Effect of surface treatment of CAD/CAM resin composites on the shear bond strength of self-adhesive resin cement

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This study examined the effects of sandblasting, hydrofluoric acid etching and priming on the shear bond strength of self-adhesive resin cement between seven different CAD/CAM resin composites and a resin composite core material at 24-h after cement mixing. Five surface treatments [control (C), sandblasting (S), priming (P), sandblasting with priming (SP), and 9% HF etching with priming (HFP)] were performed respectively for disc specimens of CAD/CAM blocks. There were no significant differences in bond strength among the C, S, and P, except for one block (p>0.05). SP showed a greater bond strength than S. Weibull moduli were not changed significantly among all treatments for all blocks, whereas the strengths with 5% and 95% failure probability of SP and HFP showed greater values than the others. The bond strengths of HFP were comparable to those of SP. Priming after sandblasting or HF etching could be effective to increase the bond strength of CAD/CAM blocks.

Keywords: CAD/CAM resin composite, Shear bond strength, Resin cement, Sandblasting, Hydrofluoric acid etching

INTRODUCTION

Fabrication of ceramic and resin composite prosthetics using CAD/CAM systems has increased in recent years. Ceramics and resin composites are chosen to provide an esthetically-pleasing and natural appearance in the oral cavity. In addition to esthetic properties, one of the most important considerations in material selection is the occlusion force during function. Because of the heavy loads on molar regions during occlusion, ceramic materials which have superior mechanical properties are chosen for crowns made by CAD/CAM\(^4,5\), and many suitable ceramic blocks with good mechanical properties are commercially available.

Resin composites for CAD/CAM fabrication having good esthetic qualities are also now available\(^6,7\). The matrix resins of CAD/CAM resin composites are made from Bis-MEPP, UDMA, TEGDMA, etc., and offer a greater degree of polymerization compared to the resin composites used for restorative fillings due to heat polymerization under high pressure\(^7,8\). Manufacturers have increased the filler loading and mechanical strength of CAD/CAM resin composites by employing nanofiller particles, strengthening the bonding between matrix resin and filler particles, improved silane coupling surface treatment, etc. However, some mechanical properties including flexural strength, elastic modulus and hardness of CAD/CAM resin composites remain lower than those of CAD/CAM lithium disilicate and lithium silicate/zirconia\(^9\). Lauvahutanon et al.\(^9\) revealed that the flexural strength of resin composite blocks was far inferior to the reported value of lithium disilicate glass ceramic and densely sintered zirconia for CAD/CAM, noting that the CAD/CAM composite resin blocks investigated were suitable when limited to single premolar crowns. Crowns made from CAD/CAM resin composites are thus useful in the molar region for patients with a metal allergy or without occlusal contact. However, because of manufacturers’ continuous efforts to improve materials, the current products are expected to withstand heavy occlusal loading, not only in premolars but also in the molar region.

For tooth-colored CAD/CAM crowns, tooth-colored core build-up materials such as resin composites offer a natural appearance in the oral cavity\(^9\). Recent clinical study\(^11\) reported that CAD/CAM resin composite crowns on metal abutments showed larger numbers of failures by detaching from abutment teeth in the early period after cementation, compared with those on resin composite cores. Differences in the elastic modulus and thermal properties such as the thermal expansion coefficient among the metal core, resin cement and crown materials would increase the mechanical stress, potentially breaking the adhesion interface\(^7,12,13\). Resin composite materials for core build-up have similar elastic and thermal properties to those of resin cements and CAD/CAM resin composite crowns\(^4,6,14\), alleviating this issue. Thus, considering both the esthetic benefits and physical and mechanical characteristics, resin composite core build-up materials are a logical choice for use with CAD/CAM resin composite crowns.

CAD/CAM resin composites contain less residual monomer than restorative filling composites. Their higher degree of polymerization results from heat-activated curing under pressure, and they thus have a more stable surface condition. Surface treatments are thus required to ensure adhesion with cement\(^7,9\). Manufacturers recommended and supply priming agents
Crown detachment from an abutment tooth in clinical cases was reported to be more likely without sandblasting or priming of the inner surface of the crown\(^{15,16}\). The longevity of crowns in the oral cavity depends on adequate adhesion being achieved between the crown and abutment tooth. Sandblasting was reported to increase the bond strength of CAD/CAM resin composite blocks\(^{17-19}\), while a specific priming agent for self-adhesive cement was reported to improve the chemical bond between resin blocks and resin cement. However, it was found that priming without sandblasting did not result in a sufficient increase in bonding strength. An appropriately rough surface increases the adhesion area and thereby improves the bonding strength by micromechanical interlocking. Sandblasting is currently the primary method to roughen adhesion surfaces. However, sandblasted surfaces display a variety of morphologies depending on the sandblasting conditions and techniques used.

Hydrofluoric acid (HF) dissolves the SiO\(_2\) in ceramics, making it an effective pretreatment method to roughen ceramic surfaces\(^{20,21}\). Resin composites also contain SiO\(_2\) fillers, and HF treatment was reported to roughen resin composite surfaces prior to bonding\(^{18,22}\). However, few studies have examined HF pretreatment of CAD/CAM resin composite blocks\(^{18,23,24}\) and, to the best of our knowledge, no reports are available on the shear bond strength between CAD/CAM resin composites treated with HF and resin composites for core build-up.

The aim of this study was to examine the effect of sandblasting, hydrofluoric acid etching and priming on the bond strength of self-adhesive resin cement between seven different CAD/CAM resin composites and resin composites for core build-up. Our hypothesis was that HF treatment of CAD/CAM resin composites is comparable with sandblasting treatment for increasing the bond strength. This study examined whether a surface condition suitable for bonding could be obtained by chemically treating the inorganic filler components of CAD/CAM resin composite blocks.

