Effect of ethyl cellulose coating as an evaluation agent against contamination on the bond strength of zirconia restorations: An in vitro study

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Zirconia-based ceramics have gained popularity as a framework or anatomic contour material because of the demand for esthetics. 1 Zirconia has excellent biocompatibility and strength, with reasonable esthetics, and is produced by computer-aided design and computer-aided manufacturing (CAD-CAM) technologies. 2-5 Although an optimal cementation protocol for zirconia ceramic restorations has yet to be established, airborne-particle abrasion with 50-μm alumina oxide (Al2O3) as a surface conditioning procedure and cementation with an adhesive system containing phosphate ester monomers such as 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) has been shown to result in stable, high-strength bonding. 6-7

ABSTRACT

Statement of problem. During the trial placement of zirconia restorations, contamination of the bonding surface is inevitable. Although cleaning methods for contaminated surfaces have been described, a method of preventing saliva contamination of the bonding surface of zirconia restorations is lacking.

Purpose. The purpose of this in vitro study was to investigate an ethyl cellulose coating as an evaluation agent to mitigate the effects of saliva contamination on the bond strength of zirconia restorations.

Material and methods. Experimental groups representing different cleaning methods of tetragonal yttria-stabilized tetragonal zirconia polycrystal (4Y-TZP) surfaces were investigated for shear bond strength with a resin luting agent, and the failure mode was analyzed. The 9.0×7.0×5.0-mm zirconia blocks (n=72) were assigned as follows: Group N: uncontaminated control; Group CU: contaminated with saliva, followed by ultrasonic cleaning with ethanol; Group CI: contaminated with saliva, followed by application of a zirconia cleaner; Group PCW: preapplication of a zirconia primer, contaminated with saliva, followed by cleaning with water spray; Group ECU: precoating with the ethyl cellulose agent, contaminated with saliva, followed by removal of the agent in an ultrasonic bath with ethanol. Each group was divided into 3 subgroups (immediate, short-term aging, and long-term aging), and the shear bond strength was measured (n=24). To analyze the bonding surface characteristics, the contact angle was measured (n=5). The surfaces of the zirconia specimens in each experimental group were evaluated by using a field emission scanning electron microscope (n=5). Time-of-flight secondary ion mass spectroscopy was used for the chemical analysis of the conditioned surfaces (n=3). A 2-way analysis of variance (ANOVA) with main effect model for shear bond strength results and a 1-way ANOVA for contact angle data were performed as statistical analysis, followed by the Bonferroni post hoc test (α=.05).

Results. The shear bond strength was significantly higher in the ECU group than in the groups with the other cleaning methods (P<.05). After the removal of ethyl cellulose with ethanol, the contact angle and surface topography were found to be similar to those of the control group, and no saliva contaminants were identified in the spectroscopy analysis.

Conclusions. Coating with ethyl cellulose may protect the bonding surface of zirconia restorations from salivary contamination better than cleaning a contaminated surface. (J Prosthet Dent 2022; -

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A clean surface, free of contaminants, is the primary prerequisite for effective bonding between adhesives and adherends. In clinical situations, the intaglio surface of the restoration is inevitably contaminated with saliva, blood, gingival crevicular fluid, and silicone evaluation paste during the trial placement procedure, and saliva contamination has been shown to be the main cause of reduced bond strength. The phosphate component of oral fluid and the acid etchant has a high affinity for zirconia, potentially leading to interference with the chemical reaction between the bonding surface and adhesive molecules such as 10-MDP. Residual organic contaminants compromise the quality of adhesion between the tooth and the restoration, which may negatively affect its longevity.

The intaglio surface of zirconia restorations can be cleaned after contamination with airborne-particle abrasion. However, excessive stress-generating surface conditioning has been reported to change the structural stability and increase susceptibility to long-term degradation, but alternative cleaning methods, such as ultrasonic cleaning, have been reported to provide insufficient decontamination. Alkaline cleaning agents such as Ivoclean (Ivoclar Vivadent AG) have been recently developed. These highly caustic cleaning agents with hypersaturated solutions of zirconia oxide particles decompose proteins and absorb phosphate contaminants but can leave particles on the surface.

Although short-term studies have reported favorable clinical outcomes with such cleaning agents, long-term studies are lacking. Moreover, whether the surface after application of the cleaning agents is similar to that of the original, uncontaminated abraded surface, especially with respect to surface free energy, is uncertain. A previous study reported that if saliva contamination occurs after 10-MDP application, rinsing off the saliva with water would preserve bond strength because the primer made the zirconia surface more hydrophobic. This practical method has also been used clinically.

