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Research Article

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The effect of continuous application of MDP-containing primer and luting resin cement on bond strength to tribochemical silica-coated Y-TZP

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Conceptualization: Lim MJ, Lee KW; Data curation: Lim MJ; Formal analysis: Lim MJ, Yu MK; Funding acquisition: Lee KW; Methodology: Lim MJ, Lee KW; Resources: Lim MJ, Yu MK; Supervision: Yu MK, Lee KW; Visualization: Lim MJ; Writing - original draft: Lim MJ; Writing - review & editing: Lim MJ, Yu MK, Lee KW.

ABSTRACT

Objectives: This study investigated the effect of continuous application of 10-methacryloyloxydecyldihydrogen phosphate (MDP)-containing primer and luting resin cement on bond strength to tribochemical silica-coated yttria-stabilized tetragonal zirconia polycrystal (Y-TZP).
Materials and Methods: Forty bovine teeth and Y-TZP specimens were prepared. The dentin specimens were embedded in molds, with one side of the dentin exposed for cementation with the zirconia specimen. The Y-TZP specimen was prepared in the form of a cylinder with a diameter of 3 mm and a height of 10 mm. The bonding surface of the Y-TZP specimen was sandblasted with silica-coated aluminium oxide particles. The forty tribochemical silica-coated Y-TZP specimens were cemented to the bovine dentin (4 groups; $n = 10$) with either an MDP-free primer or an MDP-containing primer and either an MDP-free resin cement or an MDP-containing resin cement. After a shear bond strength (SBS) test, the data were analyzed using 1-way analysis of variance and the Tukey test ($\alpha = 0.05$).
Results: The group with MDP-free primer and resin cement showed significantly lower SBS values than the MDP-containing groups ($p < 0.05$). Among the MDP-containing groups, the group with MDP-containing primer and resin cement showed significantly higher SBS values than the other groups ($p < 0.05$).
Conclusions: The combination of MDP-containing primer and luting cement following tribochemical silica coating to Y-TZP was the best choice among the alternatives tested in this study.

Keywords: Luting cement; MDP; Primer; Y-TZP

INTRODUCTION

Zirconia has superior physical properties among oxide ceramics, with good esthetics and biocompatibility, and received considerable attention when it was introduced in dentistry [1,2]. Zirconia has since started to be used in esthetics and restoration dentistry, and it is now used extensively, from inlay/onlay and single crown treatments to bridge and implant abutments and partial fixed dental prostheses [3-5].

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The most commonly used zirconia ceramic is yttria-stabilized tetragonal zirconia polycrystal (Y-TZP), which is made by stabilizing tetragonal zirconia polycrystalline by adding 3–8 mol% yttrium oxides (Y_2O_3) [6,7]. However, zirconia has a polycrystalline structure that does not contain amorphous silica glass and is acid-resistant. Therefore, surface treatments for traditional porcelain, such as hydrofluoric acid etching and/or silane primer application, are not effective for increasing the adhesion between the zirconia restoration and the teeth [8,9].

A number of studies have been conducted to solve the problem of adhesion between zirconia restorations and teeth [10-15]. Most studies have focused on tribochemical silica coating and 10-methacryloyloxydecyldihydrogen phosphate (MDP) primer. The tribochemical silica coating technique includes sandblasting the silica-coated alumina particles on the zirconia surface to increase the surface roughness. Additionally, the tribochemical silica coating creates a layer that is chemically more reactive to the luting resin cement through a silane primer [16,17]. The MDP-containing primer can chemically bind strongly to the hydroxyl groups on the zirconia surface. Blatz *et al.* [18] reported superior long-term bonding strength to zirconia restorations when using MDP-containing silane primer.

The proper selection of luting agents is very important for effective bonding to zirconia restorations. Phosphate monomer-based luting agents have been proposed for cementation of zirconia restorations [19,20]. Multi-step MDP-containing luting resin cements have been previously investigated, and they showed satisfactory results [21,22].

Recently, new universal adhesives containing MDP and new single-step self-adhesive resin cements containing MDP have been marketed and are becoming a common choice for cementation of zirconia restorations [23,24]. Few studies have been conducted on new universal adhesives containing MDP as a zirconia primer and new self-adhesive resin cements containing MDP as a luting agent for zirconia.