### MATERIALS AND METHODS

#### Materials

Seven commercially-available resin composite for CAD/CAM blocks and a resin composite for core build-up (dual-cured resin composite, Unifil Core EM, GC, Tokyo, Japan) were used. A self-adhesive resin (dual-cured resin cement, G-CEM ONE, GC) cement was used for cementing. The compositions of CAD/CAM resin blocks reported by the manufacturers, lot numbers and other information are shown in Table 1. Table 2 lists the compositions of the resin composite for core build-up, priming agent (G-Multi Primer, GC), phosphoric acid gel, hydrofluoric acid gel, and the self-adhesive resin cement.

#### Methods

1. Specimen preparation

1) Resin composite for CAD/CAM blocks

CAD/CAM resin composite blocks were cut from a slab by diamond cutter, embedded into epoxy resin (EPOFIX, Struers, Tokyo, Japan) in a plastic mold, and stored at room temperature for 24 h. After the embedded resin blocks were removed from the mold, the bonding surfaces were polished using No. 600 SiC abrasive paper with water and carefully rinsed under running water. The rinsed blocks were cleaned with deionized water in an ultrasonic bath for 10 min.

### Table 1  CAD/CAM resin blocks examined in this study

| Block                  | Manufacturer         | Lot No.  | Color | Code  | Composition                      |
|------------------------|----------------------|----------|-------|-------|----------------------------------|
| Cerasmart 300          | GC                   | 1708231  | A3-LT | CS300 | Monomer: Bis-MEPP, UDMA, Filler: SiO\(_2\), Ba glass, Filler wt\%: 78% |
| Cerasmart 270          | GC                   | 1706231  | A3-LT | CS270 | Monomer: Bis-MEPP, UDMA Filler: SiO\(_2\), Ba glass, Filler wt\%: 78% |
| Shofu Block HC         | Shofu                | 0117310  | A3-LT | HC    | Monomer: UDMA, TEGDMA Filler: SiO\(_2\), Microfumed SiO\(_2\), Zirconium silicate, Filler wt\%: 61% |
| KZR-CAD HR2            | Yamakin             | 20021713 | A3    | KZR   | Monomer: UDMA, TEGDMA Filler: SiO\(_2\), Al\(_2\)O\(_3\)-ZrO\(_2\), SiO\(_2\), Filler wt\%: 73% |
| Katana Avencia Block   | Kuraray-Noritake     | 000510   | A3-LT | AVE   | Monomer: UDMA Filler: SiO\(_2\), Filler wt\%: 62% |
| Estelite Block         | Tokuyama Dental      | 044067   | A3-LT | EST   | Monomer: UDMA, TEGDMA Filler: SiO\(_2\), Zirconia filler, Filler wt\%: 75% |
| Vita Enamic for Kavo Arctica | Kavo Dental   | 60870    | 3M2-T | ENA   | Monomer: UDMA, TEGDMA Filler: Feldspar ceramic enriched with Al\(_2\)O\(_3\), Filler wt\%: 86% |
Table 2  Materials used in this study

| Material                          | Manufacturer | Composition                                                                 | Lot No.  |
|----------------------------------|--------------|-----------------------------------------------------------------------------|----------|
| Adherend: resin composite for core build-up |              | Base paste: fluoroaluminosilicate glass, urethane dimethacrylate (UDMA)   |          |
| UniFil Core EM                   | GC           | Catalyst paste: fluoroaluminosilicate glass, urethane dimethacrylate (UDMA)|          |
| Primer                           |              | vinyl silane, phosphoric methacrylate monomer, thiophosphoric monomer, methacrylic acid ester, ethyl alchol |          |
| G-Multi Primer GC                | GC           | Paste A: fluoroaluminosilicate glass, methacrylic acid ester, initiator    |          |
| Self-adhesive resin cement       |              | Paste B: silica filler, methacrylic acid ester, phosphoric methacrylate monomer, initiator |          |
| G-CEM ONE                        | GC           | 15–40% phosphoric acid                                                     |          |
| Etching                          |              | BDW3M                                                                       |          |

Table 3  Surface treatments applied to the CAD/CAM resin composite blocks

| Abbreviation | Surface treatment                      |
|--------------|---------------------------------------|
| C            | Control                               |
| S            | Sandblasting                          |
| P            | Priming                               |
| SP           | Sandblasting with priming              |
| HFP          | HF etching with priming                |

2) Resin composite for core build-up
Disc specimens (6 mm diameter and 4 mm thick) of the resin composite for core build-up were prepared using a Teflon mold. The mold was set on plastic strips (CELLULOID STRIPS, GC) on a glass plate. After filling the resin composite into the mold, the plastic strip, and then another glass plate were placed on the mold. The resin composite was cured using a curing lamp (G-light Prima-II Plus, GC) for 6 s each at the top and bottom surfaces. The resin composite filled in the Teflon mold was cured again in a laboratory light curing unit (LAVO LIGHT LV-III, GC) for 10 min to accelerate polymerization. The disc specimens were then removed from the mold and the surfaces were finished using No. 600 SiC abrasive paper with water. The polished surfaces were rinsed under running water and then cleaned with deionized water in an ultrasonic bath for 10 min.

2. Surface treatments
Five different surface treatments were applied to CAD/CAM resin composites before bonding (Table 3). The surface treatment groups were designated C, S, P, SP and HFP. The details of each treatment procedure are described below.

C: control; Phosphoric acid gel was applied to the surface of CAD/CAM resin composites for 30 s. The surface was then rinsed using deionized water and dried in room temperature air.

S: sandblasting; The surface of CAD/CAM resin composites was sandblasted for 5 s at an air pressure of 0.3 MPa by alumina particles (70 μm, Hi-ALUMINA, Shofu, Kyoto, Japan). They were washed under running water followed by ultrasonic cleaning with deionized water for 10 min. Phosphoric acid gel was then applied to the surface for 30 s, rinsed with deionized water, and dried in room temperature air.

P: priming; After the surface of CAD/CAM resin composites was treated by C procedures, the priming agent was applied to the surface according to the manufacturer’s instructions.

SP: sandblasting with priming; After the surface of CAD/CAM resin composites was treated by
S procedures, the priming agent was applied to the surface according to the manufacturer’s instructions.

