Effect of various surface treatments on the bond strength of resin luting agent and the surface roughness and surface energy of CAD/CAM materials

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The aim of this study was to examine the effect of various surface treatments on the bond strength of a resin luting agent and the surface roughness and surface energy of computer-aided design and computer-aided manufacturing (CAD/CAM) materials. Four types of CAD/CAM blocks (Shofu Block HC: BHC; GC Cerasmart: CER; VITA Enamic: ENA; and Lava Ultimate: LAV) were used. All blocks were randomly divided into eight groups based on the surface treatment as follows: no surface treatment (C), airborne-particle abrasion (AA), hydrofluoric acid etching (HA), silane coupling agent application (SL), AA/SL, HA/SL, AA/HA, and AA/HA/SL. The microtensile bond strength (µTBS), surface roughness and surface energy were measured. Three-way ANOVA revealed that all surface treatments significantly influenced the µTBS between the resin luting agent and all types of CAD/CAM materials; however, the effect of each surface treatment on surface roughness and energy was dependent on the CAD/CAM materials.

Keywords: CAD/CAM material, Resin luting agent, Microtensile bond strength, Surface roughness, Surface energy

INTRODUCTION

In recent years, the features of ceramics and resin composites have been combined to produce a resin-ceramic computer-aided design and computer-aided manufacturing (CAD/CAM) material10. A resin-ceramic material is composed of 60–86% refractory inorganic particles by weight, irrespective of the presence of a predominant organic phase2,3. Due to the presence of the organic phase, the elastic modulus of this material is similar to that of dentin, which is easier to mill and adjust than glass-matrix or polycrystalline ceramics5. Recently, resin-ceramic materials were classified into three categories according to the types of ceramic particles: resin nanoceramics (e.g., Lava ultimate (LAV), 3M ESPE, St. Paul, MN, USA; Cerasmart (CER), GC, Tokyo, Japan), glass ceramics (e.g., Enamic (ENA), Vita Zahndfabrik, Bad Säckingen, Germany), and zirconia-silica ceramics (e.g., Shofu Block HC (BHC), Shofu, Kyoto, Japan)9. Thus, the composition of CAD/CAM blocks varies and may influence the morphology of blocks subjected to physical surface treatment, such as airborne-particle abrasion (AA) and hydrofluoric acid etching (HA).

Surface treatments of resin-ceramic materials have been tested in many studies. AA, HA and silane coupling agent application (SL) are the main treatments that have been used to improve the bond strength of resin-ceramic materials to resin luting agents5-9. However, few studies have investigated the effect of the composition, surface roughness and surface energy of CAD/CAM blocks on the bond strength between CAD/CAM materials and resin luting agents after various surface treatments. Furthermore, surface treatment using a combination of AA and HA has not been reported in previous studies in which surface treatments of CAD/CAM materials were investigated.

Manufacturers have developed appropriate surface treatment protocols for their own resin-ceramic materials. AA, HA and SL have been specified for use in these instructions10-13. AA roughens the surface of resin-ceramic materials to increase adhesion area and surface energy5,14. HA dissolves the silica-based matrix to create microporous particles on the surface of a resin-ceramic material; as a result, the surface energy and wettability increase5,14. Both AA and HA possess the ability to mechanically increase the bond strength between the resin-ceramic material and resin luting agent. SL provides a chemical bond between the adhesive resin and the matrix of silica-based material15. The effect of these surface treatments on bond strength may be related to the composition of CAD/CAM blocks because of the difference in morphology after each surface treatment.

AA followed by HA may be effective for the adhesion of resin-ceramic CAD/CAM materials to resin luting agents. The surface area of the resin-ceramic material, which consists of an organic and an inorganic substance, could be remarkably increased by AA followed by HA. Thus, the increased adhesion area on the resin-ceramic material after these treatments may improve its bond strength to the resin luting agent when applying SL. The morphological alteration of each CAD/CAM block after this surface treatment combination may vary, which may affect the bond strength between the CAD/CAM block and the resin luting agent. However, few studies have investigated the surface roughness and energy of CAD/CAM blocks after various surface treatments. Furthermore, the relationship between...
surface roughness, surface energy, and bond strength of resin luting agents to CAD/CAM materials has not yet been clarified.