The purpose of this study was to investigate the effect of continuous application of MDP-containing primer and MDP-containing luting resin cement on shear bond strength (SBS) between bovine dentin and tribochemical silica-coated Y-TZP. The null hypothesis was that the continuous application of MDP-containing primer and luting resin cement would have no effect on SBS between bovine dentin and tribochemical silica-coated Y-TZP.

MATERIALS AND METHODS

The materials listed in **Table 1** were used in this study. Pre-sintered Y-TZP blocks (NexxZr T, Sagemax, Federal Way, WA, USA), which have a 5% shrinkage, were used instead of pure zirconium dioxide. Primer and luting resin cement were selected for MDP-free and MDP-containing materials, depending on the presence of the MDP component.

Specimen preparation

Forty bovine incisors and Y-TZP specimens were prepared for the SBS test. The bovine incisors were cleaned using a scaler and stored in distilled water at 4°C. The bovine teeth were then cut to obtain dentin specimens of suitable sizes for polytetrafluoroethylene molds with an inner diameter of 7 mm, an outer diameter of 11 mm, and a height of 10 mm. The dentin specimens were embedded in the mold using polymethylmethacrylate resin, with one side of the dentin exposed for cementation with the zirconia specimen. The Y-TZP blocks

Table 1. Materials used, manufacturers, and main components

Material/trade name	Manufacturer	Main components
Y-TZP ceramic/NexxZr T	Sagemax, Federal Way, WA, USA	91.6% ZrO_2, 5% Y_2O_3, 3% HfO_2
Tribochemical silica/Rocatec Plus	3M ESPE, St. Paul, MN, USA	Aluminum oxide (110 μm) modified with silica
MDP-free primer/ESPE Sil	3M ESPE, St. Paul, MN, USA	3-TMSPMA, ethanol
MDP-containing primer/G-premio bond	GC Corp., Tokyo, Japan	MDP, 4-MET, MEPS methacrylate monomer, acetone, water, initiator, silica
MDP-free cement/Rely X ARC	3M ESPE, St. Paul, MN, USA	Paste A: Bis-GMA, TEGDMA, zirconia/silica filler, pigments, amine and photo-initiator system Paste B: zirconia/silica filler, benzoyl peroxide
MDP-containing cement/G-cem LinkAce	GC Corp., Tokyo, Japan	Paste A: fluoro-alumino-silicate glass, UDMA, dimethacrylate, silicon dioxide Paste B: phosphoric acid monomer, silicon dioxide, UDMA, dimethacrylate

Y-TZP, yttria-stabilized tetragonal zirconia polycrystal; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; 3-TMSPMA, 3-trimethoxysilylpropyl methacrylate; 4-MET, 4-methacryloyloxyethyl trimellitate; MEPS, methacryloyloxyalkyl thiophosphate methylmethacrylate; Bis-GMA, bisphenyl A glycidyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

were milled by computer-assisted design/computer-assisted manufacturing, and then finally sintered to form a cylindrical specimen (3 mm in diameter and 10 mm in height).

The bonding surface of all specimens, including bovine teeth and Y-TZP, was polished using 1,000, 1,500, and 2,000 grit silicon carbide papers, ultrasonically cleaned in distilled water for 5 minutes, and dried.

Surface treatment and luting procedure

The bonding surface of the Y-TZP specimens was sandblasted with silica-coated 110 μm aluminum oxide particles (Rocatec plus, 3M ESPE, St. Paul, MN, USA) from a distance of 10 mm for 10 seconds at 0.3 MPa.

The specimens were randomly divided into 4 groups as follows (n = 10):

1) The SR group: The MDP-free primer (ESPE Sil, 3M ESPE) was applied to the surface of tribochemical silica-coated Y-TZP with a disposable brush, followed by air-drying. The MDP-containing primer (G-premio bond, GC Corp., Tokyo, Japan) was applied to the bovine dentin surface with a rubbing motion for 10 seconds, and the surface was then dried thoroughly for 5 seconds with air under maximum air pressure. After the primer became a thin film with the appearance of frosted glass, it was light-cured for 10 seconds. The luting procedure was performed by applying the MDP-free resin cement (Rely X ARC, 3M ESPE).

2) The GR group: The MDP-containing primer (G-premio bond, GC Corp.) was applied to the surface of tribochemical silica-coated Y-TZP and the bovine dentin surface with a rubbing motion for 10 seconds, followed by thorough drying for 5 seconds with air under maximum air pressure. After the primer became a thin film with the appearance of frosted glass, it was light-cured for 10 seconds. The luting procedure was performed by applying the MDP-free resin cement (Rely X ARC, 3M ESPE).