HFP: HF etching with priming; An aqueous solution of 9% HF was applied to the surface of CAD/CAM resin composites for 90 s, and then the surface was rinsed thoroughly under running water and cleaned in an ultrasonic bath with deionized water for 10 min. Then the priming agent was applied on the surface according to the manufacturer’s instructions.

Fifteen specimens were prepared for each experimental condition. The surface roughness (Ra, μm) after each surface treatment was measured using a surface roughness measuring instrument (Surfcom 470A, TOKYO SEIMITSU, Tokyo, Japan). For the specimen groups subjected to priming, the surface roughness was determined just before primer application.

3. Shear bond strength measurement
Before the bonding procedure, disc specimens made from the resin composite for core build-up were cleaned with phosphoric acid gel for 30 s, rinsed with deionized water, and dried. The surfaces of disc specimens were treated by priming prior to bonding, except for C. Masking tape (100 μm thick) with a 5 mm diameter circular hole was placed on the surface of each CAD/CAM resin composite to standardize the bonding area. Self-adhesive resin cement was mixed for 10 s using a plastic spatula on a mixing pad, according to the manufacturer’s instructions. The cement mixture was placed on the block surface, and the disc specimen of the core composite was placed centrally on the circular bonding area and fixed by finger pressure for 5 s. Any excess cement around the disc specimens was removed using a dental explorer after preliminary light curing for 6 s by curing lamp (G-Light Prima-II Plus, GC). The resin cement was further cured for an additional 6 s using the curing lamp. The specimens for the shear bond test were kept in a moisture box maintained at 95±5% relative humidity and 37±2°C.

The shear bond strength of the cement between the CAD/CAM resin composite and the core composite was determined at 24 h after bonding. ANOVA indicated significant effects (F=2.304, p<0.001) (Table 4). HFP had higher shear bond strength than C, S, and P (p<0.05). HFP had no significant difference in shear bond strength among C, S, and P and SP (p>0.05).

4. SEM observation after surface treatment
In order to examine the surface morphologies after the surface treatment, control specimens (C), sandblasted specimens (SP before priming) and etched specimens (HFP before priming) were observed using a scanning electron microscope (magnification ×1,000 and ×3,000, JSM-6360LV, JEOL, Tokyo, Japan). The specimens were coated with gold film (20 nm thick) and observed at 20 kV of acceleration voltage.

5. Failure mode analysis after shear bond test
After the shear bond test, the fracture surfaces were examined using a stereomicroscope (magnification ×100, STM-5, Olympus, Tokyo, Japan) and classified as either adhesive failure at the interface region, cohesive failure in the adherent region, or mixed failure mode of adhesive and cohesive failures25.

Cohesive failure was defined as fracture inside the resin composites of more than two-thirds of bonding area after the shear bond test. When two-thirds or more of the adhesive surface was observed on debonded surface of the resin composite block or the resin composite core, it was classified as adhesive failure. The others were classified as mixed failure. The observation results were statistically analyzed using the Chi-squared test. The p-value less than 0.05 were considered as significant.

RESULTS
Shear bond strength
Figure 1a, b shows the shear bond strengths of self-adhesive resin cement between CAD/CAM resin composites and the resin composite for core build-up at 24 h after bonding. ANOVA indicated significant effects for the resin blocks (F=35.490, p<0.001) and surface treatment (F=97.203, p<0.001) on shear bond strength, and significant interactions were observed between the CAD/CAM resin composite and surface treatment (F=2.304, p<0.001) (Table 4).

1. Effect of surface treatments on shear bond strengths of each block
1) CS300
There were no significant differences in shear bond strength among C, S, P and SP (p>0.05). HFP had higher shear bond strength than C, S and P (p<0.05). No significant difference in shear bond strength was observed between SP and HFP (p>0.05).

2) CS270
No significant differences were found in shear bond strength among C, S and P (p>0.05). SP and HFP showed higher shear bond strengths than C, S and P (p<0.05) except for between S and HFP. No significant difference in shear bond strength was observed between SP and HFP (p>0.05).
Fig. 1  
a: Shear bond strengths between each CAD/CAM resin composite block and resin composite core material after different surface treatments. Same lower case letters indicate significant differences in bond strength of each resin composite block ($p<0.05$).

b: Shear bond strengths between CAD/CAM resin composite block and resin composite core material after each surface treatment. Same upper case letters indicate significant differences in bond strength after each surface treatment ($p<0.05$).

3) HC
There were no significant differences in shear bond strength among C, S, P and SP ($p>0.05$). HFP indicated higher shear bond strength than C, S and P ($p<0.05$). No significant differences were observed between SP and HFP ($p>0.05$).

4) KZR
No significant differences in shear bond strength were observed among C, S and P ($p>0.05$). SP indicated higher shear bond strength than C ($p<0.05$). HFP showed higher shear bond strengths than C, S and P ($p<0.05$). No significant difference in shear bond strength was observed between SP and HFP ($p>0.05$).
Table 4  Summary of analysis of variance in shear bond strengths

| Source                     | Type III sum of squares | df | Mean Square | F-value | p-value |
|----------------------------|-------------------------|----|-------------|---------|---------|
| Resin blocks (RB)          | 672.490                 | 6  | 112.082     | 35.490  | <0.001  |
| Surface treatments (ST)    | 1,227.910               | 4  | 306.978     | 97.203  | <0.001  |
| RB×ST                     | 174.612                 | 24 | 7.276       | 2.304   | <0.001  |
| Error                     | 1,547.467               | 490| 3.158       | —       | —       |
| Total                     | 3,622.479               | 524| —           | —       | —       |

5) AVE
SP and HFP showed higher shear bond strength than C and P (p<0.05). S had higher shear bond strength than C (p<0.05). There were no significant differences in shear bond strength between S and P, S and SP, S and HFP (p>0.05). No significant difference in shear bond strength was observed between SP and HFP (p>0.05).

6) EST
No significant differences were found in shear bond strength among C, S and P (p>0.05). SP and HFP had higher shear bond strength than C and P (p<0.05). There was no significant difference in shear bond strength between SP and HFP (p>0.05).

