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The Effect of Surface Conditioning Methods on the Bond Strength of CAD/CAM Composite Resin with Dentine

Xue Bing YAN[1], Deng Gao LIU[2], Cheng Wen CHAI[3], Yong WANG[4], Pei Jun LÜ[4]

Objective: *To evaluate the effect of surface conditioning methods on the bond strength of a self-developed dental CAD/CAM composite resin material (polymethylmethacrylate/nano* SiO_2*-*ZrO_2*) and dentine, and to select appropriate resin cements from three resin luting agents.*
Methods: *A total of 210 cylindrical composite resin blocks were divided into 7 groups (n = 30) and treated by 7 different surface conditions: (1) no treatment, (2) etching with phosphoric acid and application of adhesive agent, (3) silane coupling agent (S), (4) etching with hydrofluoric acid (HF) and silanisation, (5) sandblasting (Sa), (6) Sa + S, and (7) Sa + HF + S. Each group was further divided into three subgroups for application of three resin cements (RelyX ARC, Panavia F, or Variolink II) to bond the treated composite blocks and dentine. Subsequently, the shear bond strengths were measured and the failure mode examined. The composite surface treatments of 400-grit silicon carbide paper, PA, HF, Sa and Sa + HF were examined with a scanning electron microscope to determine the effect of these conditions.*
Results: *For the same resin cement, the three sandblasting treatment groups showed the highest bond strengths except for the Sa + S and Sa group when using Variolink II. For the same surface treatment, there were no differences among the three resin cements. After sandblasting treatment, the occurrence of adhesive failures at the composite–luting cement interface was greatly reduced. Sandblasting presented the greatest topographical relief with evident irregular morphological change.*
Conclusion: *Sandblasting treatment was the main factor responsible for improving the bond strength of indirect composite resin and dentine.*
Key words: *CAD/CAM, composite resin, dentine, shear bond strength, surface treatment*

At present, computer technology has revolutionised restorative dentistry, and numerous dental CAD/CAM systems have been developed and marketed for the fabrication of dental restorations. The machinable materials became commercially available at approximately the same time as the CAD/CAM system. Though CAD/CAM ceramic material has good mechanical properties, the material is susceptible to brittle fracture under stress-bearing conditions in the oral cavity. CAD/CAM composite blocks are produced as an alternative to ceramic blocks. Indirect composite inlays are now more routinely used because composite blocks show some advantages over ceramic blocks. In particular, they can be finished and polished, and subjected to add-on adjustments with relative ease. They are less harsh on natural dentition with regard to wear, and provide higher bond strengths

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[1] Department of VIP Dental Service, Outpatient Dental Center, Peking University School and Hospital of Stomatology, Beijing, P.R. China.

[2] Department of Oral Radiology, Peking University School and Hospital of Stomatology, Beijing, P.R. China.

[3] Department of Chemistry, Beijing University of Science and Technology, Beijing, P.R. China.

[4] Research Center of Engineering and Technology for Dental Computing, Ministry of Health, Peking University School and Hospital of Stomatology, Beijing, P.R. China.

Corresponding authors: Dr Pei Jun LÜ, Research Center of Engineering and Technology for Dental Computing, Ministry of Health, Peking University School and Hospital of Stomatology, Beijing 100081, P.R. China. Tel: 86-10-62179977 ext 2553; Fax: 86-10-62173402; E-mail: kqcadc@bjmu.edu.cn.
Yong WANG, Research Center of Engineering and Technology for Dental Computing, Ministry of Health, Peking University School and Hospital of Stomatology, Beijing 100081, P.R. China. Tel: 86-10-62179977 ext 2553; Fax: 86-10-62173402; E-mail: kqcadc@bjmu.edu.cn.

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and fracture resistance because of the similarity of the modulus of elasticity between the composite and the luting agent[1-3]. In addition, several *in vitro* studies found a more uniform stress distribution throughout teeth restored with indirect composite inlays, compared with ceramic inlays[4]. Short-term clinical trials and lifetime predictions reveal favourable success rates[5].

