Influence of surface treatment of contaminated zirconia on surface free energy and resin cement bonding

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Influences of contamination and cleaning methods on the bonding of resin cement to zirconia ceramics were examined. Airborne particle-abraded zirconia (IPS e.max ZirCAD) specimens were contaminated with saliva and cleaned with tap water (SC) or by application of 37% phosphoric acid (PA), Ivoclean (IC), or additional airborne particle abrasion (AB). Specimens without contamination served as controls. After application of Monobond Plus to the surface of the specimens, resin cement was mixed and inserted into a mold. Surface free energies of the specimens were determined by measuring contact angles. Surface treatment and storage conditions significantly influenced bond strength, while there was no significant interaction between the two factors. Surface free energies of the SC and IC groups were significantly lower than those of the other groups. Additional AB of saliva-contaminated zirconia increased the strength of bonding with the resin cement as well as increased surface free energy.

Keywords: Zirconia ceramics, Resin cement, Bond strength, Surface free energy

INTRODUCTION

Increasing demand for aesthetic restorations in clinical dentistry has resulted in the development of tooth-colored restorative systems with improved mechanical properties for the restorations in posterior lesions. In recent years, highly purified zirconium dioxide (zirconia) ceramics have become increasingly popular in clinical dentistry6). Because of their excellent bio compatibility, improved strength, and aesthetics, zirconia ceramics have been used as a framework material for all-ceramic restorations9).

Selection of an appropriate adhesive system to obtain good adhesion between the tooth and indirect restoration is recognized to play a crucial role in the success of restorations9). When bonding ceramic to tooth substrates, it is important to ensure optimal bond strength at two different interfaces: dentin-resin cement and ceramic-resin cement interfaces. This is because these two interfaces determine the final bond strength of the restoration. All-ceramic restorations finished for cementation are usually already abraded by airborne particles in a dental laboratory to ensure micromechanical as well as chemical bonding with the luting agent10). After try-in of the restoration in the oral environment, the ceramic surface might become contaminated by saliva or blood. Saliva contamination is one of the main reasons for decreased bond strength of the restoration to restored teeth. Water or phosphoric acid gel is generally used to clean the restoration. The effect of ceramic surface contamination with saliva and subsequent cleaning of the surface with phosphoric acid has been evaluated previously10). Comparing two different surface cleaning methods, cleaning with phosphoric acid resulted in higher bond strength values than cleaning with acetone. However, the bond strength was evaluated without considering the influence of long-term water storage or the number of thermal cycles so that the results provided no indication of bonding durability. In addition, a new type of cleaning agent containing sodium hydroxide and zirconium oxide particles has been developed, but there is no consensus regarding the use of this type of priming agent.

Wettability of the conditioned adherent surface with resin cement is important for the bonding of ceramics regardless of the mechanism of bonding, i.e., chemical, micromechanical interlocking, or a combination5). The strength of the bond between zirconia ceramics and the resin cement depends on several factors, including the surface treatment of zirconia and the ability of the resin cement to wet the adherent surface7). Measurements of contact angles on the adherent surfaces provide information about surface free energies that relate to the bonding characteristics of the solids8). To date, neither the effect of saliva contamination nor the evaluation of effective cleaning methods have been investigated by surface free energy measurements8). Therefore, in the present study, surface free energy measurement was used to identify the presence of saliva on zirconia ceramic surfaces after simulation of saliva contamination.

The purpose of this study was to examine the influence of saliva contamination and cleaning methods on the surface free energy of ceramics and the bond strength of resin cement to zirconia ceramics. The null hypothesis was that surface free energy and resin cement bonding are not affected by saliva contamination.

