Influence of different surface treatments on bond strength of novel CAD/CAM restorative materials to resin cement

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PURPOSE. To evaluate the effects of different surface treatments on the bond strength of novel CAD/CAM restorative materials to resin cement by four point bending test. **MATERIALS AND METHODS.** The CAD/CAM materials under investigation were e.max CAD, Mark II, Lava Ultimate, and Enamic. A total of 400 bar specimens (4×1.2×12 mm) (n=10) milled from the CAD/CAM blocks underwent various pretreatments (no pretreatment (C), hydrofluoric acid (A), hydrofluoric acid + universal adhesive (Scotchbond) (AS), sandblasting (Sb), and sandblasting + universal adhesive (SbS)). The bars were luted end-to-end on the prepared surfaces with a dual curing adhesive resin cement (Variolink N, Ivoclar Vivadent) on the custom-made stainless steel mold. Ten test specimens for each treatment and material combination were performed with four point bending test method. Data were analyzed using ANOVA and Tukey's test. **RESULTS.** The surface treatment and type of CAD/CAM restorative material showed a significant effect on the four point bending strength (FPBS) ($P<.001$). For LDC, AS surface treatment showed the highest FPBS results (100.31 ± 10.7 MPa) and the lowest values were obtained in RNC (23.63 ± 9.0 MPa) for control group. SEM analyses showed that the surface topography of CAD/CAM restorative materials was modified after treatments. **CONCLUSION.** The surface treatment of sandblasting or HF acid etching in combination with a universal adhesive containing MDP can be suggested for the adhesive cementation of the novel CAD/CAM restorative materials. ***[J Adv Prosthodont 2017;9:439-46]***

KEYWORDS: Adhesive cementation; Four point bending test; Resin nano ceramic; Polymer infiltrated ceramic network; Surface treatments; Universal adhesive

INTRODUCTION

Patients' increasing demands for aesthetics and biosafety in metal-free prosthesis have driven the need for the development of new dental material and new processing technologies.[1-3] Over the past 25 years, computer-aided design and computer-aided manufacturing (CAD/CAM) have become popular in dentistry, as CAD/CAM technology offers the use of safe, aesthetically satisfying and stable materials, increased productivity in laboratory processes, less time in the fabrication of restorations, quality control, and the marginal adaptation of restorations.[2-4] Further, CAD/CAM ceramic restorations are made from a highly uniform and quality ceramic without the material variations seen in laboratory-fabricated restorations.[3,5]

Aesthetic CAD/CAM-processed indirect dental restorations are fabricated using two main types of materials: 1) lithium disilicate ceramic and 2) partially ceramic and partially resin composite (hybrid materials).[5] Ceramics and composites both have advantages and disadvantages. Recently, a composite-ceramic restorative material with marketing named as resin nanoceramic (RNC), which combines the benefits of a highly cross-linked resin matrix and ceramic, was introduced by 3M. Lava Ultimate is made of zirconia-silica nano-fillers in the

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form of dispersed or aggregated particles (79 wt %) and urethane dimethacrylate (UDMA) as the matrix.[6,7]

Polymer-infiltrated ceramic network (PICN), also called hybrid ceramic by the manufacturer, is an inter-penetrating phase material like In-Ceram, which was introduced more than two decades ago. The first PICN was produced by VITA and was called Enamic. This material resulted from the infiltration of a pre-sintered glass-ceramic network conditioned by a coupling agent with triethylene glycol dimethacrylate by capillary action.[8-10] In Enamic, the glass interpenetrating phase of In-Ceram is replaced by a polymer to form PICN. Novel PICN and RNC CAD/CAM materials have many advantages compared to ceramics, including reduced brittleness, rigidity, and hardness coupled with improved flexibility, fracture toughness, and machinability.[8,9]