Recently, polymethylmethacrylate (PMMA)/nano SiO_2-ZrO_2 prefabricated composite resin blocks (14 mm in diameter and 18 mm in height), which were used for CAD/CAM, were developed for indirect inlay fabrication by a cooperative research group from Peking University School and Hospital of Stomatology and Tsinghua University (led by Professor Pei Jun LÜ and Professor Fu Zhai CUI). PMMA (55 Wt%) was selected as the resin matrix, and nano-SiO_2 particles (30 Wt%) sized from 20 nm to 30 nm and ZrO_2 particles (15 Wt%) sized from 100 nm to 200 nm were selected as filler particles. The method of *in situ* polymerisation was carried out to prepare the machinable PMMA/nano SiO_2-ZrO_2 composite resin for dental CAD/CAM. The three-point bending strength, elastic modulus, and micro-hardness of this composite resin were 112 MPa, 3413 MPa, and 28.4 MPa, respectively.

In contrast to the advantages of indirect composite restoration, bonding to the tooth structure is still a challenging issue, due to the fact that an indirect restorative procedure will increase the interfaces for bonding, one at the tooth structure and the other at the fitting surface of the restoration. In order to establish a strong and durable bond, which is necessary for the biomechanical aspect of the tooth-restoration system, appropriate treatment of the respective surfaces is crucial. Researchers have described several composite surface treatments to optimise the bond strength of composite and resin cement[3,6,7] or composite repair[8-10]. Table 1 summarises some of the studies in detail. The proposed surface treating methods include roughening, etching the substrate surface with phosphate acid or hydrofluoric acid (HF) gel, silane coupling agent, adhesive resins, air-borne particle abrasion, or the combined use of silica coating and silane.

Though etching with hydrofluoric acid followed by the application of the silane coupling agent was a well-known method[3], most researchers recommended roughening the composite surface by sandblasting or silica coating followed by silanisation as a predictable means to enhance the bond strength[6,8-10]. Despite this, for the new CAD/CAM composite, the effect of composite surface treatment on the adhesive properties of an indirect composite needs further research. In particular, the bond strength between the prefabricated composite of different surface treatments and the hard tissue of the tooth has been addressed by only a few authors[11].

Various investigations have shown that reliable resin bonds might improve the marginal seal and increase the fracture resistance of composite restorations[12,13]. With contemporary adhesive cements and a new generation of bonding systems, achieving a strong and durable bond between the tooth structure and indirect restoration

Table 1 Summary of research on the bond strength of different surface treatments of composite resin

Study	Test method	Mode	Treatment	Result
Stokes[6] 1993	Shear test	Composite-resin cement	Smooth, Sa, Sa + HF, Sa + S, Sa + HF + S, HF, HF + S, S	5.9–19.9Mpa; the highest bond strength was achieved by Sa + HF + S, Sa + HF and Sa + S treatment
Yoshida[7] 2001	Shear test	Composite-resin cement-composite	S, Adh	21.5–34.25 MPa; the application of silane improved the bond strength
El Zohairy[3] 2003	Microtensile test	Composite-resin cement	Control, Adh, HF + S, HF + S + Adh	14.1–59.4 MPa; Adh use improved the bond strength; HF + S + Adh achieved higher bond strength
Ozcan[8] 2006	Microtensile test	Composite-composite	Silica coating + S, PA + Adh	6.1–52.3 MPa; chairside silica coating and silanisation provided higher bond strength
Papacchini[9] 2007	Microtensile test	Composite-composite	Sa + PA, HF + HCl, diamond bur + PA, diamond bur	37.3–43.2 MPa; sandblasting resulted in the strongest bond
Brendeke[10] 2007	Shear test	Composite-composite	Silica coating + S, S + Adh	4.6–11.65 MPa; chairside silica coating and silanisation provided the highest bond strength

S, silane; Adh, adhesive agent; PA, phosphoric acid; HF, hydrofluoride acid; HCl, hydrochloric acid; Sa, sandblasting

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becomes feasible. However, the chemical composition of selected cementing agents and corresponding adhesive systems influence the composite–dentine bond. The RelyX™ ARC system (3M ESPE, St. Paul, MN, USA) is typically used for the conventional cementation of tooth-coloured restorations, with a Bis-GMA-based resin luting agent and a Bis-GMA monomer adhesive (Single Bond). However, this requires multiple steps of total etching, adhesion, and moisture control. The recently developed self-etch resin luting agent Panavia™ F (Kuraray Co. Ltd, Osaka, Japan) (containing the MDP prime agent) demonstrates strong bonds to dentine, metal, zirconia[14] and aluminous ceramics[15]. The dual-cured resin cement Variolink® II (Ivoclar Vivadent, Schaan, Liechtenstein) seems to have positive bond effects on ceramics.

