



The effects of different surface treatments on the shear bond strengths of two dual-cure resin cements to CAD/CAM restorative materials

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PURPOSE. The aim of the present study was to investigate the effects of surface treatments on the bond strengths between polymer-containing restorative materials and two dual-cure resin cements. **MATERIALS AND METHODS.** In the present study, rectangular samples prepared from Lava Ultimate (LU) and Vita Enamic (VE) blocks were used. The specimen surfaces were treated using CoJet sandblasting, 50 µm Al₂O₃ sandblasting, % 9 HF (hydrofluoric) acid, ER,Cr:YSGG laser treatment, and Z-Prime. Dual-cure resin cements (TheraCem and 3M RelyX U 200) were applied on each specimen's treated surface. A micro-tensile device was used to evaluate shear bond strength. Statistical analysis was performed using the SAS 9.4v3. **RESULTS.** While the bond strength using TheraCem with LU or VE was not statistically significant ($P=.164$), the bond strength using U200 with VE was statistically significant ($P=.006$). In the TheraCem applied VE groups, Z-Prime and HF acid were statistically different from CoJet, Laser, and Sandblast groups. In comparison of TheraCem used LU group, there was a statistically significant difference between HF acid and other surface treatments. **CONCLUSION.** The bonding performance between the restorative materials and cements were material type-dependent and surface treatment had a large effect on the bond strength. Within the limitations of the study, the use of both U200 and TheraCem may be suggested if Z-prime was applied to intaglio surfaces of VE. The cementation of LU using TheraCem is suitable after HF acid conditioning of the restoration surfaces. [*J Adv Prosthodont 2020;12:189-96*]

KEYWORDS: Resin cement; Polymer; Computer-aided design and computer-aided manufacturing (CAD/CAM), Alkaline cement; Surface treatment

INTRODUCTION

In contemporary dentistry, almost all dental biomaterials that replace tooth substance loss focus on preserving the remaining tooth structure; this is called the minimally invasive technique.¹ The minimally invasive technique concept shows that the most important factor that retains the restorations in the prepared tooth is adhesive bonding.² Also, the

clinical success and good prognosis of indirect dental restorations largely depend on the satisfactory bonding between the adhesive interfaces to improve the adhesive features and prognosis of indirect restorative treatments. Strong bond strength may affect the durability of indirect restorative materials by distributing homogeneous forces between the remaining tooth structure and the adhesive interfaces and intaglio surfaces of the restoration. There are many studies comparing the surface treatments that affect the bond strength of traditional and reinforced ceramics with resin cements, and the findings have been comprehensively revealed by conducting various review and meta-analysis studies.^{3,4} However, the effect of various surface treatments of polymer-infiltrated ceramic-network (PICN) and resin nano ceramic materials (hybrid materials) on resin bond strength has not been investigated enough. Current studies on the subject have conflicting findings.⁵⁻⁷ Manso and Carvalho⁸ found that since resin-matrix ceramics are the latest to be introduced to the dental market, limited studies

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have been performed for the optimal clinical protocol regarding the surface treatments for ideal bonding and the results were also material-dependent.

Self-adhesive resin cements were created as practical and efficient solutions for the cementation of adhesive restorations. Most of the current dual-cure resin cements in the dental market have an acidic monomer in their structure.⁸ It has been found that residual acidic monomers may compromise the sufficient curing of the cement, causing unfavorable outcomes on the physical characteristics of the cement. Amount of remaining acidic monomers and the characteristics of the polar functional groups of resin cement have an important role in the different levels of sorption and solubility.^{9,10} Not only the number of acidic groups in the initial stage but also the remaining acidic groups after the setting reaction are important for water sorption and solubility of the resin cement.¹⁰⁻¹² Resin cements with a strong neutralization reaction for clinical conditions have been suggested because they may cause low hygroscopic expansion stresses.^{13,14} In this respect, it can be speculated that more alkaline dual-cure resin cement may exhibit more ideal physical properties than acidic resin cements. Thus, the aim of the present study to investigate the effects of surface treatments on the bond strengths between polymer containing restorative materials and two dual-cure resin cements; one of the cements is a recently introduced dual-cure resin cement that claims to have alkaline properties.