3) The SG group: The MDP-free primer (ESPE Sil, 3M ESPE) was applied to the surface of tribochemical silica-coated Y-TZP and the MDP-containing primer (G-premio bond, GC Corp.) was applied to the bovine dentin surface in the same manner as above. The luting procedure was performed by applying the MDP-containing resin cement (G-cem LinkAce, GC Corp.).

4) The GG group: The MDP-containing primer (G-premio bond, GC Corp.) was applied to the surface of tribochemical silica-coated Y-TZP and the bovine dentin surface was applied as above. The luting procedure was performed by applying the MDP-containing resin cement (G-cem LinkAce, GC Corp.).

Table 2. Experimental groups according to the materials (primer and luting cement) used

Group	Surface treatment	Primer	Luting cement
SR		MDP-free primer*	MDP-free cement‡
GR	Tribochemical silica coating	MDP-containing primer†	MDP-free cement‡
SG		MDP-free primer*	MDP-containing cement§
GG		MDP-containing primer†	MDP-containing cement§

SR, the group with MDP-free primer pretreatment and MDP-free resin cement; GR, the group with MDP-containing primer and MDP-free resin cement; SG, the group with MDP-free primer and MDP-containing resin cement; GG, the group with MDP-containing primer pretreatment and MDP-containing resin cement; MDP, 10-methacryloyloxydecyldihydrogen phosphate.
*ESPE Sil; †G-premio bond; ‡Rely X ARC; §G-cem LinkAce.

The luting procedure was performed under a constant load of 5 newtons (N). Tack curing was done for 3 seconds and the excess cement was carefully removed with an explorer. While maintaining the load, all surfaces were light-cured for 20 seconds each. The specimens subjected to the luting procedure were stored in water at 36°C for 24 hours before the SBS test. **Table 2** summarizes the experimental groups, surface treatments, primers, and luting resin cements applied on the Y-TZP surface.

SBS test and analysis of failure modes

Each specimen was mounted in the 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 occurred (**Figure 1**). The SBS was calculated using the following formula:

$$\text{SBS (MPa)} = \text{load (N)}/\text{area (mm}^2\text{)}$$

The load was measured by recording the force at which failure occurred.

After measuring the SBS, the debonded surface was analyzed using a stereomicroscope (DM 2500M, Leica Microsystems, Wetzlar, Germany) to determine the mode of failure between the Y-TZP and bovine dentin. The failure mode was classified as adhesive, cohesive, or mixed, and the ratio of each failure mode was calculated. Representative morphologies of debonded specimens were investigated by scanning electron microscopy (SEM; S-5500, Hitachi, Tokyo, Japan) after sputtering using a gold-palladium alloy conductive layer (Ion sputter E-1030, Hitachi).

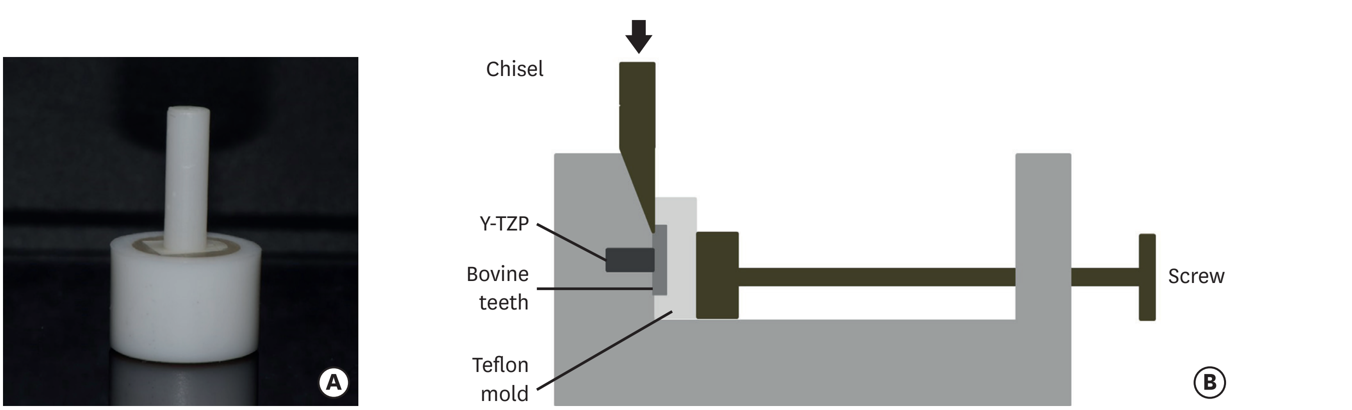

Figure 1. (A) Shear bond strength (SBS) test machine; (B) Representative image of the SBS test, with a specimen mounted in the jig of the machine. Arrow, application of a load by a universal testing machine.
Y-TZP, yttria-stabilized tetragonal zirconia polycrystal.