The purpose of the present study was to evaluate the effect of surface conditioning methods on the bond strength of CAD/CAM composite resin and dentine with a shear test and to select an appropriate resin cement from three dual-cured resin luting agents.

Materials and Methods

Tooth preparation

A total of 210 freshly extracted, non-carious permanent human molars were selected for the present study. The calculus and residual periodontal tissue were removed using a surgical knife, scaler and curette. All teeth were stored in 0.9% saline solution at 4°C for 10 to 20 days. The occlusal enamel was removed using a slow-speed saw (Isomet™, Buehler, Lake Bluff, IL, USA) under water. The teeth were placed in a steel mould and embedded in methylmethacrylate resin. The dentine bonding surfaces were then grinded with a polishing machine (Weiyi M-2, Yangjiang Weiyi Polishing Material, Yangjiang, China) using 600-grit silicon carbide (SiC) paper under running water to create a smear layer of clinically relevant thickness on the surface of the coronal dentine. The bonding surfaces were examined under a stereoscopic microscope (Nikon SMZ10, Tokyo, Japan) to ensure that they were free of retained enamel.

Specimen groups and bonding procedures

A total of 210 cylindrical composite blocks (5 mm in diameter, 3 mm in thickness) were fabricated from the prefabricated composite blocks. The bonding surfaces were wet grinded with a polishing machine using 400-grit SiC paper. All the blocks were then ultrasonically cleaned for 5 min in distilled water and air-dried.

Before cementation, all the specimens were randomly divided into seven main groups (n = 30 for each group) and conditioned by one of the following:

- group 1: no further surface treatment was applied to this control group (no treatment)
- phosphoric acid (Total Etch, Ivoclar-Vivadent) for 1 min, washed thoroughly, dried, and a thin layer of adhesive was applied (provided by the manufacturer of each cement) (PA + Adh)
- group 3: silane solution (provided by the manufacturer of each cement, Table 1) was applied for 60 s (S)
- group 4: the specimens were etched with 8% HF (Ivoclar-Vivadent) for 2 min, washed thoroughly for 1 min under running water, dried, and then silane was applied as described in group 3 (HF + S)
- group 5: air-borne particle abrasion with 50 µm Al_2O_3 particles was applied at a pressure of 2.8 bars for 10 s using an air-abrasion device (Korostar Z, Bego, Bremen, Germany), the tip of the micro-etcher was kept 10 mm away from the surface of the specimens, and the specimens were rinsed under running water to remove the debris (Sa)
- group 6: the specimens were sandblasted as described in group 5, and then the silane coupling agent was applied as described in group 3 (Sa + S)
- group 7: the specimens were sandblasted, and then HF and silane were applied (Sa + HF + S).

For each group, three resin cements (RelyX ARC, Panavia F or Variolink II) were applied to bond the treated composite blocks and dentine (n = 10). A 3 mm^2 bonding area of approximately 100 µm thickness was limited by a piece of polyethylene tape. The adhesive interface was light cured (XL 3000, 3M ESPE, 450 to 500 mW/cm^2 output) under a load of 5 N from four directions for 20 to 40 s on each side. Before the complete hardening of the resin luting agents, any excess cement was removed.

All of the bonding procedures were carried out in accordance with the manufacturer's instructions by the same operator throughout the experiments.

Shear strength tests

After 24 h of storage in distilled water at 37°C, each specimen was embedded in an acrylic resin mould and arranged in an ISO/TR 11405 shear testing jig. The shear bond strengths (SBS) of the specimens were measured with a universal mechanical testing machine (AGS500, Shimadzu, Kyoto, Japan) at a crosshead speed of 0.5 mm/min. The calculated shear bond strength was determined by dividing the force at which bond failure occurred by the bonding area. The broken specimens