7) ENA
There were no significant differences in shear bond strength among C, S and P (p>0.05). SP and HFP showed higher shear bond strengths than C, S and P (p<0.05). There was no significant difference in shear bond strength between SP and HFP (p>0.05).

2. Effect of differences in resin blocks on shear bond strength after surface treatments
Effect of differences in resin blocks on each surface treatment are shown in Fig. 1b.
1) Surface treatment group C
Blocks EST, ENA and CS300 had greater shear bond strengths than blocks CS270 or HC.

2) Surface treatment group S
Blocks AVE and EST showed greater bond strength than blocks CS300, CS270 and HC (p<0.05). Significant lower strength was observed for block HC than blocks ENA and KZR (p<0.05).

3) Surface treatment group P
Blocks EST, KZR and ENA were significantly greater bond strengths than blocks CS270 or HC (p<0.05). Shear bond strength of AVE was significantly greater than that of HC (p<0.05).

4) Surface treatment group SP
Blocks EST, AVE, ENA and KZR showed significantly greater shear bond strengths than block HC (p<0.05). Block EST had greater bond strength than CS300 (p<0.05).

5) Surface treatment group HFP
Blocks EST, KZR and ENA revealed significantly greater shear bond strengths than block CS270 (p<0.05). Block EST had significantly greater bond strength than HC (p<0.05).

Weibull analysis
Figure 2 shows the Weibull plots of shear bond strength data. Table 5 lists Weibull moduli, scale parameters, and the strengths with 5% and 95% failure probability.

No significant statistical differences were found in Weibull moduli among the 5 different surface treatments (p>0.05) for all blocks. However, the scale parameters and the strengths with 5% and 95% failure probability of the shear bond strengths of SP and HFP were higher than those of C, S and P. A majority of C revealed the lowest strength with 5% and 95% failure probabilities among all treatments in all blocks.

SEM observation after surface treatment
Figures 3 and 4 show representative SEM pictures of control specimens and specimens after sandblasting or HF etching. Both sandblasting and HF etching had rough surface textures compared to control. The surface irregularity created by HF varied among the resin blocks used. For block HC, the surface morphologies after HF were different pattern from others. There were some gaps around spherical fillers in matrix resin after HF treatment. This might have been related to the SiO₂ filler particle size, particle shape, particle distribution in the matrix resin or SiO₂ filler content in the matrix resin. The surface texture of sandblasting was roughest.

Relationship between surface roughness and shear bond strength
Tables 6 and 7 summarize the surface roughness values after each surface treatment and the statistical results.

Figure 5 shows the relationship between surface roughness and shear bond strength.
ANOVA indicated significant effects for the resin blocks (F=17.331, p<0.001) and surface treatment (F=3.457.661, p<0.001) on surface roughness. Significant interactions were observed between the resin blocks and surface treatment (F=11.335, p<0.001) (Table 7).

1. Effect of surface treatments on surface roughness of each block
All blocks did not show significant differences in surface
Fig. 2  Weibull plots of shear bond strength between CAD/CAM resin composite blocks and resin composite core material.

**Table 5**  Weibull analysis of shear bond strength between CAD/CAM resin composite block and resin composite core material

| Block | Surface treatment | Weibull modulus±95% CI | Weibull scale parameter±95% CI (MPa) | 5% Failure probability (MPa) | 95% Failure probability (MPa) |
|-------|-------------------|------------------------|-------------------------------------|-----------------------------|-----------------------------|
| CS300 | C                 | 4.4±0.8                | 5.9±1.2                             | 3.0                         | 7.6                         |
|       | S                 | 5.2±0.6                | 6.2±1.1                             | 3.5                         | 7.6                         |
|       | P                 | 4.9±1.3                | 6.3±1.3                             | 3.4                         | 7.8                         |
|       | SP                | 4.2±1.5                | 8.0±1.4                             | 3.9                         | 10.4                        |
|       | HFP               | 3.9±1.4                | 10.4±1.4                            | 4.9                         | 13.8                        |
|       | C                 | 6.3±0.9                | 4.2±1.1                             | 2.6                         | 5.0                         |
|       | S                 | 5.9±0.4                | 5.5±1.1                             | 3.3                         | 6.7                         |
| CS270 | P                 | 5.3±0.5                | 4.8±1.1                             | 2.8                         | 5.9                         |
|       | SP                | 5.3±0.8                | 8.8±1.1                             | 5.0                         | 10.9                        |
|       | HFP               | 6.7±0.6                | 7.4±1.1                             | 4.8                         | 8.7                         |
|       | C                 | 3.0±0.8                | 4.3±1.5                             | 1.6                         | 6.2                         |
|       | S                 | 5.6±1.4                | 4.0±1.2                             | 2.3                         | 4.9                         |
| HC    | P                 | 4.1±0.8                | 4.6±1.2                             | 2.2                         | 6.0                         |
|       | SP                | 4.0±0.6                | 5.9±1.2                             | 2.8                         | 7.7                         |
|       | HFP               | 3.9±0.3                | 7.9±1.1                             | 3.7                         | 10.4                        |
|       | C                 | 6.0±0.8                | 4.9±1.1                             | 3.0                         | 5.9                         |
|       | S                 | 4.2±0.7                | 6.9±1.2                             | 3.4                         | 9.0                         |
| KZR   | P                 | 5.1±1.2                | 6.9±1.2                             | 3.9                         | 8.6                         |
|       | SP                | 4.1±0.6                | 10.0±1.2                            | 4.8                         | 13.0                        |
|       | HFP               | 4.2±0.4                | 10.4±1.1                            | 5.2                         | 13.5                        |
|       | C                 | 3.5±0.7                | 5.7±1.3                             | 2.5                         | 7.8                         |
|       | S                 | 3.9±0.8                | 9.2±1.3                             | 4.3                         | 12.2                        |
| AVE   | P                 | 4.7±0.9                | 6.6±1.2                             | 3.5                         | 8.3                         |
|       | SP                | 4.8±0.6                | 10.8±1.1                            | 5.8                         | 13.5                        |
|       | HFP               | 4.1±0.5                | 9.5±1.1                             | 4.6                         | 12.4                        |
|       | C                 | 3.2±0.7                | 6.9±1.3                             | 2.7                         | 9.7                         |
|       | S                 | 5.8±0.7                | 8.6±1.1                             | 5.1                         | 10.4                        |
| EST   | P                 | 3.7±0.6                | 7.5±1.2                             | 3.4                         | 10.1                        |
|       | SP                | 4.3±0.9                | 11.7±1.2                            | 5.9                         | 15.0                        |
|       | HFP               | 4.9±0.5                | 11.1±1.1                            | 6.1                         | 13.9                        |
|       | C                 | 4.1±0.8                | 6.7±1.2                             | 3.2                         | 8.8                         |
|       | S                 | 9.5±1.3                | 7.2±1.1                             | 5.3                         | 8.0                         |
| ENA   | P                 | 4.3±1.3                | 6.9±1.3                             | 3.5                         | 8.9                         |
|       | SP                | 4.8±0.6                | 10.7±1.1                            | 5.7                         | 13.4                        |
|       | HFP               | 5.1±0.9                | 10.3±1.2                            | 5.8                         | 12.7                        |
roughness between C and P or between S and SP ($p>0.05$).
1) CS300, CS270, HC, AVE and EST
Surface roughness after S and SP was significantly greater than other treatments ($p<0.05$). No significant differences in surface roughness were found among HFP, P and C ($p>0.05$).