Ethyl cellulose (EC) is a derivative of cellulose, which is produced by substituting the hydroxyl groups of the repeated glucose of the cellulose with ethyl ether groups. EC is used industrially as a thin-film coating material for paper and medical pills and as a thickener in cosmetics. The major physicochemical properties of EC include insolubility in water, high solubility in organic solvents, chemical stability, excellent biocompatibility, and high resistance to tearing. These qualities suggest that EC can be adopted as an evaluation coating agent for zirconia-based ceramics. If the bonding surface is covered with an EC agent before the trial placement, it might be protected from contaminants. In addition, EC can be easily removed by organic solvents after trial placement, contributing to the efficient bonding of zirconia restorations.

This in vitro study aimed to determine whether an EC coating agent had a protective effect on the bonding surface of zirconia restorations subsequently contaminated with saliva. The null hypothesis was that the bond strength of the EC-protected zirconia surface would be similar to that of an uncontaminated zirconia surface and contaminated surfaces treated with various cleaning methods.

**MATERIAL AND METHODS**

The compositions and batch numbers of the materials used in this study are listed in Table 1. The EC coating agent was 1.5 wt% EC (Daejung Chemical) dissolved in 98.5 wt% ethanol. The ratio of EC to ethanol was determined through preliminary experiments. Because EC has low solubility in ethanol at room temperature, it was heated to 43 °C and completely dissolved by using a magnetic stirrer. After complete dissolution, the heated coating agent was cooled and stored at 18°C for 72 hours before use.

Three hundred sixty 9.0×7.0×5.0-mm zirconia specimens were cut from presintered zirconia disks (4Y-TZP, Zircon; Kuwotech) by using a high-speed diamond saw (Isomet 5000; Buehler). The surfaces of the specimens to be bonded were serially polished with 320-, 600-, and 1000-grit silicon carbide (SiC) papers. The specimens were then ultrasonically cleaned for 3 minutes in pure ethanol, rinsed with distilled water, and air-dried.

The saliva used in the experiments was obtained from one of the authors (M.P.) according to Kim et al. All experiments were performed with whole fresh saliva collected at the same time. Except for the control group, all specimens were immersed in the collected saliva for 1 minute. The specimens were assigned to 5 study groups (n=72) according to the decontamination methods. The assigned experimental groups are shown in Figure 1.
After the specimens were treated with their corresponding decontamination method, a zirconia primer (Z-prime plus; Bisco) was applied, which represents the method commonly used in the clinic to prevent salivary contamination, to all groups except Group PCW. The primer was allowed to react for 5 minutes, and the solvent was thoroughly air-dried.

Rods of a resin luting agent (Ø2.38×2.1 mm) were formed on the zirconia surface by using a bonding device (Ultradent Dental Products, Inc) with a cylindrical Teflon mold. The resin luting agent (Duo-Link Universal; Bisco) was poured into the mold, covered with a Mylar strip, and then photopolymerized (SmartLite focus; Dentsply Sirona) for 60 seconds at a...
distance of 1 mm from the light tip at an irradiance of 1300 mW/cm².

All specimens were soaked in distilled water at 37 °C for 24 hours. The specimens of each group were assigned to 3 subgroups (n=24) of artificial aging conditions: immediate; stored in distilled water at 100 °C for 6 hours, equivalent to 6 months in an oral environment; or stored in distilled water at 100 °C for 24 hours, equivalent to 2.2 years in an oral environment. Artificial aging was conducted in a constant-temperature water bath (WH-4; Nasco Korea Corp.).

All specimens were tested in the shear mode by using a universal testing machine (AGS-X; Shimadzu) at a crosshead speed of 1 mm/min. The load was applied to the adhesive interface with a knife edge until failure occurred. The maximum stress required to produce a fracture was recorded (MPa) as the shear bond strength (SBS). After testing, the specimens were examined under an optical microscope (SZN745 stereo microscope; Sunny) at ×40 magnification to identify the location of bond failure. Failure patterns were classified as adhesive failure that occurred at the interface between the zirconia ceramic and resin luting agent, cohesive failure that occurred in the resin luting agent or zirconia ceramic, and mixed failure, a combination of adhesive and cohesive failures.

