical silica coating,[16,18,19,21,22,24] and application of silane coupling agent.[16,17,20,22,23] The application of an intermediate adhesive also improves the repair bond strength.[16,17,21-23]

CAD/CAM materials are one of the fastest developing field in dental materials. Despite their increasing use, no repair protocol has yet been agreed upon. Thus, the aim of this study was to investigate the effect of different surface treatments and the additional silane application in the repair of four different CAD/CAM materials using a silane-containing universal adhesive. The hypotheses were (a) the additional silane application would improve the repair bond strength values compared to the use of silane-containing universal adhesive alone, and (b) the CAD/CAM material type and different surface treatments would not influence the repair bond strength.

MATERIALS AND METHODS

Four different type of CAD/CAM indirect restorative blocks, namely (a) flexible hybrid ceramic (FHC; CeraSmart; GC Corp., Tokyo, Japan), (b) resin nanoceramic (RNC; Lava Ultimate; 3M ESPE, St. Paul, MN, USA), (c) polymer infiltrated ceramic network (PICN; Vita Enamic, VITA Zahnfabrik H. Rauter, Bad Sakingen, Germany), and (d) feldspar ceramic (FC; Vitablocks Mark II, VITA Zahnfabrik H. Rauter, Bad Sakingen, Germany) were used in this *in vitro* study. The brands, batch numbers, manufacturers, and chemical compositions of the tested materials are presented in Table 1.

The CAD/CAM blocks were cut into 3-mm thick slices using a precision cutter (IsoMet High Speed Pro; Buechler, Lake Bluff, IL, USA). Thirty-two specimens were obtained for each CAD/CAM block (in total, 128 specimens), and then embedded in a self-cured acrylic resin (Integra; BG Dental, Ankara, Turkey) with the surfaces to be tested facing upwards. The embedded specimens were ground using water-cooled 600-grit silicon carbide (SiC) paper. All specimens were aged by thermal cycling (5000 thermal cycles between 5°C and 55°C with a dwelling time of 30 seconds) prior to repair, and then specimens were randomly assigned to one of the following subgroups (n = 8):

Group 1 (Control, no treatment): no surface treatment was done to the CAD/CAM material surface as a control.

Group 2 (HF; hydrofluoric acid etching): 9% hydrofluoric acid (Ultradent, South Jordan, UT, USA) was used for etching the CAD/CAM material surface for 60 seconds. HF was rinsed in distilled water, and then air dried.

Table 1. Materials used in the study

Material	Batch Number	Type	Composition
CeraSmart (FHC; GC Corp., Tokyo, Japan)	150625A	Flexible hybrid ceramic	Bis-MEPP, UDMA, Dimethacrylate. Filler: SiO_2, barium glass, 71% by weight.
Lava Ultimate (RNC; 3M ESPE, St. Paul, MN, USA)	N619802	Resin nanoceramic	Bis-GMA, UDMA, Bis-EMA, TEGDMA. Filler: SiO_2, ZrO_2, Si/ZrO_2 cluster, 80% by weight.
Vita Enamic (PICN; Vita Zahnfabrik H. Rauter, Bad Sackingen, Germany)	51540	Polymer infiltrated ceramic network	UDMA, TEGDMA. Filler: Feldspar ceramic enriched with aluminum oxide, 86% by weight.
Vitablocks Mark II (FC; Vita Zahnfabrik H. Rauter, Bad Sackingen, Germany)	35360	Feldspar ceramic	Fine-particle feldspar ceramic.
Single Bond Universal (3M ESPE, St. Paul, MN, USA)	665259	Universal adhesive	2-HEMA, 10-MDP, dimethacrylate resins, Vitrebond™ copolymer, silane, filler, ethanol, water, initiators. pH: 2.7

Abbreviations: 2-hydroxyethyl methacrylate (HEMA), 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), Bisphenol A polyethethylene glycol diether dimethacrylate (Bis-EMA), Bisphenol A diglycidylmethacrylate (Bis-GMA), 2,2-Bis (4-methacryloxypolyethoxyphenyl) propane (Bis-MEPP), triethylene glycol dimethacrylate (TEGDMA), Urethane dimethacrylate (UDMA).

Group 3 (AlO; air-borne particle abrasion): Air-borne particle abrasion treatment was performed on CAD/CAM material surfaces using a sandblaster (Airsonic® mini sandblaster; Hager Werken, Duisburg, Germany) 10 mm above from the specimen surface at 2.5 bar pressure with aluminum-oxide particles (Cobra; 50 μm aluminum oxide, Renfert GmbH, Hilzingen, Germany). The specimens were cleaned in distilled water using an ultrasonic bath for 5 minutes, and were then air dried.[25]

Group 4 (TSC; Tribochemical silica coating): Tribochemical silica coating was performed using the same sandblaster 10 mm above from the specimen surface at 2.5 bar pressure. For TSC, alumina coated by silica particles (CoJet sand; 30 μm, 3M ESPE, Seefeld, Germany) were used. After the TSC treatment, the specimens were cleaned in distilled water using an ultrasonic bath for 5 minutes, and were then air dried.[25]