Statistical analysis

Statistical analyses were performed using statistical software (SPSS version 18.0, SPSS Inc., Chicago, IL, USA). The mean and standard deviation values of the SBS were analyzed using 1-way analysis of variance, and the all-pairwise multiple-comparison procedure (Tukey test) was used for identifying significant differences among the groups. The p values less than 0.05 were considered to indicate statistical significance in all tests.

RESULTS

The SBS values between bovine dentin and Y-TZP for the 4 groups are depicted in **Figure 2**. The SR group (8.31 ± 0.96 MPa) with MDP-free primer pretreatment and MDP-free resin cement showed significantly lower SBS values than the MDP-containing groups: GR, 11.12 ± 1.66 MPa; SG, 10.88 ± 0.95 MPa; and GG, 15.21 ± 2.61 MPa ($p < 0.05$). Among the MDP-containing groups, the GG group with MDP-containing primer pretreatment and MDP-containing resin cement showed significantly higher SBS values than the other groups ($p < 0.05$). In contrast, there was no statistically significant difference between the GR group with MDP-containing primer and MDP-free resin cement and the SG group with MDP-free primer and MDP-containing resin cement.

Figure 3 shows the failure modes in the debonded area between bovine dentin and Y-TZP. A schematic diagram below the graph showing the failure modes in **Figure 3** illustrates the debonding patterns at the zirconia-resin cement-bovine dentin interface. SEM images of the debonded Y-TZP surface and bovine dentin surface after the SBS test are shown in **Figure 4**. The SR group showed 50% adhesive failure at the zirconia-resin cement interface and 50% mixed failure. Compared to the SR group, adhesive failure in the GR and SR groups was less common, and mixed failure was more common. The GG group showed 10% adhesive failure at the zirconia-resin cement interface, 70% mixed failure, and 20% cohesive failure within the resin cement. Cohesive failure was only observed in the GG group.

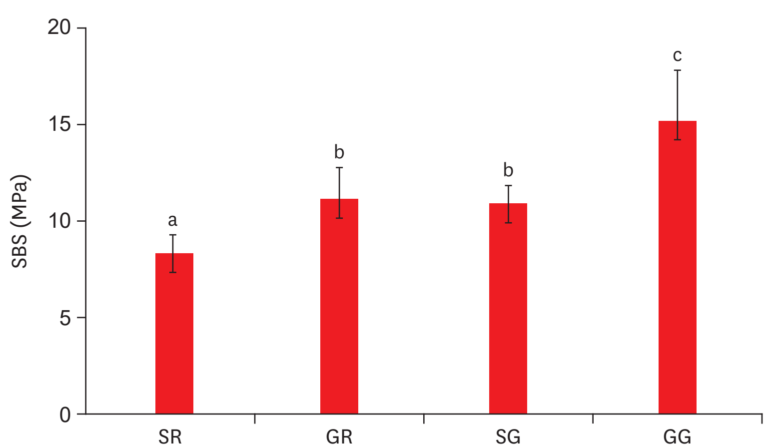

Figure 2. The shear bond strength (SBS) of yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) bonded to bovine dentin with different primers and luting cements. Groups indicated with different letters showed significant differences ($p < 0.05$).
SR, the group with MDP-free primer pretreatment and MDP-free resin cement; GR, the group with MDP-containing primer and MDP-free resin cement; SG, the group with MDP-free primer and MDP-containing resin cement; GG, the group with MDP-containing primer pretreatment and MDP-containing resin cement; MDP, 10-methacryloyloxydecyldihydrogen phosphate.