2) KZR and ENA
Treatments S and SP showed significantly greater surface roughness than P and C ($p<0.05$). Surface roughness after SP was greater than HFP which revealed greater surface roughness than C ($p<0.05$).

2. Effect of differences in resin blocks on surface roughness after surface treatments
1) Surface treatment group C
For control (C), surface roughness of AVE or KZR was significantly lower than those of CS300, CS270 and HC ($p<0.05$).

2) Surface treatment group S
After sandblasting (S), HC revealed significantly greater surface roughness than EST, CS300, KZR and
**Table 6** Surface roughness of CAD/CAM resin composite blocks after surface treatments

| Block      | C (μm) | S (μm) | P (μm) | SP (μm) | HFP (μm) |
|------------|--------|--------|--------|---------|----------|
| CS300      | 0.14 (0.05)^abA | 1.59 (0.26)^bcG | 0.11 (0.04)^b | 1.66 (0.28)^abG | 0.18 (0.04)^cD |
| CS270      | 0.13 (0.04)^abcF | 1.71 (0.14)^abA | 0.13 (0.06)^cd | 1.66 (0.22)^bcE | 0.21 (0.06)^bcF |
| HC         | 0.16 (0.05)^abcDG | 1.98 (0.12)^abDPGH | 0.13 (0.04)^abHI | 1.93 (0.31)^abAD | 0.25 (0.08)^cdD |
| KZR        | 0.08 (0.02)^abcEFG | 1.46 (0.23)^abcEFG | 0.09 (0.03)^d | 1.54 (0.26)^cABC | 0.19 (0.03)^cD |
| AVE        | 0.07 (0.03)^abcABD | 1.77 (0.16)^abBE | 0.08 (0.04)^abcAB | 1.97 (0.29)^abBCFG | 0.12 (0.03)^cABD |
| EST        | 0.11 (0.05)^cd | 1.64 (0.17)^abcHI | 0.09 (0.05)^abc | 1.61 (0.15)^abcF | 0.20 (0.06)^cGH |
| ENA        | 0.12 (0.04)^abc | 1.32 (0.26)^abcABD | 0.13 (0.04)^abcA | 1.58 (0.11)^abcDE | 0.46 (0.11)^abcFGH |

μm, Mean(SD)

Same lower case letters indicate significant differences in surface roughness of each resin composite block (p<0.05).

Same upper case letters indicate significant differences in surface roughness after each surface treatment (p<0.05).

**Table 7** Summary of analysis of variance in surface roughness after surface treatments

| Source          | Type III sum of squares | df  | Mean square | F-value | p-value |
|-----------------|-------------------------|-----|-------------|---------|---------|
| Resin blocks (RB) | 2.204                   | 6   | 0.367       | 17.331  | <0.001  |
| Surface treatments (ST) | 293.114                | 4   | 73.279      | 3,457.661 | <0.001  |
| RB×ST           | 5.765                   | 24  | 0.240       | 11.335  | <0.001  |
| Error           | 10.385                  | 490 | 0.021       | —       | —       |
| Total           | 311.468                 | 524 | —           | —       | —       |

**Fig. 5** Relationships between surface roughness and shear bond strength of CAD/CAM resin composite blocks.

ENA (p<0.05). Block AVE was found to be significantly greater surface roughness than blocks KZR and ENA (p<0.05). Surface roughness of CS270 was significantly greater than that of ENA (p<0.05).

3) Surface treatment group P

Block AVE showed significantly lower surface roughness than blocks ENA and HC (p<0.05).
4) Surface treatment group SP
Block AVE revealed significantly greater surface roughness than blocks CS300, EST, ENA and KZR ($p<0.05$). Significantly greater surface roughness was found in block HC than blocks ENA and KZR ($p<0.05$).

5) Surface treatment group HFP
Surface roughness of ENA was significantly greater than those of CS300, CS270, EST, KZR and AVE ($p<0.05$). Block AVE showed significantly lower surface roughness than HC, CS270 and EST ($p<0.05$).

There were significant positive correlations between surface roughness after surface treatment and shear bond strength for blocks CS270, AVE, EST and ENA ($p<0.05$). No significant correlations were observed for blocks CS300, HC and KZR ($p>0.05$).

Failure mode analysis after bond strength tests
Figure 6 shows the failure mode distribution of blocks after shear bond testing. The Chi-squared test indicated that the surface treatment process affected the failure mode distribution for blocks CS270, AVE, EST and ENA ($p<0.05$, Table 8).