MATERIALS AND METHODS

Specimen preparation
Materials tested in this study are summarized in Table 1. Yttrium-stabilized zirconia (IPS e.max ZirCAD,
Ivoclar Vivadent, Schaan, Liechtenstein) plates were cut from pre-sintered blocks using a water-cooled precision diamond saw (Isomet 1000 Precision Saw, Buehler, Lake Bluff, IL, USA) to produce zirconia specimens with the required dimensions (10 mm×10 mm×2 mm). The zirconia plates were cut 20% larger than the desired dimensions to take into consideration shrinkage and were sintered at 1,500°C for 90 min (Programat S1, Ivoclar Vivadent). Each ceramic specimen was then mounted in cold-curing acrylic resin (Trey Resin II, Shofu Inc., Kyoto, Japan) and placed in tap water to reduce the temperature rise due to the exothermic polymerization reaction of the acrylic resin. All specimens were ground with 600-grit silicon carbide paper and airborne particle abraded with 50-μm grain size Al₂O₃ particles at 0.2 MPa pressure for 20 s at a distance of 10 mm. The specimens were then cleaned in an ultrasonic bath with distilled water for 10 min.

The cleaned samples were randomly divided into five groups, each consisting of 10 specimens. The contamination and cleaning procedures for each group are summarized in Table 2, and these specimens were used for bond strength and surface free energy measurements.

Group 1 (control): The specimens were not contaminated but were abraded and cleaned as mentioned above.

Group 2 (SC): After saliva contamination, the surface was rinsed with tap water for 30 s and air dried for 10 s.

Group 3 (PA): After saliva contamination, the surface was treated with Total Etch (Ivoclar Vivadent) for 30 s, rinsed with tap water for 30 s, and air dried for 10 s.

Group 4 (IC): After saliva contamination, the surface was treated with Ivoclean (Ivoclar Vivadent) for 20 s, rinsed with tap water for 30 s, and air dried for 10 s.

Group 5 (AB): After saliva contamination, the surface was subjected to additional airborne particle abrasion for 20 s, rinsed with water spray using a three-way syringe for 30 s, and air dried for 10 s.

For saliva contamination, saliva was obtained from one healthy male donor (the principal investigator) who

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**Table 1** List of materials used in this study and their main compositions according to the manufacturers

| System (Code)      | Main composition                                 | Manufacturer                  | Lot No.   |
|--------------------|-------------------------------------------------|-------------------------------|-----------|
| IPS e.max ZirCAD   | ZrO₂, Y₂O₃, HfO₂, Al₂O₃                         | Ivoclar Vivadent (Schaan, Lichtenshtein) | M47562    |
| Total Etch         | phosphoric acid, water, silica                  | Ivoclar Vivadent              | P14739    |
| Ivoclean           | sodium hydroxide, ZrO₂, water, polyethylene glycol, pigments | Ivoclar Vivadent              | NP66483   |
| Monobond Plus      | MDP, silane methacrylate, ethanol, sulfide methacrylate | Ivoclar Vivadent              | N7525     |
| Multilink Automix  | Base: dimethacrylates, ytterbium trifluoride, glass filler, silicon dioxide, initiators, stabilizers, pigments  | Ivoclar Vivadent              | N77855    |

*Multipurpose*: MDP: 10-methacryloyldecyl dihydrogen phosphate, HEMA: 2-hydroxyethyl methacrylate.

**Table 2** Study design for bond strength testing between resin cement and zirconia ceramics with different surface treatments

| Code | Pretreatment | Treatment | Priming |
|------|--------------|-----------|---------|
|      | Sand blasting| Saliva contamination | Phosphoric acid | Ivoclean | Re-sand blasting | Monobond Plus |
| Control | + | – | – | – | – | + |
| SC | + | + | – | – | – | + |
| PA | + | + | + | – | – | + |
| IC | + | + | – | + | – | + |
| AB | + | + | – | – | + | + |
had refrained from eating or drinking for 2 h before the collection process. The specimen was put into the saliva at 37°C for 60 s, then the specimen was rinsed with tap water for 15 s and air dried for 30 s. The study protocol was approved by the ethics committee of Nihon University School of Dentistry (#2011-19).

Surface free energy measurement
The surface free energies of five zirconia specimens per treatment group were determined by measuring the contact angle on the surface for three test liquids: distilled water, 1-bromonaphthalen, and ethylene glycol, each of which has known surface free energy parameters. The contact angle meter (Drop Master DM500, Kyowa Interface Science Co., Saitama, Japan) was fitted with a charge-coupled device camera, which allowed automatic measurements of the contact angles.