The aim of this study was to examine the effect of various surface treatments on CAD/CAM blocks in terms of the bond strength of the resin luting agent and the surface roughness and surface energy of the CAD/CAM materials. The null hypotheses were that the various surface treatments would not influence the bond strength of the resin luting agent or the surface roughness and surface energy of the CAD/CAM materials.

**MATERIALS AND METHODS**

The composition and filler contents of the materials used in this study are shown in Table 1. Figure 1 shows a schematic of the procedure used to produce the specimens (rectangular microbars) for microtensile bond strength (µTBS) testing.

**Plate preparation**

For each material, 3 CAD/CAM blocks (12×14×18 mm) were used. Each CAD/CAM block was cut into 4 plates with dimensions of 12×14×4.5 mm using a diamond wafering blade and a linear precision saw (Isomet, 4000, Buehler, Lake bluff, IL, USA) under water coolant. The surfaces of the plates were ground with abrasive silicon carbide papers (CarbiMet, 600 grit, Buehler) with a water coolant. The surface of each plate was divided into 4 equal sections with two rectangular shallow grooves.

### Table 1  Materials used in this study

| Material         | Code | Category                                                                 | Composition                                                                 | Filler content (wt%) | Lot number | Manufacturer               |
|------------------|------|---------------------------------------------------------------------------|-----------------------------------------------------------------------------|----------------------|------------|---------------------------|
| Block HC         | BHC  | Zirconia-silica ceramic in a resin-interpenetrating matrix                | UDMA, TEGDMA, silica powder, micro fume silicate, zirconium silicate          | 61                   | 1217850    | SHOFU, Kyoto, Japan        |
| Cerasmart        | CER  | Resin nanoceramic                                                        | Bis-MEPP, UDMA, DMA, silica, barium glass                                    | 71                   | 1812041    | GC, Tokyo, Japan           |
| Enamic           | ENA  | Glass ceramic in a resin-interpenetrating matrix                         | UDMA, TEGDMA, SiO₂, Al₂O₃, Na₂O, K₂O, B₂O₃, ZrO₂, CaO                       | 86                   | 63460      | Vita Zahnfabrik, Bad Säckingen, Germany |
| Lava ultimate    | LAV  | Resin nanoceramic                                                        | Bis-GMA, UDMA, Bis-EMA, TEGDMA, SiO₂, ZrO₅, aggregated SiO₂/ZrO₂ cluster    | 80                   | N953902    | 3M ESPE, St Paul, MN, USA  |
| Panania V5       | —    | Dual-cured self-adhesive resin luting agent                              | Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, initiators, accelerators, silanated barium glass filler, silanated fluoroaluminosilicate glass filler, colloidal silica, silanated aluminum oxide filler, dl camphorquinone, pigments | —                   | CD0068     | Kuraray Noritake, Tokyo, Japan |
| Jet blast II     | —    | Aluminum oxide                                                          | Al₂O₃ (50 µm)                                                               | —                    | —          | Morita, Tokyo, Japan       |
| IPS ceramic      | —    | Hydrofluoric acid                                                        | 4.5% hydrofluoric acid                                                      | —                    | V37045     | Ivoclar Vivadent, Schaan, Liechtenstein |
| Clearfil         | —    | Silane coupling agent                                                   | 3-trimethoxysilylpropyl methacrylate, MDP, ethanol                           | —                    | 7D0028     | Kuraray Noritake           |

Bis-GMA: bisphenol-A-diglycidyl methacrylate; UDMA: urethane dimethacrylate; Bis-EMA: ethoxylated bisphenol-A dimethacrylate; TEGDMA: triethyleneglycol dimethacrylate; Bis-MEPP: 2,2-bis-(4-methacyrloxypropyloxyphenyl) propane; DMA: dodecyl dimethacrylate; MDP: 10-methacryloyloxydecyl dihydrogen phosphate
using a diamond wafering blade and a linear precision saw. All plates were cleaned using an ultrasonic cleaner (1510J-MT, Bransonic, Danbury, CT, USA) for 5 min to remove debris and contaminants and then were dried with an air syringe. In total, 12 plates were prepared for each resin-ceramic CAD/CAM material.