Six Ø10.0×7.0×2.0-mm zirconia disks were fabricated by a dental laboratory technician for contact angle measurement, with 5 assigned to the experimental groups as described previously, that is, 1 to each group, after the specified protocol of each group had been completed. The sixth disk was assigned to another group (Group P, positive control), which was only airborne-particle abraded, without contamination, and treated with zirconia primer. The disks were serially polished with 320-, 400-, and 600-grit SiC papers. A drop of distilled water (10 μL) was placed onto the zirconia disks and maintained until equilibrium was reached. The contact angle was measured by using a contact angle analyzer (Phoenix 10 contact angle tester; SEO) with the SurfaceWare 8 software program (n=5) under ambient conditions (temperature: 20 ±2 °C; relative humidity: 40 ±3%). The contact angle of the water droplet was calculated by averaging the contact angles on the left and right sides. The drop volume was 1 μL, and the dropping distance was 10 mm.

The structure of the zirconia surfaces was observed by using a field emission scanning electron microscope (FE-SEM) (S-4700; Hitachi High Technologies Co) when the surface treatment of the 6 groups had been completed (n=5). The specimens were dried, gold sputter-coated, and examined by using an FE-SEM at an operating voltage of 10 keV. For Group ECU, the EC agent thickness was measured and averaged over 5 points along the layer of the SEM image.

### Table 2. Mean ± standard deviation of shear bond strength (MPa)

| Group   | Immediate | 100 °C 6 h | 100 °C 24 h | Mean ± SD |
|---------|-----------|------------|------------|-----------|
| Group N | 23.53 ± 3.40<sup>a</sup> | 15.16 ± 2.60<sup>b</sup> | 7.76 ± 1.17<sup>c</sup> | 15.49 ± 2.39 |
| Group CU | 12.53 ± 3.87<sup>a</sup> | 6.16 ± 1.49<sup>b</sup> | 4.26 ± 1.17<sup>c</sup> | 7.65 ± 2.17 |
| Group CI | 23.40 ± 3.67<sup>a</sup> | 14.31 ± 2.80<sup>b</sup> | 6.67 ± 2.06<sup>c</sup> | 14.99 ± 2.48 |
| Group PCW | 18.34 ± 3.82<sup>a</sup> | 12.65 ± 2.23<sup>b</sup> | 6.27 ± 1.40<sup>c</sup> | 12.42 ± 2.49 |
| Group ECU | 24.77 ± 3.58<sup>a</sup> | 15.06 ± 2.48<sup>b</sup> | 7.97 ± 1.45<sup>c</sup> | 15.93 ± 2.50 |
| Mean ± SD | 19.81 ± 4.13 | 12.19 ± 2.65 | 6.31 ± 1.30 | -- |

CI, contaminated with saliva, followed by application of zirconia cleaner; CU, contaminated with saliva, followed by ultrasonic cleaning with pure ethanol; ECU, specimens were coated once with EC agent and stored at 18 °C–21 °C for 24 hours to allow complete evaporation of solvent, and subsequently specimens were contaminated with saliva and then cleaned in ultrasonic bath using pure ethanol for 3 minutes; N, no saliva contamination (control group); PCW, surfaces had zirconia primer applied, were contaminated with saliva, and then were cleaned with water spray using 3-way syringe for 20 seconds; SD, standard deviation. n=24 Values with same lowercase letters in same column and same uppercase letters in same row not significantly different (P<.05).

A time-of-flight secondary ion mass spectrometry (TOF-SIMS; TOF.SIMS 5; IONTOF GmbH) was used for the chemical analysis of the conditioned surfaces of the specimens in each experimental group described in the contact angle measurement section (n=3) for the 6 groups. It was conducted at an energy of 15 keV after in situ gallium-ion sputtering. The sputter-depth profiles were recorded by using a 1 keV Cs<sup>+</sup> ion beam and a raster size of 500×500 μm. All specimens were rinsed with deionized water and air-dried before the analysis.

The SBS of the experimental groups was compared by using a 2-way analysis of variance (ANOVA) with the cleaning method and aging condition as factors, followed by the Bonferroni post hoc test. The main effect model was used for the analysis. Contact angle measurements were compared by using 1-way ANOVA and the Bonferroni post hoc test (P<.05 for all tests). A statistical software program (IBM SPSS Statistics, v23.0.0; IBM Corp) was used for all analyses.

