Effects of alumina airborne-particle abrasion on the surface properties of CAD/CAM composites and bond strength to resin cement

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The purpose of this study is to clarify physical and chemical changes in surfaces of CAD/CAM composites caused by alumina airborne-particle abrasion and its effect on adhesive bonding. Our study involved three dispersed filler (DF)-based and a polymer-infiltrated ceramic network (PICN)-based CAD/CAM composites. Changes in the surface morphologies of the composites were examined, and surface free energy (SFE) analysis was performed based on Owens-Wendt theory. The influence of the abrasion on the bond strengths of CAD/CAM composites to the resin cement was characterized by shear bond strength (SBS) test. The abrasion increased the roughness of the composites. The SFE analysis showed that the abrasion significantly increased the dispersive component but decreased the polar component of the SFE associated with the DF-based composites, while no change occurred for those of the PICN-based composite. The abrasion slightly improved the SBS for the DF-based composites but not that of the PICN-based composite.

Keywords: Composite resin, Polymer-infiltrated ceramic, Surface free energy, Blasting, Bonding

INTRODUCTION

Dental composite resins for indirect restoration have greatly improved due to advances in computer-aided design and manufacturing (CAD/CAM) systems. The CAD/CAM composites comprise a large amount of filler with a high degree of conversion of the matrix resin, thereby improving their mechanical properties. Owing to their excellent properties, CAD/CAM composites have been increasingly employed in crown restorative materials. However, clinical problems hinder their usage in restoration, such as debonding from the abutment tooth within short periods in the oral environment. This debonding failure is attributed to poor adhesion of the CAD/CAM composites or incompatible preparation of the crown material and abutment tooth. To overcome this issue, many fundamental studies have been conducted to elucidate the adhesive properties of the CAD/CAM composites. For the bonding protocol of the CAD/CAM composites, several studies and manufacturers' instructions suggested that composite crowns should be subjected to alumina airborne-particle abrasion followed by application of a primer containing a silane-coupling agent (hereafter silane primer). In contrast, some studies addressed that alumina airborne-particle abrasion is not ideal for the interfacial strength between the resin cement and CAD/CAM composites. Consequently, we classified CAD/CAM composites according to their microstructures and investigated the effects of the alumina airborne-particle abrasion on the bond strength of the CAD/CAM composites. Recent dental CAD/CAM composites can be classified as dispersed filler (DF)-based and polymer-infiltrated ceramic network (PICN)-based composites. DF-based composites contain ceramic fillers in the resin matrix and are widely used in dentistry for both direct and indirect restorations. The PICN-based composite is a relatively new type of CAD/CAM composite with dual networks composed of a ceramic skeleton and infiltrated resin phase. In this study, DF-based and PICN-based commercial CAD/CAM composites were used to elucidate the effects of airborne-particle abrasion on the surface properties and bond strength between the composites and resin cement. Affinities of the abraded composites to resin cement with silane primer were examined by using shear bond strength (SBS) tests. The abrasion increased the roughness of the composites. The SFE analysis showed that the abrasion significantly increased the dispersive component but decreased the polar component of the SFE associated with the DF-based composites, while no change occurred for those of the PICN-based composite. The abrasion slightly improved the SBS for the DF-based composites but not that of the PICN-based composite.

MATERIALS AND METHODS

Sample preparation

Table 1 lists the commercial CAD/CAM composite blocks used in this study: three DF-based composites (AV: KATANA AVENCIA Block, CE: CERASMART 270, and SH: SHOFU BLOCK HC) and a PICN-based composite (EN: VITA ENAMIC). The classification based on their microstructures has been performed in our previous...
glycol dimethacrylate, DMA: dimethacrylate
Bis-MEPP: 2,2-Bis(4-methacryloxypolyethoxyphenyl)propane, UDMA: urethane dimethacrylate, TEGDMA: triethylene glycol dimethacrylate, DMA: dimethacrylate

Angles according to the following equations:

\[
\theta = \arctan \left( \frac{1}{\gamma_d} \right)
\]

\[
\theta = \arctan \left( \frac{1}{\gamma_d} \right)
\]

\[
\gamma_d = \frac{\gamma_{\text{total}} - \gamma_p}{1 + \cos \theta}
\]

SFE of the test liquids were used \( \gamma_{\text{total}} \) (water: \( \gamma_{\text{total}} = 72.8 \) mJ/m\(^2\), diiodomethane: \( \gamma_{\text{total}} = 50.8 \) mJ/m\(^2\), and diiodomethane: \( \gamma_{\text{total}} = 49.5 \) mJ/m\(^2\)).

