Effects of different silica-based layer coatings on bond strength of Y-TZP to bovine dentin

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This study investigated the effects of different silica-based layer coatings on shear bond strength (SBS) between Y-TZP and bovine dentin. Three different silica-based layer coatings were applied to the Y-TZP surface: tribochemical silica coating, vitrification (glaze coating), and composite resin sintering. A silane coupling agent (SIL) was applied to the silica-coated Y-TZP surface in the presence or absence of hydrofluoric acid (HF) treatment. A one-step adhesive was then applied to the silica-coated Y-TZP and cemented to bovine dentin using MDP-free resin cement. The SBS value of the tribochemical silica coating group was lowest among the experimental groups, while the HF+SIL subgroup showed the highest SBS value after vitrification (p<0.05). While hydrofluoric acid etching did not affect the SBS value of the tribochemical silica coating group, it affected the SBS value in the vitrification and composite resin sintering groups (p<0.05).

Keywords: Vitrification, Composite resin sintering, Tribochemical silica coating

INTRODUCTION

Zirconia has attracted attention for use as a tooth-colored indirect restoration due to its biocompatibility and aesthetic and mechanical properties1-4. The most commonly used zirconia ceramic is a tetragonal zirconia polycrystalline stabilized with 3 to 8 mol% yttrium oxide (Y-TZP). Unlike feldspathic ceramics, zirconia has high crystallinity and no glassy phase. Due to this unique structure, surface pretreatment for traditional ceramic restorations, such as hydrofluoric acid etching or application of silane coupling agents, do not improve bond strength to a zirconia surface3-5.

Many studies have investigated increasing the bond strength to zirconia6-9. According to these studies, 10-methacryloyloxydecyl dihydrogen phosphate (MDP) can form strong chemical bonds with the hydroxyl groups on the zirconia surface, improving the bond strength to zirconia7,8. Blatz et al.10 reported superior long-term bonding strength to zirconia restorations when using MDP-containing silane primer. However, the other studies11-13 have reported that improved bond strength to zirconia treated with MDP-containing primers cannot be maintained for a long time due to accelerated hydrolysis of the bonding site in the mouth. In addition, the bond strength to zirconia when using MDP-containing cement has not yet reached the bond strength to silica ceramic pretreated with hydrofluoric acid treatment and silane primer14.

Alternative surface modification methods have been introduced to improve bond strength to zirconia, including selective infiltration etching (SIE)15, plasma spraying16, coating with silica-based ceramic17-19, coating with silica-like seed layers20, and experimental zirconia-silica coating21. These methods have improved mechanical retention, chemical retention, or both to increase the bond strength to zirconia.

Among these methods, those forming a silica-based layer are tribochemical silica coating20-22, vitrification23 (glaze coating), and composite resin sintering19. The silica-based layer coating creates a zirconia surface with many silicon oxides that can be made more retentive and reactive by selectively conditioning23-25, similar to the bonding mechanisms of feldspathic ceramics.

The purpose of this study was to investigate the effects of different silica-based layer coatings on shear bond strength (SBS) between Y-TZP and bovine dentin. The null hypothesis was that the various silica-based layer coatings do not affect the bond strength of Y-TZP to bovine dentin.

MATERIALS AND METHODS

The materials used in the study are listed in Table 1.

Specimen preparation
Seventy Y-TZP specimens and bovine teeth were prepared for SBS testing. A pre-sintered Y-TZP block (NexxZr T, Sagemax, Federal Way, WA, USA) was used and sintered according to the manufacturer’s instructions to obtain Y-TZP specimens of standard cylinder shape (3 mm in diameter, 10 mm in height). The bovine teeth were cut to obtain root dentin. The dentin specimens were then embedded in polymethylmethacrylate (PMMA) resin in a polytetrafluoroethylene (PTFE) mold with an inner diameter of 7 mm, an outer diameter of 11 mm, and a height of 10 mm. One side of the dentin was exposed for cementation with the Y-TZP specimen. The exposed
Table 1. Materials used, manufacturers and main components

