



Repair Bond Strength of Composite to Zirconia Ceramic Using Two Types of Zirconia Primers

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ABSTRACT

Objectives: This study aimed to assess the effect of application of two types of zirconia primers on repair bond strength of composite to zirconia ceramic.

Materials and Methods: In this in vitro, experimental study, 60 zirconia blocks were divided into five groups and subjected to the application of Z-Prime Plus (ZPP), Monobond Plus (MBP), Porcelain Bonding Resin (PBR), ZPP followed by PBR (ZPP+PBR) and MBP followed by PBR (MBP+PBR). They were then bonded to Z100 composite. The samples were then immersed in water at 37°C for 24 hours, thermocycled for 1000 cycles between 5-55°C and subjected to shear bond strength (SBS) test. The mode of failure was determined under a stereomicroscope and a scanning electron microscope (SEM).

Results: The mean bond strength was the highest in ZPP+PBR group followed by MBP+PBR, ZPP, PBR and MBP group (22.29±8.86, 15.75±2.81, 12.02±3.24, 3.60±2.92 and 2.92±1.78 MPa, respectively). The effects of type of zirconia primer and use/no use of PBR on SBS were significant (P<0.05). The frequency of adhesive failure in MBP and PBR groups was significantly higher than that in MBP+PBR and ZPP+PBR groups (P<0.05). The cohesive failure was significantly more frequent in ZPP+PBR group than in ZPP, MBP and PBR groups (P<0.05).

Conclusion: Simultaneous application of zirconia primer and PBR is the most efficient technique for repair of all-ceramic zirconia restorations with composite resin.

Keywords: Composite Resins; Zirconium Oxide; Shear Strength

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INTRODUCTION

Despite the growing popularity and optimal properties of zirconia ceramics such as their favorable biocompatibility, optimal durability, excellent esthetics, acceptable flexural strength and lack of metal in these

restorations, their use is still accompanied by some complications in the clinical setting [1-4]. Fracture or chipping of the veneering ceramic is among the primary reasons for failure of all-ceramic restorations [2,5]. Evidence shows that the core-veneering interface is among the

weakest areas of all-ceramic restorations, and these restorations are susceptible to chipping and crack formation especially in this region [6]. Retrieval and replacement of all-ceramic restorations are costly and time consuming for both patients and clinicians, and often result in patient dissatisfaction [2].

Defects of these restorations may be repaired chairside in some cases if they are not related to excessive thinning of the ceramic core or faulty tooth preparation design. Several materials and techniques have been suggested for repair of ceramic restorations with variable levels of success. Hydrofluoric acid etching of porcelain and subsequent repair of porcelain with composite resin is among the suggested techniques for porcelain repair. However, evidence shows that use of hydrofluoric acid is not suitable for alumina and zirconia restorations [7,8]. Thus, some other surface treatments have been proposed for these restorations such as air abrasion and silica coating. Air abrasion is often required to achieve a strong and durable bonding. Air abrasion associated with silica coating and silane application increases the success rate of repair of ceramic restorations [4,9-11]. However, this method did not yield satisfactory results in some zirconia ceramics following artificial aging [12-15]. But, it has been confirmed that increasing the sandblasting pressure from 0.15 MPa to 0.45 MPa increases the bond strength to zirconia ceramic from 11.2 MPa to 30 MPa, which is significant [16]. Some other studies showed that air abrasion with 50 μ aluminum oxide particles and 0.25 MPa pressure from 10 mm distance was relatively successful in roughening the surface and increasing the bond strength [17,18]. Although air abrasion can increase the mechanical retention and bond strength, the increase in retention is only mechanical and not chemical. Moreover, air abrasion causes superficial defects, which can cause problems for ceramic restorations [3,19,20]. The size of these defects varies from 15 to 40 μ m [21]. Thus, decreasing the sandblasting pressure during air abrasion in order to decrease the superficial defects and application of a material that can chemically bond to ceramic to increase the bond strength

would be favorable. Zirconia primer is a product designed for this purpose. Zirconia primers were recently introduced to chemically bond to zirconia and enhance the resin-zirconia bond strength. Moreover, they can be used for bonding to metal and alumina, and for intraoral repair of restorations [22]. Considering the recent introduction of zirconia primers to dental market, studies on their efficacy are still limited. Thus, this study aimed to assess the effect of application of two zirconia primers with/without using Porcelain Bonding Resin (PBR) on repair bond strength of composite to zirconia ceramic.

The research hypothesis was that simultaneous application of zirconia primer and PBR would be the most efficient technique for repair of all-ceramic zirconia restorations with composite resin.