Surface characterization
The surface roughness of the CAD/CAM composites was measured by a confocal laser scanning microscopy (CLSM: VKX-100, Keyence, Osaka, Japan) \((n=10)\). Surfaces characterization of the CAD/CAM composites was performed by scanning electron microscope (SEM; S-4300, Hitachi-Technologies, Tokyo, Japan). Chemical compositions and elemental mapping of the CAD/CAM composites were examined by energy-dispersive X-ray spectroscopy (EDX; Ametek, Berwyn, PA, USA). Each composite surface was coated with platinum using a sputtering device. The acceleration voltage used for the SEM-EDX observation was 10 kV and the numbers of counts for the elemental mapping was 10,000–15,000.

The SFE of the CAD/CAM composites was determined \( \theta \) via contact angle measurements. The contact angles of each composite for two test liquids (distilled water and diiodomethane (>99%, Kanto Chemical, Tokyo, Japan) were examined using a contact angle meter (DMe-211, Kyowa Interface Science, Niiza, Japan) under ambient conditions at 20±3°C \((n=10)\). Two microliters test liquid was dropped on the composite surface to capture its contact angle. The SFEs based on Owens-Wendt theory were calculated with analytical software (FAMAS, Kyowa Interface Science) by using the measured contact angles according to the following equations:

\[
\gamma_{\text{total}} = \gamma_p + \gamma_d
\]

\[
\gamma_{\text{total}} = \gamma_p + \gamma_d
\]

where \( \theta \) represents the measured contact angle for the test liquids, the subscript indexes \( L_1 \) and \( L_2 \) mean the test liquids of water and diiodomethane, respectively; \( \gamma_{\text{total}} \), \( \gamma_p \), and \( \gamma_d \) are the total SFE, and polar (hydrogen), and dispersive components of SFE for the composites, respectively. The previously reported values for the SFE of the test liquids were used \( \gamma_{\text{total}} \) (water: \( \gamma_{\text{total}} = 72.8 \) mJ/m\(^2\), diiodomethane: \( \gamma_{\text{total}} = 50.8 \) mJ/m\(^2\), and diiodomethane: \( \gamma_{\text{total}} = 49.5 \) mJ/m\(^2\)).

Shear bond strength (SBS) tests
Shear bond strengths of the CAD/CAM composites to the resin cement were measured by a conventional SBS test. A constant adhesion area on the composite surface was examined by using a double-faced tape. The silane-coupling agent was applied on the composite surface, and then a resin cement (Resicem, SHOFU) was loaded on the surface through the Teflon tube. The loaded cement was polymerized using a light irradiator (\( \mu \) LIGHT II N, J. Morita) for 5 min. The cement-cured sample was kept at ambient conditions for 60 min; subsequently, the Teflon tube was detached from the sample. The cement-bonded composite was stored in distilled water at \( 37°C \) for 24 h.

The resultant sample was subjected to the SBS

| Product name | Product company | Abbreviation | Monomer Composition | Filler Composition | Filler weight (%) | Microstructural type |
|--------------|-----------------|--------------|---------------------|-------------------|------------------|---------------------|
| KATANA AVENCIA Block | Kuraray Noritake Dental | AV | UDMA, Methacrylic monomer, other | Silica (40 nm), Alumina (20 nm) | 62 | Nano dispersed-filler structure |
| CERASMART270 | GC | CE | Bis-MEPP, UDMA, DMA | Silica (20 nm), Barium glass (300 nm) | 77 | Submicron dispersed-filler structure |
| SHOFU BLOCK HC | SHOFU | SH | UDMA, TEGDMA, other | Silica, Zirconium silicate | 62 | Micron dispersed-filler structure |
| VITA ENAMIC | VITA Zahnfabrik | EN | UDMA, TEGDMA | Feldspar ceramic | 86 | Polymer-infiltrated-ceramic- network structure |