### RESULTS

This study was sufficiently powered (1-β=0.94) to demonstrate significant differences among the 5 experimental groups. The SBS values of all experimental groups are shown in Table 2. The 2-way ANOVA analysis showed that the artificial aging condition and cleaning method significantly affected the SBS (P<.001). The interaction between these 2 factors was statistically significant (P<.001). The SBS data were analyzed and interpreted by using the main effect model, and, in all experimental groups, the bond strength decreased with the aging time (P<.05). In the immediate test and the test after 6 hours of artificial aging, the bond strengths in Groups N, CI, and ECU were significantly higher than those in Groups CU and PCW (P<.05), and the N, CI, and ECU groups were statistically similar (P>.05). After 24 hours of artificial aging, specimens in Groups N and ECU exhibited higher bond strengths than those in the other groups (P<.05). Figure 2 shows the distribution of...
The failure patterns of the specimens. Failure mode analysis showed that mixed failure was predominant in the experimental groups. As the aging time increased, the proportion of mixed failure decreased, and the rate of adhesive failure increased in all experimental groups.

The contact angles are presented in Table 3. The water contact angle of zirconia ceramics changed significantly depending on the cleaning methods used. The specimens in the 5 experimental groups showed significantly lower contact angles than those in group P (positive control) (P<.05). Groups N and ECU had the lowest contact angle among all experimental groups (P<.05), but no statistical difference was observed between the results from Groups N and ECU (P=.663).

Figure 3 shows representative SEM images of the zirconia ceramic surfaces after treatment with different surface cleaning methods. Airborne-particle abrasion created relatively rough surfaces in Group N specimens (Fig. 3A). An amorphous, thin film partially covered the surfaces of the Group CU specimens (Fig. 3B). In the image of Group CI (Fig. 3C), small spherical agglomerated particles were observed on the surfaces of the specimens. As opposed to the specimens treated with the MDP primer alone (Fig. 3D), amorphous particles were partially observed on the smooth surfaces of the specimens in Group PCW (Fig. 3E).

Figure 4 shows SEM images after applying the EC coating agent and after ultrasonic cleaning with pure ethanol. In Group ECU (Fig. 4B), rough surfaces resembling those present in Group N were observed after the removal of the EC coating agent during ultrasonic cleaning with ethanol. It was confirmed that the coating...
Figure 3. Scanning electron microscope images of zirconia specimens after cleaning using various methods (original magnification ×5000). A, After airborne-particle abrasion (Group N). B, After saliva contamination (Group CU). Surface of specimen appeared coated with amorphous materials. C, After cleaning agent (Ivoclean; Ivoclar Vivadent AG) application (Group CI). Spherical agglomerates observed to remain on surface. D, After zirconia primer application (Group P). Airborne-particle abraded surface coated with zirconia primer to provide uniform and smooth surface. E, After saliva contamination in preprimed group (Group PCW). Small amount of amorphous material remained on primer treated surface, presumed to be component of saliva not removed during washing after saliva contamination.

Figure 4. Scanning electron microscope images of specimen surface after EC thin-film coating and cleaning (original magnification ×5000). A, Surface coated with EC. Homogeneous and smooth surface observed. B, Surface of specimen after ultrasonic cleaning. Surface similar to that obtained immediately after airborne-particle abrasion, and coated EC completely removed. C, Cross section of specimen coated with EC thin film. White arrows indicate EC film layer.

agent completely decomposed after the cleaning. Figure 4C shows a cross-sectional image of the agent-coated zirconia specimen. The thickness of the EC agent was also measured by using the SEM image, and the mean ± standard deviation thickness was approximately 37 ±19 μm.

Figure 5 shows the negative ion spectra in the 0 to 200 mass region for each experimental group. The spectra of the Group N (Fig. 5A) and Group ECU specimens (Fig. 5F) were similar. In all experimental groups, strong peaks were observed in mass regions 13 (CH\(^{-}\)), 16 (O\(^{-}\)), 17 (OH\(^{-}\)), and 25 (C\(_2\)H\(^{-}\)), presumably because of contamination with organic matter in the air and water absorption. Figure 5B shows distinctive peaks at mass regions 63 (PO\(_2\)\(^{-}\)), 79 (PO\(_3\)\(^{-}\)), and 97 (H\(_2\)PO\(_4\)\(^{-}\)) in Group P, which were treated only with zirconia primer. These distinctive peaks were observed when chemical bonding occurred between the zirconia surface and zirconia primer.\(^{36,39}\) In Group CU, compared with Group N, strong peaks were observed in regions 26 (CN\(^{-}\)), 42
Mass regions 59 and 71 indicated sugar headgroups that were mainly from the fragments of glycolipids. The peaks at mass regions 26 and 42 were assumed to be caused by the protein fragments of saliva. The peak at region 79 was assumed to be caused by PO$_3^-$, which is a component of saliva and a component of MDP-containing zirconia primer. In Group CI (Fig. 5D), distinctive peaks were observed in mass regions 26, 41, 42, 45, 71, and 79. E, Saliva contamination after application of zirconia primer (Group PCW). Peaks detected in mass regions 42, 45, 79, and 159. F, Ultrasonic cleaning after EC thin-film coating (Group ECU). Similar to surface obtained immediately after airborne-particle abrasion.