Surface treatments
Half of the plates in each resin-ceramic material were treated with airborne particles (50 µm Al₂O₃ particles) using a sand-blasting machine (Jet blast II, Morita, Tokyo, Japan). After a razor blade was positioned at the groove to shield the other side of the plate surface, half of the plate surface was etched with hydrofluoric acid. Subsequently, a razor was perpendicularly positioned on the plate, and silane coupling agent was applied to half of the plate surface. Accordingly, 8 surfaces on 2 plates of each resin-ceramic material were treated using the different protocols as follows:

1) Absence of any surface treatments, i.e., control (C).
2) Abrasion with airborne particles for 20 s, followed by rinsing and cleaning with an ultrasonic cleaner (AA). The pressure of the abrasion differs for each material type according to the manufacturer’s instructions.
3) Etching with a 4.5% hydrofluoric acid gel for 60 s, followed by rinsing and cleaning with an ultrasonic cleaner (HA).
4) Applying a thin coat of silane coupling agent for 60 s, followed by air-drying for 60 s (SL).
5) AA followed by SL (AA/SL).
6) HA followed by SL (HA/SL).
7) AA followed by HA (AA/HA).
8) AA followed by HA and SL (AA/HA/SL).

Luting procedures
Two ceramic plates were bonded using a self-adhesive resin luting agent, and the areas with the same surface treatment were aligned opposing each other; during bonding, the plates were subjected to a constant load of 1 kg. After loading for 2 min, all four sides of the sandwiched plate were light-cured for 40 s using a light-emitting diode (LED) curing unit (Pencure, 2000, Morita) to photopolymerize the resin luting agent. After loading for 10 min, the load was removed, and all four sides of the sandwiched plate were further light-cured for 60 s. The sandwiched plates were stored in distilled water at 37°C for 24 h prior to the preparation of rectangular microbars.

µTBS test
Six sandwiched plates of each CAD/CAM material were cut into rectangular microbars (1×1×8 mm) using a diamond wafering blade and a linear precision saw under water coolant. Forty-eight microbars were obtained for each sandwiched plate and stored in distilled water at 37°C for 6 days. Twenty-eight outer-positioned microbars were discarded. Eighteen microbars were obtained for each group from 3 sandwiched plates. The widths of the microbars were measured before the µTBS test was performed. Microbars with widths of less than 0.95 mm or more than 1.05 mm were excluded from this study. A total of 15 microbars were selected for each group (n=15). The microbars were attached to the testing jig (Bencor-multi-T, Danville Engineering, San Ramon, CA, USA) using cyanoacrylate (Model Repair Pink, Dentsply Sankin, Tochigi, Japan). The µTBS was measured at a 1.0 mm/min crosshead speed using a tabletop material tester (EZ Test 500N, Shimadzu, Kyoto, Japan).

Failure mode analysis
The fracture surfaces of the microbars were observed using a stereomicroscope (Leica EZ4D, Microsystems CMS, Wetzlar, Germany) at 35× magnification to determine the failure mode based on the criteria for classification as shown in Table 2.

Scanning electron microscopy (SEM) observation
The surfaces of the resin-ceramic materials with each
treatment were sputter-coated with palladium and platinum and observed using an acceleration voltage of 15 kV at 5,000× magnification. The representative debonded surfaces of the microbars were sputter-coated and observed at 70× magnification (S-800, Hitachi, Tokyo, Japan).

Measurement of surface roughness
The CAD/CAM blocks were cut into specimens with dimensions of 4×5×2 mm and treated with the same protocol as that used for the µTBS test. The surface topography of the specimens with each surface treatment was analyzed using a profilometer (Surfcom, S96A, Tokyo Seimitsu, Tokyo, Japan). To evaluate the surface roughness, the arithmetic mean deviation of the roughness profile (Ra) was calculated at a tracing speed of 0.3 mm/s and a cut-off speed of 0.8 mm/s.

Measurement of surface energy
Specimens from the surface roughness measurements were investigated for surface energy using the sessile drop technique. The contact angles were measured with a contact angle meter (Face, Kyowa interface Science, Saitama, Japan). For every specimen, the contact angle was measured with H2O and diiodomethane and calculated by the model of Owens-Wendt16.