Bis-MEPP: 2,2-Bis(4-methacryloyloxypropoxyphenyl)propane, UDMA: urethane dimethacrylate, TEGDMA: triethylene glycol dimethacrylate, DMA: dimethacrylate
test to obtain the bond strength at the initial stage. On the other hand, for examining the durability by thermocycling, the cement-bonded composite was alternately immersed in cold water at 5°C and hot water at 55°C, which was repeated 20,000 times with a 60-s dwell time at each step. After thermocycling, the SBS test for the sample was performed using a mechanical testing machine (AGS-H, Shimadzu, Kyoto, Japan) with a crosshead speed of 1.0 mm/min.

The debonded surface after the SBS test was observed by an optical microscope with 50× magnification to classify the failure modes (adhesive failure at the cement–CAD/CAM composite interface (A), cohesive failure within the CAD/CAM composite (C), and mixed failure of A and C (M)).

**Results**

**Surface modification of the CAD/CAM composites by alumina airborne-particle abrasion**

Figure 1 shows the surface roughness of each CAD/CAM composite before and after alumina airborne-particle abrasion. The surface roughness for each composite was significantly increased by airborne-particle abrasion; the roughness values for AV, CE, and SH increased 4 times, and those for EN increased 4 times, and those for EN increased about twice.

Figure 2 shows SEM images and EDX elemental mappings for the no-abraded composites. The microstructures correspond to AV, CE, and SH with DF structure, and EN with PICN structure. The EDX elemental mapping shows that Si is dispersed throughout the DF-based composites (AV, CE, and SH) as the main component of the fillers. Al of the alumina fillers is distributed throughout in AV and CE. Each DF-based composite has C, which is the main component of the resin matrix, distributed throughout the matrix. For EN, the elements represent specific distributions derived from the PICN structure; Si and Al are localized in the ceramic skeleton, and C, in the infiltrated resin phase, respectively. Figure 3 shows SEM images and EDX elemental mappings for the alumina airborne-particle abraded composites. From the SEM images, the roughened surface with cracks can be observed for each composite. Si, Al, and C are distributed throughout all composites, and no remarkable Al localization is observed at all. These results mean that for all of the composites, no alumina particles remained on the composite after the airborne-particle abrasion. We note that the Al elements found in the EDX elemental mappings of AV, CE, and EN presented in Fig. 3 are due to the alumina fillers contained in the composites themselves, rather than due to the alumina particles remained by the abrasion.

**Statistical analysis**

Statistical analysis was carried out by using a software EZR (Saitama Medical Center, Jichi Medical University, Saitama, Japan). Students' t-tests were employed for determining the statistical difference between two groups for the surface roughness and SFE analysis results. One-way analysis of variance (ANOVA) was applied for multiple comparisons in the groups for the SBS results. Tukey's post hoc testing was performed in the groups when the results of ANOVA were statistically significant. All statistical analysis adopted a significance level (p) of 0.05.

**Influence of alumina airborne-particle abrasion on the SBS of the CAD/CAM composites**

Figure 5(a) shows SBS results of the composites before thermocycling. For AV and CE, no statistical differences were obtained in the SBSs of the composites. For SH, the SBS was the highest for the group with silanization+airborne-particle abrasion. For EN, the SBS for the groups with silanization, airborne-particle abrasion, and silanization+airborne-particle abrasion are significantly higher than that with no treatment.

Table 2 lists the failure modes of the samples. In AV, CE, and SH, the number of cohesive failures increases in the following order: the group with no
Fig. 2  SEM images and EDX elemental mappings of the no-abraded CAD/CAM composites. AV: KATANA AVENCIA Block, CE: CERASMART 270, SH: SHOFU BLOCK HC, EN: VITA ENAMIC.

Fig. 3  SEM images and EDX elemental mappings of the alumina airborne-particle abraded CAD/CAM composites. AV: KATANA AVENCIA Block, CE: CERASMART 270, SH: SHOFU BLOCK HC, EN: VITA ENAMIC.
treatment, silanization, airborne-particle abrasion, and silanization+airborne-particle abrasion. For EN, on the other hand, the cohesive failure was the highest for the group with silanization.