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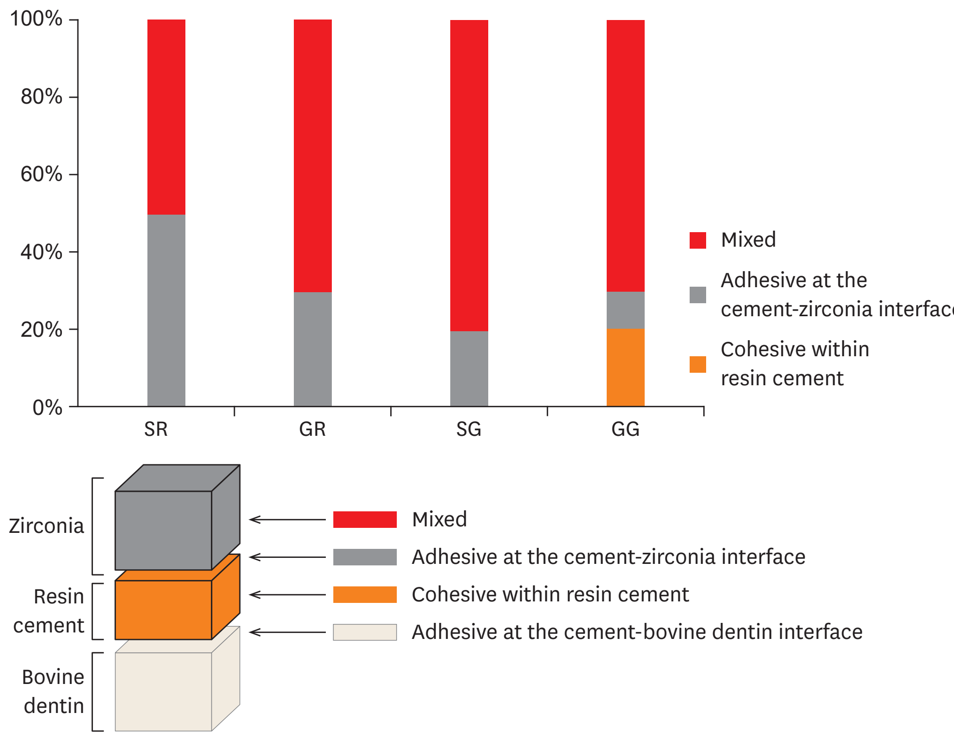

Figure 3. Schematic diagram and illustration of the percentages of the failure modes after the shear bond strength (SBS) test of yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) bonded to bovine dentin.
SR, the group with MDP-free primer pretreatment and MDP-free resin cement; GR, the group with MDP-containing primer and MDP-free resin cement; SG, the group with MDP-free primer and MDP-containing resin cement; GG, the group with MDP-containing primer pretreatment and MDP-containing resin cement; MDP, 10-methacryloyloxydecyldihydrogen phosphate.

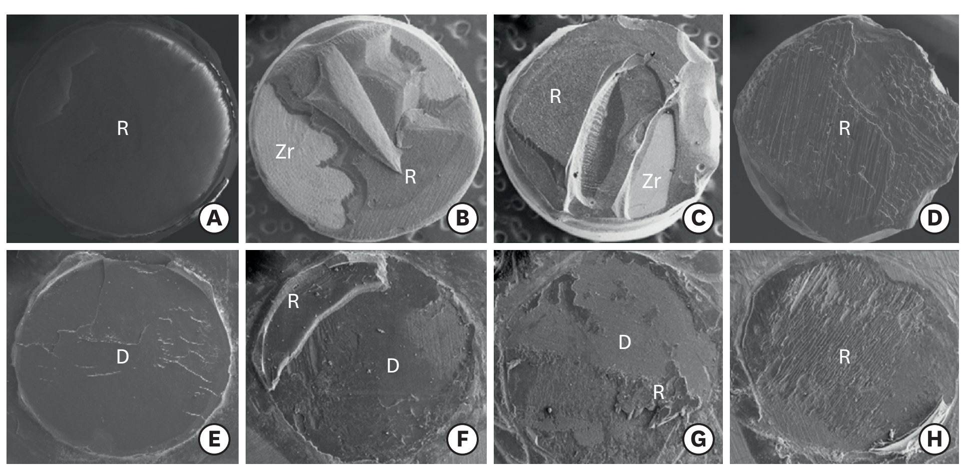

Figure 4. Scanning electron microscopy images (×30 magnification) of debonded yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) surface (A-D) and bovine dentin surface (E-H) after the shear bond strength (SBS) test. (A, E) Adhesive failure at the zirconia-resin cement interface in the SR group; (B, F) Mixed failure in the GR group; (C, G) Mixed failure in the SG group; (D, H) Cohesive failure within resin cement in the GG group.
Zr, Y-TZP surface; R, resin cement; D, bovine dentin; SR, the group with MDP-free primer pretreatment and MDP-free resin cement; GR, the group with MDP-containing primer and MDP-free resin cement; SG, the group with MDP-free primer and MDP-containing resin cement; GG, the group with MDP-containing primer pretreatment and MDP-containing resin cement; MDP, 10-methacryloyloxydecyldihydrogen phosphate.

