Microtensile Bond Strength of Self-Adhesive Resin Cements to CAD/CAM Resin-Matrix Ceramics Prepared with Different Surface Treatments

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Purpose: To investigate the effects of different combinations of self-adhesive resin cements and surface treatments on the microtensile bond strength (μTBS) of different CAD/CAM resin-matrix ceramics. Materials and Methods: Two different types of CAD/CAM resin-matrix ceramics (LAVA Ultimate [LU] and Vita Enamic [VE]) were used. Slices 5 mm in thickness were obtained from each CAD/CAM material. Samples of each ceramic were randomly separated into the following surface treatment groups: (1) Control group with no surface treatment; (2) 9.5% hydrofluoric acid (HF) + universal adhesive (UA, Single Bond Universal, 3M ESPE); and (3) Sandblasting with 50-μm aluminum oxide particles (SN) + UA. Two different types of recently developed resin cement (RelyX U200, 3M ESPE; SET PP, SDI Dental Limited) were applied to the treated ceramic slices in each group. After 24 hours, parallel sections were removed from the specimens, and microbeam-shaped sticks (1.0 × 1.0 × 10 mm) were prepared. The μTBS test was performed, and the data were statistically analyzed. Results: Statistical analyses revealed differences among the study groups (P < .05). The control groups of each resin cement exhibited lower μTBS values than the groups that received surface treatment (P < .05). The effect of surface treatment on μTBS (partial eta-squared [ηp²] = 0.381) was more significant than the effects of ceramic (ηp² = 0.267) and self-adhesive resin cement (ηp² = 0.184). Conclusion: Surface treatment is the most important factor affecting the μTBS of resin cement to CAD/CAM materials, followed by the type of resin-matrix ceramic and the type of resin cement, respectively. Int J Prosthodont 2019;32:433–438. doi: 10.11607/ijp.6268

Presently, with the development of technology, computer-aided design/computer-assisted manufacturing (CAD/CAM) systems are increasingly preferred in dentistry because of their superior advantages compared to conventional clinical procedures. The continuous addition of new material for CAD/CAM use is inevitable because of this growing demand. In the recent decade, CAD/CAM resin-matrix ceramic blocks have been introduced to combine the advantages of different materials together. A resin nanoceramic (LAVA Ultimate [LU], 3M ESPE) was first introduced for this purpose in 2012, demonstrating improved mechanical properties. This material contains nanoceramic particles (80 wt%) embedded in a polymer matrix. Afterwards, a glass-ceramic in a resin interpenetrating matrix (hybrid ceramic, VITA ENAMIC [VE], Vita Zahnfabrik) was introduced in early 2013. VE includes a preinserted ceramic network (86 wt%) infiltrated by a monomer mixture and exhibits improved mechanical properties due to the polymeric network. The advantages of these resin-based materials are still the subject of research. In particular, the adhesion of these CAD/CAM resin-matrix ceramics to the tooth tissues and/or to the resin cement is of great importance in terms of the clinical success and longevity of an indirect restoration made with these materials.
A durable bond between the three components of an adhesion complex—tooth tissues, resin cement, and/or ceramic—is a key factor for the long-term clinical success of a restoration.6,7 Optimal adhesion is required to obtain a high bond strength. The traditional adhesive technique involves the multi-step pretreatment of tooth tissues and the ceramic surface, including etching, priming, and/or bonding. Self-adhesive resin cement was designed to reduce these conventional steps.8

The first self-adhesive resin cement was introduced to the market in 2002 to combine the advantages of adhesive and conventional resin cement.9 Since then, self-adhesive resin cements have been increasingly marketed by different companies. Although there has been basic research to investigate the chemical or physical properties of these new materials, there is a need for more information about the bond strength of these materials, especially with regard to CAD/CAM resin-matrix ceramics. Therefore, the objective of this study was to assess the microtensile bond strength (μTBS) of two different types of self-adhesive resin cement to recently developed CAD/CAM resin-matrix ceramics prepared with various surface modifications. The null hypotheses were as follows: (1) there would be no difference in the μTBS of self-adhesive resin cements to the CAD/CAM resin-matrix ceramics; and (2) the type of surface treatment would not affect the μTBS of self-adhesive resin cements to the CAD/CAM resin-matrix ceramics.