Statistical analysis
The data from each CAD/CAM material were statistically analyzed using three-way ANOVA to determine the effect of three surface treatments on the bond strength between the resin luting agent and the CAD/CAM materials, the surface roughness, and the surface energy at a significance level of 0.05. Statistical comparison of the bond strength across 8 experimental groups was performed through one-way ANOVA at a significance level of 0.05. Failure modes were analyzed by Fisher’s exact test, which was performed among 8 surface treatments within each material, at a significance level of 0.05.

RESULTS
The µTBS test results, surface roughness measurements and surface energy measurements for each CAD/CAM material are shown in Tables 3, 4 and 5, respectively.

µTBS of the resin luting agent to the CAD/CAM materials
The results of three-way ANOVA are as follows. AA, HA, and SL significantly influenced the µTBS between the resin luting agent and all types of CAD/CAM materials (p<0.01). The effect of the interaction between AA and HA was detected in the µTBS of the resin luting agent to BHC and LAV (p<0.02). The effect of the interaction between HA and SL was detected in the µTBS of the resin luting agent to BHC (p=0.013). The effect of the interaction between AA and SL was detected in the µTBS of the resin luting agent to CER and ENA (p<0.01). The effect of the interaction among AA, HA and SL was detected in the µTBS of the resin luting agent to LAV (p=0.001).

The results of one-way ANOVA were as follows. For BHC, the µTBS of the SL group was significantly lower than that of the other groups except C (p<0.01). There were no significant differences in µTBS between AA/HA and AA/HA/SL, between AA and AA/SL, and among AA, HA, SL and AA/HA/SL (p>0.05). For CER, there were no significant differences in µTBS among AA, HA, SL and AA/SL, and among AA/SL, HA/SL, AA/HA and AA/HA/SL (p>0.05). For ENA, the µTBS of AA/HA/SL was significantly higher than that of the other groups (p<0.01). The µTBS of AA was significantly lower than that of the other groups except C (p<0.01). There were no significant differences in µTBS between AA/SL and HA/SL and between SL and AA/HA (p>0.05). For LAV, the µTBSs of AA and SL were significantly lower than those of the other groups except C (p<0.01). There were no significant differences in µTBS between AA and SL, between HA and AA/SL, and among HA/SL, AA/HA and AA/HA/SL (p>0.05).

Failure mode analysis
The results of the failure mode analysis are displayed in Fig. 2. Regarding failure analysis, the highest percentages of adhesive failure were found in C. The percentages of mixed failure increased in AA and HA. For AA/SL and HA/SL, the percentages of mixed failure were not increased, although the bond strengths were higher than those of AA and HA. More cohesive failures were found in ENA. The failure mode was associated with the surface treatment in all materials (p<0.04).

SEM findings
The SEM images of the treated resin-ceramic materials are presented in Fig. 3. The surface topography of the C group revealed the polymer matrix and the ceramic filler. The surface of specimens treated by AA showed crevices and pits. Cracks were found in the fillers of the BHC specimens (Fig. 3 BHC-AA). The specimens treated with HA showed a surface texture change and irregular gaps and micropores. The ceramic phases

| Table 2 Criteria for judging each failure mode |
|--------------------------|---------------------------------------------|
| Failure mode             | Definition                                  |
| Cohesive failure         | Failure occurred exclusively within the resin-ceramic material |
| Mixed failure            | Failure continued from the adhesive into the resin-ceramic material |
| Adhesive failure         | Failure occurred entirely within the adhesive area |

16. Owens W., Wendt S., 1956. J. Appl. Polym. Sci. 1, 877-892.
Table 3  Microtensile bond strength (MPa) of the CAD/CAM materials after various surface treatments

