Effect of etching with low concentration hydrofluoric acid on the bond strength of CAD/CAM resin block

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INTRODUCTION

Dental restoration using a computer-aided design/computer-aided manufacturing (CAD/CAM) system has become the standard for esthetic dental treatment. Several materials, such as feldspathic porcelain, leucite-based glass ceramics, lithium disilicate glass ceramics, zirconia, and composite resin, can be used with CAD/CAM1-3). One such material is a composite resin block comprising a glass phase and a matrix resin phase. This block is an esthetic material with properties realized by controlling the temperature and pressure, increasing the compounding amount of filler, and using high levels of polymerization; therefore, these blocks can be effectively used for crowns and inlays4). Previous studies have reported that CAD/CAM resin blocks have higher fracture resistance, fatigue strength, and bond strength than glass ceramics4-7). Despite the rapidly increasing demand for these blocks, suitable pre-surface treatments are still being investigated. Several studies have considered the appropriate surface treatment for such blocks. For example, mechanical treatment methods such as airborne-particle abrasion3,8-14) and tribochemical treatment12), and chemical treatment methods such as silanization3,8,11) and hydrofluoric acid etching9,15), and phosphoric acid etching9,15), and hydrofluoric acid (HF) etching have been considered9,10,11,14).

There are many reports on the effectiveness of airborne-particle abrasion. However, the effect of airborne-particle abrasion on the surface of the CAD/CAM resin block has not yet been fully elucidated. It has been suggested that the block surface may be damaged by airborne-particle abrasion. CAD/CAM resin blocks have a lower Vickers hardness than glass ceramics, making it preferable to use a lower airborne-particle abrasion pressure (0.2-0.3 MPa)10); however, some reports found that the surface is not uniform under this pressure. Another study found that alumina particles remain on the block surface and inhibit adhesion17).

The necessity for silanization has also been outlined in many studies. When a silane coupling agent is applied to the surface of a block, a siloxane bond (Si-O-Si) is created through a dehydration–condensation reaction with the inorganic filler on the block surface. The reaction promotes the union of dissimilar materials8,11,18). Phosphoric acid treatment, which is being studied as a chemical bonding pretreatment performed in combination with silanization, is considered effective only for cleaning and has no effect on the bond strength. A CAD/CAM resin block, which is a composite material, is detrimental to adhesion because it contains two phases (a filler phase and a matrix resin) and is highly polymerized and free of residual monomers18). Therefore, a chemical pretreatment used in conjunction with silanization has also been investigated. Multiple studies have reported that phosphoric acid etching has the effect of cleaning without improving the bond strength15,19). Conversely, HF etching is effective in improving the adhesion strength by reacting with the glass component in the CAD/CAM resin block8,11,13). However, the resin block is a composite material whose composition differs from that of glass ceramics, and only a small amount of the silica reacts with HF. The structure, size, and blending amount of the silica filler varies with the type of block. Nevertheless, HF used for research is usually limited to the concentration that is commercially available for glass ceramics, i.e., ~9%. Considering the composition and structure of the CAD/CAM block, it is
conceivable that the HF concentration suitable for the glass ceramics will affect the properties of the material. However, there have been no reports investigating the concentration of HF used for etching CAD/CAM resin blocks. In this study, we investigated the concentration of HF etching suitable for CAD/CAM resin blocks and further examined the effect of HF. The purpose of this study was to evaluate the effect of HF etching on these blocks, and to verify the following hypotheses:

1. Compared with phosphoric acid etching, HF etching for the surface of CAD/CAM resin blocks improves the bond strength.
2. The concentration of HF etching suitable for these blocks differs from that of glass ceramics, and low concentration HF is suitable.

### MATERIALS AND METHODS

The materials assessed are listed in Table 1 and the experimental procedure is schematically illustrated in Fig. 1. Four types of CAD/CAM resin blocks were cut into slices (thickness: φ2.0 mm) using a low-speed cutting saw with a diamond disk (Isomet 11-1196 Low Speed Saw, Buehler, Lake Bluff, IL, USA). Each block was polished with #600 grit silicon carbide paper and ultrasonically cleaned for 300 s.