Figure 5(b) shows SBS results of the composites after thermocycling. For AV, the SBS for the group with airborne-particle abrasion+silanization is significantly higher than that with no treatment. For CE, no statistical difference is observed between the groups with no treatment and airborne-particle abrasion+silanization. For SH, the SBS for the group with airborne-particle abrasion+silanization is significantly higher than that with no treatment.

Fig. 3 Shear bond strengths (SBSs) of the CAD/CAM composites with no treatment, silanization, airborne-particle abrasion, and silanization+airborne-particle abrasion. For EN, on the other hand, the cohesive failure was the highest for the group with silanization.

The different letters denote a significant difference in the groups in each composite (p<0.05, Tukey test, n=10). AV: KATANA AVENCIA Block, CE: CERASMART 270, SH: SHOFU BLOCK HC, EN: VITA ENAMIC.
Abrasion+silanization is significantly higher than that with no treatment. For EN, the SBS for the group with silanization is the highest in the groups. As shown in Table 2, the number of cohesive failures for the DF-based composites (AV, CE, and SH) increases in the following order: the group with no treatment, silanization, airborne-particle abrasion, and airborne-particle abrasion+silanization. For the PICN-based composite (EN), on the other hand, the highest number of cohesive failures can be found in the group with silanization.

**DISCUSSION**

We discuss the surface modification effect of airborne-particle abrasion on the CAD/CAM composites based on their microstructures. The physical changes on the composite surfaces are explained based on the morphological evaluations. As shown in Fig. 1, the surface roughness of each composite significantly increased by the airborne-particle abrasion. The surface roughness for AV, CE, and SH increased 4 times whereas that for EN increased about twice. This indicates that the PICN-based composite surface is more difficult to abrade than that of the DF-based composite, because the former composite is harder than the latter. The SEM result in Fig. 3 indicates that the airborne-particle abrasion roughens the composite surfaces due to the developed cracks, and no alumina particles remain on their surface. Our results are compatible with other reports on the same and various other composites.

The SFE analysis is used to estimate the chemical state of a material surface. We reported that the SFE is strongly correlated to the microstructure of the CAD/CAM composite, affecting its adhesion to the resin cement in our previous report. In the DF-based composites (AV, CE, and SH), the dispersive and polar components of the SFE increased and decreased, respectively, due to the airborne-particle abrasion. In contrast, the dispersive and polar components of the SFE for the PICN-based composite (EN) remained nearly the same after the airborne-particle abrasion. We interpret these changes in SFE by focusing on the silanol (Si-OH) groups of the fillers on the composite surface because they are active sites for the silane-coupling agent in the silane primer and strongly affect the interfacial interactions between the composite and resin cement.

If many silanol groups exist on the fillers of the composite surface, the polar component of the composites must be large because of the high polarity of silanol groups. In many silanol groups exist on the fillers of the composite surface, the polar component of the composites must be large because of the high polarity of silanol groups. In contrast, the increment in the dispersive component and decrement in the polar component imply that the airborne-particle abrasion reduces the density of silanol groups on the composite surface. It is known that silica has fewer silanol groups inside in comparison to its surface. Even if the fillers in the composites fracture due to the impact of the airborne-particle abrasion and subsequently get exposed, the silanol groups hardly exist at the fractured interfaces. Thus, SFE analysis results indicate that the airborne-particle abrasion causes chemical changes on the DF-based composites by reducing the density of silanol groups, while no chemical change occurs on the surface of PICN-based composite upon airborne-particle abrasion.