A contact profilometer (Surtronic S128, Taylor Hobson Ltd., Leicester, England) with a 5-μm diamond stylus was used for surface roughness measurements (n = 8). The profilometer was calibrated before the measurements with the reference specimen provided by the manufacturer. Following surface treatments, starting from the center of each specimen five measurements were taken in different directions (cut-off length: 0.25 mm). The average roughness values (Ra) of the treated surfaces were recorded in μm.[25]

The topographic images of the CAD/CAM specimens were obtained over a 10 μm × 10 μm square area of treated surfaces using the scanning probe microscopy (SPM) imaging capability of the TriboIndenter (TI 950; Hysitron Inc., Eden Prairie, MN, USA), fitted with a sharp probe of 20 nm tip radius at a low imaging force of 0.5 μN. For nanoindenter, two samples were prepared for each group (n = 2).

Following the surface roughness measurements, the CAD/CAM specimens were further divided into two subgroups according to additional silane application. A silane coupling agent (S; Clearfil Ceramic Primer Plus; Kuraray Noritake Dental Inc., Tokyo, Japan) was applied to half of the specimens with an applicator brush in accordance with the manufacturer's recommendations (n = 4). Subsequently, a universal adhesive (SBU; Single Bond Universal; 3M ESPE, St. Paul, MN, USA) was applied to the CAD/CAM specimen surfaces, and light-cured using a LED curing unit (Valo Grand; 1000 mW/cm^2, Ultradent, South Jordan, UT, USA) according to the manufacturer's recommendations. The light intensity was controlled periodically. Small transparent microtubules were carefully cut from polyvinyl tube with inner diameter 1 mm and height 0.5 mm and to ensure parallel ends using a gauge. After the adhesive application, each specimen surface received two resin microtubules (n = 8 with additional silane application and n = 8 without additional silane application in each subgroup). Each microtubule was adjusted over the treated specimen surface, and gently filled with composite resin material (Filtek Ultimate Flowable Restorative, 3M ESPE, St. Paul, MN, USA). In this way, very small cylinders of composite resin were bonded to the treated surfaces as a repair. Light polymerization of the composite resins was performed according to the manufacturers' instructions using LED curing unit for 20 seconds. Polymerized specimens were kept in distilled water at 37°C for 24 hours.

A shear force was applied to the adhesive interface using a μSBS testing device (MOD Dental, Esetron Smart Robotechnologies, Ankara, Turkey) at a crosshead speed of 0.5 mm/minute. The load at failure was recorded in MPa. The failure modes were examined under a stereomicroscope at 30× magnification (Olympus SZ61, Munster, Germany). Failure modes were categorized as adhesive (A; failure at the repair-substrate bonded interfaces), cohesive (C; failure at least parts of the substrate or the repair composite), or mixed (M; A + C).

For scanning electron microscope (SEM) observations, two samples were prepared for each surface treatment group as described before (in total, 8 specimens). The specimens were sputter-coated with gold (Polaron SC7620 sputter coater, ThermoVG Scientific), and were examined under a scanning electron microscope (JEOL 5500; JEOL Inc., Peabody, MA, USA) at 10 kV accelerating voltage. Observations were performed under 2000× magnification.

The mean and standard deviations were calculated, and normality of data distribution was tested using Kolmogorov-Smirnov and Shapiro-Wilk tests. According to the normality test, the data were normally distributed. Therefore, μSBS data were analyzed using three-way analysis of variance (ANOVA) to determine the effects of CAD/CAM material type, different surface treatments and additional silane application, and the interaction of these three factors on μSBS values. Surface roughness data were also analyzed using two-way ANOVA test. Pairwise analyzes were performed using Tukey. All analyzes were performed using SPSS statistical software (SPSS Version 22, IBM, Chicago, IL, USA) and $P < .05$ considered as statistically significant.

RESULTS

The mean and standard deviations of repair μSBS values for CAD/CAM materials after surface treatments and additional silane application are presented in Table 2. Three-way ANOVA clearly revealed that the CAD/CAM material type ($F = 131.648$, $P < .001$), different surface treatments ($F = 282.400$, $P < .001$), and additional silane application ($F = 869.915$, $P < .001$) exhibited significant influence on repair μSBS values (Table 3). In addition, the interaction among the three factors were significant (Table 3, $P < .001$).