**MATERIALS AND METHODS**

Two different types of CAD/CAM resin-matrix ceramic blocks, composed of VE or LU, were selected for this study. Descriptions of the materials are given in Table 1.

<table>
<thead>
<tr>
<th>Brand name</th>
<th>Manufacturer</th>
<th>Description</th>
<th>Filler loading (wt%)</th>
<th>Shade</th>
<th>Lot No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>LAVA Ultimate</td>
<td>3M ESPE</td>
<td>Resin composite reinforced with nanoparticles (20 nm silica, 4–11 nm zirconia, and 0.6–10 μm nanoclusters)</td>
<td>80</td>
<td>A2-LT</td>
<td>N661407</td>
</tr>
<tr>
<td>VITA ENAMIC</td>
<td>VITA Zahnfabrik</td>
<td>Glass-ceramic in a resin interpenetrating matrix (hybrid ceramic)</td>
<td>86</td>
<td>1M1-T</td>
<td>49490</td>
</tr>
<tr>
<td>Relux U200</td>
<td>3M ESPE</td>
<td>Self-adhesive resin cement</td>
<td>70</td>
<td>A2</td>
<td>636547</td>
</tr>
<tr>
<td>SET PP</td>
<td>SDI Dental Limited</td>
<td>Self-adhesive resin cement</td>
<td>65</td>
<td>A2</td>
<td>51509081</td>
</tr>
<tr>
<td>Single Bond Universal</td>
<td>3M ESPE</td>
<td>Universal adhesive, includes 10-MDP, pH of 2.7 (extra mild)</td>
<td>–</td>
<td>–</td>
<td>590662</td>
</tr>
<tr>
<td>Porcelain Etchant</td>
<td>Bisco</td>
<td>9.5% hydrofluoric acid</td>
<td>–</td>
<td>–</td>
<td>1400001790</td>
</tr>
<tr>
<td>Airsonic Aluminium-Oxyd-Pulver</td>
<td>Hager &amp; Werken</td>
<td>50-μm aluminum oxide particles</td>
<td>–</td>
<td>–</td>
<td>605084</td>
</tr>
</tbody>
</table>

10-MDP = 10-methacryloyloxydecyl dihydrogen phosphate

HF (9.5% Porcelain Etchant, Bisco) was applied to the specimens for 1 minute, and then the specimens were rinsed with air-water spray for 1 minute, cleaned in an ultrasonic bath filled with distilled water for 5 minutes, and air dried. Sandblasting procedures were performed with a sandblaster machine (Macro Cab, Danville Engineering) at approximately 5 mm from the resin-matrix ceramic surface for 20 seconds. The specimens were sandblasted with 50-μm Al₂O₃ (Airsonic Aluminum-Oxyd Pulver, Hager & Werken) for 20 seconds, and a pressure of 2 bar was maintained for air abrasion. All sandblasted specimens were then cleaned in an ultrasonic bath filled with distilled water for 5 minutes and then air dried. After surface pretreatments, universal adhesive (Single Bond Universal, 3M ESPE) was directly applied onto the surface of the specimen for 20 seconds and then polymerized with a light-emitting diode (LED) curing device (Elipar DeepCure-S, 3M ESPE; Light output: 1,470 mW/cm²) for 20 seconds according to the manufacturer’s directions.

After the surface procedures, each ceramic slice was placed in a 10-mm-thick mylar strip using a Tofflemire retainer with the bonding surface on the top. Resin cement (Relux U200 [3M ESPE] or SET PP [SDI Dental]) was used until the space on the ceramic slice was filled. The resin cement surfaces were closed using a mylar strip.
and polymerization was completed with a high-intensity LED curing unit (Elipar DeepCure-S) for 20 seconds for each surface (top and four sides) of the resin cement. An additional curing step was provided on the top and sides for 20 seconds after removing the Tofflemire retainer. Each sample was embedded in an epoxy resin and stored in distilled water at 37°C for 24 hours. The epoxy resin blocks were placed on the water-cooled cutting device (Isomet Low Speed Saw, Buehler) with a diamond saw. The blocks were sectioned under water cooling to maintain numerous thin microbeam-shaped sticks (approximately 1.0 × 1.0 × 10 mm) using the nontrimming technique. Four sticks were obtained from each ceramic resin cement block, and 15 were provided for each group. During sectioning of the sticks, specimens were stored in distilled water in order to avoid deterioration of the samples.