The null hypothesis was that various surface treatments have no effect on the bond strength to the hybrid restor-

ative materials and the bond strength value is not affected by the cement type.

MATERIALS AND METHODS

A conventional self-adhesive resin cement (Rely X U200, 3M ESPE Dental Products, St. Paul, MN, USA), a new generation alkaline dual-cure resin cement (TheraCem, BISCO Dental Products, Schaumburg, IL, USA), a resin ceramic material (Vita Enamic, Vita Zahnfabrik, Cuxhaven, Germany), and a nano-ceramic composite CAD/CAM material (Lava Ultimate, 3M ESPE Dental Products, St. Paul, MN, USA) were evaluated in the present study. Table 1 shows the materials investigated in the present study.

A total of 240 LU and VE specimens were cut transversely under water irrigation to get rectangular specimens with 2 mm thickness using a diamond disc (IsoMed 1000, Buehler, Lake Bluff, IL, USA). The specimens that had more than 0.05 mm variation from the 2 mm specimen thickness after the measurement via a digital caliper (Surftest SJ 201, Mitutoyo, Tokyo, Japan) were excluded from the study, and a new specimen was prepared. After wet ground, the specimens were placed in a polyvinylchloride cylinder mold, and the mold was filled with auto polymerizing acrylic resin (Paladent, Heraeus Kulzer GmbH & Co. KG, Wehrheim, Germany). One surface of all samples was processed using 600 grit silicon carbide paper (3M ESPE, St. Paul, MN, USA). All specimens were cleaned ultrasonically for 4 minutes. Both LU (n = 120) and VE

Table 1. Materials used in the present study

| Material | Type | Manufacturer | Lot no. | Composition |
|---|---|---|---------|--|
| Vita Enamic | Polymer infiltrating ceramic network (PICN) | Vita Zahnfabrik, Cuxhaven, Germany | 41470 | 86 wt% feldspar ceramic, 14 wt% polymer |
| Lava Ultimate | Nano-ceramic composite (NCC) | 3M ESPE Dental Products, St. Paul, MN, USA | 613657 | 80 wt% nanoceramic, 20 wt% resin |
| 3M RelyX U 200 | Self-adhesive resin cement | 3M ESPE Dental Products, St. Paul, MN, USA | 714236 | Matrix: hydroxyethylmethacrylate (HEMA), BisGMA Fillers: fluoroaluminosilicate glass, zirconia silica |
| Theracem | Alkaline dual cure resin cement | Bisco Dental Products, Schaumburg, IL, USA | 43275 | 10-methacryloyloxydecyl dihydrogen phosphate |
| Ultradent Porcelain Etch (HF etchant) | Hydrofluoric acid | Ultradent Porcelain Etch, South Jordan, UT, USA | BCB97 | 9% hydrofluoric acid |
| Paladent | Auto polymerizing acrylic | Heraeus Kulzer GmbH & Co. KG, Wehrheim, Germany | 13168 | Powder: polymethyl methacrylate Liquid: methyl methacrylate, N,N dimethyl p-toluidine |
| CoJet Sand | - | 3M ESPE Dental Products, St. Paul, MN, USA | 124563 | 30 µm Al ₂ O ₃ grains modified by silicon dioxide particles |
| Al ₂ O ₃ sandblasting | - | Bego GmbH | | 50 mm Al ₂ O ₃ particles |
| Z-Prime™ Plus | Zirconia - Alumina - Metal Primer | Bisco Dental Products, Schaumburg, IL, USA | 57354 | Bisphenol dimethacrylate/ hydroxyethyl methacrylate/ethanol |

specimens (n = 120) were grouped randomly in four groups (n = 60) for the two resin cements. Each of the four CAD/CAM groups was further subdivided into 24 subgroups (n = 10) according to the resin cement and surface treatment method. The grouping of the specimens is demonstrated in Fig. 1. Surface treatments applied to the specimens are described below:

Control: No surface treatment applied.