Many studies and systematic reviews have identified that fractures are the main reason of clinical failures of aesthetic CAD/CAM restorations.[11-15] Preventing fracture is crucial for the clinical survival of CAD/CAM restorations; in the past, fractures have generally occurred in the marginal areas of Class I and Class II restorations and consist mostly of chips and bulk fractures.[11,12,14,15] The successful adhesive cementation of aesthetic restorations can eliminate the risk of fractures.[16] Enhancing the bond strength between aesthetic CAD/CAM restorations and cement is essential to improve fracture resistance and to preserve the marginal integrity of restorations.[17,18] To manage a sufficient bond to resin or ceramic-based CAD/CAM restorative materials, mechanical or chemical pre-treatments are important.[19,20] Chemical bonds between methacrylate monomers are widely understood bonding mechanisms between resin cement and resin-based restorative material.[19] Silane coupling agents have been used to wet ceramic and polymeric resin surfaces in order to enhance adhesive bonding.[19,21] Recently, multimode universal adhesives have been used in dental bond restoration.[22] The universal adhesive contains methacrylate-based monomers that may improve the bond between CAD/CAM materials and cement. Further, micromechanical pre-treatments achieved by hydrofluoric acid etching and/or grit blasting can ensure the success of adhesive bonding.[16] A number of previous studies have investigated Lava Ultimate and Enamic bonding through tensile and shear tests.[16,19,23]

Recently, flexural (bending) tests are preferred to determine the strength of adhesive bonds, as it is important to maintain uniform stress at the interface of two bonding materials' surfaces in order to impede critical errors, which can affect test results.[24-26] Flexural strength of ceramic and composite materials can be determined using three or four point loading. Beam-shaped specimens with a rectangular cross section is supported by two points and the load is applied vertically at either one point (three point flexure test) or two points (four point flexure test). The stress concentration of a three point test is small and concentrated under the center of the loading point, whereas the stress concentration of a four point test is over a larger region, avoiding premature failure. In contrast to three point flexural tests, four point flexural tests should be used for heterogeneous materials.[26] Although many studies have investigated bond strength between resin cement and novel CAD/CAM restorative materials, there have been limited studies on using four point bending strength (FPBS) tests to evaluate the bond strength between resin cements and CAD/CAM resin-ceramic materials.[19,23,27-29] Therefore, the purpose of this *in vitro* study was to evaluate the effect of different surface treatments on bond strength between dual-curing adhesive resin cement and different CAD/CAM hybrid restorative materials using FPBS tests. The null hypothesis of the present study was that the surface treatments and restorative materials type would not affect the bond strength of CAD/CAM restorative materials to resin cement.

MATERIALS AND METHODS

The materials used in this study and their compositions are shown in Table 1 and specimen preparation is schematically explained in Fig. 1. Specimens with the dimensions of 4 mm × 1.2 mm × 12 mm were cut from four different CAD/CAM restorative materials blocks: 1) e.max CAD LT [LDC], 2) Lava Ultimate [RNC], 3) Mark II [FGC], and 4) Enamic [PICN]. A total of 400 rectangular bar specimens (100 for each material) were obtained from blocks with a low-speed diamond saw (Micracut 201, Metkon, Bursa, Turkey) at 300 rpm/min under water cooling.

Lithium disilicate specimens were crystallized at max. 820°C for 16 minutes in a ceramic furnace (Programat P300, IvoclarVivadent, Schaan, Liechtenstein) according to the manufacturer's instructions. The specimens were cleaned ultrasonically (JP-4820 Heatable Ultrasonic Cleaner, Skymen, China) for 15 minutes at room temperature. There was no requirement for any further processing for other CAD/CAM materials.

The following treatments were applied to the bonding surface of the specimens;

Control (C): No surface treatment (n = 10).

Acid Etching (A): Etch with <9.5% hydrofluoric (HF) acid gel (Bisco, Schaumburg, IL, USA) for 60 seconds in groups RNC, PICN, FGC and for 20 seconds in group LDC. Rinse and dry for 60 seconds.

Acid Etching + Universal Adhesive(AS): Etch with <9.5% HF acid gel for 60 seconds in groups RNC, PICN, FGC and for 20 seconds in group LDC. Rinse and dry for 60 seconds. Apply Scotchbond Universal Adhesive (3M/ESPE, St. Paul, MN, USA) for 20 seconds, dry for 5 seconds, and light cure for 10 seconds.

Sandblasting (Sb): Sandblast (Renfert GmbH, USA) with 50 µm Al_2O_3 at 2.8 bar pressure for 10 seconds at a distance of 10 mm.