For the DF-based composites (AV, CE, and SH), the SBS results in Fig. 5 show that the airborne-particle abrasion tends to increase mean SBS values. In addition, the number of cohesive failures was increased by the airborne-particle abrasion. The airborne-particle abrasion improves the bond strength of DF-based composites due to the increment in the surface roughness of the composites. Meanwhile, combined use of airborne-particle abrasion and silanization have no synergistic effect on the bond strength of the DF-based composites. Although the increment in the surface roughness increases the SBS, the reduction of silanol groups does not improve the SBS due to the decrease in the silanization process. For the PICN-based composite (EN), the highest SBS can be found in the groups with silanization, as shown in Fig. 5. Meanwhile, no differences were found between the SBSs for the groups with the airborne-
particle abrasion and no treatment. Thus, the airborne-particle abrasion has no positive effect on SBS for the PICN-based composite. The SFE analysis suggests that airborne-particle abrasion does not chemically change the surface of the PICN-based composite. The results of the surface roughness measurement and the SEM observation indicate that the airborne-particle abrasion increases surface roughness with generating the cracks on the surface. These results mean that the airborne-particle abrasion enhances the SBS by interlocking between the roughened composite surface and the resin cement, while negatively affecting the mechanical strength due to the surface cracks, which decrease the SBS. Therefore, the airborne-particle abrasion should be avoided in the PICN-based composite.

Our results prove the effectiveness of airborne-particle abrasion as a pretreatment for the CAD/CAM composites, but since airborne-particle abrasion does not work effectively for all composites, it is necessary to select an appropriate surface pretreatment by considering the specific microstructure of the composite. From a clinical point of view, the use of alumina airborne-particle abrasion as a bonding pretreatment is recommended for the DF-based composite but not for the PICN-based composite.

CONCLUSION

We investigated the impact of alumina airborne-particle abrasion on the CAD/CAM composites. The affinity of the composites to the resin cement depends on their microstructure. For the DF-based composites (KATANA AVENCIA Block, CERASMART 270, and SHOFU BLOCK HC), the alumina airborne-particle abrasion increases the surface roughness and the dispersive component of the SFE, while it decreases the polar component of the SFE. These changes in SFE would be attributed to the reduction of the surface silanol groups. On the other hand, airborne-particle abrasion increases the surface roughness of the PICN-based composite (VITA ENAMIC), while it does not affect its SFE. As a pretreatment technique, airborne-particle abrasion is effective for the DF-based composites, while it is not for the PICN-based composite. No synergistic effects of airborne-particle abrasion and silanization were observed in the SBSs for either DF- or PICN-based composites.

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CONFLICTS OF INTEREST

The authors declare no conflicts of interest.