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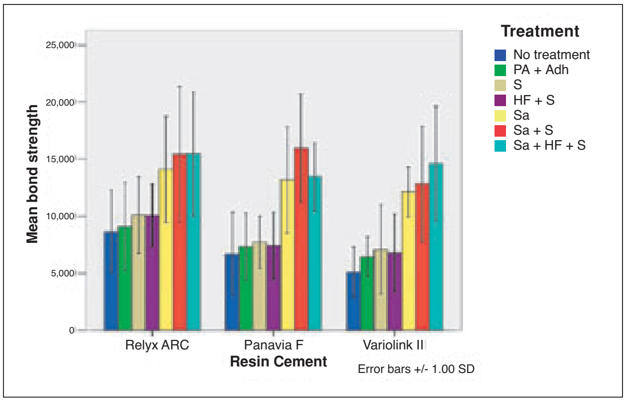

Fig 1 The mean shear bond strength values (MPa) and standard deviations for seven surface treatments and three resin cements in bar graphs.

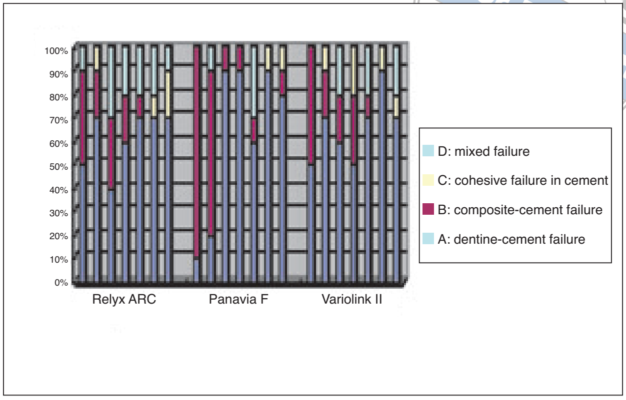

Fig 2 Graphical presentation of the proportional prevalence of fracture modes among different groups.

were examined with a ×50 magnification stereomicroscope (Nikon SMZ645) to determine the failure mode. The fractured surfaces were classified into one of the following types: A, adhesive failure at the dentine–cement interface; B, adhesive failure at the composite–cement interface; C, cohesive failure in the resin cement; and D, mixed A and B.

Fourier transform infrared spectroscopy analysis

In an additional experiment, Fourier transform infrared spectroscopy (FTIR-8400S, Shimadzu) was used to analyse the mechanism of the silane-coupling agent. The reflection FT-IR microscopy was operated under the following conditions: 4000 to 400 cm^{-1} range, 4 cm^{-1} resolution, 20 ± 1°C temperature and 85 to 90% humidity. Spectroscopy was performed for seven specimens, including the untreated composite, three silane-coupling agents, and three composites treated by corresponding silanisation.

SEM analysis

Five additional composite blocks of surface treatments with 400-grit SiC paper (as control), PA, HF, Sa, and Sa + HF were made, and the surface topography was examined under the scanning electron microscope (SEM, Phillips SEM XL 20, Eindhoven, Holland) to determine the effect of these conditions. The specimens were prepared, carbon sputtered and examined under SEM. The surface structures were viewed and photographed at original magnifications of ×2000.

Statistical analysis

The data were expressed as the mean and standard deviation for each group analysed. Statistical analysis was performed using SPSS 11.0 (SPSS, Chicago, IL, USA) software for Windows. A two-way ANOVA ($\alpha = 0.05$) was performed with the bond strength as the dependent variable, and surface treatment and cement type as independent factors.

Results

Shear bond strengths

SBS test results are shown in Figure 1. The two-way ANOVA revealed a significant influence of the surface treatments ($P < 0.001$) and the resin cements on the test results ($P = 0.001$). Further analysis was done by means of a least significant difference test of one-way ANOVA. The results show that for RelyX ARC and Panavia F, the three sandblasting treatment groups had significantly higher bond strength than the three groups of chemical treatment and the no treatment group ($P < 0.05$). No statistically significant differences were found among the three sandblasting treatment groups, or among the four non-sandblasting treatment groups. For Variolink II, the Sa + HF + S treatment group gave the highest bond strength compared with the four non-sandblasting treatment groups but not significantly different compared to Sa and Sa + S treatment groups. There were no significant differences among S, HF + S, Sa and Sa + S treatment groups. For the same surface treatment, there were no significant differences among the three resin cements.