For each test liquid, the equilibrium contact angle (θ) was measured using the sessile drop method at 23±1°C for five specimens in each treatment group. Contact angle measurement was done soon after the test liquid was applied. The surface free energy parameters of the solids were then determined on the basis of the fundamental concepts of wetting. The Young-Dupré equation describes the work of adhesion for a solid (S) and a liquid (L) that are in contact (WSL), the interfacial free energy between the solid and the liquid (γSL), and the surface free energy of the liquid and the solid (γL and γS, respectively), as follows:

\[ W_{SL} = \gamma_{SL} - \gamma_{S} - \gamma_{L} \]

By extending the Fowkes equation, γSL is expressed as follows:

\[ \gamma_{SL} = \gamma_{L} + \gamma_{S} - 2(\gamma_{L}^d \cdot \gamma_{S}^d)^{1/2} - 2(\gamma_{L}^p \cdot \gamma_{S}^p)^{1/2} - 2(\gamma_{L}^h \cdot \gamma_{S}^h)^{1/2} \]

where \( \gamma_{L}^d, \gamma_{S}^d, \gamma_{L}^p, \gamma_{S}^p, \gamma_{L}^h, \) and \( \gamma_{S}^h \) are components of the surface free energy (γ) arising from the dispersion force, polar (permanent and induced) force, and hydrogen bonding force, respectively. θ values were determined for the three test liquids, and the surface free energy parameters of the treated dentin surfaces were calculated on the basis of the equations using add-on software and an interface measurement and analysis system (FAMAS, Kyowa Interface Science).

Preparation of bonding specimen
A piece of double-sided adhesive tape, with a 4-mm-diameter hole, was firmly attached to define the adhesive area of the cement for bonding. Monobond Plus (Ivoclar Vivadent) was applied to the specimen surface using a microbrush and allowed to stand for 60 s, and then the surface was thoroughly dried in air. A Teflon mold (height, 2.0 mm; diameter, 4.0 mm) was used to shape the resin cement and to hold it in place on the specimen surface. The auto-mixed cement was condensed into the mold and light cured for 20 s using a halogen curing unit (Optilux 501, Kerr Corp., Orange, CA, USA). A piece of double-sided adhesive tape, with a 4-mm-diameter hole, was firmly attached to define the adhesive area of the cement for bonding. Monobond Plus (Ivoclar Vivadent) was applied to the specimen surface using a microbrush and allowed to stand for 60 s, and then the surface was thoroughly dried in air. A Teflon mold (height, 2.0 mm; diameter, 4.0 mm) was used to shape the resin cement and to hold it in place on the specimen surface. The auto-mixed cement was condensed into the mold and light cured for 20 s using a halogen curing unit (Optilux 501, Kerr Corp., Orange, CA, USA).

All specimens were stored in water at 37°C for 24 h, after which they were randomly allocated to three groups (n=10 per group) for thermocycling: (1) no thermal cycling; (2) 10,000 thermal cycles between 5°C and 60°C (10,000TC); and (3) 30,000 thermal cycles between 5°C and 60°C (30,000TC). Thermocycling was conducted using a thermocycling machine (Thermal Shock Tester TTS-1 LM, Thomas Kagaku Corp., Tokyo, Japan). Each cycle consisted of water bath incubation for 30 s, with a transfer time of 5 s.

Bond strength test
Each group for each cement was tested in a shear mode using a shear testing apparatus in a universal testing machine (Instron Type 4204, Instron Corp., Canton, MA, USA) at a crosshead speed of 1.0 mm/min. The shear bond strength values (in MPa) were calculated from the peak load at failure divided by the specimen surface area. After testing, the specimens were examined under an optical microscope (SZH-131, Olympus Co., Tokyo, Japan) at a magnification of ×10 to define the location of bond failure. The type of failure was determined on the basis of the percentage of substrate-free material as adhesive failure or as cohesive failure in cement.