| Surface treatment     | CAD/CAM material |
|-----------------------|------------------|
|                       | BHC              | CER              | ENA              | LAV              |
| No treatment          | 2.02 (1.93)      | 13.84 (2.91)     | 9.47 (2.23)      | 4.01 (1.47)      |
| AA                    | 14.10 (5.57)a    | 32.57 (7.19)a    | 20.35 (2.69)     | 7.73 (1.39)a     |
| HF etching (HA)       | 31.32 (5.96)b    | 30.70 (8.86)a    | 24.88 (3.23)     | 19.73 (4.21)b    |
| Silane (SL)           | 7.33 (2.85)      | 30.11 (8.04)a    | 34.59 (7.01)a    | 6.86 (2.25)a     |
| AA/SL                 | 20.87 (6.49)a    | 36.94 (8.25)ab   | 44.30 (4.90)b    | 26.71 (2.53)c    |
| HF/SL                 | 30.36 (6.92)b    | 41.05 (4.23)b    | 45.13 (10.31)b   | 24.65 (5.89)c    |
| AA/HA                 | 36.17 (4.63)bc   | 36.65 (3.09)b    | 29.44 (2.82)a    | 24.65 (5.89)c    |
| AA/HA/SL              | 39.86 (4.07)c    | 44.89 (4.31)b    | 61.57 (5.58)     | 27.44 (6.83)c    |

Mean (S.D.) In the same column, same superscript letters mean no statistical significance ($p>0.05$).

Table 4  Surface roughness (µm) of the CAD/CAM materials after surface treatments

| Surface treatment     | CAD/CAM material |
|-----------------------|------------------|
|                       | BHC              | CER              | ENA              | LAV              |
| No treatment          | 0.23 (0.02)      | 0.23 (0.04)      | 0.25 (0.04)      | 0.18 (0.03)      |
| AA                    | 1.80 (0.28)      | 0.97 (0.21)      | 0.59 (0.12)      | 1.43 (0.14)      |
| HF etching (HA)       | 0.35 (0.12)      | 0.27 (0.08)      | 0.56 (0.10)      | 0.22 (0.03)      |
| Silane (SL)           | 0.23 (0.02)      | 0.27 (0.06)      | 0.19 (0.04)      | 0.21 (0.02)      |
| AA/SL                 | 1.92 (0.29)      | 1.00 (0.19)      | 0.62 (0.10)      | 1.38 (0.20)      |
| HA/SL                 | 0.33 (0.07)      | 0.22 (0.04)      | 0.60 (0.07)      | 0.22 (0.05)      |
| AA/HA                 | 1.93 (0.31)      | 1.00 (0.14)      | 0.70 (0.11)      | 1.23 (0.13)      |
| AA/HA/SL              | 1.88 (0.20)      | 0.92 (0.13)      | 0.79 (0.10)      | 1.36 (0.20)      |

Mean (S.D.)

Table 5  Surface energy (mJ/m²) of the CAD/CAM materials after various surface treatments

| Surface treatment     | CAD/CAM material |
|-----------------------|------------------|
|                       | BHC              | CER              | ENA              | LAV              |
| No treatment          | 103.10 (4.23)    | 89.31 (4.05)     | 111.89 (3.93)    | 108.66 (3.75)    |
| AA                    | 80.85 (4.02)     | 79.74 (3.41)     | 117.05 (3.45)    | 99.96 (1.52)     |
| HF etching (HA)       | 91.10 (10.62)    | 116.51 (4.68)    | 117.02 (4.12)    | 119.30 (8.39)    |
| Silane (SL)           | 118.81 (5.25)    | 114.82 (7.97)    | 103.21 (5.15)    | 128.91 (15.76)   |
| AA/SL                 | 87.92 (11.73)    | 89.30 (6.11)     | 96.29 (6.42)     | 150.14 (2.49)    |
| HA/SL                 | 94.10 (5.01)     | 108.99 (12.61)   | 147.64 (4.22)    | 100.99 (6.00)    |
| AA/HA                 | 78.25 (8.20)     | 115.78 (6.35)    | 123.51 (1.85)    | 102.19 (6.34)    |
| AA/HA/SL              | 98.54 (5.84)     | 80.74 (5.39)     | 140.53 (2.79)    | 112.88 (10.37)   |

Mean (S.D.)

of the ENA specimens appeared to be dissolved by the HA treatment. For the BHC and LAV specimens, the filler sizes of the HA-treated specimens appeared smaller than those of the C specimens. However, the CER fillers nearly disappeared from the specimen surface. The surfaces of the AA/HA-treated specimens showed a combination of macroscale crevices and pits with microscale micropores and gaps between the filler,
Fig. 2  Number of specimens exhibiting each failure mode.
Specimens exhibiting pretest failure were excluded. The p-values are from Fisher’s exact test. In each material, the failure mode results are associated with the surface treatment at a significance level of 0.05.