CoJet: All the specimens were treated by CoJet system (3M ESPE, St. Paul, MN, USA) that spewed out of 30 µm Al₂O₃ grains modified by silicon dioxide particles (CoJet Sand, 3M ESPE, St. Paul, MN, USA). The treatment was carried out perpendicularly for 15 seconds at a distance of 10 mm to the sample surface. Samples were ultrasonically cleaned and air dried.

Sandblast: Samples were sandblasted with 50 µm Al₂O₃ using an air abrasion device (Ney, Blastmate II, Yucaipa, CA, USA). The process was carried out at a distance of 10 mm for 15 seconds with a pressure of 2 bar. A holder was used to fix the distance between the device and the sample and ensure that the particles were perpendicular to the surface. Samples were ultrasonically cleaned and air dried.

HF acid: The etching was performed with 9% HF acid (Ultradent Products, South Jordan, UT, USA). After 1 minute of application, HF acid was removed using plenty of water. Air drying was applied to the sample surfaces.

Z-Prime: Z-Prime Plus (Bisco Dental Products, Schaumburg, IL, USA) was applied to the sample surfaces using a micro brush. In applying, the manufacturer's instructions were taken into account.

Laser: ER,Cr:YSGG laser (Waterlase iPlus; Biolase

Technology Inc., Irvine, CA, USA) with 2780 nm wavelength was applied to the samples, with 10 Hz repetition rate, pulse duration of 140 - 200 milliseconds, 71 J/cm² energy density, and 2 W power. A laser optic fiber with 600 µm diameter was set perpendicular to the CAD/CAM sample surface. The treatment was performed on the sample surface area from a distance of 1 mm for 20 seconds. The water flow was 65% and the air flow was 55%. After rinsing with distilled water, the samples were air-dried.

Scanning electron microscope (SEM) (Zeiss-Leo 1430 SEM; Angstrom Scientific Inc., Ramsey, NJ, USA) was used to observe the surfaces after surface treatments. Existing samples could not be used in this process. The reason for this was that the acrylic blocks, where the samples were embedded, were not suitable for electron microscope examination. For the SEM images, the above-mentioned sample preparation steps were applied exactly and the samples were prepared to be suitable for examination in the electron microscope where the acrylic block was thinner. Samples covered with gold and palladium layer (Polaron SC7620 Sputter Coater; VG Microtech, West Sussex, UK). After finishing the surface treatments of all specimens, SEM images of example specimen were obtained. Secondary electron emission detector has been used for surface characterization.

The dual-cured resin cements used in the present study were applied on the treated surface of each CAD/CAM materials with a 1 mm diameter and 2 mm length teflon mold. The teflon mold was applied to the center of each of the VE and LU specimens. An LED device (Ultradent Products, South Jordan, UT, USA) with 395 - 480 nm light intensity and 1000 mW/cm² power was used for 40 s to res-