#### Chemical elemental analysis

The change in the elemental composition of the block surface caused by HF etching was measured. HF concentrations of 0.5, 1.0, 2.0, 3.0, 3.5, 4.0, and 9.5%, which is commercially available for glass ceramics, were employed. The surface of each block was examined via scanning electron microscopy (SEM; TM-3000, Hitachi, Tokyo, Japan) and energy dispersive X-ray spectroscopy (EDX; TM-3000+SwiftED3000, Hitachi, Tokyo, Japan). EDX analysis (accelerating voltage: 15 kV, analysis time: 100 s) was performed on an arbitrary surface of each block (n=10).

#### Three-dimensional (3D) laser microscopy observations

The surface roughness of the cut block before and after each surface treatment was analyzed with a 3D laser microscope (VK-X250 3D Laser Scanning Confocal Microscope, Keyence, Osaka, Japan).

#### SEM observations

Each prepared test specimen was placed in an acyclic ring (10×10×2 mm) and then embedded and fixed by injecting a self-curing resin (Palapress Vario, Heraeus

| Material | Product [Code] (Manufacturer/Lot) | Compositions | Mass% |
|----------|-----------------------------------|--------------|-------|
| CAD/CAM resin block | CERASMART [CS] (GC/1512211) | Silica, Barium glass | 71 |
| | Shofu HC [HC] (Shofu/091401) | Silica powder, micro fumed silica, Zirconium silicate | 61 |
| | KATANA [KA] (Kurary Noritake Dental/000106) | Mixed filler with colloidal silica, aluminum | 62 |
| | ENAMIC [EN] (VITA/40330) | Feldspar ceramic enriched with aluminum oxide | 86 |
| Pre adhesive surface treatment | Phosphoric acid etching (Kerr/31297) | 37% Phosphoric acid | |
| | 0.5, 1, 2, 3, 3.5, 4% Hydrofluoric acid | 0.5, 1, 2, 3, 3.5, 4% Hydrofluoric acid Polyacrylamidomethylpropane sulfonic acid | |
| | Porcelain etchant gel (BISCO/1500005145) | 9.5% Hydrofluoric acid Polyacrylamidomethylpropane sulfonic acid | |
| | Rocatec pre (3M ESPE/) | Alumina oxide 110 μm | |
| Surface treatment | Clearfil Photo Bond (Kuraray Noritake Dental/3A0026, 380024) | Catalyst: BisGMA, HEMA, MDP, hydrophobic aliphatic methacrylate, CQ Universal: ethanol, catalysts, accelerators | |
| | Clearfil Porcelain Bond Activator (Kuraray Noritake Dental/3P0029) | γ-MPTS, hydrophobic aromatic dimethacrylate | |
| Composite resin | Clearfil Majesty Flow (Kuraray Noritake Dental/1J0018) | TEGDMA, barium glass, microfiller, photoinitiator | |

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Table 1 Materials used in this study
Kulzer, Hanau, Germany) into the ring. Two types of adhesion pretreatment were then performed, namely:

a) The specimens underwent airborne-particle abrasion (Basic Quattro, Renfert, Hilzingen, Germany) from a distance of 10 mm with 110 μm aluminum oxide particles perpendicular to the surface (duration: 5 s, pressure: 0.2–0.3 MPa).

b) The specimens were etched with 9.5% HF for 90 s and subsequently cleaned for 60 s using an ultrasonic device.

Following adhesive application, the resin composite was incrementally filled on the surfaces. The specimens were cut perpendicular to the adhesion surface, and the adhesion interface was analyzed via SEM (S-4700, Hitachi) at 5 kV. The specimens were dried in a vacuum desiccator and sputtered (HPC-1S, Vacuum Device, Mito, Japan) with osmium for 30 s. For SEM observation, the samples were mounted with carbon adhesive tape on a specimen holder and coated with platinum.