In total, 12 groups were included in the study. In each group, the samples were subjected to the μTBS test. Each specimen was luted using a cyanoacrylate adhesive (Pattex 2K Professional Rapid Adhesive, Henkel) at each end after mounting on the microtensile testing device (Micro Tensile Tester, BISCO). Reverse pressure was applied at a crosshead speed of 1 mm/minute until a fracture took place in the stick, and the fracture value was recorded in Newtons (N). The fractured sample was meticulously taken out of the testing device using a sharp spatula. The dimensions (a and b) of each fractured stick were measured using a digital caliper (Insize) from the area nearest to the adhesion surface to calculate the μTBS of the fracture load in MPa. The fracture value was then converted to MPa using the formula MPa = N / (a × b) for each sample.

Three additional ceramic slices were prepared from each resin-matrix ceramic, and on one of these ceramic slices, no pretreatment was performed. HF and SN pretreatments were performed on the other ceramic slices as described above to observe the surface changes. For evaluation with scanning electron microscopy (SEM) (QUANTA 400F Field Emission SEM, Thermo Fisher Scientific), all specimens were dried and coated with gold.

Normality of the data was assessed with Kolmogorov-Smirnov and Shapiro-Wilk tests. The data were analyzed statistically with one-way analysis of variance (ANOVA) and Tukey Honestly Significant Difference tests (SPSS Version 20, SPSS). Two-way ANOVA was performed to test the effect of study parameters, including resin cement, surface treatment, and resin-matrix ceramic, on the μTBS values separately and together.

### RESULTS

The mean μTBS values of the groups and the statistical differences are presented in Table 2. Significant differences were found between the study groups (P < .05). The highest μTBS value was obtained for LU when treated with HF + UA and bonded to RelyX U200 (38.7 ± 6.4). This group was statistically significant compared to all other experimental groups (P < .05), followed by groups 3 (31.7 ± 4.5), 6 (30.8 ± 5.4), and 8 (28.3 ± 4.2), respectively, although there was no statistical difference between these three groups (P > .05).

No statistically significant difference was observed between the control groups of the two different types of self-adhesive resin cement (P > .05). When RelyX U200 self-adhesive resin cement was used, the control groups exhibited statistically lower values than the groups that received surface treatments for both of the resin-matrix ceramics (P < .05). The groups in which RelyX U200 resin cement was applied to the surface-treated ceramics (groups 2, 3, 5, and 6) showed statistically higher values than the groups in which SET PP resin cement was used on the treated ceramics (groups 9, 11, and 12), with the exception of group 8 (Table 2).

When SET PP self-adhesive resin cement was used, the surface-treated LU groups (groups 8 and 9) showed statistically higher values than the other groups (P < .05). There was no significant difference detected between groups 8 (28.3 ± 4.2) and 9 (25.3 ± 5) (P > .05). Within the SET PP subgroups, the control groups exhibited statistically similar values to the other