Scanning electron microscopy (SEM) observation
Ultrastructural observation of the ceramic surface was performed by SEM images. All SEM specimens were dehydrated and transferred to a critical point dryer for 30 min. In a vacuum evaporator (Quick Coater Type SC-701, Sanyu Denshi Inc., Tokyo, Japan), the surfaces were coated with a thin film of Au and were observed under a scanning electron microscope (ERA 8800FE, Elionix Ltd., Tokyo, Japan) at an operating voltage of 10 kV.

Statistical analysis
Results were analyzed by two-way analysis of variance (ANOVA) followed by Tukey’s honestly significant difference test to compare the surface treatments (α=0.05) for each cement. All statistical analyses were performed using the Sigma Stat software system version 3.1 (SPSS Inc., Chicago, IL, USA).

RESULTS
The contact angles for the three test liquid are shown in Table 3. From these values, the surface free energies and their components resulting from the different zirconia surface treatments were calculated as shown in Fig. 1. For all of the treated enamel surfaces, \( \gamma_{Sh} \) values remained relatively constant (37.2–42.6 mN/m). \( \gamma_{Sp} \) and \( \gamma_{Sh} \) values of the SC group decreased. \( \gamma_{Sp} \) and \( \gamma_{Sh} \) values of the PA group increased substantially but remained significantly lower than those of the control group. No significant differences were found for the IC and AB groups compared with the control group.

The influence of surface treatment on the shear bond strength of the resin cement to zirconia ceramics is shown in Table 4. Two-way ANOVA revealed that surface treatment (p<0.01) and storage conditions (p=0.03) had a significant influence on bond strength, while there was no significant interaction between the two factors.
(p=0.449). The control, IC, and AB groups showed relatively higher bond strength, and no significant differences were found among these three groups under different storage conditions. The bond strength was significantly lower in the SC group than in the control group (p<0.001). The PA group showed significantly higher bond strength than the SC group, but it showed significantly lower strength than the control, IC, and AB groups (p<0.01). Adhesive failure was the predominant failure mode for all specimens (Fig. 2), and a correlation was observed between the bond strength and the failure mode, indicating that bond strengths lower than 13.5 MPa resulted in adhesive failures.

SEM observations are shown in Fig. 3a–e. Relatively rough surfaces created by airborne particle abrasion were observed in the control group (Fig. 3a). The surface contaminated with saliva was covered by a thin film of amorphous deposits (Fig. 3b). In contrast, surface in the PA group was roughened and appeared similar to that in the control group, but with small amounts of minute

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**Table 3** Influence of surface treatment of saliva-contaminated zirconia on contact angles (°) of three test liquids

| Treatment | Water | 1-Bromnaphthalin | Diiodomethane |
|-----------|-------|-----------------|---------------|
| Control   | 24.0 (1.1) | 11.0 (1.1) | 22.8 (1.2) |
| SC        | 58.0 (1.0) | 25.8 (1.0) | 47.5 (0.8) |
| PA        | 45.0 (1.0) | 19.7 (1.0) | 32.9 (1.1) |
| IC        | 24.5 (1.2) | 8.1 (1.9) | 23.1 (1.0) |
| AB        | 25.1 (1.1) | 9.5 (1.2) | 28.1 (0.8) |

n=5, ( ): S.D. Values connected by horizontal lines indicate not significantly different (p>0.05).

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**Table 4** Influence of surface treatment of saliva-contaminated zirconia on bond strength (MPa) of resin cement