Fig. 3  SEM observation (5,000×) of the BHC, CER, ENA, and LAV samples.
The images with -C are the surfaces after sandpaper polishing; the images with -HA are the surfaces after hydrofluoric acid etching; the images with -AA are the surfaces after airborne-particle abrasion treatment; and the images with -AA/HA are the surfaces after airborne-particle abrasion treatment followed by hydrofluoric acid etching. The white arrows show gaps between the filler and polymer matrix. The white dotted line indicates a crack in the filler.
Various surface treatments, including AA, HA, and SL, and their combinations significantly influenced the µTBS of the resin luting agent to resin-ceramic materials. However, in this study, the effects of various surface treatments on the surface roughness and energy of the CAD/CAM materials were dependent on the type of CAD/CAM material. In accordance, the null hypothesis for the bond strength between the resin luting agent and the CAD/CAM materials was rejected, whereas the null hypothesis for the surface roughness and energy of the CAD/CAM materials was partially accepted.

HA (etching with a 4.5% hydrofluoric acid gel) increased the bond strength between the resin luting agent and all resin-ceramic materials, but a greater effect was recognized in BHC and LAV specimens. Peumans et al. published corresponding results15). CER was also influenced by HA, but the bond strength was not different from that of the other individual treatments, similar to a study by Lise et al.17) ENA was influenced by HA, but the bond strength was lower than that of SL. However, Cekic-Nagas et al. reported that HA had no effect on the bond strength in CER, ENA and LAV specimens18). The main compositions of these materials were silica and silicate compounds4), and HA partially dissolved those silicas. The etched surface became a hexafluoro-silicate compound, which can be washed out by water to become microporous. In the SEM results of this study, there was a noticeable change in the etched surfaces of CER and ENA, and the roughened surfaces seemed to have contributed to the improved bond strength between the CAD/CAM materials and the resin luting agent. However, after the various surface treatments, only the Ra value of ENA was significantly influenced by HA. In contrast, the surface energy of all the CAD/CAM materials was significantly influenced by HA after the various surface treatments. Previous studies reported that the contact angle of these materials decreased, whereas the surface roughness and energy increased, which allowed good wettability, thereby attaining high bond strength for the resin luting agent19-21). Hence, the bond strength between a resin luting agent and CAD/
CAM materials might significantly relate to the surface energy of the latter after various surface treatments. In this study, SEM showed only a few changes in the BHC and LAV surfaces after HA, even though HA influenced the bond strength between the resin luting agent and these materials. Experimental groups including HA had higher bond strengths than the other groups without HA. Moreover, there were voids in the resin luting agent on all debonded surfaces of the LAV microbars treated with HA. These voids might not form from air trapped during the mixing or bonding procedure because the experimental groups, including the HA treatment (HA, HA/SL, AA/HA, and AA/HA/SL) groups, were simultaneously bonded with the same resin luting agent using the same resin-ceramic plates, but these voids were found in the specimens treated with HA. The reason for this phenomenon was not clarified.

AA (abrasion with airborne particles for 20 s) increased the bond strength between the resin luting agent and all resin-ceramic materials and significantly influenced the surface roughness of all CAD/CAM materials. AA increases the surface roughness, surface area, and surface energy in many resin-ceramic materials and improves the bond strength of resin luting agents. Peumans et al. reported corresponding results. AA provides a higher bond strength than that of the control groups, similar to the results in many studies. However, some studies have reported contrary opinions. Duzyol et al. reported that AA should be avoided in LAV. Yoshihara et al. reported low bond strength in AA-damaged CER, LAV and BHC material surfaces, especially in BHC. The greatest influence of AA was found in CER. The manufacturer suggested using a pressure of abrasion of 0.15 MPa, while the pressure of abrasion for the other materials ranges from 0.1–0.3 MPa. CER has a lower filler mass percentage and flexural modulus than LAV and ENA. A low modulus may cause CER to be more easily influenced than other materials. However, a low pressure (0.15 MPa) may be desirable for all materials because this reduces the damage to resin-ceramic surfaces. In our study, the filler mass percentage and flexural modulus of BHC were less than those of CER, but BHC was less influenced by AA than CER. Yoshihara et al. reported that AA damaged the subsurface cracking of BHC; thus, the bond strength was lowered. The polymeric network of ENA, a feldspathic ceramic with a microcrack source from AA, is believed to render the material less brittle than classic ceramics, as inferred from its higher Weibull modulus.