**DISCUSSION**

The results of this study determined that the EC coating did not reduce adhesion to zirconia. In the SBS test after aging, Groups N and ECU exhibited a higher bond strength than the other groups ($P<.05$), suggesting that the contamination cleaning and prepriming methods tested did not restore the bond strength to zirconia after contamination with saliva such as would occur during trial placement. Therefore, the null hypothesis was partially rejected because Group ECU showed no significant difference in bond strength compared with the uncontaminated control, whereas it did demonstrate a
higher bond strength than groups cleaned by other methods. 

Previous studies have focused on methods of removing contaminants attached to the bonding surface of zirconia restorations.²⁰⁻⁴¹ Ishii et al²⁰ examined the influence of saliva contamination and cleaning methods on the bond strength of a resin luting agent to zirconia and the surface free energy of zirconia. Yoshida⁴¹ compared different cleaning regimens of saliva-contaminated zirconia and reported that a commercial cleaning agent was effective in removing saliva contaminants. The current study focused on EC coating as a method of preventing the contamination of the bonding surfaces of zirconia restorations during trial placement. 

Airborne-particle abrasion increases roughness and surface energy, which decreases the contact angle between the water and the bonding surface.⁶,⁴²,⁴³ After the removal of EC, the contact angle decreased to a level equal to that of Group N. In Group PCW, even if the contaminant was removed from the primed zirconia surface, it did not return to the contact angle of the prepriming group (Group P), which showed an increased contact angle because of the hydrophobicity of the surface. 

A decrease in zirconia bond strength after saliva contamination is caused by the chemical bonding between the activated zirconia surface and phosphorous components such as phospholipids in the saliva and the adsorption of residual organic matter, especially salivary protein, on the restoration surface.¹⁰,³⁴,⁴⁴ Groups CU and PCW exhibited strong 26 (CN), 42 (CNO⁻), and 79 (PO₃⁻) peaks in the TOF-SIMS analysis, which were not observed in Groups N or ECU (Figs. 5).⁴⁵⁻⁴⁷ suggesting that the residual proteins and phosphorus components of saliva remained on the primed surface of the zirconia.

In addition, the present study determined that the applied EC coating agent did not leave residue on the bonding surface after its removal. The TOF-SIMS results showed that Group ECU exhibited peaks similar to those of Group N. Additionally, in the FE-SEM images, after the removal of EC by the organic solvent, an airborne-particle abraded rough surface was retrieved without any evidence of residue from the EC agent (Fig. 4B). Group ECU showed an EC layer approximately 30-μm thick, as shown in Figure 4C, which is below the clinically acceptable cement space (100 μm) of the CAD-CAM system.⁴⁶⁻⁴⁹ The thickness may be acceptable to avoid interference with the fit of the restoration during the trial placement procedure. However, further assessments related to the marginal fit and accuracy of adaptation after EC coating are needed to test the feasibility of clinical use.

The purpose of this study was to determine whether coating the zirconia surface with a thin film of EC before the trial placement procedure would protect it from deleterious effects produced by direct contact with saliva contaminants. Limitations of the present study included its in vitro design and that only organic contaminants were evaluated. Other contaminant sources at trial placement, including gypsum and silicone, should be evaluated in further studies. Biological toxicity was not evaluated and would also be required for clinical adoption. Toxic chemical components such as 20% toluol might be involved in the EC synthesis process.²⁷ The EC coating method should be further optimized and adopted for clinical use in future studies and clinical trials.

**CONCLUSIONS**

Based on the findings of this in vitro study, the following conclusions were drawn:

1. An EC-coated contaminated zirconia surface exhibited better bond strength than with different cleaning methods.
2. The EC-coated specimens showed good surface recovery after contamination, without leaving a residue.

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