Sandblasting + UniversalAdhesive (SbS): Sandblast with 50 µm Al_2O_3 at 2.8 bar pressure for 10 seconds at a distance of 10 mm, apply Scotchbond Universal Adhesive for 20 seconds, dry for 5 seconds, and light cure for 10 seconds.

A custom made stainless steel mold was used for cementation procedure to standardize the cement thickness to 0.1

Table 1. Composition of materials used in this study

Product name	Manufacturer	Composition
Lava Ultimate (RNC) (Resin nanoceramic block)	3M/ESPE, St. Paul, MN, USA	80% nanoceramic, 20% resin, silica nanomers (20 nm), zirconia nanomers (4 - 11 nm), nano group particles (0.6 - 10 μm), silane bonding agent
Vita Mark II (FGC) (Feldspathic block)	Vita Zahnfabrik, Bad Säckingen, Germany	Feldspathic glass ceramic
IPS e-max CAD (LDC) (Lithium disilicate block)	IvoclarVivadent, Schaan, Liechtenstein	Lithium disilicate crystals precipitated in glass matrix
Enamic (PICN) (Interpenetrating phase composite blocks)	VitaZahnfabrik, Bad Säckingen, Germany	Acrylate polymer (14%), ceramic network (86%)
Scotchbond (Universal adhesive)	3M/ESPE, St. Paul, MN, USA	MDP phosphate monomer, dimethacrylate resin, HEMA, Vitrebond copolymer, filler, ethanol, water, initiators, silane
Variolink N (Adhesive resin cement)	IvoclarVivadent, Schaan, Liechtenstein	Barium glass filler, dimethacrylates, dispersed silica, ytterbium trifluoride, initiators and stabilizers, pigments
Bisco (Ceramic etching gel)	Bisco, Schaumburg, IL, USA	9.5% hydrofluoric acid

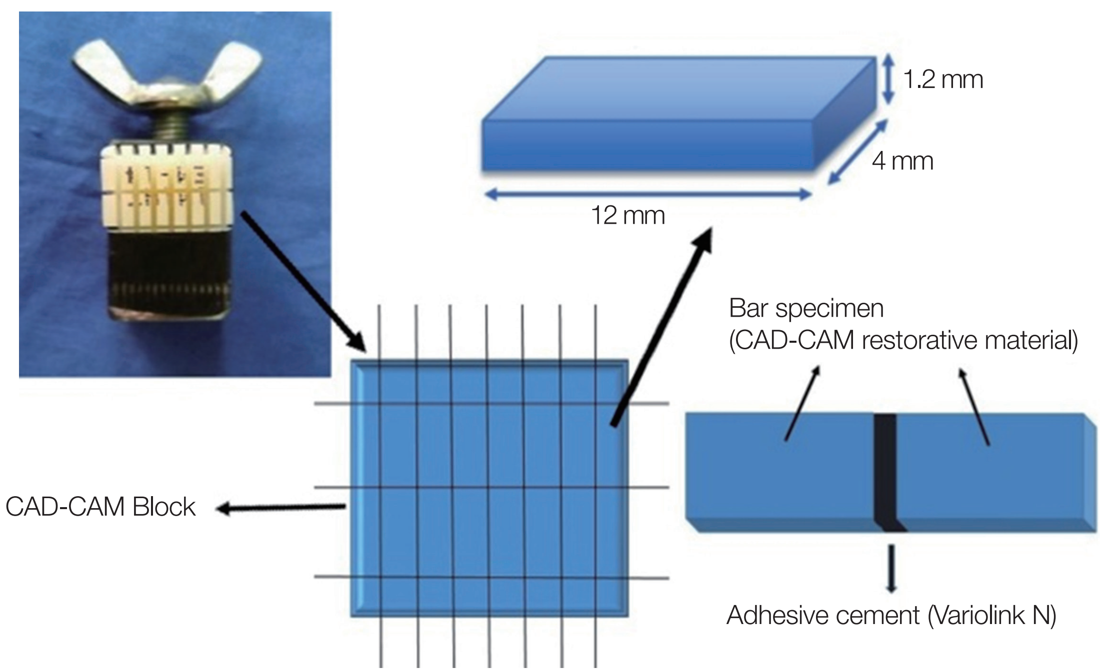

Fig. 1. Schematic drawing illustrating the study set-up and specimen preparation procedure.