The results of the independent t-test revealed that additional silane application yielded significantly higher μSBS values (Table 2, $P < .05$). According to the post-hoc analysis, all surface treatments showed statistically significant increases in repair μSBS values compared to the control group, irrespective of the additional silane application (Table 4, $P < .05$). For RNC, the most influential parameters were AlO and TSC treatments (AlO: 25.58, TSC: 28.96; AlO+S: 29.23, TSC+S: 34.01; $P < .05$). Similarly, higher repair μSBS values for FHC were obtained with AlO and

Table 2. Means and standard deviations of μSBS data according to the silane application

Material	Surface Treatment	Silane Application	Mean (SD)	Median	Min.	Max.	*P* Value
CeraSmart (FHC)	Control	No	11.33 (1.52)	11.67	8.80	13.01	< .001***
		Yes	19.70 (1.99)	19.93	16.85	22.41	
	HF	No	15.71 (2.24)	15.93	12.00	18.40	< .001***
		Yes	27.22 (2.14)	27.53	22.42	29.10	
	AlO	No	20.52 (2.24)	20.38	17.53	25.00	< .001***
		Yes	31.36 (3.15)	30.88	26.30	35.42	
	TSC	No	24.24 (3.45)	23.62	19.52	30.47	< .001***
		Yes	32.87 (3.56)	32.87	26.00	38.60	
Lava Ultimate (RNC)	Control	No	12.22 (2.51)	12.94	7.20	14.53	< .001***
		Yes	21.35 (2.41)	21.74	16.88	24.30	
	HF	No	18.46 (1.69)	18.11	15.72	20.88	< .001***
		Yes	27.96 (1.79)	28.63	24.20	29.37	
	AlO	No	25.58 (2.40)	25.47	22.42	30.20	.04*
		Yes	29.23 (1.78)	29.47	26.50	31.30	
	TSC	No	28.96 (1.79)	28.43	27.19	31.58	< .001***
		Yes	34.01 (2.33)	33.74	29.87	37.10	
Vita Enamic (PICN)	Control	No	10.97 (1.56)	11.47	8.56	13.15	< .001***
		Yes	23.05 (2.78)	24.65	17.80	25.35	
	HF	No	23.22 (4.65)	23.20	16.76	29.75	.01*
		Yes	32.29 (3.51)	30.34	29.00	37.65	
	AlO	No	20.66 (2.35)	21.10	16.34	23.65	< .001***
		Yes	30.62 (1.96)	30.83	27.50	33.47	
	TSC	No	24.87 (4.17)	24.61	16.45	30.00	.001***
		Yes	32.34 (2.72)	33.69	27.80	34.46	
Vitablocks Mark II (FC)	Control	No	6.29 (1.25)	6.50	4.50	7.78	< .001***
		Yes	13.36 (1.10)	13.51	11.79	14.81	
	HF	No	16.35 (4.16)	15.54	11.16	22.00	.01*
		Yes	23.97 (3.15)	24.70	17.90	28.10	
	AlO	No	10.48 (1.08)	10.15	8.74	11.89	< .001***
		Yes	28.31 (1.38)	24.91	22.64	26.73	
	TSC	No	12.69 (1.37)	12.66	10.75	14.56	< .001***
		Yes	24.87 (1.58)	28.33	26.66	30.54	

Independent t-test, ***$P < .001$ and *$P < .05$.

TSC treatments (AlO: 20.52, TSC: 24.24; AlO+S: 31.36, TSC+S: 32.87; $P < .05$). For FC, AlO and TSC treatments provided a slight improvement in terms of μSBS values, but this improvement was not statistically significant for AlO (Control: 6.29, AlO: 10.48, TSC: 12.69; $P < .05$). On the other hand, HF treatment significantly improved repair μSBS, particularly with additional silane application (Control: 6.29, HF: 16.35; $P < .05$). However, although higher repair μSBS values were obtained after HF treatment with additional silane application, there was no significant difference between AlO and TSC treatments and HF treatment (Control: 13.36, HF: 28.31, AlO: 24.87, TSC: 23.97; $P > .05$). For PICN, surface treatments also positively contributed to repair μSBS values compared to the control group ($P > .05$). In addition, it is noteworthy that PICN was the only CAD/CAM block in which there was no significant difference among surface treatments ($P > .05$).

Failure mode distribution for CAD/CAM materials after surface treatments and additional silane application is listed in Table 5. For the composite blocks (FHC and RNC), the

Table 3. Influence of material type, surface treatment and additional silane application on μSBS results according to the three-way ANOVA

Source	Type III sum of squares	df	Mean square	F	Sig.
Corrected Model	14743.666[a]	31	475.602	73.603	.000***
Intercept	127822.785	1	127822.785	19781.497	.000***
Material	2552.024	3	850.675	131.648	.000***
Treatment	5474.390	3	1824.797	282.400	.000***
Silane	5621.157	1	5621.157	869.915	.000***
material * treatment	521.859	9	57.984	8.973	.000***
material * silane	159.255	3	53.085	8.215	.000***
treatment * silane	3.120	3	1.040	.161	.923
material * treatment * silane	411.861	9	45.762	7.082	.000***
Error	1447.429	224	6.462		
Total	144013.879	256			
Corrected Total	16191.094	255			

[a] R Squared = .911 (Adjusted R Squared = .898), ***$P < .001$.