The application of SL influenced the bond strength of all resin-ceramic materials. SL did not significantly influence the surface roughness of all CAD/CAM materials; however, SL significantly influenced the surface energy of the CAD/CAM materials except CER. The SL treatment uses an adhesive promotor that bonds the silica-based ceramic to the resin luting agent, improving the surface energy and wettability. All SL groups exhibited higher bond strengths than those of the C groups in all materials, which agreed with a study by Peumans et al. but differed from a study by Demirtag and Culhaoglu. The highest influence was found in ENA, while the lowest influence was found in LAV. ENA is a resin-infiltrated feldspathic ceramic that is more similar to a ceramic than to other resin-ceramic materials, namely, those similar to resin composites. ENA is composed of two networks: a ceramic network (86% by weight) and a polymer network (14% by weight). HA has a very high ceramic percentage, and most of its composition is SiO₂ (60% by weight). More glass or SiO₂ provides more area to react with the SL. Accordingly, ENA is influenced more by the SL than by HA and AA, in agreement with a study by Peumans et al. The ceramic content of BHC (60% by weight) was the lowest, with few silica components reacting with SL. The respective manufacturer has suggested using a special primer, the HC primer, which was specifically designed for this material. The components are resin monomer, acetone, polymerization initiator, etc., without the SL. LAV had a high ceramic content (80% by weight); however, the influence SL was quite low and was less than that of BHC. Both SL and AA treatments were low in LAV. SL significantly influenced the surface energy of the CAD/CAM materials except CER.

From the results of the µTBS test, the effect of the interaction between AA and HA, that between HA and SL and that between AA and SL were detected in the µTBS of the resin luting agent to BHC and LAV, that to BHC and that to CER and ENA, respectively. Moreover, the effect of the interaction among AA, HA and SL was detected in the µTBS of the resin luting agent to only LAV. Thus, the effect of the interaction between the respective surface treatments was dependent on the type of the CAD/CAM materials. In general, a positive AA/SL interaction was found because both the roughness and surface energy were increased by AA. SL demonstrated more chemical interactions. The present study revealed that the AA/ SL groups showed higher bond strength compared with that of the AA or SL groups in all CAD/CAM materials. However, a significant AA/SL interaction was recognized in CER and ENA, but not in BHC and LAV. Peuman et al. reported that a positive AA/SL interaction was found in only ENA and not in LAV. The HA/SL groups showed higher bond strength compared with that of the SL group in all CAD/CAM materials and the HA group in the CAD/CAM materials except BHC; however, in the present study, a significant HA/SL interaction was not detected in the CAD/CAM materials except BHC, which showed a negative HA/SL interaction. These results agreed with the previous study reported by Peuman et al., in which the HA/SL interaction was negative for BHC. Etching with HA resulted in the dissolution of the embedded glass fillers in the laboratory composites, and no SL molecules were retained. Therefore, SL had no impact on the bond strength. The HA/SL group showed high surface roughness but low surface energy compared with that of the control (no treatment) in BHC. In general, decreasing the surface energy facilitated the wettability of the SL. HA also promoted a hydroxyl group on the etched surface of the glass ceramic that...
improved the chemical bond to the SL. However, the HA/SL interaction negatively influenced BHC, probably because BHC had a lower filler mass percentage than that of the other CAD/CAM materials. HA provided a porous resin-ceramic surface with increased surface roughness but decreased the filler surface chemically reacted with SL. In the present study, the AA/HA groups showed higher bond strength than that of the AA or HA group in all CAD/CAM materials; however, a significant AA/HA interaction was detected in BHC and LAV, but not in CER and ENA. A negative AA/HA interaction was detected for the surface energy in BHC and LAV. Therefore, for BHC and LAV, the significant increase in bond strength in the AA/HA group might be related to a significant decrease in surface energy after surface treatment with a combination of AA and HA. For all types of materials, the AA/HA/SL groups had the highest bond strengths, although a positive interaction was found in only LAV. All surface treatments (AA, HA and SL) influenced the bond strength. This combination allowed the treatments to influence each other. AA/HA/SL contributed significantly more than did the other groups for all the materials because AA and HA facilitated the SL. However, this combination is time consuming because more steps are needed. Although there were no statistically significant differences between AA/HA and AA/HA/SL, the application of SL may be necessary because SL improves the durability of the bonding in conventional ceramics and indirect composites. However, the effectiveness of the long-term bond in AA/HA/SL requires further study.