### Table 2  Mean Microtensile Bond Strength (μTBS) Values of the Study Groups

<table>
<thead>
<tr>
<th>Group</th>
<th>Resin cement</th>
<th>Resin-matrix ceramic</th>
<th>Surface treatment</th>
<th>No. of samples</th>
<th>μTBS (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>RelyX U200</td>
<td>LAVA Ultimate</td>
<td>None</td>
<td>15</td>
<td>19.1±4.9</td>
</tr>
<tr>
<td>2</td>
<td>HF + UA</td>
<td></td>
<td></td>
<td>15</td>
<td>38.7±6.4</td>
</tr>
<tr>
<td>3</td>
<td>SN + UA</td>
<td></td>
<td></td>
<td>15</td>
<td>31.7±4.5</td>
</tr>
<tr>
<td>4</td>
<td>VITA ENAMIC</td>
<td></td>
<td>None</td>
<td>15</td>
<td>17.6±4.6</td>
</tr>
<tr>
<td>5</td>
<td>HF + UA</td>
<td></td>
<td></td>
<td>15</td>
<td>23.7±4.1</td>
</tr>
<tr>
<td>6</td>
<td>SN + UA</td>
<td></td>
<td></td>
<td>15</td>
<td>30.8±5.4</td>
</tr>
<tr>
<td>7</td>
<td>SET PP</td>
<td>LAVA Ultimate</td>
<td>None</td>
<td>15</td>
<td>22.1±4.8</td>
</tr>
<tr>
<td>8</td>
<td>HF + UA</td>
<td></td>
<td></td>
<td>15</td>
<td>28.3±4.2</td>
</tr>
<tr>
<td>9</td>
<td>SN + UA</td>
<td></td>
<td></td>
<td>15</td>
<td>25.3±4.4</td>
</tr>
<tr>
<td>10</td>
<td>VITA ENAMIC</td>
<td></td>
<td>None</td>
<td>15</td>
<td>19.8±4.9</td>
</tr>
<tr>
<td>11</td>
<td>HF + UA</td>
<td></td>
<td></td>
<td>15</td>
<td>20.9±4.5</td>
</tr>
<tr>
<td>12</td>
<td>SN + UA</td>
<td></td>
<td></td>
<td>15</td>
<td>18.9±5.9</td>
</tr>
</tbody>
</table>

HF = Hydrofluoric acid; SN = sandblasting; UA = universal adhesive. Values denoted with the same superscript letter were statistically homogenous (Tukey Honestly Significant Difference test, α = .05).
The combined effect of resin-matrix ceramic and resin cement type was not statistically significant ($P > .05$).

The comparison of the three different surface treatments independent of types of resin-matrix ceramic and resin cement is presented in Table 4. The control group with no treatment exhibited statistically lower values than the treatment groups ($P < .05$). Cumulatively, no significant difference between the two treatment groups was found ($P > .05$).

**DISCUSSION**

This study was performed to evaluate the μTBS of different self-adhesive resin cements to different resin-matrix ceramic CAD/CAM materials with different surface treatments. Two different types of recently developed CAD/CAM resin-matrix ceramic blocks and two different types of resin cement were employed in this in vitro study to examine their properties.

For a successful restoration, the power of bond strength between the ceramic restoration, resin cement, and tooth tissues has been emphasized.\textsuperscript{10–13} Self-adhesive resin cements provide a better bond strength to dentin than conventional cements.\textsuperscript{8} However, there have not been enough studies reporting how these resin cements affect the bond strengths of contemporary CAD/CAM materials when different surface treatments are applied.
declared no significant difference between the bond strength values of the resin cement bonded to a resin-infiltrated ceramic treated with either HF or SN pretreatments. Nevertheless, Elsaka reported that HF increased the bond strength to the resin-infiltrated hybrid glass-ceramic block. The different results can be attributed to the different methodologies and materials used. Although the results of the literature are somewhat contradictory, the great majority of studies have found and recommended both HF and SN pretreatments. Nevertheless, further research, especially in clinical settings, is needed to determine which pretreatment will be better to use for the specific material of a restoration.

It has been reported that the higher the filler content of the resin cement, the higher the bond strength of the material. The inorganic filler load of RelyX U200 resin cement is approximately 70% by weight, and the filler load of SET PP is about 65%. This difference between the filler loads may explain why the μTBS values of RelyX U200 resin cement, especially when used with LU, were higher than that of SET PP resin cement. This result is in agreement with the results of Cekic-Nagas et al, indicating that resin cements with a higher inorganic filler content exhibit higher bond strength than those with lower inorganic content.

Additionally, surface pretreatments, such as grinding, sandblasting, and/or acid etching, are typically used to increase the bond strength between the restoration and resin cement. Therefore, two surface pretreatments, etching with HF and sandblasting with Al2O3 particles, were used in this study before the application of the UA. In a recent study, the importance of applying the chemical silane after SN or HF etching has been emphasized to improve the bond strength between the resin-matrix ceramic restoration and resin cement. Therefore, a UA, which is also considered to be a silane, was preferred, and the groups receiving the pretreatment alone were not included in this study.