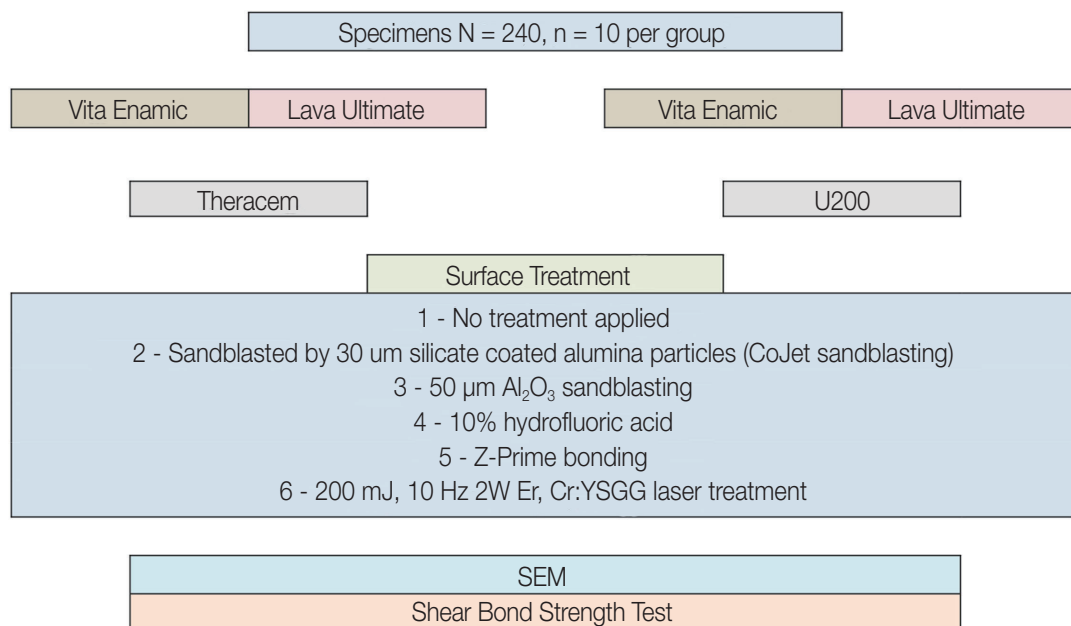


Fig. 1. Schematic representation of the study.

in cement light curing. The specimens were kept for 24 h in the water at 37°C after the completion of the resin cement polymerization procedure. The bond strength test was performed by applying force at a crosshead speed of 0.5 mm/min perpendicular to the sample surface through a micro tensile device (Esetron Mekatronik, Ankara, Turkey). The maximum load that caused the failure and debonding between the bonding interfaces for each of the specimens

was recorded in Newtons. The determination of the bonding failure data in megapascals (MPa) was performed by dividing the maximum force applied before bonding by the total area of the resin cement (Fig. 2).

Evaluation of failure modes was performed by a researcher at 40× magnification via stereomicroscope (DV4; Stemi, Göttingen, Germany). Three different failure modes were determined: cohesive (failure in restorative material), adhesive (failure in resin cement) mixed (failure between restorative material and resin cement). SAS software (SAS 9.4v3, Cary, NC, USA) was used to perform statistical analysis. A P value of < .05 was considered to be statistically significant.

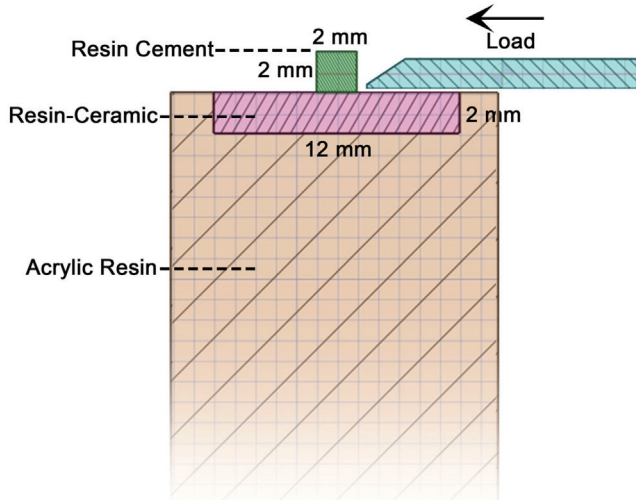


Fig. 2. Schematic diagram of shear bond strength test.

RESULTS

Three-way ANOVA with Duncan’s multiple comparison test and Bonferroni’s correction pairwise test were used to analyze the data. First, Levene’s test was used to assess the homogeneity of the variances, which is a precondition for parametric tests, while the Shapiro-Wilk test was used to check the assumption of normality. The assumption of homogeneity of variance was violated. Therefore, data were log-transformed to achieve the assumption of the parametric analysis. The non-transformed data means are presented in Table 2. Table 3 shows the failure mode types of samples. In general, the most failure type was the mixed type, but an increase was observed in cohesive type in HF acid and