All blocks showed adhesive failure with C treatment. For S treatment, blocks CS300, CS270, HC, KZR and ENA showed adhesive failure. Although a few specimens of blocks AVE and EST showed cohesive and/or mixed failure with S treatment, adhesive failure was still the dominant failure mode in AVE and EST. For P treatment, all of the blocks showed adhesive failure, except for block ENA which showed cohesive and mixed failure modes.

Blocks CS300, HC and KZR showed only adhesive failure with SP treatment. Cohesive failure was dominantly found with SP treatment for blocks CS270,

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![Figure 6](image-url)  
Fig. 6 Failure mode distribution of different surface treatments after shear bond strength test between CAD/CAM resin composite block and resin composite core material.

| Block | Chi-square | $p$-value | Significance |
|-------|------------|-----------|--------------|
| CS300 | 0          | 1         | not significant |
| CS270 | 62.90323   | <0.001    | significant |
| HC    | 12.5       | 0.13025   | not significant |
| KZR   | 0          | 1         | not significant |
| AVE   | 43.36134   | <0.001    | significant |
| EST   | 33.51852   | <0.001    | significant |
| ENA   | 59.77564   | <0.001    | significant |
AVE, EST and ENA. All of the specimens of blocks CS300, CS270 and KZR showed adhesive failure with HFP treatment, and blocks HC and EST dominantly had cohesive failure. In contrast, block ENA showed only cohesive failure. Cohesive failure mode was found in nine specimens of block AVE after HFP treatment.

**DISCUSSION**

It is known that two experimental setups have been commonly used to investigate the bond strength of dental materials. The setup composes of a single adhesive interface between two materials as the case of adhesive and substrate materials\(^{24,26-30}\). Other is the setup with two adhesive interfaces among three materials as the case of two substrate bonded with one adhesive\(^{18,22,23,31-33}\). Since a dental luting cement is the material which is applied with a form of very thin layer, it could be hard to determine the true adhesive strength between the cement layer and the substrate such as CAD/CAM resin composite or core build-up materials in this study. The present study focused to examine the shear bond strength using the experimental setup that CAD/CAM resin composite was cemented with resin composite abutment using resin cement.

This study examined the effects of surface treatments on bond strength between seven different resin composites for CAD/CAM and a core build-up resin composite using a self-adhesive resin cement. Since we wished to clarify the surface treatment effects on different CAD/CAM resin composites, only one adhesive resin cement and one priming agent, from the same manufacturer and of common clinically-used materials, were employed. The priming agent (G-Multi primer) contained MDP (10-methacryloyloxyeyecyl dihydrogen phosphate), MDTP (10-methacryloyloxyeyecyl dihydrogen triphosphate) and γ-MPTS as adhesive components. The self-adhesive resin cement (G-CEM ONE) contained MDP and methacrylic acid ester for adhesion. It is well known that γ-MPTS is an effective priming agent to bond SiO\(_2\) filler particles in resin composites, and that MDP or methacrylic acid ester provides a priming effect on the resin matrix in composites\(^{17,19,22,23,34}\).

Ceramic surface treatment using hydrofluoric acid is an ordinary treatment method to enhance the bond strength of repaired ceramic restoration or cemented crown. Güngör et al.\(^{36}\) and Elsaka\(^{37}\) both used 9% hydrofluoric acid to resin-ceramic hybrid materials for pretreatment of repairing system. Elsaka\(^{37}\) reported that the 90-s etching time was found to enhance the bond strength of hybrid ceramic material. According to these previous reports, this study used 9% hydrofluoric acid for 90 s to CAD/CAM restorative materials.

In the recent previous studies, the surface treatment using hydrofluoric acid has been recognized as the most accepted surface treatment for glass ceramics\(^{20,38}\), but also resin composites\(^{38}\), or hybrid ceramics\(^{21,29,30}\), to enhance the bond strength with resin cement. According to these previous studies in which HF etching was recommended to be followed by silanization before bonding, the present study carried out HF etching with priming agent which contained silane coupling component, γ-MPTS. Thus, it would be needed to note the limitation of experimental results for HF etching group in this study.

There were no significant differences in shear bond strength values between priming (P) and control (C) treatments for all blocks. Although the primer was not used in the control (C) group, the monomers such as MDP and methacrylic acid ester in the self-adhesive resin cement could have acted as priming agents for chemical bonding. This may help increasing the bond strength of the control without priming when the self-adhesive resin cement is used. The silane coupling agent, γ-MPTS, in the priming agent might not significantly influence bonding to the CAD/CAM resin composite surface without sandblasting. In addition, as seen in Figs. 3 and 4, the control specimens have scratches because of polishing procedures. These scratches might contribute to lift the bond strength of the control groups by mechanical interlocking effect.

Only in block AVE was the bond strength of sandblasting (S) greater than that of the control (C). This suggested that a single surface treatment, either sandblasting for mechanical interlocking or priming agent for chemical bonding, was not effective to increase the bond strength compared to the control (C), except in block AVE. The surface appearance in block AVE after sandblasting demonstrated more sharp ridges and riffs than in blocks CS300, CS270, HC and KZR. We consider this surface morphology to have been a factor in the higher bond strength observed after pretreatment by sandblasting (S) in block AVE compared to the control (C).

Yoshihara et al.\(^{31}\) examined the bond strength of self-adhesive resin cement between zirconia specimens and composite CAD/CAM blocks treated with different surface treatments. They reported that greater bond strength was found in the group treated with sandblasting before priming than with solely sandblasting. The present results may suggest a trend that the application of priming after sandblasting would improve the shear bond strength. These results were similar to those reported by Yoshihara et al.\(^{31}\). This is possibly explained by the following mechanisms: sandblasting of the CAD/CAM resin composite block.
yields surface roughness, i.e., the surface area to be bonded increases and exposes more filler particles on the resin matrix surface, and then the priming agent, especially γ-MPTS, reacts with the filler particle surfaces increased by sandblasting to cause more chemical bonding between the CAD/CAM resin composite block and resin cement. Thus, greater bond strength could be obtained by combining sandblasting and priming.