| Treatment | 24 h | Group | 10,000 TC | Group | 30,000 TC | Group |
|-----------|------|-------|-----------|-------|-----------|-------|
| Control   | 16.4 (2.9) | [8/2/0] | 15.0 (1.5) | [8/2/0] | 13.5 (2.1) | [8/2/0] |
| Fracture mode |        | a     |           | a     |           | b     |
| SC        | 5.7 (0.5) | [10/0/0] | 4.5 (0.8) | [10/0/0] | 2.9 (0.9) | [10/0/0] |
| Fracture mode |        | e     |           | e     |           | f     |
| PA        | 11.4 (2.5) | [10/0/0] | 7.9 (2.1) | [10/0/0] | 5.3 (1.8) | [10/0/0] |
| Fracture mode |        | c     |           | d     |           | e     |
| IC        | 14.9 (2.2) | [9/1/0] | 13.8 (1.9) | [9/1/0] | 11.9 (2.1) | [9/1/0] |
| Fracture mode |        | a     |           | b     |           | b, c |
| AB        | 16.4 (1.9) | [8/2/0] | 15.7 (2.0) | [8/2/0] | 13.0 (2.0) | [8/2/0] |
| Fracture mode |        | a     |           | a     |           | b     |

n=10, ( ): S.D. Values with the same letter are not significantly different (p>0.05).

Fracture mode: [adhesive/cohesive in resin cement/cohesive in zirconia]

TC: Thermal cycles
Fig. 2  Representative SEM photomicrographs of the cement surfaces debonded from zirconia ceramics after bond strength test. All groups showed adhesive failure between zirconia ceramics and resin cement surfaces.

Fig. 3  Representative SEM photomicrographs of the surface of zirconia ceramics after different surface treatments. (a) Control, (b) saliva contamination (SC), (c) phosphoric acid etching (PA), (d) Ivoclean treatment (IC), and (e) additional airborne particle abrasion (AB). In the control group, relatively rough surfaces created by airborne particle abrasion were observed (a). The surface contaminated with saliva was covered by a thin film of amorphous deposits (b). Residual small aggregated particles were observed on the treated surfaces in the IC group (Fig. 3d), and rough surfaces, very similar to those in the control group, were observed in the AB group (Fig. 3e).

**DISCUSSION**

Optimal interaction between the resin cement and the ceramic surface is important to enable the cement to spread across the entire ceramic surface and thus
establish optimum adhesion. Factors that influence the wetting of a solid by a liquid include the relative surface free energy of the solid and the surface tension of the liquid. The wetting of the adherent surface by an adhesive could be indicated by the contact angle. Measurements of the contact angle on adherent surfaces provide information about the surface free energies that relate to the bonding characteristics of solids. The surface free energy of a solid ($\gamma_s$) is defined as the sum of the dispersion, hydrogen bonding, and polar forces. The dispersion force ($\gamma_d$) represents London interactions between apolar molecules. The polar (non-dispersion) force ($\gamma_p$) represents electric and metallic interactions, in addition to the dipolar interactions. In addition to these two parameters of $\gamma_s$, the hydrogen bonding force ($\gamma_h$), which relates to water and hydroxyl components, was calculated in this study. Because hydration of the adherent surface is of major importance to the wettability behavior related to bonding, polar interactions, including dipole and hydrogen bonding characteristics, should be accurately estimated for the interaction with water. A separate estimation of the dipole (polar) interactions, apart from the hydrogen bonding interactions, might provide novel insight into the mechanisms contributing to wettability as well as the bonding characteristics. The $\gamma_s$ value must be maximized to achieve optimal wettability, and the liquid should exhibit a lower contact angle to the solid.