Figure 2 shows a graphical presentation of the proportional prevalence of failure modes for different treatment methods and resin cements. With regard to the failure modes, cohesive failures within dentine or composite resin were not found in all loaded specimens. The overall percentages of the four different failure modes were

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63.3%, 21.4%, 5.8% and 9.5% for A, B, C and D, respectively. Adhesive failure at the dentine–cement interface was a predominant type of failure except for the no treatment and PA + Adh group using Panavia F. There were similar failure modes among the three resin cements. Going from the control group and chemical treatment groups to the sandblasting groups, there was a gradual reduction in the occurrence rate of adhesive failures at the composite–luting cement interface. In the Sa + S group, no composite–cement interface failures were found.

Fourier transform infrared spectroscopy analysis

Figure 3 shows the representative FT-IR spectra. For each of the three silane coupling agents, the spectra of the composite, the coupling agent, and the silanised composite were comparatively analysed. New peaks were detected in the three silane coupling agent treatments, which belong to the stretching and bending vibrations of Si-O-Si groups (1087 to 1112 cm^{-1}, indicated by the blue arrow).

SEM observations

The SEM analysis showed the representative micrographs of composite surfaces after different surface treatments. Grinding by 400-grit SiC paper produced slight surface scratches (Fig 4a). Phosphoric acid etching had no effect on the topography, but a cleaning effect (Fig 4b). HF etching resulted in a moderate amount of surface relief with the presence of pores (Fig 4c). It should be noted that sandblasting presented the greatest topographical relief with evident irregular morphological changes (Fig 4d), whereas sandblasting combined with

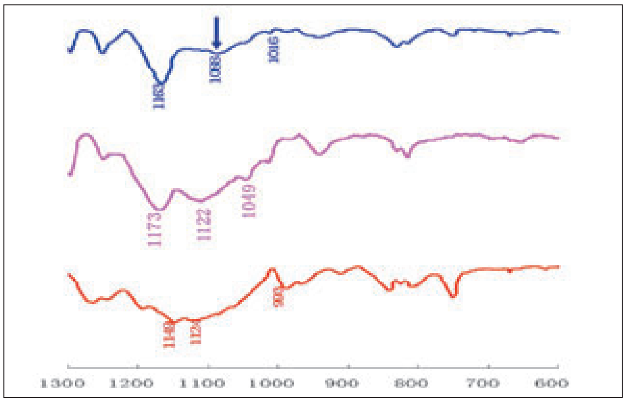

Fig 3 The comparative presentations of FT-IR spectra obtained from the composite (red line), the silane coupling agent (RelyX Ceramic Primer, pink line) and the silanised composite (blue line).