Shear bond strength test
The embedded specimens that were etched for 30 s using 37% phosphoric acid were used as the control group. The experimental group included the specimens that were etched for 90 s with different concentrations of HF (n=12). The treated specimens were subsequently cleaned for 60 s in an ultrasonic device. Following adhesive application, the resin composite was incrementally applied to the surfaces. The flowable composite resin was packed into a stainless steel mold (10×10×2 mm) with round holes. All specimens were then stored in water for 24 h at 37°C. Half of the specimens were thermocycled in water at temperatures of 5–55°C with a 60 s dwell time per bath for 10,000 cycles (K178, Tokyo Giken, Tokyo, Japan). The shear bond strength of each specimen was then measured with a universal testing machine (Type1125, Instron, Caton, MA, USA) operating at a crosshead speed of 0.5 mm/min.

Statistical analysis
The shear bond strength values were statistically analyzed through three-way analysis of variance (ANOVA) and Tukey's multiple comparison test. Tukey's multiple comparison test was used to determine any significant differences between the groups. The level of statistical significance was set at 5%. The sample size (n=12) was based on a power analysis study designed for the detection of medium effect size differences. Furthermore, the data from each group were examined and analyzed using computer software (G*power3.1, Heinrich-Heine-Universität, Düsseldorf, Germany).

Failure mode analysis
The failure mode was assessed via SEM (TM-3000, Hitachi) examination of the fractured specimen surfaces after shear bond strength testing. The fracture modes were classified as adhesive failure, mixed failure or cohesive failure.

RESULTS

Chemical elemental analysis
Figure 2 shows the EDX analysis results, where the mean changes in the element quantities on the block surfaces before and after HF etching were measured. Silicon (Si) was detected in all CAD/CAM resin blocks.
Fig. 2  Elemental composition as determined via EDX analysis of the CAD/CAM resin block before and after HF etching.
Si (silicon), F (fluorine), Zr (zirconium), Al (aluminum), Ba (barium). CS, CERASMART; EN, VITA ENAMIC; HC, Shofu HC; KA, KATANA

Fig. 3  The surface topography of the CAD/CAM resin block.
The surface roughness of each block was changed via HF etching. However, the degree of change in the surface roughness differed with each block. CS, CERASMART; EN, VITA ENAMIC; HC, Shofu HC; KA, KATANA

|       | CS | HC | KA | EN |
|-------|----|----|----|----|
| Control | ![Image] | ![Image] | ![Image] | ![Image] |
| 0.5%   | ![Image] | ![Image] | ![Image] | ![Image] |
| 1.0%   | ![Image] | ![Image] | ![Image] | ![Image] |
| 2.5%   | ![Image] | ![Image] | ![Image] | ![Image] |
| 3.0%   | ![Image] | ![Image] | ![Image] | ![Image] |
| 3.5%   | ![Image] | ![Image] | ![Image] | ![Image] |
| 4.0%   | ![Image] | ![Image] | ![Image] | ![Image] |
In addition, zirconium (Zr) was detected in CS and HC, but barium (Ba) occurred in the CS group only. Trace amounts of aluminum (Al) occurred in each block. The concentrations prepared for this study (0.5%, 1.0, 2.0, 3.0, 3.5, 4.0%) and 9.5% HF, which is commercially available for surface treatment of glass ceramics, were used for all blocks. Moreover, the fraction of Si in each block decreased significantly with the 9.5% HF etching surface treatment. The fraction of Si in each block (except for the Si fraction of the CS group) changed only slightly with HF etching at a concentration of 0.5–4.0%. In the CS group, the percentage of Si decreased with increasing HF concentration for concentrations ≥1%.

3D laser microscopy observations
The 3D laser microscopy images in Fig. 3 show the surface roughness. Although abrasion was generated on the block surface after polishing, a fine structural change occurred on the surface after HF etching. The surface roughness of each block increased with HF etching. In the CS, HC, and KA groups, owing to varying HF concentrations, the surface roughness changed only slightly compared with that of the group that underwent #600 grit polishing only. However, the surface roughness of the EN group increased with increasing HF concentration.