Table 4. Means and standard deviations of μSBS data and post-hoc analysis for pairwise comparison

Silane Application	Surface Treatment	CAD/CAM Indirect Restoratives			
		FHC	RNC	PICN	FC
No	Control	11.33 ± 1.52 $^{A, a}$	12.22 ± 2.51 $^{A, a}$	10.97 ± 1.56 $^{AB, a}$	6.29 ± 1.25 $^{B, a}$
	HF	15.71 ± 2.24 $^{A, ab}$	18.46 ± 1.69 $^{AB, b}$	23.22 ± 4.65 $^{B, b}$	16.35 ± 4.16 $^{A, b}$
	AlO	20.52 ± 2.24 $^{A, bc}$	25.58 ± 2.40 $^{B, c}$	20.66 ± 2.35 $^{A, b}$	10.48 ± 1.08 $^{C, ac}$
	TSC	24.24 ± 3.45 $^{A, c}$	28.96 ± 1.79 $^{A, c}$	24.87 ± 4.17 $^{A, b}$	12.69 ± 1.37 $^{B, bc}$
Yes	Control	19.70 ± 1.99 $^{X, x}$	21.35 ± 2.41 $^{X, x}$	23.05 ± 2.78 $^{X, x}$	13.36 ± 1.10 $^{Y, x}$
	HF	27.22 ± 2.14 $^{X, y}$	27.96 ± 1.79 $^{XY, y}$	32.29 ± 3.51 $^{Y, y}$	28.31 ± 1.38 $^{XY, y}$
	AlO	31.36 ± 3.15 $^{X, yz}$	29.23 ± 1.78 $^{XY, yz}$	30.62 ± 1.96 $^{X, y}$	24.87 ± 1.58 $^{Y, y}$
	TSC	32.87 ± 3.56 $^{X, z}$	34.01 ± 2.33 $^{X, z}$	32.34 ± 2.72 $^{X, y}$	23.97 ± 3.15 $^{Y, y}$

Means followed by similar capital letters are not significantly different (surface treatment comparison). Means followed by similar lowercase letters (comparison between material type) are not significantly different (a, b, c for with additional silane application; x, y, z for without additional silane application).

predominant mode of failure was adhesive failure at the interface. An increase in the rate of mixed failure was observed for the specimens with additional silane application. In composite blocks, it was seen that AlO and TSC treatments caused a slightly more mixed failure than HF treatment. For PICN and FC blocks, the predominant mode of failure was also adhesive failure, but unlike FHC and RNC blocks, HF treatment increased the mixed mode of failure. In addition, the highest percent of cohesive failures were evident in additional silane application subgroups for all materials (Table 5).

The surface roughness (Ra) values of groups are presented in Table 6. Surface treatment and CAD/CAM material type influenced the Ra values and a significant interaction between those two factors was detected according to two-way ANOVA ($P < .001$). After the surface treatments, the surface roughness values of each CAD/CAM material were increased significantly according to the contact profilometer results ($P < .05$). No significant difference was found between the control (no treatment) groups among all CAD/CAM materials ($P > .05$). The highest surface roughness values were obtained with AlO and TSC treatments in all indirect restoratives, except FC ($P < .05$); HF treatment was produced the highest surface roughness values for FC (1.15). In addition, no significant difference was found between AlO and TSC treatments in all CAD/CAM materials ($P > .05$), except PICN. AlO treatment (1.15) produced higher surface roughness values than TSC (0.83) for PICN

Table 5. Failure mode distribution

Material	Surface Treatment	Silane Application	Adhesive	Cohesive	Mixed
CeraSmart (FHC)	Control	No	87.5	0	12.5
		Yes	75	12.5	12.5
	HF	No	75	0	25
		Yes	62.5	12.5	25
	AlO	No	62.5	0	37.5
		Yes	50	12.5	37.5
	TSC	No	50	12.5	37.5
		Yes	25	25	50
Lava Ultimate (RNC)	Control	No	87.5	0	12.5
		Yes	62.6	12.5	25
	HF	No	75	0	25
		Yes	62.5	0	37.5
	AlO	No	50	12.5	37.5
		Yes	37.5	25	37.5
	TSC	No	50	25	25
		Yes	37.5	25	37.5
Vita Enamic (PICN)	Control	No	100	0	0
		Yes	87.5	0	12.5
	HF	No	50	12.5	37.5
		Yes	37.5	25	37.5
	AlO	No	62.5	12.5	25
		Yes	50	25	25
	TSC	No	62.5	0	37.5
		Yes	50	25	25
Vitablocks Mark II (FC)	Control	No	87.5	0	12.5
		Yes	75	12.5	12.5
	HF	No	50	12.5	37.5
		Yes	25	25	50
	AlO	No	62.5	12.5	25
		Yes	50	12.5	37.5
	TSC	No	62.5	12.5	25
		Yes	50	12.5	37.5

Table 6. Mean and standard deviation of surface roughness (Ra ± SD) values of CAD/CAM indirect restoratives with different surface treatments