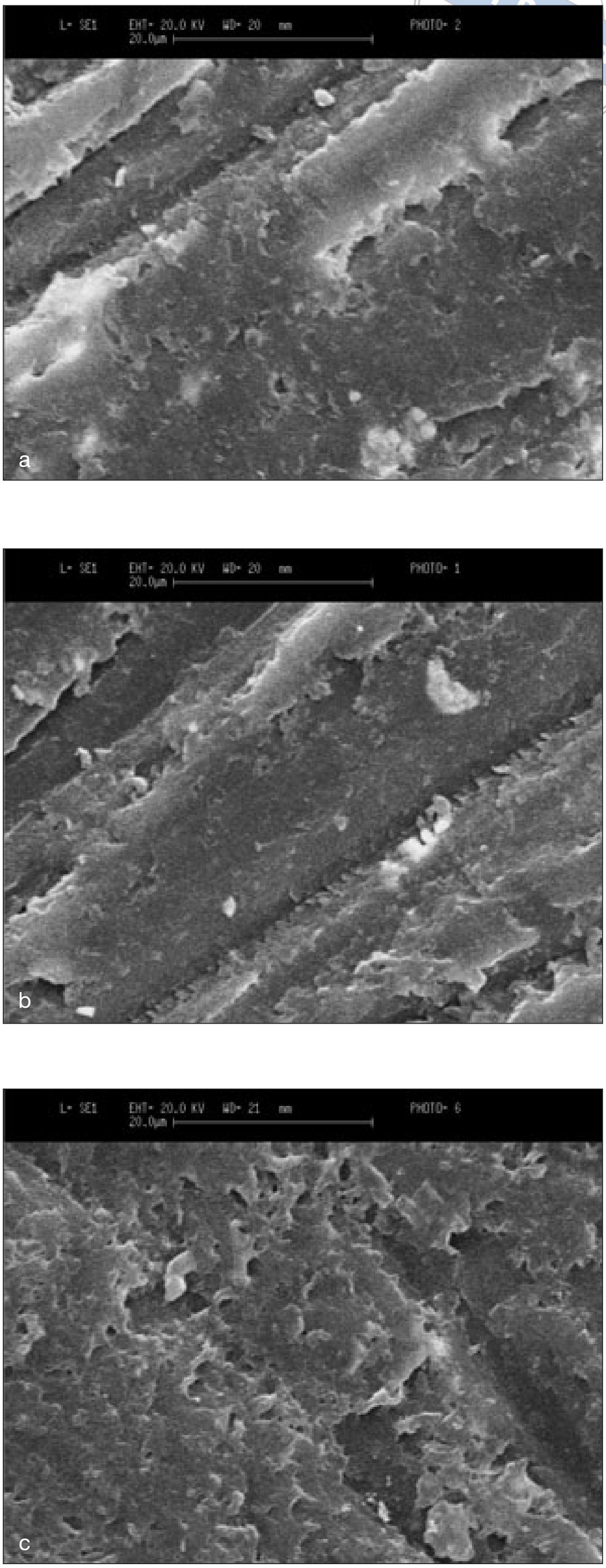

Fig 4 SEM images. a) composite surface ground with 400-grit SiC paper as a control. b) phosphoric acid etched composite surface. c) HF-etched composite surface (original magnification, ×2000).

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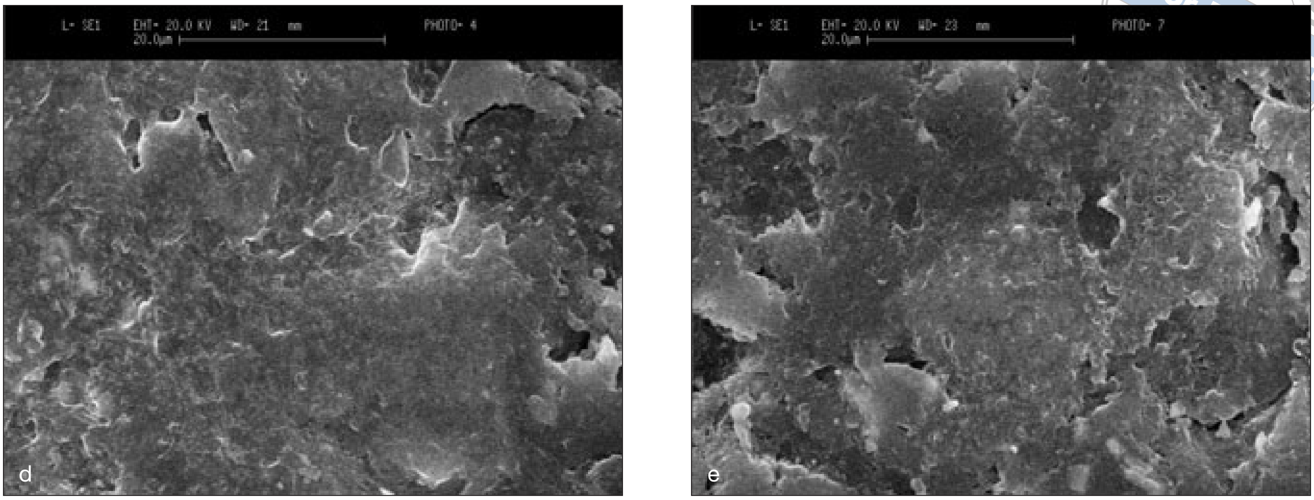

Fig 4 (cont) SEM images. d) sandblasted composite surface. e) sandblasted and HF-etched composite surface. The specimen shows fewer rough surface morphological changes with a few pores (original magnification, ×2000).

HF exhibited few rough surface morphological changes with some pores (Fig 4e).

Discussion

For indirect composite restorations, researchers have focused on the bond strength between composite restoration and resin cement. However, little information related to the bond strength between composite restorations and dentine is available. Long-term stable composite–dentine bonds rely on chemical bonds and micromechanical interlocking at the interface. The surface treatments of CAD/CAM composite performed in the present study were selected on the basis of valid conditioning methods suggested by previous studies to improve the bonding performance of indirect composite restorations. The methods involved different chemical procedures, mechanical procedures or a combination of both. The test included two substrates and two interfaces, which was more complex and reflected more clinical situations.