| Material/Trade name                        | Manufacturer                  | Main components                                      |
|------------------------------------------|-------------------------------|-------------------------------------------------------|
| Y-TZP ceramic/                          | Sagemax, Federal Way, WA, USA | 91.6% ZrO₂, 5% Y₂O₃, 3% HfO₂                          |
| NexxZr T (Shade: A1)                     |                               |                                                       |
| Tribochemical silica/                    | 3M ESPE, St. Paul, MN, USA    | Aluminum oxide 110 µm modified with silica           |
| Rocatec Plus                             |                               |                                                       |
| Vitrification/                           | Ivoclar Vivadent, Schaan, Liechtenstein | 60–70% Ceramic powder and pigments                  |
| IPS e.max Ceram glaze                    |                               | 30–40% Glycole and glycerin                          |
| Composite resin/                         | 3M ESPE                       |                                                       |
| Filtek supreme XT                        |                               |                                                       |
| Silane coupling agent/                   | Dentzply, Konstanz, Germany   | 3-TMSPMA, Ethanol, Acetone                            |
| Calibra                                  |                               |                                                       |
| One-step adhesive/                       | Bisco, Schaumburg, IL, USA    | MDP, Bis-GMA, HEMA, Ethanol, Water, Initiators       |
| All-Bond Universal                       |                               |                                                       |
| Resin cement/                            | Bisco                         | Base: Bis-GMA, TEGDMA, UDMA, glass filler             |
| Duo-link Universal                       |                               | Catalyst: Bis-GMA, TEGDMA, glass filler              |

Dentin surface for adhesion was polished using 1,000, 1,500, and 2,000 grit silicon carbide paper; ultrasonically cleaned in distilled water for 5 min; and dried. Prior to the SBS test the bovine dentin specimens were stored in distilled water at 37°C for 24 h to achieve thorough wetting.

Silica-based layer coating on the Y-TZP surface

Untreated sintered Y-TZP specimens were used as control and other experimental groups were coated with three different silica-based layers as follows.

Tribochemical silica coating: Tribochemical silica airborne-particle (Rocatec plus, 3M ESPE, St. Paul, MN, USA) abraded on the bonding surface of the Y-TZP specimen from a distance of 10 mm for 10 s at 0.3 MPa.

Vitrification (glaze coating): The glaze coating material (IPS e.max Ceram glaze, Ivoclar Vivadent, Schaan, Liechtenstein) was applied to the bonding surface of the Y-TZP specimen using a brush. The Y-TZP specimen was then sintered in a dental porcelain furnace at a rate of 60°C/min from 403 to 703°C.

Composite resin sintering: The flowable composite resin (Filtek supreme XT Flowable, 3M EPSE) was applied to the bonding surface of the Y-TZP specimen with a micro-brush. The Y-TZP specimen was then sintered in a dental porcelain furnace at a heating rate of 15°C/min and a maximum temperature of 1,100°C for 10 min. The specimen was then cooled at room temperature for 100 min.

Pretreatment procedure

The Y-TZP specimens of each silica-based layer coating were further subdivided into two subgroups according to etching and priming conditions. (A) SIL: A silane coupling agent (SIL) (Calibra, Dentsply, Konstanz, Germany) was applied to the silica-based layer coating surface of the Y-TZP specimen. The silane coupling agent was dried, reacted for 1 min, and then gently air-dried with oil-free air. After silanization, a one-step adhesive system (All-Bond Universal, Bisco, Schaumburg, IL, USA) was used as a primer. (B) HF+SIL: The silica-based layer coating surface of the Y-TZP specimen was etched with 9.5% hydrofluoric acid (HF) gel for 1 min, rinsed with air-water spray, and dried. The silane coupling agent and one-step adhesive system were then applied in the same way as in the SIL group.