Table 2. Mean and the standard deviations of the material and combinations with different treatments

| | TheraCem | | U200 | |
|--------------|---------------------------|---------------------------|---------------------------|---------------------------|
| | VE | LU | VE | LU |
| Control | 8.3 ± 2.6 ^{Ba} | 7.7 ± 2.0 ^{Ca} | 9.8 ± 3.43 ^{Ba} | 9.4 ± 2.9 ^{Ba} |
| CoJet | 13.6 ± 8.8 ^{Ba} | 12.2 ± 3.1 ^{Ca} | 9.5 ± 1.88 ^{Ba} | 12.9 ± 4.8 ^{Ba} |
| Sandblasting | 13.5 ± 4.0 ^{Bab} | 10.8 ± 2.8 ^{Cb} | 15.0 ± 3.8 ^{Ba} | 12.0 ± 4.0 ^{Bab} |
| HF acid | 19.9 ± 6.1 ^{Aa} | 23.3 ± 4.3 ^{Aa} | 14.5 ± 4.4 ^{Bb} | 12.5 ± 6.0 ^{Bb} |
| Z-Prime | 21.0 ± 4.8 ^{Aa} | 16.2 ± 5.0 ^{Bbc} | 20.1 ± 8.0 ^{Aab} | 14.7 ± 4.3 ^{Ac} |
| Laser | 12.5 ± 3.7 ^{Ba} | 10.9 ± 2.4 ^{Cab} | 13.5 ± 5.1 ^{Ba} | 9.1 ± 5.4 ^{Bb} |

**For each column values with same upper-case letters indicate no statistically significant differences (P > .05)

**For each row values with same lower-case letters indicate no statistically significant differences (P > .05)

Table 3. The failure mode distribution of specimens after the bond strength test

| | TheraCem | | | U200 | | |
|-----------|------------------|------------------|---------------|------------------|------------------|---------------|
| | Cohesive (VE/LU) | Adhesive (VE/LU) | Mixed (VE/LU) | Cohesive (VE/LU) | Adhesive (VE/LU) | Mixed (VE/LU) |
| Control | 0/0 | 1/0 | 9/10 | 2/1 | 1/2 | 7/7 |
| CoJet | 1/0 | 1/1 | 8/9 | 2/1 | 1/2 | 7/7 |
| Sandblast | 0/0 | 2/1 | 8/9 | 1/1 | 1/1 | 8/8 |
| HF acid | 4/3 | 2/3 | 4/4 | 0/0 | 1/0 | 9/10 |
| Z-Prime | 4/2 | 2/1 | 4/7 | 4/2 | 2/2 | 4/6 |
| Laser | 0/0 | 1/1 | 9/9 | 1/1 | 0/0 | 9/9 |

Zprime applied groups.

The main effects of the resin cement ($P = .02$), restorative material ($P = .003$), and the surface treatment ($P < .0001$) were found to be statistically significant according to variance analysis. Resin cements and surface treatments interaction effects were statistically significant. Resin cements and surface treatments interaction effects were statistically significant ($P < .0001$). Two-way interaction between the restorative material and the surface treatment was also statistically significant. ($P = .007$). However, the restorative material and resin cement interaction effect ($P = .33$) and the three-way interaction effect ($P = .17$) were not statistically significant. Table 2 shows the mean values and standard deviations.

The bond strength using TheraCem with LU or VE was not statistically significant ($P = .164$). On the other hand,

using U200 with LU and VE was statistically significant ($P = .006$). In the TheraCem applied VE groups, Z-Prime and HF acid were statistically different from CoJet sandblasting, Laser, and Sandblast groups. Comparison of TheraCem used LU groups showed that the bond strength values of HF acid and other surface treatments were statistically significant. In the U200 applied VE or LU groups, a statistically significant difference was observed between Z-Prime groups and the other surface treatments in both VE and LU.

SEM images showing the effects of surface treatments were given in Fig. 3. It was seen that all surface treatments caused changes on the surface topography of the samples compared to the control group. Different surface treatments caused different effects on the surface of the samples. When SEM images of CoJet sandblasted, Al_2O_3 sand-