Since there were statistically significant differences between the groups in the study, the first null hypothesis of the study was rejected. While HF pretreatment increased the μTBS values for the LU resin nanoceramic, it decreased those values for the VE hybrid ceramic. In this case, SN can be recommended for resin-infiltrated hybrid glass-ceramics. Similarly, HF application may be recommended for resin nanoceramics reinforced with nanoparticles such as silica and zirconia. Therefore, the results support the rejection of the second null hypothesis, that the type of surface treatment does not affect the μTBS of self-adhesive resin cements to CAD/CAM resin-matrix ceramics. SEM images also support these findings. SEM analyses revealed an irregular surface was generated for both of the treatment methods (Figs 1 and 2). However, when the images of VE after HF pretreatment (Fig 2b) are examined, broken ceramic crystals can be clearly observed. Therefore, HF pretreatment can weaken the hybrid ceramic. The fact that the surface pretreatments cause different effects on two different resin-matrix ceramics can be related to the different contents of the materials.

Conflicting results have been presented in the literature about the bond strength between resin-matrix ceramic restorations and resin cement. Barutcigil et al declared no significant difference between the bond strength values of the resin cement bonded to a resin-infiltrated ceramic treated with either HF or SN pretreatments. Nevertheless, Elsaka reported that HF increased the bond strength to the resin-infiltrated hybrid glass-ceramic block. The different results can be attributed to the different methodologies and materials used. Although the results of the literature are somewhat contradictory, the great majority of studies have found and recommended both HF and SN pretreatments. Nevertheless, further research, especially in clinical settings, is needed to determine which pretreatment will be better to use for the specific material of a restoration.

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The ηp2 value gives the correlation between the dependent variable and the study parameter. It normally consists of values ranging from 0 to 1. Surface treatment affected the μTBS values more than the other two parameters, the type of resin cement and the resin-matrix ceramic, in this study. Furthermore, resin cement and resin-matrix ceramic had no effect on the μTBS values together. Therefore, it can be said that the treatment to be applied on the adhesion surface of the restoration might be more important than the type of material used. Peumans et al also emphasized the importance of surface treatment in the μTBS values of CAD/CAM glass-ceramics.
The results concluded that the bond strengths in the treated surface groups were higher than in the nontreated groups. Therefore, a process must be applied on the resin-matrix ceramic surface. However, there was no difference between the two surface treatments in total regardless of the type of CAD/CAM material and resin cement. This result could be related to the appropriate concentration of HF, as well as to the particle size of Al₂O₃. In the present study, 9.5% HF was used, which is close to the concentration used by Vidotti et al., who showed that a 9% concentration was able to sufficiently dissolve the glass phase in ceramics. Furthermore, 50-μm Al₂O₃ particles were used in the present study and were found to be more effective for the bond strength values of CAD/CAM resin-matrix ceramic blocks than smaller particles in a recent study. In agreement with the results of the current study, Lise et al. obtained higher μTBS values for the samples roughened by either SN or HF than for the nontreated control groups.

The majority of the papers reporting μTBS results included no thermocycling (94%) and 24 hours of storage time prior to testing (86%). Since these methods could be considered cost-effective, fast, and practical, long-term results were not included in this study. However, Ozcan et al. reported that aging conditions are mandatory in terms of giving an opinion about the adhesion quality, especially for clinical situations. Therefore, future studies are needed to find out whether adhesion quality is influenced by aging conditions.

This study has some limitations. A single UA was used, despite the availability of many adhesive materials. Furthermore, the effect of the application period of the surface pretreatments was not considered. Additionally, the present study did not report any results about the long-term bond strength of the study materials using thermal cycling. Thus, further studies are needed to investigate the effects of other adhesive strategies, adhesives, and application times on the restoration lifetime.

CONCLUSIONS

The following conclusions can be drawn:

- Surface treatment should be applied to the adhesive surface of the CAD/CAM resin-matrix ceramic before cementation, independent of the type of self-adhesive resin cement.
- The type of surface treatment is a more important factor affecting the bond strength of resin cement to the resin-matrix ceramic than the type of material used.
- SN pretreatment is preferred for CAD/CAM hybrid ceramics with a high content of ceramic.
- HF pretreatment is recommended for CAD/CAM resin nanoceramic reinforced with nanoparticles.

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REFERENCES


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