mm (Fig. 2). The distance of separated fragments was 4 mm to remove the excess cement. Each pair of pretreated specimens were luted end-to-end with a dual-cure resin cement (Variolink N, Ivoclar Vivadent, Schaan, Liechtenstein) according to the manufacturer's instructions in the mold. After screwing down the mold to standardize the cement thickness, the excess cement was removed. The luted area was then covered with an oxygen-inhibiting material (Liquid Strip, Ivoclar Vivadent, Schaan, Liechtenstein). Light curing was performed for 20 seconds using T-Led (Elca Technologies, Imola, Italy) device with a light output of no less than 550 mW/cm^2.

The device was designed in accordance with the dimension of samples used for FPBS test method according to ISO standards (ceramic materials ISO 6872:2015) (Fig. 3). The FPBS test was performed using a universal testing machine (Lloyd LRX; Lloyd Instruments, Ametec Inc., Berwyn, PA, USA) at a crosshead speed of 1.0 mm/min.

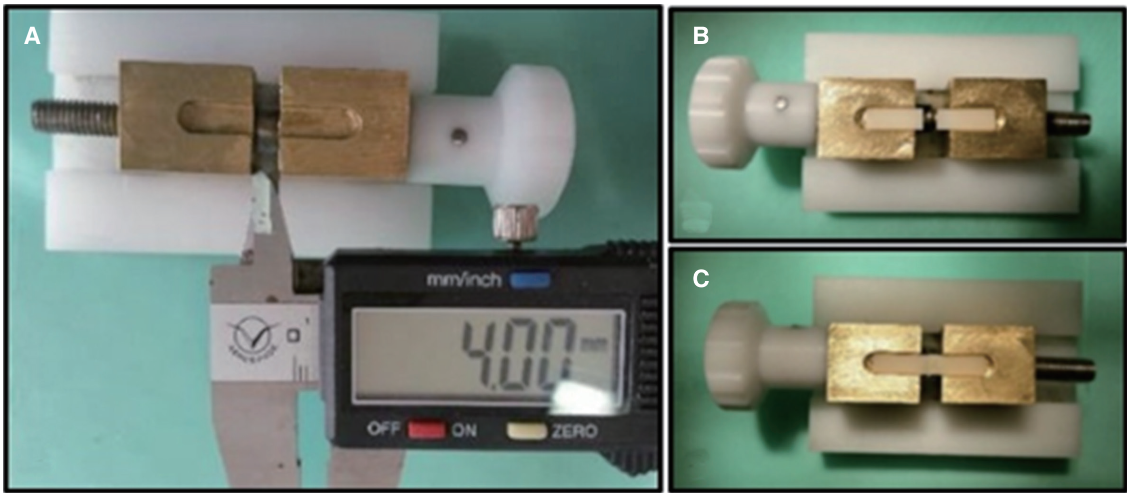

Fig. 2. (A) A custom made stainless steel mold was used for cementation. After screwing down the instrument for cementation, the distance of separated two fragments was 4 mm; (B) Setting the specimens end to end to the mold; (C) Cementation of a pair of specimens.

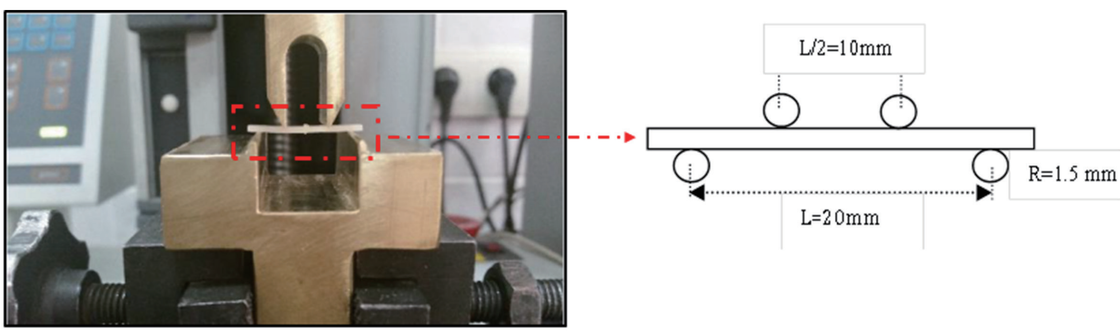

Fig. 3. The custom made device for four point bending test and experimental setup of the test.