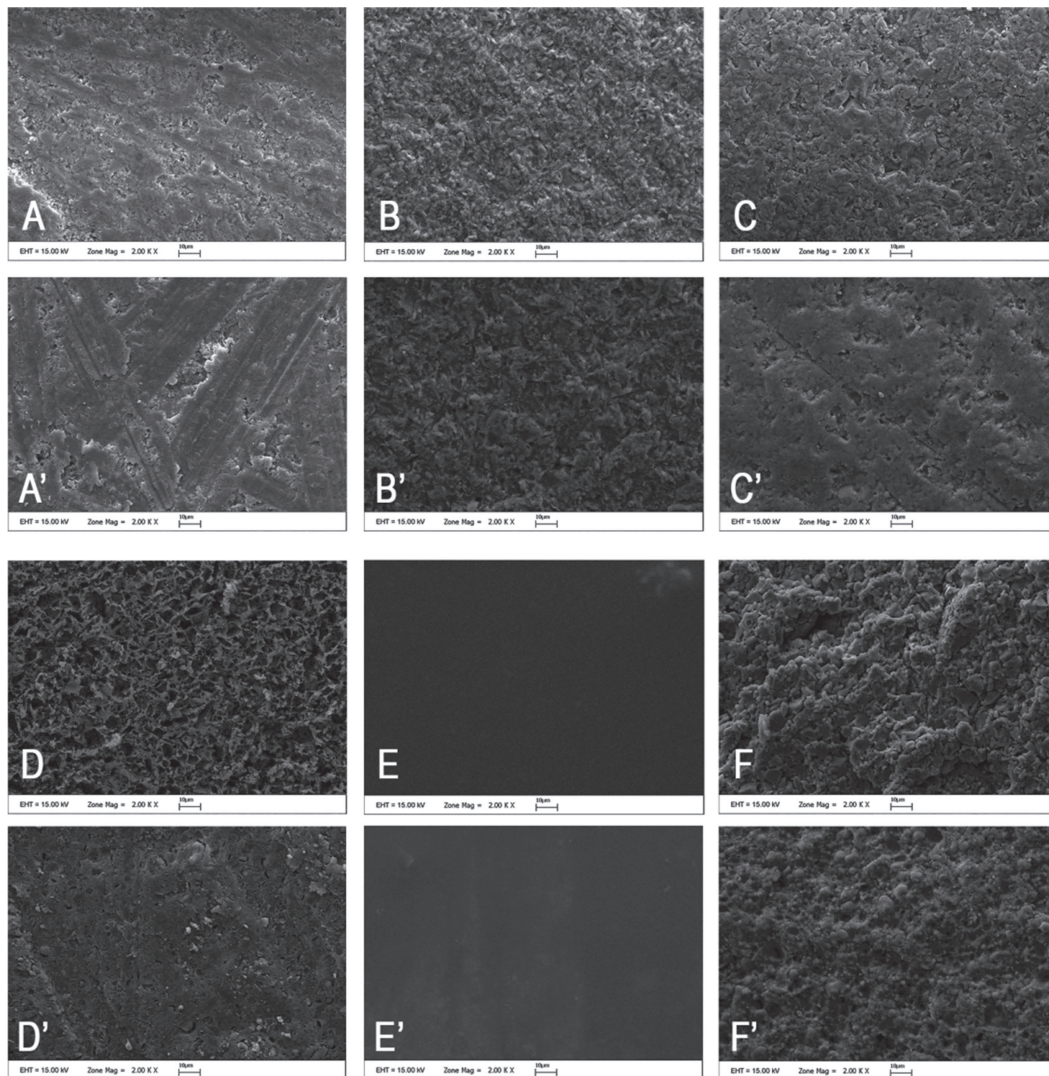


Fig. 3. Representative SEM photomicrographs of treated VE and LU surfaces (original magnification: 2000 \times). Capital letters indicate VE examples, capital letters with quotation marks indicate LU examples. (A,A') Untreated specimens -control group, (B,B') CoJet sandblasted specimens, (C,C') Al_2O_3 sandblasted specimens, (D,D') HF acid etched specimens, (E,E') Z-Prime applied specimens, (F,F') Laser applied specimens.

blasted, and HF acid-etched samples were examined, it was seen that VE sample surfaces had more frequent and deeper pits than LU sample surfaces.

DISCUSSION

Bond strength values showed a statistically significant change after the application of various surface treatments to VE and LU surfaces. The cement types had effects on the bond strength values and the surface treatment methods affected the results. The results were statistically significant. Thus, the study's null hypothesis was rejected.