For BHC, treatment with AA/SL has been suggested by the manufacturer, but treatment with HA significantly increased the µTBS in the present study. For CER and LAV, manufacturers have suggested AA/SL and HA/SL. Compared with a single application of each surface treatment, the combinations contributed to a higher bond strength; however, the bond strength of AA/HA/SL was higher than that of AA/SL or HA/SL. HA treatment significantly increased the µTBS in LAV. For ENA, surface treatments with HA/SL have been suggested by the manufacturer. In the present study, the SL group showed significantly higher µTBS than the AA or HA group in ENA. Moreover, similar to the HA/SL treatment, the AA/SL treatment demonstrated a high bond strength. HA treatment has been avoided in some countries because of the harmfulness of hydrofluoric acid in the human body. Therefore, for ENA, AA/SL is recommended because of its bonding performance and safety, and individual SL treatment is also recommended.

From the SEM images, when compared to the HA etching treatment, the AA treatment roughened the resin-ceramic surface on a large scale. All manufacturers suggested using alumina powder with a particle size of 25–50 µm. AA removed both the resin matrix and filler from the surface. However, HA partially removed the inorganic filler with filler sizes ranging from 0.004 to 0.6 µm. AA not only increased the roughness, surface area and surface energy but also facilitated HA. In this study, the AA/HA groups had the statistically highest bond strengths in BHC, CER and LAV, although there was a negative interaction in BHC.

The percentage of adhesive failure was high when no treatment was used because of the low bond strength. AA and HA increased the percentages of mixed failure, which might be a result of the high bond strength due to mechanical interlocking and chemical interaction. However, the percentages of mixed failure did not increase when AA or HA was followed by SL. We speculated that this result occurred because the SL treatment increased the surface energy and made the entire surface equally bonded. More cohesive failure was found in ENA, which could be explained by the high brittleness of this material.

In this study, two adhesive interfaces exist between the resin-ceramic and resin luting agent because two resin-ceramic plates were bonded together as a sandwich. Although fracture occurred at either side, the obtained bond strengths were measured from the adhesive interface. The sandwich specimens were light-cured from four directions to obtain the highest rate of polymerization of the resin luting agent; however, this light-curing method may not simulate the clinical situation.

Adhesion to a tooth abutment should provide retention and marginal seal to achieve clinical durability. A bond strength test helps manufacturers determine the performance of their luting agent and dental material, from which they can make a prognosis on the clinical durability. However, few studies have correlated in vitro bond strength data with clinical outcomes. Publications often find a correlation between dentin bond strength and the retention rate of Class V restorations. In the meta-analysis, the results of the regression analysis for the pooled data revealed that µTBS tests correlated accurately with both marginal discoloration after 6 months of water storage and retention rate at 5 years. However, µTBS is currently recommended as a good measure of dental resin composite retention. The µTBS has some advantages over macroscopic bond strength testing, including more economical use of material (multiple micro-specimens from one block of material), better stress distribution at the target interface, avoiding cohesive failure in the material substrate, and better control of substrate variables.

Further studies are required to evaluate the outcome in the long term, the specimen with sizes and thicknesses in accordance with the clinical situation, and the effect of surface treatments with different resin luting agents, AA, HA, and SL, on the bond strength.

CONCLUSION

AA, HA, and SL significantly influenced the bond strengths between the resin luting agent and the CAD/CAM materials used in this study. In addition, combinations of these surface treatments positively affected the bond strengths. However, the effect of the surface treatments and their combinations on the surface
roughness and energy of the CAD/CAM materials were dependent on the type of CAD/CAM materials.

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