SEM observations
Figure 4 shows the SEM images. A CAD/CAM resin block and a composite resin are shown on the left side and right side, respectively. The image of each block revealed a clear difference between the adhesion interface resulting from airborne-particle abrasion and

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Fig. 4  SEM observation of the adhesive interface between the CAD/CAM resin block and composite resin (×2,000). White arrow: Adhesive interface between the CAD/CAM resin block and composite resin. 1) CERASMART (CS), 2) Shofu HC (HC), 3) KATANA (KA), 4) VITA ENAMIC (EN). a: airborne-particle abrasion; b: etched 9.5% HF.
HF etching. Airborne-particle abrasion yielded a rough uneven surface. However, in the case of HF etching, a fine structural change occurred on the block surface and an adhesive layer was observed between the block surface and the composite resin.

Shear bond strength test and failure mode analysis

Mean shear bond strength values (MPa) and standard deviations (SD) of the tested materials are shown in Table 2 and Fig. 5. ANOVA revealed that the HF concentration ($p<0.01$, $F=17.9895$), TC ($p<0.01$, $F=17.3080$) had significant effects on shear bond strength.

Table 2  Shear bond strength (MPa) and types of failure mode

|       | Control     | 0.5%       | 1.0%       | 2.0%       | 3.0%       | 3.5%       | 4.0%       |
|-------|-------------|------------|------------|------------|------------|------------|------------|
|       |             |            |            |            |            |            |            |
| CS    | Before TC   | 27.4 (8.3) | 27.3 (5.5) | 39.2 (20.5)| 35.3 (5.5) | 31.7 (7.0) | 30.8 (5.2) | 34.3 (6.3) |
|       |             | 9/3/0      | 8/3/1      | 7/1/4      | 8/1/3      | 10/2/0     | 10/2/0     | 10/2/0     |
|       | After TC    | 9.5 (2.4)  | 11.3 (7.9) | 19.3 (6.4) | 22.6 (9.5) | 13.9 (3.7) | 17.6 (7.5) | 13.3 (5.6) |
|       |             | 12/0/0     | 11/1/0     | 10/2/0     | 15/0/0     | 12/0/0     | 12/0/0     | 12/0/0     |
| HC    | Before TC   | 20.8 (10.4)| 18.4 (4.8) | 28.1 (15.0)| 38.8 (16.5)| 42.2 (17.8)| 39.9 (18.1)| 34.1 (13.9)|
|       |             | 9/2/1      | 12/0/0     | 10/1/1     | 4/2/6      | 1/2/9      | 1/1/10     | 1/2/9      |
|       | After TC    | 9.6 (3.8)  | 11.7 (3.8) | 18.9 (19.0)| 27.7 (23.3)| 52.9 (29.4)| 52.0 (20.4)| 48.0 (38.4)|
|       |             | 12/0/0     | 12/0/0     | 10/2/0     | 7/2/3      | 2/5/5      | 0/8/4      | 0/7/5      |
| KA    | Before TC   | 32.0 (10.4)| 30.2 (4.8) | 25.4 (15.0)| 26.0 (16.5)| 32.4 (17.8)| 35.9 (18.1)| 37.7 (13.9)|
|       |             | 9/3/0      | 10/1/1     | 11/0/1     | 8/2/2      | 4/1/7      | 7/1/2      | 1/2/9      |
|       | After TC    | 27.7 (15.4)| 12.3 (8.1) | 14.6 (7.7) | 23.3 (9.3) | 30.9 (7.9) | 51.5 (30.8)| 41.9 (31.1)|
|       |             | 8/2/2      | 12/0/0     | 12/0/0     | 9/2/1      | 7/3/2      | 5/2/5      | 6/2/4      |
| EN    | Before TC   | 25.9 (3.8) | 21.7 (5.0) | 24.3 (3.9) | 20.5 (2.1) | 21.0 (3.2) | 22.3 (3.8) | 19.0 (8.8) |
|       |             | 5/4/3      | 2/5/5      | 5/2/5      | 6/3/3      | 6/2/4      | 6/2/4      | 8/2/2      |
|       | After TC    | 16.4 (4.2) | 33.1 (12.6)| 30.6 (14.9)| 27.4 (12.1)| 23.2 (10.1)| 25.8 (8.5) | 26.5 (8.8) |
|       |             | 6/6/0      | 1/1/10     | 0/1/11     | 2/3/7      | 3/2/7      | 3/3/6      | 4/3/5      |

Data are shown as mean (standard deviation). $n=12$

The number below the average data is the number of failure modes. From left to right: adhesive failure / mixed failure / cohesive failure.