The one-step adhesive system was also applied to the bonding surface of bovine dentin as a dentin adhesive. MDP-free resin cement (Duo-Link Universal, Bisco) was mixed and applied to the pre-treated surface of the Y-TZP specimen. Under a constant load of 5 newtons (N), light curing was performed for 3 s for tack curing, and excess cement was carefully removed with an explorer. While maintaining the load, all surfaces were light-cured for 20 s. The specimens that underwent luting procedures were stored in water at 36°C for 24 h before the SBS test.

SBS test and analyses of failure modes

Each specimen was mounted in a jig of a universal testing machine (Model 5543, Instron, Canton, MA, USA), and shear force was loaded onto it using a chisel-shaped
metal rod at a constant crosshead speed of 0.5 mm/min until failure (Fig. 1). The SBS was calculated using the following formula: SBS(MPa)=load(N)/area(mm²). The load was measured as the force at which the failure occurred.

After measuring the SBS, the debonded surfaces were analyzed using a stereomicroscope (DM 2500M, Leica Microsystems, Wetzlar, Germany) to determine the failure mode between the Y-TZP and bovine dentine. The failure modes were categorized into four types:

- Type 1, adhesive mode: failure between Y-TZP and cement
- Type 2, mixed mode: failure between Y-TZP and cement and within cement
- Type 3, cohesive mode: failure within cement
- Type 4, adhesive mode: failure between cement and bovine dentin

The proportion of each failure type was calculated. Representative morphologies of debonded specimens were investigated.

**SEM and EDS analyses of silica-based layer coatings**

One specimen from each silica-based layer coating group was coated with gold-palladium alloy using a sputter-coating technique (Ion sputter E-1030, Hitachi, Tokyo, Japan) and examined by SEM (SU8230, Hitachi) to observe the morphology of the silica-based layer coating. Moreover, SEM coupled with energy-dispersive spectroscopy (EDS; X-max® large area silicon drift detector, Oxford Instruments, Abingdon, UK) was used for identifying and quantifying the elemental compositions of the silica-based layer-coated areas. EDS spectra were conducted under 10kV voltage condition. Elemental mapping by EDS was used to record the two-dimensional elemental composition of the surfaces of defined sample section.

**Statistical analyses**

Statistical analyses were performed using statistical software (SPSS Version 18.0, SPSS, Chicago, IL, USA). Mean and standard deviation (SD) values of SBS (n=10) were analyzed using two-way analysis of variance (ANOVA), and the pairwise multiple comparison procedure (Tukey’s test) was used for determining significant differences among the groups. A p-value less than 0.05 was considered statistically significant in all tests.

**RESULTS**

**SBS test and failure modes**

Mean SBS values and SDs are summarized in Table 2.

| Silica-based layer coating methods       | Pretreatment | Bond strength* |
|-----------------------------------------|--------------|----------------|
| Control                                 | No pretreatment | 2.54±0.89A |
| Tribochemical silica coating            | SIL          | 8.60±3.05B |
|                                         | HF+SIL       | 8.50±2.41B |
| Vitrification (Glaze coating)           | SIL          | 13.59±3.14C |
|                                         | HF+SIL       | 19.84±4.35B |
| Composite resin sintering               | SIL          | 12.78±2.75C |
|                                         | HF+SIL       | 9.54±3.71B |

SIL: silane coupling agent application followed by primer application, HF+SIL: etching with hydrofluoric acid plus SIL

*Identical uppercase letters indicate no statistically significant differences (p>0.05).
Two-way ANOVA showed that the silica-based layer coating and etching conditions had a significant effect on SBS. The interactions of the silica-based layer coating and etching conditions were also statistically significant.

The SBS values of the tribochemical silica coating group were lowest among the experimental groups, while the HF+SIL subgroup showed the highest SBS value after vitrification. While hydrofluoric acid etching did not affect the SBS value in the tribochemical silica coating group, it affected the SBS values in the vitrification and composite resin sintering groups. In the vitrification group, the SBS value of the HF+SIL subgroup was significantly higher than that of the SIL subgroup. In the composite resin sintering group, hydrofluoric acid etching led to the opposite result, with the SBS value of the SIL subgroup significantly higher than that of the HF+SIL subgroup.