In the dental literature, few studies compared the alkaline resin cements and cements releasing more acidic monomers. Chen *et al.*¹⁵ compared the bond strength of alkaline resin cement (TheraCem), a resin-modified glass ionomer cement (FujiCem 2), and a self-adhesive resin cement (UniCem 2) to zirconia. In contrast to the present study, Chen *et al.* showed a stronger bond strength with TheraCem. However, Chen *et al.*¹⁵ evaluated zirconia in their study, whereas the present study examined polymer-based materials. Ihsan and Mohammed¹⁶ evaluated the bond strength between orthodontic brackets and cements, and found no significant difference between TheraCem and a traditional orthodontic bonding system (Transbond™ XT Primer/Transbond™ XT composite resin). The difference among the bond strength values of different studies may originate from the cement types, methods, and bonded surface differences. In the limits of the present study, the alkaline and other self-adhesive resin cement demonstrated similar bond strengths with two different CAD/CAM materials except in the hydrofluoric acid groups. Alkaline cement (TheraCem) showed better values than the other cement (U200) in the HF acid applied groups.

Many studies have been conducted comparing the effects of surface treatments on bond strength. Altan *et al.*¹⁷ reported that Al₂O₃ sandblasting and CoJet application is more effective on monolithic zirconia than Y-TZP zirconia block in terms of bond strength. In the same study, HF acid application on Vita Suprinity was shown to be more effective than Al₂O₃ sandblasting and CoJet methods. This can be regarded as an indication that the effect of the surface treatment on the bond strength is directly related to the type and content of the material. In the present study, the most effective methods for the bond strength were HF acid and Z-Prime with Theracem. In Altan *et al.*'s study, HF acid was also more effective in Vita Suprinity than other methods similar to VE in the present study. It may be explained by the higher ceramic contents of Vita Suprinity and VE among the studied materials in both studies since HF acid application is still a gold standard for glass ceramic surface treatment. In U200 groups, the most effective surface treatment method was Z-Prime and not HF acid. It may be explained by the different chemical structures of Theracem and U200 (the alkaline features of Theracem). Çevik *et al.*¹⁸ reported that HF acid application was quite ineffective in the treatment of feldspathic porcelain surface compared to

Al₂O₃ sandblasting. In the present study, HF acid application significantly increased the bond strength in the TheraCem group compared to Sandblasting, but this difference was not observed in the U200 group. This shows that the effect of material type-surface treatment-resin adhesive type relationship on bond strength can be more complex. Different resin adhesives can perform differently in similar surface treatments. The complex chemical structures and different effect of their constituents of cements should be evaluated in further studies.

A comprehensive review and meta-analysis study was carried out about the effects of laser use on bond strength of ceramic materials.⁴ The findings of García-Sanz *et al.* showed that all laser applications in the literature including Er, Cr: YSGG laser significantly increased the bond strength compared to the control group. In the same study, laser applications were compared with Al₂O₃ sandblasting and it was reported that the findings were quite heterogeneous.⁴ In the present study, when the control, laser, and Al₂O₃ sandblasting groups were compared, the average values in all laser applied groups were higher than the control groups except the U200-LU group. This may be explained by the similar type effects of lasers in ceramics. In the present study, there was no statistically significant difference among Al₂O₃ sandblasting, laser, and control groups. The findings of the study are remarkable in this aspect and are attributed to difference of restorative materials among the studies.

In a study in which the effects of surface treatments on bond strength of two different hybrid blocks (LU and Cerasmart (Cerasmart GC, Tokyo, Japan) with composite resin were compared, Er, Cr: YSGG laser application and sandblasting provided the highest bond value. No difference was observed between the bond strength values of hybrid blocks.⁷ In the present study, results showed that while HF acid affected the bond strength more than sandblasting in TheraCem groups, the results were similar in the HF and sandblasted groups in the U200 groups.