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DISCUSSION

Conventional Al_2O_3 sandblasting has been widely used on zirconia as a surface treatment. However, it is likely to produce surface defects depending on the pressure and the size of the particles, and to compromise the mechanical strength of Y-TZP at high pressure [25,26]. In contrast, for tribochemical silica coating, not only does mechanical bonding result from an increase in the surface roughness, but chemical bonding can also be achieved via siloxane bonds [27,28]. Therefore, the Y-TZP surface treatment method was chosen as a roughening procedure for tribochemical silica coating instead of conventional Al_2O_3 sandblasting in this study. However, controversy exists regarding tribochemical silica coating. According to Matinlinna *et al.* [29], the silica content on the Y-TZP bonding surface after tribochemical silica coating is too low to achieve sufficient silanization. Moreover, siloxane bonds may be sensitive to hydrolytic degradation in conditions involving contact with moisture, such as water storage and thermocycling, thus affecting the long-term stability of the adhesive interface [16,30].

G-cem LinkAce, one of the luting resin cements used in this study, is a self-adhesive, dual-cure, and MDP-containing resin cement. According to the manufacturer's instructions, application of this resin cement is very simple and can be accomplished in a single clinical step without any pre-treatment. However, previous studies have reported that some self-adhesive resin cements had a low bonding strength when applied to intact zirconia surfaces where neither tribochemical silica coating nor application of primer had been done [24,27,28]. For this reason, the luting resin cement used in the study was applied to the Y-TZP surface pretreated with tribochemical silica coating and primer application.

The MDP monomer consists of 3 different functional groups: the methacryloyl, decyl, and dihydrogen phosphate groups [31]. The methacryloyl group acts as an essential component for the copolymerization of the MDP monomer, while the dihydrogen phosphate group chemically bonds to the zirconium oxides [32]. More specifically, 2 hydrogen groups derived from the phosphate group react with zirconium oxides to form a stable Zr-O-P covalent bond [19,20]. According to Byeon *et al.* [33], the Zr-O bond was confirmed using XPS analysis, proving that a chemical bond between MDP and Y-TZP occurred. This can explain why the MDP-containing groups showed significantly higher bond strengths than the MDP-free group.

In this study, the MDP monomer was applied twice on the bonding surface of tribochemical silica-coated Y-TZP by primer and luting resin cement. The MDP monomer of the first applied primer would be expected to be a major contributor to the chemical bond between MDP and Y-TZP. However, since the primed layer on the bonding surface of Y-TZP could act as a semi-permeable membrane after polymerization, it could be expected that the MDP monomer of luting cement is recharged with a primed layer even after the solvent is volatilized. As a result, the MDP-containing experimental group in both the primer and the luting resin cement would have provided a higher supply of dihydrogen functional groups capable of chemically bonding with zirconium oxide than the MDP-containing experimental group in either the primer or luting cement. For this reason, the group with a combination of MDP-containing primer and luting resin cement showed the highest shear bond strength, with statistical significance. Therefore, the null hypothesis was rejected.

The failure modes in the debonded area between Y-TZP and bovine dentin reflect the SBS [33]. According to Cavalcanti *et al.* [34] and Zandparsa *et al.* [35], mixed failure at the interface between zirconia and resin cement occurred predominantly, in contrast to mixed failure, at the interface between luting resin cement and tooth substrate, and a markedly higher rate of adhesive failure is associated with weak bond strength between zirconia and the luting resin cement. Thus, in MDP-containing groups such as the GR, SG, and GG groups, the proportion of adhesive failure was lower, but the rate of mixed failure was higher than in the MDP-free SR group. This means that there is a close correlation between SBS and the failure mode.

This study has the following limitations. Water storage and thermocycling, which could affect the bonding strength of Y-TZP, were not considered. Further research is needed on other surface treatments of Y-TZP, the adhesion of self-adhesive MDP-containing resin cements to zirconia, and long-term water storage conditions and thermocycling as challenges to bonding surfaces.

CONCLUSIONS

The combination of MDP-containing primer and luting cement following tribochemical silica coating was the best choice among the alternatives tested in this study for bonding Y-TZP to bovine dentin.

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