The FPBS values were calculated with the following formula:

FPBS = "3PL / 4wb^2"

P = the load at failure in Newtons

L = the centre-to-centre distance between outer support rollers (20 mm)

w = the width of the specimen (4 mm)

b = the thickness of the specimen (1.2 mm)

After FPBS test, failure modes of the specimen surfaces were examined under a stereomicroscope (Leica MZ12, Meyer Ins., Bannockburn, IL, USA) at ×20 magnification and recorded as adhesive, cohesive, or mixed failure type.

Two-way analysis of variance (two-way ANOVA) was used to determine significant differences of FPBS values among the different CAD/CAM restorative materials and surface treatments. All mean FPBS values were compared using the Tukey-HSD multiple comparison test.

RESULTS

The FPBS values of the groups were compared and significant differences were found among the groups. According to the two-way ANOVA, the material type, surface treatments, and their interaction were statistically significant (P < .001) (Table 2). The mean FPBS values (in MPa) with standard errors for each group are presented in Table 3.

In general, control groups for each material showed lower values than the pretreatment groups. Among the control groups, e-max CAD (64.48 ± 10.3 MPa) and Mark II (64.16 ± 11.6 MPa) showed statistically higher FPBS values than Lava Ultimate (23.63 ± 9.0 MPa) and Enamic (37.88 ± 17.6 MPa) (P < .001). AS and SbS groups mostly showed the highest FBPS values for all of the CAD/CAM restorative materials.

Regarding the e-max CAD groups, there was no significant difference in FPBS values between AS (100.31 ± 10.7

Table 2. Results of two-way ANOVA test

Variable (Source)	Sum of squares	df	Mean squares	F	*P*
Material type	34700.641	3	11566.880	68.433	.000*
Surface treatment	57470.113	4	14367.528	85.003	.000*
Interaction	34542.117	12	2878.510	17.030	.000*
Error	30424.294	180	169.024		
Total	1133746.487	200			

*Significantly different at $P < .001$.

Table 3. Mean ± standard error of the FPBS values (MPa) derived from CAD/CAM restorative material/surface treatment combinations

	LDC (e.max CAD)	RNC (Lava Ultimate)	FGC (Mark II)	PICN (Enamic)
C (Control)	64.48 ± 10.3^{cA}	23.63 ± 9.0^{cB}	64.16 ± 11.6^{bA}	37.88 ± 17.6^{cB}
A (Acid Etching)	93.43 ± 20.7^{abA}	28.04 ± 8.9^{cB}	74.20 ± 5.7^{abA}	40.65 ± 14.2^{bB}
AS (Acid Etching + Universal Adhesive)	100.31 ± 10.7^{aA}	73.07 ± 10.8^{bB}	92.05 ± 15.7^{aAB}	84.00 ± 11.5^{aAB}
Sb (Sandblasting)	69.67 ± 8.7^{cB}	27.81 ± 7.7^{cC}	92.74 ± 13.4^{aA}	64.53 ± 18.7^{bB}
SbS (Sandblasting + Universal Adhesive)	74.57 ± 9.3^{bcB}	100.19 ± 19.7^{aA}	94.10 ± 12.3^{aAB}	98.06 ± 9.3^{aA}

Mean values represented with same superscript uppercase letters (row) or lowercase letters (column) are not significantly different according to Tukey-HSD multiple comparison test ($P < .001$).