Surface Treatment	CAD/CAM Indirect Restoratives			
	FHC	RNC	PICN	FC
Control	0.56 ± 0.08 $^{A, a}$	0.62 ± 0.07 $^{A, a}$	0.51 ± 0.10 $^{A, a}$	0.56 ± 0.04 $^{A, a}$
HF	1.20 ± 0.08 $^{A, b}$	1.18 ± 0.08 $^{AB, b}$	1.01 ± 0.14 $^{B, bc}$	1.14 ± 0.12 $^{A, b}$
AlO	1.47 ± 0.08 $^{A, c}$	1.55 ± 0.08 $^{A, c}$	1.13 ± 0.09 $^{B, c}$	0.94 ± 0.13 $^{C, c}$
TSC	1.61 ± 0.07 $^{A, c}$	1.69 ± 0.16 $^{A, c}$	0.88 ± 0.13 $^{B, b}$	0.90 ± 0.09 $^{B, c}$

Mean values represented with same superscript uppercase letters (row) or lowercase letters (column) are not significant according to Tukey test ($P > .05$).

($P > .05$).

It is observed that HF treatment created deep grooves for both PICN and FC. AlO and TSC treatments produced similar surface topography in accordance with surface roughness measurements.

Example images from the SEM evaluation are shown in Fig. 1 to Fig. 4. According to SEM micrographs, all CAD/CAM material surfaces were smooth before the surface treatments. However, the surface topography of each CAD/CAM material significantly altered after surface treatments. These alterations in surface roughness were clearly observed on SEM micrographs. Evident undercuts, ridges and grooves on the surface of each material could be easily identified after surface treatments (Fig. 1, Fig. 2, Fig. 3, Fig. 4). HF treatment produced rougher surface than AlO and TSC treatments in profilometry measurements. The profilometry measurements were confirmed through the SEM micrograph of the FC, which appeared much rougher than AlO and TSC ceramic surfaces (Fig. 4H, Fig. 4F).

DISCUSSION

The bond strength between CAD/CAM indirect restorative material and composite resin is crucial for repair of fractures when the prognosis of the repair is concerned. In this *in vitro* study, the effect of different surface treatments and the additional silane application on the repair of four different CAD/CAM materials using a silane-containing universal adhesive was investigated. When the findings of our study were examined, additional silane application yielded better repair μSBS values than silane-containing universal adhesive application alone (Table 2). Therefore, the first hypothesis -

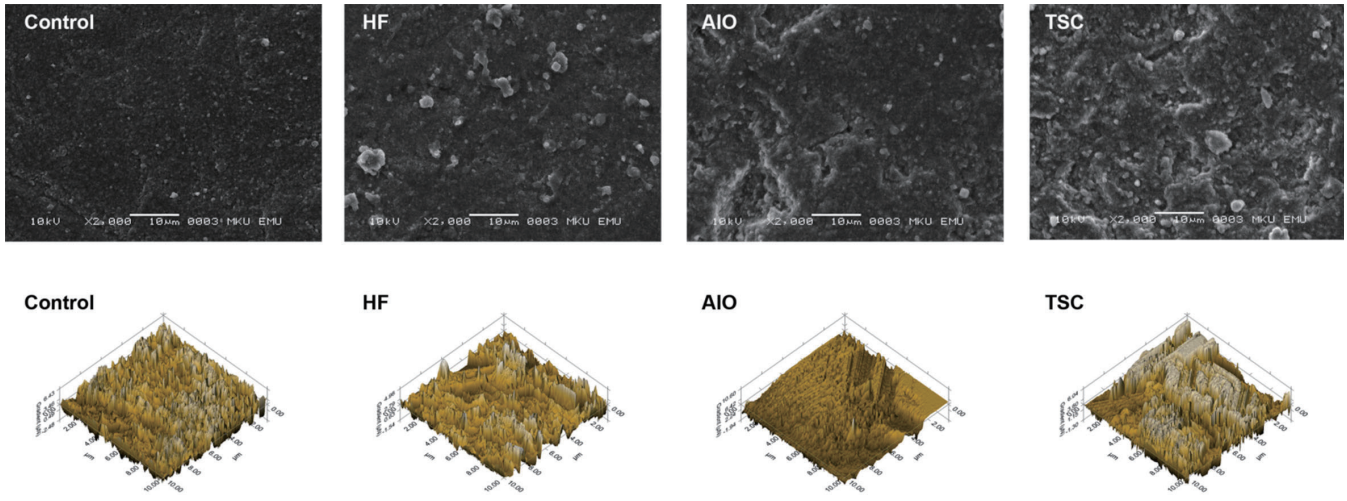

Fig. 1. SEM and surface topography images of FHC material.