REFERENCES

1) Ruse ND, Sadoun MJ. Resin-composite blocks for dental CAD/CAM applications. J Dent Res 2014; 93: 1232-1234.
2) Mainjot AK, Dupont NM, Oudkerk JC, Dewael TY, Sadoun MJ. From artisanal to CAD-CAM blocks: state of the art of indirect composites. J Dent Res 2016; 95: 487-495.
3) Amesti-Garaizabal A, Agustin-Panadero R, Verdejo-Sola B, Fons-Font A, Fernandez-Estevan L, Montiel-Company J, et al. Fracture resistance of partial indirect restorations made with CAD/CAM technology. A systematic review and meta-analysis. J Clin Med 2019; 8: 1932.
4) Stawarzczuk B, Liebermann A, Eichberger M, Guth JF. Evaluation of mechanical and optical behavior of current esthetic dental restorative CAD/CAM composites. J Mech Behav Biomed Mater 2015; 55: 1-11.
5) Awada A, Nathanson D. Mechanical properties of resin-ceramic CAD/CAM restorative materials. J Prostheth Dent 2015; 114: 587-593.
6) Chavali R, Nejat AH, Lawson NC. Machinability of CAD-CAM materials. J Prostheth Dent 2016; 118: 194-199.
7) Lawson NC, Bansal R, Burgess JO. Wear, strength, modulus and hardness of CAD/CAM restorative materials. Dent Mater 2016; 32; e275-e283.
8) Flury S, Diebold E, Peutzfeldt A, Lussi A. Effect of artificial toothbrushing and water storage on the surface roughness and micromechanical properties of tooth-colored CAD-CAM materials. J Prostheth Dent 2017; 117: 767-774.
9) Schlenz MA, Schmidt A, Rehmann P, Niem T, Westmann B. Microleakage of composite crowns luted on CAD/CAM-milled human molars: a new method for standardized in vitro tests. Clin Oral Investigat 2019; 23: 511-517.
10) Rosentritt M, Preis V, Behr M, Kriftka S. In-vitro performance of CAD/CAM crowns with insufficient preparation design. J Mech Behav Biomed Mater 2019; 90: 269-274.
11) Yamaguchi S, Lee C, Karnaer O, Ban S, Mine A, Imazato S. Predicting the debonding of CAD/CAM composite resin crowns with AI. J Dent Res 2019; 98: 1234-1238.
12) Vanoorbeek S, Vandamme K, Lijnen I, Naert I Computer-aided designed/computer-assisted manufactured composite resin versus ceramic single-tooth restorations: a 3-year clinical study. Int J Prosthodont 2010; 23: 225-230.
13) Zimmermann M, Koller C, Reymus M, Meli A, Hickel R. Clinical evaluation of indirect particle-filled composite resin CAD/CAM partial crowns after 24 months. J Prosthodont 2018; 27: 694-699.
14) Miura S, Kasahara S, Yamauchi S, Katsuda Y, Harada A, Aida J, et al. A possible risk of CAD/CAM-produced composite resin premolar crowns on a removable partial denture abutment tooth: a 5-year retrospective cohort study. J Prosthodont Res 2019; 63; 78-84.
15) Yano HT, Ikeda H, Nagamatsu Y, Masaki C, Hosokawa R, Shimizu H. Correlation between microstructure of CAD/CAM composites and the silanization effect on adhesive bonding. J Mech Behav Biomed Mater 2020; 101: 103441.
16) Shinagawa J, Inoue G, Nikaido T, Ikeda M, Burrow MF, Tagami J. Bonding effectiveness of self-adhesive and conventional-type adhesive resin cements to CAD/CAM resin composite. Dent Mater J 2019; 38; 28-32.
17) Kawaguchi A, Matsumoto M, Higashi M, Miura J, Minamino T, Kabetani T, et al. Bonding effectiveness of self-adhesive and conventional-type adhesive resin cements to CAD/CAM resin blocks. Part 2: Effect of ultrasonic and acid cleaning. Dent Mater J 2016; 35: 29-36.
18) Higashi M, Matsumoto M, Kawaguchi A, Miura J, Minamino T, Kabetani T, et al. Bonding effectiveness of self-adhesive and conventional-type adhesive resin cements to CAD/CAM resin blocks. Part 1: Effects of sandblasting and silanization. Dent Mater J 2016; 35: 21-28.
19) Stawarzczuk B, Krawczuk A, Ilie N. Tensile bond strength of resin composite repair in vitro using different surface preparation conditionings to an aged CAD/CAM resin...
20) Kassotakis EM, Stavridakis M, Bortotolo T, Ardu S, Krejci I. Evaluation of the effect of different surface treatments on luting CAD/CAM composite resin overlay workpieces. J Adhes Dent 2015; 17: 521-528.

21) Peumans M, Valjakova EB, De Munck J, Mishevska CB, Van Meerbeek B. Bonding effectiveness of luting composites to different CAD/CAM materials. J Adhes Dent 2016; 18: 289-302.

22) Flury S, Schmidt SZ, Peutzfeldt A, Lussi A. Dentin bond strength of two resin-ceramic computer-aided design/computer-aided manufacturing (CAD/CAM) materials and five cements after six months storage. Dent Mater J 2016; 35: 728-735.

23) Lise DP, Van Ende A, De Munck J, Vieira L, Baratieri LN, Van Meerbeek B. Microtensile bond strength of composite cement to novel CAD/CAM materials as a function of surface treatment and aging. Oper Dent 2017; 42: 73-81.

24) Mine A, Kabetani T, Kawaguchi-Uemura A, Hagino R, Higashi M, Tajiri Y, et al. Effectiveness of current adhesive systems when bonding to CAD/CAM indirect resin materials: A review of 32 publications. Jpn Dent Sci Rev 2019; 55: 41-50.

25) Kawaguchi-Uemura A, Mine A, Matsumoto M, Tajiri Y, Hagini R, et al. Adhesion procedure for CAD/CAM resin crown bonding: Reduction of bond strengths due to artificial saliva contamination. J Prosthodont Res 2018; 62: 177-183.

26) Tekce N, Tuncer S, Demirci M. The effect of sandblasting duration on the bond durability of dual-cure adhesive cement to CAD/CAM resin restoratives. J Adv Prosthodont 2018; 10: 211-217.