It was reported that there was a significant increase in bond strength compared with the non-treated control groups when the composite substrate was treated with a thin layer of adhesive[3,7]. This positive influence could be related to the effect of producing micromechanical retention through monomer penetration into the matrix microcracks, enhancing the composite–cement bond by chemical bonds to the exposed filler particles[16] as well as increasing the wettability of the treated surface. However, this favourable result was not detected in the present study. The fact that the bond strength was not significantly improved may be related to the surface bond properties of the CAD/CAM composite. The greater hydrophobic performance impeded the surface wetting of adhesive agents in which the solvents of the three adhesive agents were water, ethanol, or a mixture of water and ethanol.

Silanes are bifunctional molecules that bind silicon dioxide with OH groups on the ceramic surface. They also have a degradable functional group that copolymerises with the resin's organic matrix[17]. The application of the silane coupling agent to a pretreated ceramic surface provides a chemical covalent hydrogen bond and is a major factor in creating a sufficient resin bond to silica-based ceramics[18]. A controversial issue is whether the silane coupling agent is required in a composite–dentine bond. Some scholars consider that silanisation can improve the bond strengths of composite and resin cement, even after long-time thermal cycling[7]. However, there is no clear evidence that reveals the mechanism. In the present study, new Si-O-Si group peaks in the representative FT-IR spectra were found. This confirmed the effective mechanism of silanisation. Although the new bonds were beneficial for the improvement of bond strength, the increase of chemical bonds was small due to the limited content of silicon dioxide (20 to 30%). Consequently, silanisation could only slightly improve the bonding strength.

D'Arcangelo[11] verified that HF + S treatment did not produce significant changes in tensile bond strength between composite and dentine, although SEM showed a moderate amount of surface relief with the presence of pores. In the present study, a similar effect was observed with HF + S treatment. This can be explained by the possibility that application of hydrofluoric acid not only

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resulted in the complete dissolution of exposed glass particles, but also produced a softening and porosity in the composite resin matrix[16]. El Zohairy et al[3] used three resin cements to treat CAD/CAM composite surfaces prior to bonding and found significant differences among them. In the present study, for the HF + S treatment group, there was no statistical difference among the three resin cements.

The three sandblasting treatment groups exhibited the highest bond strengths except for the Sa + S and Sa group when using Variolink II (not significantly smaller than other treatment groups). The SEM evaluation of the sandblasting treatment showed the greatest surface relief with irregularly rough morphological changes, which was important for creating interlocking and wetting for silanisation. The results of this and other previously published studies suggest that sandblasting treatment is the main factor responsible for improving the retentive properties of indirect composite restorations[11].

In this study, no statistically significant differences were found among the three sandblasting treatment groups. In addition, the SEM image of Sa + HF treatment showed fewer rough surface morphological changes than that of Sa treatment. Considering the possible hazardous effects of HF etching, the insignificant improvement by Sa + HF + S treatment and that the fewest composite–cement interface failures occurred in the Sa + S group (0%), sandblasting followed by silanisation is recommended as a reliable means for increasing the bond strength between the CAD/CAM composite material and dentine. When using sandblasting followed by silanisation treatment, the three resin cements showed similar bond effects.

Some articles reported that shear tests often led to non-uniform distribution of the stress at the adhesive area and produced cohesive bulk fracture of the substrate away from the bonding interface. However, in the present study, the types of failure excluded the cohesion failure within dentine or the composite resin. It can be attributed to the bond strength values (4 to 26 MPa, mean 10 MPa, suitable for the shear test)[19] and the low elastic modulus of the composite substrate[3].

In the present study, the percentage of adhesive failure at the dentine–cement interface was higher than that at the composite–cement interface. It indicated that there may be a more effective bond at the composite–cement interface compared to the dentine–cement interface. The fact that the occurrence of adhesive failures at the composite–luting cement interface was greatly reduced after sandblasting treatment further confirms that sandblasting treatment is more important for the improvement of bond strength than chemical treatments.

Future studies are warranted to compare this material to other CAD/CAM composites and to test the long-term effects of bond strength of the CAD/CAM composite to dentine.

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