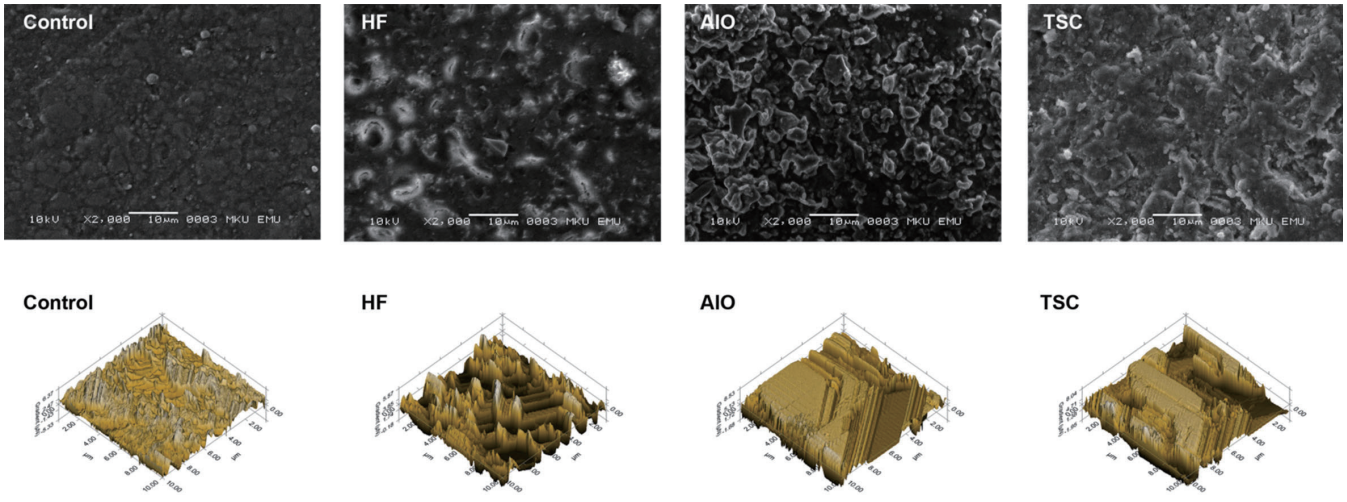

Fig. 2. SEM and surface topography images of RNC material.

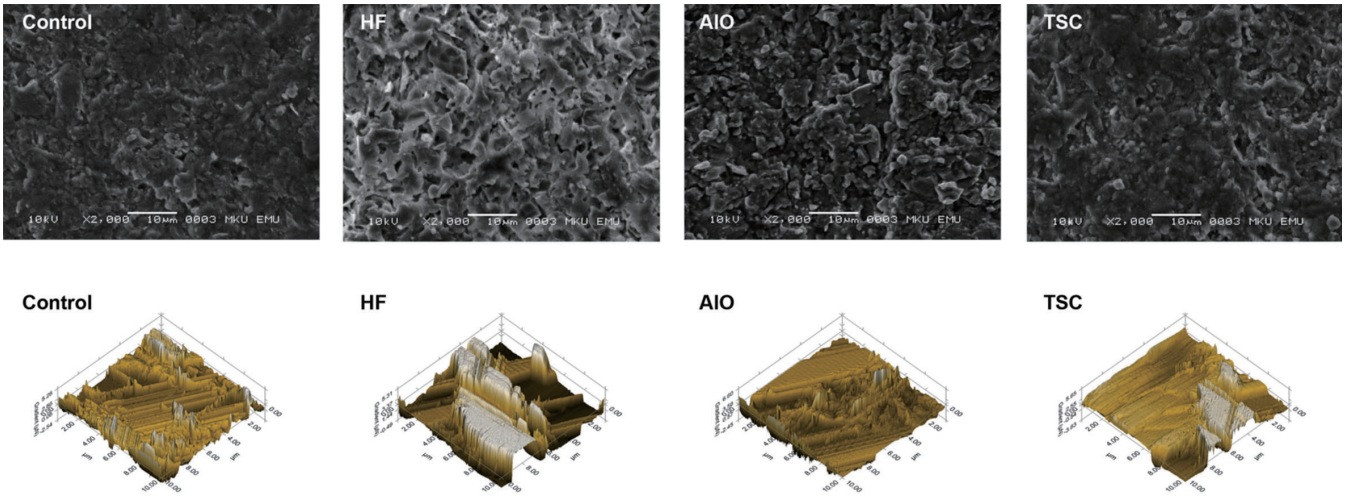

Fig. 3. SEM and surface topography images of PICN material.