Table 4. Distribution of failure types (adhesive/cohesive/mixed)

	C	A	AS	Sb	SbS
LDC (e.max CAD)	7/0/3	1/6/3	0/8/2	4/1/5	4/1/5
RNC (Lava Ultimate)	9/0/1	6/0/4	2/5/3	7/1/2	1/6/3
FGC (Mark II)	5/0/5	1/5/4	1/6/3	2/4/4	0/5/5
PICN (Enamic)	6/1/3	4/0/6	0/6/4	3/4/3	2/7/1

MPa) and A groups (93.43 ± 20.7 MPa) ($P > .05$) and these groups showed significantly higher FPBS results than the other groups ($P < .001$). Sandblasting and universal adhesive treatment (SbS) was the most effective pretreatment method for Lava Ultimate (100.19 ± 19.7 MPa). However, acid etching (28.04 ± 8.9 MPa) and sandblasting (27.81 ± 7.7 MPa) showed no significant differences from the control group (23.63 ± 9.0 MPa) and showed the lowest values for Lava Ultimate ($P > .05$). For Enamic, there was no significant difference in FPBS values between AS (84.00 ± 11.5 MPa) and SbS (98.06 ± 9.3 MPa) groups ($P > .05$). For Mark II, there was no significant difference among the different surface treatments ($P > .05$).

Failure pattern distribution of different CAD/CAM restorative materials and different surface treatments were represented in Table 4. Regarding the failure modes; according to Chi-square Test and Fisher's Exact Test statistical analyses, there was no significant interaction between the distribution of failure modes and CAD/CAM restorative materials ($P = .610$).

DISCUSSION

Based on the results, the null hypothesis that none of the material types and surface treatments would affect the bond strength of CAD/CAM restorative materials to resin cement was rejected.

Some studies have pointed out that most resin-ceramic bonding tests create non-uniform stress across the bonding area, thus affecting the reliability of results.[30] For example, shear tests generally tend to show cohesive failure due to the creation of inhomogeneous stress and its distribution across the bonding area.[17,31] Shear stress is also seen in three point bending strength tests. Three point bending test employs specimens that bend under compression load, promoting tensile stresses in the lower surfaces that are more likely related to the fracture initiation. In four point bending, there is no shear component between inner supports and there is no stress concentration near the points of loading. FPBS tests create a uniform tensile stress distribution across the bonding area.[32-34] In the current study, FPBS tests were used to evaluate the resin-ceramic bond strength.

Many studies have evaluated the bond strength between novel CAD/CAM restorative materials and different resin cements using different surface treatments,[8,16,17,23,27,35-37] yet there is no common view on whether the combination of mechanical and chemical surface treatments is more effective than mechanical pre-treatments alone on resin cement bonding with Lava Ultimate and Enamic. Elsaka revealed that, while HF acid etching or sandblasting in combination with a silane pre-treatment was more effective than mechanical surface treatment alone for Enamic, there were no significant differences among various surface treatments for Lava Ultimate.[23] Conversely, Peumans *et al.* confirmed that pre-treatments with silane increased bond strength values significantly for both Lava Ultimate and Enamic.[27] The discrepancy between Elsaka and Peuman. *et al* could be attributed to the different concentrations of HF acid gel used in the studies; while Elsaka used 9% HF acid gel, Peumans *et al.* used 5% HF acid gel. Frankenberger *et al.* also used 5% HF acid gel in their study to evaluate the adhesive luting of new CAD/CAM materials but found different results from Peumans *et al.*[16,27] Whereas Peumans *et al.* confirmed that pre-treatments with silane increased the adhesive luting of Lava Ultimate and Enamic significantly, Frankenberger *et al.* found no significant differences among the pre-treated groups with and without silane.[16,27] This discrepancy may be caused by the different types of chemical agents; Peuman *et al.* used 10-methacryloyloxydecyl dihydrogen phosphate (MDP).[27] Further, Cekic-Nagas *et al.* stated that resin cements containing MDP monomers have higher bond strengths than other adhesive cements, as indicated in a previous study.[36,38]

Scotchbond Universal Adhesive was used in the current study, and 9.5% HF acid etching and sandblasting in combination with the Scotchbond-treated groups of Lava Ultimate and Enamic showed significantly higher FPBS values than acid etching and sandblasting alone for both materials. Therefore, sandblasting did not affect the FPBS values of Lava Ultimate like HF acid etching in the current study. However, the group that applied Scotchbond after sandblasting showed higher FPBS values (100.19 ± 19.7 MPa) than those which applied Scotchbond after acid etching pretreatment (73.07 ± 10.8 MPa); the difference between the two groups was statistically significant. MDP containing agents like Scotchbond could have a contributory effect on bond strength of resin cements.[36] Sandblasting increased the FPBS values more when treated with Scotchbond. Therefore, for Lava Ultimate, sandblasting is preferable for HF acid etching when used with a universal adhesive. Likewise, acid etching pre-treatment is not suggested for Lava Ultimate by the manufacturer, possibly due to the zirconia nanomer filler in the RNC.