El-saka et al. reported the micro-tensile bond strength (μTBS) between CAD/CAM restorative materials with different surface treatments and resin composite using self-adhesive resin cement. Their results revealed higher μTBS in the group treated with HF before silane coupling agent than in those treated with sandblasting alone or sandblasting before silane coupling agent. Hori et al. investigated the effect of surface treatment of an indirect resin composite with 1% HF for up to 10 min on the bond strength of self-adhesive resin cement. Application of HF significantly increased the bond strength compared to the specimens without HF. Although there were no statistical differences in bond strength among the HF-treated groups, the specimens treated for 5 min showed greater bond strength than the others.

It is well known that HF can dissolve SiO2 glass ceramics. Venturini et al. reported that HF acid etching induce an increased wettability on the resin cement, in response to an increase in surface area. Increased wettability is associated with a lower contact angle and greater bonding potential among substrates. HF treatment of CAD/CAM resin composite makes the surface rough by dissolving SiO2 filler particles on the surface. Because the polymer network in resin composites is not sensitive to reaction by HF, the remained polymer network after HF treatment provides the sites of chemical adhesion with resin cement. Although the surface treated by sandblasting also has a rough appearance, its appearance was different from that treated with HF (Figs. 3 and 4). The many small surface dents observed in specimens treated with HF were likely derived from dissolved filler particles in the resin matrix, while sandblasting produced a much coarser surface (Figs. 3 and 4). Extremely uneven and deep irregularity of the adherend surface would reduce the bond strength compared to a surface which has adequate roughness, because air bubbles would remain in deep dents and/or the cement would not spread evenly spread over the surface. Hori et al. reported that the bond strength between indirect resin composite and indirect resin composite using resin cement treated with application of 1% HF for 10 min was lower than those for 30 s or 5 min. Prolongation of HF treatment time is known to increase the roughness of treated surfaces. This indicates that there is an optimal roughness for bonding, allowing sufficient penetration of adhesives into surface dents.

The sandblasting treatment for resin composite materials are well known to affect the surface roughness and the bond strength. The previous studies examined varied range of sandblasting conditions such as 0.1 to 0.5 MPa of air pressure and 5 to 60 s of sandblasting time etc. These previous studies indicated that the optimum sandblasting conditions existed to enhance the bond strength of resin composites using resin cements. Although the air pressure of sandblasting is recommended from 0.1–0.2 MPa (AVE and EST) and 0.2–0.3 MPa (HC and KZR) by the manufacturers, the current study fixed the sandblasting air pressure to be 0.3 MPa for 5 s for all blocks. This is because the conditions used provided the range of 1.32 to 1.98 μm of surface roughness (Table 6). These roughness values are considered to be within or close to an equivalent range of recommended sandblasting conditions for the CAD/CAM blocks reported in the previous studies.

The surface roughness values of CAD/CAM resin composite after HF treatment (HFP) ranged from 0.12 to 0.46 μm, while those after sandblasting treatment (SP) ranged from 1.32 to 1.98 μm. SEM photographs clearly showed the difference in appearance between HF-treated surfaces and sandblasted surfaces (Figs. 3 and 4). The sandblasted groups (S and SP) showed more roughness but did not always have greater bond strength than the HF etching group (HFP). These results indicated that a rougher surface did not necessarily result in greater bond strength. Surface roughening affects bond strength, but is not the only factor.

There were statistical differences in surface roughness among the blocks even though same surface treatment was carried out. The surface morphologies after polishing of blocks were related to their hardness or composition of each block. In sandblasting, blasted alumina particles gouge the resin composite block surface and debond filler particles from the resin matrix or expose them on the surface. In HF treatment, HF dissolves SiO2 filler particles from the surface, leaving numerous pits. Because the priming agent and the resin cement can penetrate into these pits, bond strength similar to that obtained after sandblast treatment could be achieved even though the surface roughness values after HF etching were lower than those after sandblasting. In addition, surface roughening by HF etching would be influenced by the particle sizes, shapes and dispersion conditions of inorganic fillers, the filler contents, etc.

Resin block ENA after HF treatment (HFP) showed the greatest surface roughness value and a different surface morphology among the blocks under SEM examination (Fig. 4). The bulk structure of block ENA is different from other resin blocks because ENA is made from a frame of feldspar ceramic with an infiltrated ceramic components such as SiO2. The surface of ENA is thus prone to etching by HF treatment and had the greatest surface roughness value among the resin blocks, because HF dissolved SiO2 glass ceramics in matrix phase.

The results of the present study suggested that combining surface treatments can yield greater bond strength of self-adhesive resin cement to a CAD/CAM resin composite, by roughening the surface for...
mechanical bonding and priming for chemical bonding. No statistical differences were found in the bond strengths of blocks treated with HF and priming (HFP) from those with sandblasting and priming (SP). This surface texture obtained by HF treatment is considered to have advantage of bonding with resin cement. The present results indicate that HF etching, which is used for ceramic prosthesis surfaces, is also effective for increasing micromechanical interlocking when bonding CAD/CAM resin composites. Sandblasting generates more roughness than HF etching, but carries a risk of damage to the surface structure by creating cracks that could adversely affect bonding of restorative materials. HF etching is therefore considered to be a useful surface treatment for resin composites. Because the remained polymer network conditioned by silane containing primer has the advantage to make polymer-resin cement layer for bonding.

Bond strength data are, in general, likely to show a wide range. For example, in this study, the shear bond strengths of block CS300 etched with HF and primed ranged from 6.1 to 18.9 MPa, with a mean strength and standard deviation of 9.4±2.9 MPa. In addition to comparisons of mean bond strength data, the present study employed Weibull analysis of the results to assess the effectiveness of surface treatments on CAD/CAM resin composite blocks and the reliability of bonding. Weibull analysis provides parameters such as the Weibull modulus, the scale parameter, and the bond strengths with 5% or 95% failure probability. The strengths with 5% or 95% failure probability can be used to examine the surface treatment effects on bond strength of CAD/CAM resin composites based on the probabilistic fracture mechanics.