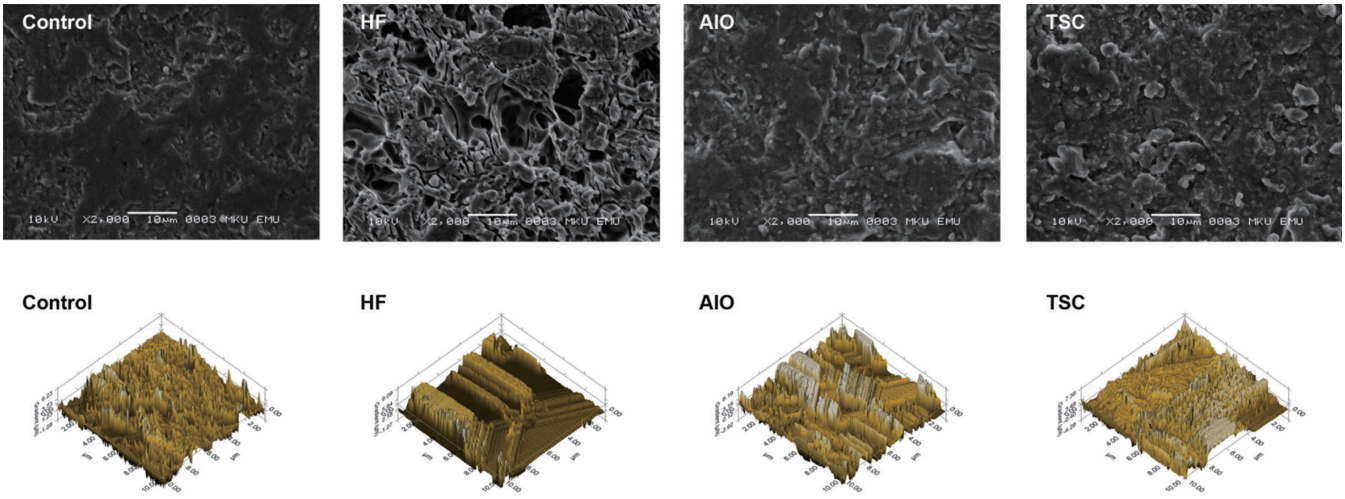

Fig. 4. SEM and surface topography images of FC material.

that the additional silane application would improve the repair bond strength values compared to the use of silane-containing universal adhesive alone - could be accepted. On the other hand, three-way ANOVA clearly revealed that the CAD/CAM material type and different surface treatments exhibited significant influence on repair μSBS values (Table 3). Thus, the second hypothesis - that the CAD/CAM material type and different surface treatments would not influence the repair bond strength - was rejected.

Failures that require repair usually occur after a certain period of clinical usage. Aging of the materials before repair is important to simulate oral conditions in laboratory studies examining the repair potential of restorative materials. Although there is no gold standard for the aging of CAD/CAM materials, thermal cycling is applied in most of the studies.[19,22,25] Therefore, specimens were submitted to thermal cycling (5000 cycles in two water baths of 55°C and 5°C with a dwell time of 30 s in each bath) to simulate the oral environment. Volumetric changes resulting from the thermal cycling may cause mechanical stress and form microcracks in the material, which may decrease the bond strength. Moreover, decrease in bond strength with aging has been reported in different studies.[19,22,25] In our study, the effect of surface treatments on bond strength were investigated rather than the effect of aging protocol. Hence, all samples were treated equally regarding the aging protocol to provide comparability.

Universal adhesives reduce the complexity of clinical application procedures and provide ease of application for the clinicians.[17] One of the main ingredients of universal adhesives is a functional monomer, mostly 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP).[26,27] 10-MDP chemically interacts with metals, hydroxyapatite or filler particles,[28] in particular with ZrO_2.[29-31] SBU also contains Bis-

GMA monomer (Table 1). The Bis-GMA monomer can inhibit the reaction between the silane and the hydroxyl group of the silica-containing restorative materials.[32] In a previous study, the compatibility between the hydroxyl groups of inorganic filler particles of the substrate and the hydrolysable functional groups of the silane has been reported to influence the bond strength.[17] In our study, higher bond strength values obtained by additional silane application suggested that the amount of silane contained in the universal adhesive did not provide sufficient compatibility. Similar results were reported in a previous study, in which the highest bond strength values were obtained with additional silane application compared to the silane-containing universal adhesive alone.[33] Yoshihara *et al.*[34] tested the bond strength of silane-free universal adhesive to the ceramic substrate after mixing with silane; immediately, 1 day, 3 days and 7 days after the mixture. They reported that only the silane coupling effect of freshly prepared silane-containing universal adhesive was effective. In addition, Fourier-transform infrared (FTIR) spectroscopy analyses showed silanol-characteristic peaks only in freshly prepared adhesive/silane mixture.[34] On the other hand, SBU has low pH and contains water in its chemical composition. In a different study, it was reported that the presence of water and low pH caused silane hydrolysis.[35] Both the data from our study and the literature[33,34] suggest that silane in universal adhesives is not an alternative to additional silane application.