Helbling and Özcan⁵ applied HF acid and CoJet to the surface of various ceramic materials, including LU and VE, and compared the effects of these applications on the bond strength to resin cement. When thermal aging was not applied, the binding values were higher in the VE group compared to the LU group, and similar results were obtained for both groups when thermal aging was applied. No difference was observed between HF acid and CoJet applications.⁵ In the present study, a difference was statistically significant between HF acid and CoJet applied groups. The use of HF acid increased bond strength more than the CoJet application.

Various studies have shown the effectiveness of HF acid. Kursoğlu *et al.*¹⁹ compared the effects of Er, Cr: YSGG laser application and HF acid etching on shear bond strength values between lithium disilicate ceramic and resin cement. The laser application (1.5 and 2.5 W) caused higher bond strength values compared to the control group. HF acid application resulted in the highest bond strength val-

ues.¹⁹ Motevasselian *et al.*⁶ tested the effectiveness of Er: YAG laser, HF acid, Al₂O₃ sandblasting, and phosphoric acid applications. The highest bond strength values between VE-resin cement were observed in the HF acid group, while the lowest bond strength values were observed in the Er: YAG laser group. Frankenbergera *et al.*²⁰ suggested that HF acid application for VE and sandblasting for LU to treat the intaglio restoration surfaces. Güngör *et al.*²¹ supported similar results with Frankenbergera *et al.*²⁰ in their surface roughness studies. Also, in the studies that evaluated both HF acid etching and sandblasting, it was shown that both methods strengthened the bond strength.^{20,22,23} In the present study, contrary to the past studies, a Zirconia-Alumina-Metal primer (Z-Prime™ Plus) was used as the surface agent. Z-Prime caused the best bonding performance in all the groups except the LU groups cemented with TheraCem. The increase in failure mode rates in favor of adhesive type in Zprime groups supports this finding. According to these results, Z-Prime may be a good choice for both LU and VE since better bonding performances were observed for VE and LU with both TheraCem and U200. The results of the present study also imply that U200 may be a better choice than TheraCem if the surface treatment of VE is performed using HF acid. Further studies are needed that evaluate HF and Z-Prime together.

The was no effect of the CAD/CAM materials in these results. In the present study, HF caused a better bonding performance in the TheraCem groups. Elsaka²³ also showed that HF generated a better bonding performance than sandblasting in both LU and VE. In all the above-mentioned studies, the contents and applying procedures of HF acid were the same. However, the sandblasting period, the distance of the sandblasting probe to the specimen, and the type of sand may affect the results. Elsaka²³ generated two different groups, HF acid and sandblasting; saline was then applied to both groups. In the study of Elsaka,²³ the best surface treatment results for VE were obtained with HF + silane and sandblasting + silane. In future studies, the effects of salinization added to the surface treatment and the alkaline content of cement on the bond strength should be evaluated.

Differences between the groups in the present study imply that the bond strengths of the two resin cement to CAD/CAM material depend on the surface treatment method. This could be attributed to the different structures and constituents of the CAD/CAM blocks and resin cements, and also the material based effects of surface treatments on these materials. Above-mentioned reasons for the strong or weak bond strength were also stated in previous studies.

It has been shown that the physical behavior of the cements depends on the contact condition and period in the oral cavity. In a study with adhesive resin cement, bond strength to VE and LU were affected by the period of exposure to water.²³ Thermal aging procedures have effects on the different restorative materials and cements in different ways.²² Inadequate simulation of the oral cavity was a

limitation of the present study. The best alternative to *in vivo* material studies is clinical investigations. Thus, new *in vivo* studies are necessary that evaluate commonly used resin cements and alkaline resin cements in the future.

CONCLUSION

The results imply that the bonding performance between the restorative materials and cements are material type-dependent and surface treatment has a large effect on the bond strength. Thus, selecting the proper surface treatment and bonding agent according to the restorative material is important for successful bond strength between the adhesive interfaces. Within the limitations of the present study, the use of both U200 and TheraCem can be suggested if Z-prime is applied to intaglio surfaces of VE. If the intaglio surface conditioning is performed using HF acid, TheraCem should be preferred instead of U200. According to the results, the cementation of LU using TheraCem is suitable after HF acid conditioning of the restoration surfaces.

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