27) Duzeyl M, Sagsoz O, Polat Sagsoz N, Akgul N, Yildiz M. The effect of surface treatments on the bond strength between CAD/CAM blocks and composite resin. J Prosthodont 2016; 25: 466-471.

28) Yoshihara K, Nagaoka N, Maruo Y, Nishigawa G, Irie M, Yoshida Y, et al. Sandblasting may damage the surface of composite CAD-CAM blocks. Dent Mater 2017; 33: e124-e135.

29) Eldafrawy M, Ebroin MG, Gailly PA, Nguyen JF, Sadoun MJ, Mainjot AK. Bonding to CAD-CAM composites: an interfacial fracture toughness approach. J Dent Res 2018; 97: 60-67.

30) Owens DK, Wendt DT. Estimation of the surface free energy of polymers. J Appl Polym Sci 1969; 13: 1741-1747.

31) Keul C, Muller-Hahl M, Eichberger M, Liebmann A, Roos M, Edelhoff D, et al. Impact of different adhesives on work of adhesion between CAD/CAM polymers and resin composite cements. J Dent 2014; 42: 1105-1114.

32) Cavalcanti YW, Bertolini MM, Cury AA, da Silva WD. The effect of poly(methyl methacrylate) surface treatments on the adhesion of silicone-based resilient denture liners. J Prosthet Dent 2014; 112: 1539-1544.

33) Ueta H, Tsujimoto A, Barkmeier WW, Oouchi H, Sai K, Takamizawa T, et al. Influence of an oxygen-inhibited layer on enamel bonding of dental adhesive systems: surface free-energy perspectives. Eur J Oral Sci 2016; 124: 82-88.

34) Stasic JN, Selakovic N, Puac N, Miletic M, Malovic G, Petrovic ZL, et al. Effects of non-thermal atmospheric plasma treatment on dentin wetting and surface free energy for application of universal adhesives. Clin Oral Investig 2019; 23: 1383-1396.

35) Akazawa N, Koizumi H, Nogawa H, Kodaira A, Burrow MF, Matsumura H. Effect of etching with potassium hydrogen difluoride and ammonium hydrogen difluoride on bonding of a tri-n-butylborane initiated resin to zirconia. Dent Mater J 2019; 38: 540-546.

36) Tsujimoto A, Iwasa M, Shimamura Y, Murayama R, Takamizawa T, Miyazaki M. Enamel bonding of single-step self-etch adhesives: influence of surface energy characteristics. J Dent 2010; 38: 129-130.

37) Tsujimoto A, Barkmeier WW, Takamizawa T, Latta MA, Miyazaki M. The effect of phosphoric acid pre-etching times on bonding performance and surface free energy with single-step self-etch adhesives. Oper Dent 2016; 41: 441-449.

38) Tsujimoto A, Barkmeier WW, Takamizawa T, Latta MA, Miyazaki M. Influence of the oxygen-inhibited layer on bonding performance of dental adhesive systems: surface free energy perspectives. J Adhes Dent 2016; 18: 51-58.

39) Tamura Y, Takamizawa T, Shimamura Y, Akiba S, Yabuki C, Imai A, et al. Influence of air-powder polishing on bond strength and surface-free energy of universal adhesive systems. Dent Mater J 2017; 36: 762-769.

40) Tsujimoto A, Barkmeier WW, Takamizawa T, Wilwerding TM, Latta MA, Miyazaki M. Interfacial characteristics and bond durability of universal adhesive to various substrates. Oper Dent 2017; 42: E59-E70.

41) Matinlinna JP, Lung CYK, Tsoi JKH. Silane adhesion mechanism in dental applications and surface treatments: A review. Dent Mater 2018; 34: 1-13.

42) Yanagisawa N, Fujimoto K, Nakashima S, Kurata Y, Sanada N. Micro FT-IR study of the hydration-layer during dissolution of silica glass. Geochim Cosmochim Acta 1997; 61: 1165-1170.

43) Holyoak LS. The effect of thermal treatment of silica gel on its surface free energy components. Colloids Surf, A 1998; 134: 321-329.

44) Zhuravlev L. The surface chemistry of amorphous silica. Zhuravlev model. Colloids Surf A 2000; 173: 1-38.