The results of this *in vitro* study, further revealed that surface treatments significantly improved repair μSBS values of CAD/CAM materials. Considering the acceptable bond strength range suggested by Elsaka[24] (15 - 25 MPa), repair bond strength values obtained with additional silane application were within the acceptable range for all CAD/CAM materials, except control group of FC (13.36). On the other hand, the bond strength values of the surface treated FHC, RNC and PICN materials were also within the acceptable range when the silane-containing universal adhesive was used alone. This finding was also consistent with a previous study.[17] The results showed that, although additional silane application is required especially for FC, mechanical interlocking with irregularities created on the ceramic surface is the main factor for a strong bond. Previous studies have shown similar results.[16,36] The control group, which was flattened using SiC paper and received no surface treatment, exhibited low bond strengths (Control: 6.29, Control+S: 13.36) which were insufficient to increase reliable adhesion to the repair material.[24] According to our results, for FC, the optimum bond strength values were obtained for the combination of HF treatment and silanization (28.31). HF etching and silane-containing universal adhesive application resulted in higher bond strength values than AlO and TSC treatments when additional silane application was not possible (16.35). In spite of all the potential harmful effects of intra oral administration,[37] hydrofluoric acid is still the most preferred surface treatment for acid-sensitive ceramics.[38,39] The hydrofluoric acid selectively interacts with the glassy parts of the glass-ceramic materials and creates porous, irregular surface, thereby increasing the surface roughness (Table 6). It provides microretention and creates hydroxyl groups that provide chemical bonding with composite resin.[40] Furthermore, in the present study, HF treatment produced the highest surface roughness values for FC and SEM micrographs support this finding.

PICN is an interpenetrating phase composite material, which is formed by infiltration of 14% resin into 86% ceramic network.[1,6,7] As a result, hybrid ceramics can be etched with hydrofluoric acid, such as etchable ceramics,[19,21] or roughened by air-borne particle abrasion, such as indirect composites.[19,21,23] Since the ceramic phase is more dominant in the chemical composition of the PICN, HF treatment can be expected to result in better bond strength value than the other treatments. Campos et al. reported that HF treatment was more successful than air-borne particle abrasion for PICN in terms of bond strength. In our study, HF treatment resulted in higher bond strength values for PICN compared to air-abrasion treatment, but this difference was not statistically significant. With the current study, we have determined that after thermal cycling (5000 thermal cycles between 5°C and 55°C with a dwelling time of 30 seconds), HF treatment had significantly higher bond strength values than air-borne particle abrasion treatments. On the other hand, HF treated hybrid ceramic showed higher bond strength values than HF treated FC in the present study. This finding can be explained by the fact that the monomers in the chemical composition of the universal adhesive improve bond strength with the almost completely reacted monomers, which forms the hybrid ceramic. This increase in bond strength is similar to the repair of aged composite restorations. Although there is no unreacted methacrylate monomer in the aged composite structure, monomers in the adhesive provide a better bond between the aged and the new composite by preparing the surface of the aged substrate.[41] Similar to our results, PICN showed higher bond strength values than FC in other studies.[19,23,42]

Air-borne particle abrasion treatments exhibited the highest bond strength and surface roughness values, except feldspar ceramic. Although, TSC treatment showed slightly higher bond strength values, there was no significant difference between AlO and TSC treatments. Air-borne particle abrasion is based on the throwing of particles, which was accelerated by air pressure against to the substrate surface. The energy generated by the intensity of this impact provides the formation of a new layer on the substrate surface, which has an irregular, porous surface topography, as seen on Fig. 1 - Fig. 4. The increased surface roughness improves the interlocking between substrate and composite resin.[43] Furthermore, when alumina coated by silane particles (TSC sand) are employed, the impact generated by the air abrasion promotes the silicatization of the surface by a tribochemical reaction.[19] By increasing the surface energy, air-borne particle abrasion enables optimal wetting of silane.[44] SEM micrograph and nanoindentation image clearly demonstrated this roughening. The additionally applied silane

coupling agent on the substrate surface caused covalent bonds to form between the alumina and silica particles, and the resin material,[45,46] thereby increasing the bond strength between the resins and substrate.[38,47]

For an overall comparison between the four CAD/CAM materials repaired, FHC and RNC showed higher μSBS values when air-borne particle abrasion protocols were employed in comparison to the PICN and FC materials. This may be due to the difference in microstructure of these materials. SBU adhesive which contains 10-MDP monomer that chemically reacts to ZrO_2, providing a higher repair bond strength values for RNC, particularly after air-borne particle abrasion.[16,48] Similar to our findings, researchers concluded that the efficiency of surface treatment was highly dependent on the chemical composition of the substrate material rather than the surface treatment itself.[16,49] Beside the difference in flexural strength, resilience may allow the resin nanoceramics to reach higher repair μSBS values than feldspar ceramic before the failure. The findings of our study suggested the influence of surface treatments on repair bond strength of CAD/CAM materials is material-dependent, which were consistent with previous studies.[16,19]

CONCLUSION

Within the limitations of the present study, additional surface treatment is mandatory when the silane coupling agent is not preferred, but the best bond strength values were obtained with the combination of surface treatment and additional silane application. HF treatment provides more successful bond strength when the ceramic content increases in the material composition, whereas air-borne particle abrasion gives more successful bond strength values when the composite content increases. For feldspathic ceramic, both air-borne particle abrasion and HF etching can be used when silane is applied. HF etching is indicated when the silane-containing adhesive is applied alone. Besides, micromechanical retention with airborne-particle abrasion is essential for repairing aged CS and LU. Therefore, the success of surface treatments can vary depending on the CAD/CAM material type. The most suitable surface treatment should be determined by the clinician according to the CAD/CAM material used.

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