Figure 2 shows the failure patterns in the debonded area between Y-TZP and bovine dentin. The tribochemical silica coating group showed 70% Type 1 failure and 30% Type 2 failure regardless of hydrofluoric acid etching. Only Type 3 failure was observed in the vitrification group. Type 1 failure was reduced, and Type 2 failure was increased in the composite resin sintering group compared to the tribochemical silica coating group.

**SEM and EDS analyses**

Representative SEM images of the specimens coated with various silica-based layers on the Y-TZP surface are presented in Fig. 3. The tribochemical silica coating specimen showed a change in surface texture involving formation of micro-retentive grooves. SEM images of vitrified and composite sintered specimens showed that the zirconia surface was completely covered by the coating layer. However, a smooth surface was observed in the vitrified specimen, while a rough surface was detected in the composite resin sintered specimen.

Elemental mapping images of the specimens coated with each silica-based layer are presented in Fig. 4. Table 3 shows the elemental composition of the specimens. The sample surface of the control specimen was composed mostly of Oxygen (O) and Zirconium (Zr) elements, with only a small amount of Yttrium (Y) element. In the tribochemical silica-coated specimen, only Aluminum element was observed in addition to O, Zr, and Y elements. While several other elements were observed on the surface of the vitrified specimen, Zr element was not detected. O, Si, Y, and Zr elements were

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**Fig. 2** Failure mode analysis after bonding strength measurement of silica-coated experimental groups with or without hydrofluoric acid treatment on Y-TZP surface.

SIL: silane coupling agent followed by primer application, HF+SIL: hydrofluoric acid etching plus SIL.

**Fig. 3** SEM images (original magnification, ×1,000) of various silica-based layer coatings on Y-TZP surface.

(a) Control, (b) Tribochemical silica coating, (c) Vitrification (glaze coating), (d) Composite resin sintering.

**Fig. 4** Elemental mapping of images after coating various silica-based layers on Y-TZP surface.

(a) Control, (b) Tribochemical silica coating, (c) Vitrification (glaze coating), (d) Composite resin sintering.
Table 3 Changes in elemental composition after coating of different silica-based layers on Y-TZP surface

| Silica-based layer coating methods       | O   | Al  | Si  | Y   | Zr  | etc |
|-----------------------------------------|-----|-----|-----|-----|-----|-----|
| Control                                 | 26.52 | —   | —   | 4.28 | 69.21 | —   |
| Tribochemical silica coating            | 29.51 | 6.73 | —   | 11.00 | 52.76 | —   |
| Vitrification (Glaze coating)           | 42.66 | 2.71 | 32.65 | 9.45 | —    | 12.53 |
| Composite resin sintering               | 40.26 | —   | 33.22 | 14.32 | 12.20 | —   |

O: Oxygen, Al: Aluminum, Si: Silicon, Y: Yttrium, Zr: Zirconium

detected on the surface of the composite resin-sintered specimen. Compared with the control specimen, the proportion of Zr element decreases, the proportion of O and Y elements increases, and the Si element occupies the remaining proportion of the surface of the coated sample in the composite resin-sintered specimen.

DISCUSSIONS

For cementation of Y-TZP restoration, clinicians use various cements ranging from conventional cements (e.g., zinc phosphate cement, glass ionomer cement, and resin-modified glass ionomer cement) to resin cement\(^{26,27}\). Of these, resin cement provides greater retention\(^{28}\), lower solubility, and lower marginal leakage\(^{29}\). Although resin cement performs better than other conventional cements, bonding to Y-TZP remains a challenge\(^{30,31}\). For this reason, various surface conditioning techniques such as air-abrasion with particles of alumina or silica (tribochemical silica coating)\(^{32,33}\), use of zirconia primers\(^{34}\), glazing\(^{35}\), and deposition of silica films\(^{36,37}\) are used to improve the bond strength between Y-TZP and resin cement.

Among them, silica-based layer coating has been proposed as a promising method for conditioning Y-TZP. When the Y-TZP surface is coated with a silica-based layer, the Y-TZP surface acts like a silicon oxide-based ceramic. That is, the silica-based layer coating surface provides a chemically reactive surface and the possibility for micromechanical bonding by acid etching.