A higher Weibull modulus means that the shear bond strengths were more consistent within the same experimental conditions. The present study found no statistical differences in Weibull moduli among the treatments in all the blocks. In this Weibull analysis, the scale parameter was a bond strength with 63.2% failure treatments in all the blocks. In this Weibull analysis, the scale parameter was a bond strength with 63.2% failure probability regardless of the Weibull modulus. The scale parameter is another indicator that predicts the bond strength from scattered data. The scale parameters of sandblasting with priming (SP) and HF etching with priming (HFP) groups were found to be greater than those of other treatments in all the blocks. This suggests that the combined effects of surface roughening and priming are effective for strengthening the bonding of the blocks.

The bond strengths with 5% failure probability of sandblasting with priming (SP) and HF etching with priming (HFP) groups showed higher values than other treatment groups in all blocks. This may indicate that the sandblasting/priming and HF etching/priming treatments increased the reliability of shear bond strength at 5% proof load. The bond strength with 5% failure probability of the control (C) group was the lowest among the treatment groups examined in all blocks. Except for block KZR, the 5% failure probability strength increased in the order of priming (P) followed by sandblasting (S) in all blocks.

Blocks CS270 and AVE showed higher 5% failure probability strengths in the sandblasting with priming (SP) group than in the other treatment groups, while blocks CS300, HC, KZR, EST and ENA exhibited higher 5% failure probability strengths in the HF etching with priming (HFP) group than in the other treatment groups.

Statistical comparisons of average values of shear bond strength could not assess the strength with 5% failure probability, which assumes a 5% proof load of the bond strength. This implies that few debonding fractures would occur at load values less than the 5% failure probability strength. Although no blocks showed identical trends in the 5% failure probability strengths of the treatment groups, the sandblasting with priming (SP) and HF etching with priming (HFP) groups showed greater 5% failure probability strength in a majority of blocks than the other treatment groups. These procedures together with roughening and priming of the surface may increase the reliability of bonding.

In probabilistic fracture mechanics, the bond strength with 95% failure probability indicates the strength at which 95% of samples are assumed to fail. This implies that few samples will survive past the 95% failure probability strength when loaded. Thus, in this study, the specimens with greater than 95% failure probability bond strength suggested higher reliability of the bonding system. As with the 5% failure probability strength, either sandblasting with priming (SP) or HF etching with priming (HFP) treatment resulted in a higher 95% failure probability strength than the other treatments in all blocks. The strength values with 95% failure probability of the sandblasting with priming (SP) group were close to those of HF etching with priming (HFP) in a majority of blocks.

Figures 3 and 4 show the differences in surface appearance after treatment between sandblasting and HF etching resulting from the compositions of the blocks. These differences derive from the shapes, sizes, contents and/or distribution in matrix of filler particles. Use of the priming agent increased both the 5% and the 95% failure probability strengths of sandblasting with priming (SP) and HF etching with priming (HFP). Further investigation will be needed to explain in detail the effects of sandblasting and HF etching together with priming on the strengths with 5% or 95% failure probability.

Failure mode examination revealed that a majority of specimens in the control (C), sandblasting (S) and priming (P) groups exhibited adhesive failure at the bonding interface. The sandblasting group (S) dominantly showed adhesive failure mode, whereas the group of sandblasting followed by priming (SP) showed cohesive failure within resin blocks for blocks CS270, AVE, EST and ENA. All of the cohesive failures observed in this study occurred inside the resin blocks. Because the priming procedure after sandblasting treats both the exposed filler particles with silane coupling agent and the surrounding resin matrix surface with adhesive
monomer MDP, chemical bonding was created between the block and the cement. The bond strength of specimens primed after sandblasting (SP) may have exceeded the bulk strength of CAD/CAM resin composite blocks, so that the resin block specimens fractured cohesively. Block ENA specimens all showed cohesive failure after HF treatment (HFP). As stated above for ENA, because its structural characteristics resemble those of ceramics, the properties and failure behavior of ENA differed from the other CAD/CAM resin composite blocks. As seen in the SEM photograph of ENA (Fig. 4), HF eroded the feldspar ceramic regions of the surface and increased the surface area available to be bonded. By priming after HF treatment (HFP), the treated surface with a larger area of ceramic component was available for bonding with resin cement and could combine with the layer of cement at the adhesive interface. This is probably a reason why block ENA had a higher bond strength and showed cohesive failure in the bond strength test.

There are clinical reports of some resin composite crowns fabricated by CAD/CAM systems debonding from abutment teeth within one year after cementation. In addition to geometric factors of the abutment tooth such as abutment height, diameter or taper, the surface treatment procedures are believed to be an important factor in preventing crown detachment. The resin composites for CAD/CAM systems are highly polymerized by heat under pressure, and less residual monomer remains in the bulk as compared to direct filling resin composites \(^7\). Therefore, the surface of CAD/CAM resin composites is chemically inert \(^8\). This characteristic is known to be unfavorable for fine chemical bonding. Consequently, CAD/CAM resin composites require appropriate surface treatments for reliable bonding.

From the results of present study, the proposed hypothesis can be accepted: HF treatment of CAD/CAM resin composite is comparable with sandblasting treatment for increasing the bond strength between a CAD/CAM resin composite block and resin composite core using resin cement.

This study limited to examine the shear bond strength values at 24-h after the cementation. The durability of bonding behavior could not be extrapolated from the results of present study. Further investigation would be needed in future.

CONCLUSIONS

Under the present experimental conditions, the following conclusions were drawn:

1. Either sandblasting alone or priming alone of the CAD/CAM resin composite blocks before bonding was not effective in increasing the bond strength of self-adhesive resin cement between CAD/CAM resin composite blocks and resin composite for core build-up, except for block AVE.
2. Sandblasting with priming of CAD/CAM resin composite blocks increased the bond strength.
3. The bond strengths obtained by 9% HF etching with priming were comparable to those by sandblasting with priming.
4. Although no differences were found in Weibull moduli among all surface treatment groups, the scale parameters and the bond strengths of all blocks with 5% or 95% failure probability were greater in the CAD/CAM resin composite blocks treated with sandblasting or 9% HF etching before priming than those of the other surface treatment groups.

CONFLICT OF INTEREST

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