Factors influencing the bonding of resin cement to zirconia ceramics include the wettability of the ceramic by adhesive resin cements, roughness of the ceramic surface, ingredients of the resin cements, sensitivity of the material handling technique, and possible contamination during bonding procedures such as by saliva. In the present study, bond strengths of the cement to zirconia ceramics significantly decreased after saliva contamination (5.7 MPa) compared with the controls (16.4 MPa). After 30,000 thermal cycles, bond strength in the SC group decreased to 2.9 MPa, which was only 50.9% of the initial bond strength. The predominant failure mode for SC group was adhesive failure, indicating surface contamination of zirconia ceramics related to the decrease in bond strengths. This result is in agreement with that of previous studies, which showed reduced bond strengths after saliva contamination of ceramic surfaces. Saliva contamination adversely affects resin bonding because organic deposits remain on the restorative materials after the first few seconds of exposure to saliva. Water rinsing alone is not sufficient to remove saliva contamination. Saliva contains more than 99% water, combined with small amounts of proteins, glycoprotein sugars, amylase, and inorganic particles. After saliva contamination, non-covalent adsorption of salivary proteins occurs on the surfaces of restorative materials, creating an organic coating that cannot be removed by rinsing with water. A possible explanation for the decreased bond strength is that an invisible thin residual organic film, which was observed in SEM (Fig. 3b), covers the ceramic surface and prevents chemical bonding to zirconia ceramics, while thermal cycling then further interferes with the formation of a durable bond. Lower bond strength values and a high percentage of adhesive failure could be explained by the fracture phenomena at the surface area of zirconia ceramics. The results of surface free energy measurements showed that a significantly lower value was obtained for the surface in the SC group (46.6 mN·m$^{-1}$) than for that in the control group (72.0 mN·m$^{-1}$). Therefore, the null hypothesis that saliva contamination has no influence on the bonding of the resin cement to zirconia ceramics or on surface free energy should be rejected.

Cleaning with phosphoric acid was not effective in removing saliva contamination as shown by the fact that both the initial bond strength (11.4 MPa) and the bond strength after thermal cycling were both remarkably lower (7.9–5.3 MPa). Cleaning of saliva-contaminated zirconia ceramics with phosphoric acid improved bond strength but was not able to re-establish the same bond strength as that in the control group. And the fracture mode after the bond strength test was adhesive failure for PA and SC groups, indicating durability of resin cement bonding to zirconia ceramics was not satisfactory. Cleaning with phosphoric acid is based on the removal of residual organics, which are readily dissolved in acid. A previous study using X-ray photoelectron spectroscopy to identify the existence of saliva contamination on zirconia ceramic surfaces showed that phosphoric acid removed almost all organic contaminants. However, data obtained from the present study revealed that bond strengths in the PA group were significantly decreased after thermal cycling. The results of surface free energy measurements indicated that the PA group had a significantly lower energy than the control group (57.8 mN·m$^{-1}$ and 72.0 mN·m$^{-1}$, respectively). One possible explanation for the decrease in bond strength is that phosphoric acid changes the surface free energy of the ceramic surface for bonding, leading to a reduction in bonding properties although organic contaminants have been removed. This phenomenon was corresponding to the fracture mode after the bond strength test.

Saliva consists of phosphate groups in the form of phospholipids, which actively bond to the internal surface of restorations. According to the manufacturer’s scientific documentation, Ivoclean contains zirconia, water, polyethylene glycol, sodium hydroxide, and other additives. Phosphate contaminants on the ceramic surface are more likely to bond to the particles in Ivoclean than to the ceramic surface because the size and concentration of the particles were adjusted for this purpose. In accordance with this theory, Ivoclean might absorb the phosphate contaminants and leave behind a clean zirconium oxide surface. The initial bond strength was lower in the IC group than in the control group, but the difference was not significant. After thermal cycling, bond strength in the IC group decreased significantly, and the bond strength after 30,000 cycles was 11.9 MPa, which was 79.9% of the initial bond strength. On the other hand, no significant differences were found for the surface free energy (70.0 mN·m$^{-1}$) compared with the
control group. A possible explanation for the reduction in bond strength is the presence of residual agglomerated small particles detected in SEM images, which could reduce bonding durability after thermal cycling.

Among the cleaning methods tested, airborne particle abrasion was the most effective method for removing contaminants, as shown by bond strength and surface free energy measurements. Airborne particle abrasion might remove contaminants from ceramic surfaces and expose a fresh bonding surface by mechanical removal of superficial ceramics, leading to a durable bond between the zirconia ceramics and the resin cement, as suggested previously.

The present study indicates that a thin layer of contaminants remains on the zirconia ceramic surface after exposure to saliva, significantly impeding the bonding of the resin cement and reducing the surface free energy of the ceramics. Airborne particle abrasion was the most effective method of removing the contaminants from saliva-contaminated zirconia ceramics and creating an effective surface for resin cement bonding.

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