Cekic-Nagas *et al.* revealed that 10% HF acid gel treatment had no effect on bond strength between resin cement and CAD/CAM block composite material.[36] Similar to the results of the present study, they found that 9.5% HF gel had no effect on the FPBS values of Lava Ultimate or Enamic. However, for e.max CAD and Mark II, there was no significant difference in terms of the FPBS values. In addition, Aboushelib and Sleem evaluated the micro-tensile bond strength of e.max CAD to a resin adhesive and verified that 9.5% HF acid etching in combination with silane primer increased bond strength significantly.[35] HF acid etching in combination with Scotchbond treatment on e.max CAD had the highest FPBS value in the current study.

Most studies evaluating the bond strength between adhesive resin cement and feldspathic ceramic revealed that HF acid etching in combination with silane increases bond strength.[28,37,39] In the current study, the group acid etching in combination with Scotchbond (AS) showed higher FPBS value (92.05 ± 15.7) than the group A (74.20 ± 5.7) in the Mark II groups, but statistically no significant differences were found among the pre-treated Mark II groups. This can be attributed to Scotchbond, because Scotchbond is not a silane and may be insufficient to wet the surface of feldspathic ceramic by the resin cement.

Studies evaluating resin-ceramic bond strength are generally carried out with thermo-cycling or water storage to simulate oral conditions. While some studies have shown that aging processes destroy the effects of resin-ceramic bonding due to hydrolytic degradation, some have stated that there are no significant differences in bond strength before and after the aging process.[21,40] The current study was carried out in dry conditions, as the aim of this study was to evaluate early adhesive failure between resin cement and novel CAD/CAM restorative materials.

Each CAD/CAM restorative material is different from others in terms of its composition and properties. The properties of the restorative substrate used in *in vitro* bond strength tests can affect test results.[16] Two different ceramic materials and hybrid materials, which combine the properties of resin and ceramics, were used in the current study. The FPBS values obtained from the ceramic materials were significantly higher than the values obtained from the hybrid materials, similar to the results of Frankenberger *et al.*.[16]

Toledano *et al.*[41] reported that low bond strength test values were associated with high adhesive failures. In the current study, the control group with the lowest FPBS values regarding the surface treatment procedures was the group with the highest adhesive failure (67.5%). Mixed and cohesive failures were clinically more acceptable than adhesive failures.[41,42] The cohesive failure of the resin cement showed that the best possible bonding condition was achieved.[42] In the current study, the use of Scotchbond universal adhesive after mechanical surface treatment significantly increased both the FPBS values and the amount of cohesive failure (AS (62.5%) and SbS (47.5%)). There was no significant difference among the failure types observed in the material groups regardless of surface treatment (P = .610).

This current study only evaluated the FPBSs of four CAD/CAM restorative materials with four surface conditioning methods to one resin cement. Other bond strength test methods such as microtensile, microshear, or fatigue response of other CAD/CAM restorative materials should be investigated. Different surface conditioning methods and luting resin cements can affect the results of the bond strength of CAD/CAM restorative material to resin cement.

CONCLUSION

Considering the limitations of this study, the following conclusions may be drawn. The sandblasting or HF acid etching treatment in combination with a universal adhesive containing MDP increases the adhesive luting of Lava Ultimate and Enamic. The universal adhesive application after HF acid etching or sandblasting has a small advantage but does not significantly affect the adhesive luting of e.max CAD and Mark II. While HF acid etching is preferable to sandblasting for e.max CAD, sandblasting is preferable to HF acid etching for Mark II among micro-mechanical inter-locking treatments. The sandblasting or HF acid etching treatment in combination with a universal adhesive containing MDP can be suggested for the adhesive cementation of the novel CAD/CAM restorative materials.

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