CS, CERASMART; EN, VITA ENAMIC; HC, Shofu HC; KA, KATANA; TC, thermocycling.

Fig. 5  Shear bond strength.

The same letters of the alphabet indicate that the values are significantly different ($n=12$). CS, CERASMART; EN, VITA ENAMIC; HC, Shofu HC; KA, KATANA; TC, thermocycling.
In the CS group, both before and after thermocycling (TC), the mean values (MPa) obtained for the 2% group were significantly higher than those of the control groups. Before TC, many cohesive failure and mixed failure modes were observed in the 1 and 2% groups. The shear bond strength of each group decreased after TC and adhesive failure was observed in more than 90% of the specimens. In the HC group, the mean values of the 2 and 3% groups were considerably higher than that of the 0.5% group before TC. After TC, the mean values of the 3, 3.5, and 4% groups were significantly higher than those of the control group, 0.5, 1, and 2% groups. Both before and after TC, many cohesive and mixed failures occurred after ≥3% HF etching. The mean values of the groups comprising the KA group differed only slightly before TC. After TC, the mean value of the 3.5% group was significantly higher than those of the control, 0.5, 1, and 2% groups. Similarly, the mean value of the 4% group was significantly higher than those of the control, 0.5, and 1% groups. The 3.5 and 4% groups underwent many cohesive and mixed failures both before and after TC. The mean values of the groups comprising the EN group differed only slightly higher than those of the control group and many cohesive and mixed failures occurred in the low concentration HF group.

**DISCUSSION**

HF etching has been widely used for silicon-based glass ceramics such as feldspathic porcelain, leucite-based glass ceramics, and lithium disilicate glass ceramics. When HF is allowed to act on glass ceramics, the ensuing reaction dissolves the substrate (i.e., the substrate becomes porous), and yields micromechanical retention for the adhesive surface of glass ceramics.

\[
\text{SiO}_2 + 4\text{HF} \rightarrow \text{SiF}_4 + 2\text{H}_2\text{O} \\
4\text{SiF}_6 + 3\text{H}_2\text{O} + 2\text{HF} \rightarrow 3\text{H}_2\text{SiF}_6 + \text{H}_2\text{SiO}_3
\]

Previous studies have shown that HF reacts with the glass component of CAD/CAM resin blocks and improves the bond strength. However, the resin block is a composite material composed of filler particles and a resin matrix, and its composition differs from that of glass ceramics. We determined the optimal concentration of HF for the blocks. The EDX analysis results revealed that the fraction of Si in each block decreases significantly with 9.5% HF etching. As the concentration of HF increased, the reaction with Si progressed, possibly leading to changes in the fraction of Si on the block surface. However, the amount of Si in the HC, KA, and EN groups remained approximately the same with etching at HF concentrations of 4% or less. In CS, barium glass constitutes approximately half of the glass component of the filler. Barium glass is non-reactive with HF and, hence, the amount of barium glass may increase with a gradual decrease in the fraction of Si. Since Si reacts with the silane coupling agent, a large amount of reactable surface-layer Si is essential for obtaining a high bond strength. Therefore, little or no effect of the surface treatment on the amount of Si is desired. This suggests that, since the HF concentration used for glass ceramics causes an excessive reaction that significantly alters the surface composition, concentrations of 0.5 to 4% are more suitable for CAD/CAM resin blocks. Three-dimensional laser microscope images revealed that the surface roughness generated after HF etching of the CS, HC, and KA groups was greater than that generated after polishing only. However, almost identical surface roughness was generated at the various HF concentrations. In the EN group, the surface roughness increased with increasing concentration. EN is a resin-impregnated porcelain produced by polymerizing an alumina-reinforced feldspathic ceramic porous body impregnated with a urethane dimethacrylate/triethylene glycol dimethacrylate monomer mixture. Among the CAD/CAM resin blocks used in this study, EN has properties closest to ceramics. This structural difference influences the degree of change in the surface roughness. The EDX analysis and 3D laser microscopy results revealed no correlation between increasing surface roughness and compositional changes. However, the SEM image of each block revealed clear differences between the adhesion interfaces resulting from airborne-particle abrasion and HF etching. The airborne-particle abrasion treatment generated large and gently sloping uneven grooves on the surface layer of the block, and resin infiltrated this surface roughness. A layer of Si dissolved in HF penetrated the bonding agent. Although airborne-particle abrasion is currently used as a bonding pretreatment of CAD/CAM resin blocks, the mechanical fitting force is weak. Mechanical surface treatments such as airborne-particle abrasion may lead to chipping and changes in the adhesion surfaces. For example, the surface becomes irregular, owing to direct strikes by the alumina particles, and the bond strength decreases after TC. In addition, depending on the type of CAD/CAM resin block, airborne-particle abrasion yields no improvement in the bond strength, because the block surface is damaged by the airborne-particle abrasion. The type of structural change induced by the HF etching treatment differs from that associated with airborne-particle abrasion. HF etching, a chemical treatment, can yield finer structural changes than those resulting from mechanical treatments, such as airborne-particle abrasion. HF etching selectively dissolves the filler on the block surface, thereby exposing the crystal phase to the adhesion surface and, in turn, generating many microporosities, undercuts, and grooves. An adhesive layer with a fine structure impregnated by a bonding agent is formed on the surface, and this layer exhibits micromechanical retention, which is beneficial for adhesion. The magnitude of the structural change on the surface and the type of structural change may both lead to improvements in the bond strength. From the results of the shear bond strength test and failure mode observations, as compared with HF etching, HF etching showed the same or better effect at all concentrations in...
all blocks.