In this experiment, a silica-based layer was coated on Y-TZP surfaces by three different methods, improving bond strength. In particular, bonding strength was highest when hydrofluoric acid etching and silane coupling agent were applied after vitrification. When vitrification forms a glassy layer on the Y-TZP surface\(^{38}\), the silica-based layer resembles a glass-based ceramic surface. After vitrification, two mechanisms can explain the increased bond strength. 1) Micromechanical retention: The Y-TZP surface vitrified by silica coating was etched by hydrofluoric acid to produce strong micromorphological changes such as increased surface roughness and formation of micro-retainive grooves\(^{34,35}\). 2) Chemical bonding: Large amounts of Si elements may have improved chemical interactions with silane coupling agents\(^{36}\).

The group with silica-coated Y-TZP surface by sintering of the composite resin showed significantly higher bonding strength than the control group. After applying zirconia-silica nanoparticles embedded in uncured composite resin onto the Y-TZP surface, sintering the composite resin produces a surface as shown in Fig. 3(d). The SEM image of the composite resin sintered group showed that the zirconia-silica nanoparticles completely covered the Y-TZP surface, increasing the surface roughness. In addition, since this intermediate layer contains a large amount of Si element, as seen in the EDS analysis, it can form a chemical bond between the intermediate layer and the resin cement.

In terms of ceramic adhesion, hydrofluoric acid etching dissolves the amorphous glass phase selectively in the leucite crystalline structures and forms soluble hexafluoroasilicates. Surface irregularities occurred in the etching process, resulting in increased surface area and energy. Etching also exposes hydroxyl (OH) groups that can readily react with those of the silane coupling agent\(^{37,38}\). However, in the present study, the unusual thing about the composite resin sintering group was that the bond strength decreased when etched with hydrofluoric acid. This is presumed to be because the silica-based layer formed by sintering of the composite resin was composed only of glass, so the intermediate layer was almost completely dissolved by hydrofluoric acid etching, exposing the previously applied silica-based layer coating.

Pretreatment with tribochemical silica coating roughens the Y-TZP surface and induces a chemical reaction between silica and Y-TZP\(^{39}\). However, in this study, the tribochemical silica coating group had the lowest SBS value among the silica coating groups. In the EDS analysis, the tribochemical silica coating layer showed no Si, implying that a silica coating layer was not actually produced. This result suggests that the injection pressure of aluminum oxide particles is weakened. In this experiment, a tribochemical silica coating was applied in the chairside. As a result of examining the injection pressure, it was confirmed that the chairside pressure (0.3 MPa) was weaker than the laboratory pressure (0.4 MPa). If tribochemical coating was applied with aluminum oxide particles of 30 µm size or injection pressure in the laboratory, Si element would have been detected.
Failure modes on the bonding surfaces between Y-TZP and bovine dentin reflected the SBS values. The tribocomical silica coating group showed Type 1 failure (adhesive failure) at a high rate, supporting low bond strength between Y-TZP and resin cement. Only Type 3 failure (cohesive failure) was observed in the vitrification group, showing good bond strength between Y-TZP and resin cement.

Given the present results, hydrofluoric acid etching and application of silane coupling agent after vitrification seems to be a promising method to increase bond strength. However, to better establish new silica-based layer coating methods such as vitrification and sintering of composite resins, further problems must be investigated including bonding durability, influence of water storage and thermocycling, and interactions with MDP-containing primer or/and MDP-containing resin cement. In addition, clinical studies are needed to confirm the clinical feasibility of these procedures.

CONCLUSIONS
From the study results, a silica-based coating on the Y-TZP surface increased the bond strength between Y-TZP and bovine dentin. Therefore, the null hypothesis was rejected. In addition, among the alternatives tested in this study, the most effective way to improve bond strength is to apply hydrofluoric acid etching and silane coupling agent after vitrification on the Y-TZP surface.

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