Although the effective concentration of HF was different for each block, the optimum concentration other than CS showed significantly higher adhesion strength than phosphoric acid etching after thermal cycle, and HF etching compared to phosphoric acid etching. Hydrofluoric acid etching can be said to promote better adhesion strength and adhesion durability than phosphoric acid etching. The difference in the optimal concentration seems to be correlated with the composition and structure of each block. CS has a filler composition of 71 mass%, and the glass component of the filler is fine silica powder (particle size: 20 nm) and barium glass (particle size: 300 nm)\(^{31,32}\). In the CS group, the best bond strength was achieved at 2% both before and after TC, but nearly all the failures were adhesive failures occurring after TC. In contrast to its effect on the other blocks, the HF treatment is considered ineffective for the CS group. HC (filler composition: 61 mass%) contains the filler components: silica powder, microfumed silica acid, and zirconium silicate\(^{31,32}\). KA (filler composition: 62 mass%) contains 40 nm colloidal silica and 20 nm aluminum oxide\(^{8,15}\). The shear bond strength of the HC and KA groups increased significantly after \(\geq 3\%\) HF etching both before and after TC. Despite the differences in the type of filler, HF etching had a similar effect on the groups, owing to the similarity in the filler component ratios. In the EN group, the various concentrations before TC yielded only slightly different shear bond strengths. This group has a filler composition of 86 mass%, i.e., the highest of the blocks used in the experiment\(^{19}\). Moreover, EN (like glass ceramics) has a porous structure and, hence, the surface roughness of EN increased even with low concentration HF etching, i.e., the HF effect was exacerbated, owing to the porous structure\(^{45}\). High shear bond strengths were obtained following 0.5 and 1% HF etching after TC, and the EN group demonstrated higher bond strength durability after HF etching with lower concentrations (as opposed to higher concentrations). Other researchers have also reported that the increase in surface roughness caused by HF etching is larger in EN than in CS and HC\(^{17}\). Previous studies reported that, in the case of EN, etching with high concentrations of HF resulted in a decrease in the shear bond strength after TC\(^{11}\). The 3D laser microscopy results revealed that the surface roughness increased with increasing HF concentration, whereas the shear bond strength increased significantly with low concentration HF etching following TC. This improved the bond strength and durability, and suggests that both surface roughness and surface structural change are necessary for strong adhesion.

CONCLUSIONS

1) HF etching increases shear bond strength values between CAD/CAM resin blocks and flowable composite resin.

2) The optimum HF concentration varies with the type and composition of the resin block.

3) Low concentration HF yields only slight changes in the composition of the block surface but, nevertheless, modifies the surface.

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