MATERIALS AND METHODS

This *in vitro*, experimental study was performed on 60 zirconia blocks (Cercon; Degudent, Hanau, Germany) in five groups of 12. A square-shaped metal block measuring 11 mm in diameter and 4 mm in thickness was milled and coated with one layer of silver and fixed in a milling machine (Cercon; Degudent, Hanau, Germany). The device scanned the metal block and accordingly, a ceramic model was milled 25% larger to account for 25% shrinkage during sintering. The milled ceramic model was then sintered in Cercon heat furnace (Degudent, Hanau, Greece) at 1350°C for 7 hours. Square-shaped zirconia blocks measuring 11 mm in diameter and 4 mm in thickness were fabricated as such. For sectioning, they were mounted in epoxy resin (Oxydental Products, Inc., Irvington, NY, USA). Samples were divided into five groups of 12 blocks each (measuring 5x5x4 cm). Next, using a rectangular metal mold with 5 cm length and 2 cm width, each sectioned zirconia block was mounted in acrylic resin (De Trey Division, Dentsply Limited, Weybridge, U.K). Hand grinding was performed by a wheel-shaped diamond bur (Prima, England, London) and high-speed hand-piece. A transparent tube with 2.6 mm diameter was sectioned into pieces with 2 mm height. A transparent shield

with 1 mm thickness was placed on the acrylic-zirconia samples and it was punched at the point over the zirconia block to create a punched-out circle with 3 mm diameter. This was done to prevent leakage of bonding materials and stabilize the transparent tube during composite application. The materials used in this study are presented in Table 1. The five groups were prepared as follows.

Table 1. Materials used in this study and their composition

| Material | Composition | Batch number |
|---|--|--------------|
| Z-Prime Plus (Bisco, Schaumburg, USA) | Organophosphate, monomer/carboxylic acid monomer/other monomers | REF B-6001 |
| Mono Bond Plus (Ivoclar Vivadent) | Alcohol solution of silane methacrylate, phosphoric acid methacrylate and sulfide methacrylate | REF 626221 |
| Z100 composite (3M ESPE, St. Paul, MN, USA) | Bis-GMA/TEGDMA/zirconia/silica | 5540A3 |
| Porcelain Bonding Resin (Bisco) | HEMA-free unfilled resin | REF B-3110P |

- 1) Z-Prime Plus (ZPP) group: One layer of ZPP (Bisco, Schaumburg, USA) zirconia primer was applied on each sample by a microbrush and dried with air spray for 3-5 seconds. The transparent tube was placed over the surface and Z100 composite (3M ESPE, St. Paul, MN, USA) was applied into it and light-cured for 40 seconds by curing light (Kerr, Orange, USA).
- 2) Monobond Plus (MBP) group: The procedures were the same as those in ZPP group except for the use of MBP (Ivoclar, Vivadent, Schaan, Liechtenstein) instead of ZPP.
- 3) PBR or control group: No zirconia primer was used in this group. PBR (Bisco, Schaumburg, USA) was applied on the zirconia surface by a microbrush, dried with air spray for 3-5 seconds and cured for 20 seconds. Composite was applied as in ZPP group.
- 4) ZPP+PBR group: ZPP was first applied

on the zirconia surface as in ZPP group and PBR was then applied as in PBR group and cured. Composite was applied as in ZPP group. 5) MBP+PBR: MBP was applied as in MBP group. PBR was then used as in PBR group. Composite was applied as in ZPP group. Afterwards, the transparent mold (tube) and the transparent shield were removed with a hot spatula. Care was taken not to apply any pressure on the composite or the bonding interface.

The samples were then immersed in distilled water at 37°C for 24 hours and thermocycled (Neslab Instruments Inc., USA) between 5-55°C for 1000 cycles.

The samples were fixed in a universal testing machine (Z050; Zwick Roell, Ulm, Germany) and subjected to load application at a crosshead speed of 1 mm/minute until fracture. The shear bond strength (SBS) was determined as such. The samples were then inspected under a stereomicroscope (SMZ800; Nikon, Tokyo, Japan) at x30 magnification to determine the mode of failure.

Two samples from each group were randomly evaluated under a scanning electron microscope (SEM; CamScan MV2300; Oxford, England-Czech) after sputter gold coating by a sputter auto coater (E5200; BioRad, USA) for further assessment of the mode of failure. Evaluation of the mode of failure was done in two ways for higher accuracy.

Statistical analysis:

The mean and standard deviation of SBS and the absolute and relative frequency of modes of failure were reported. One-way ANOVA was used to compare the SBS of the five groups of ZPP, MBP, PBR, ZPP+PBR and MBP+PBR. Pairwise comparisons were carried out using the Games-Howell test. The Fisher's exact test was used to compare the modes of failure among the five groups. Level of significance was set at 0.05.

RESULTS

The mean SBS of composite to zirconia in ZPP+PBR group (22.29±8.86 MPa) was higher than that in other groups (P<0.05). The mean SBS of ZPP, MBP and PBR groups was significantly different (P<0.05, Table 2).

Table 2. Mean±standard deviation (SD) of shear bond strength (MPa) in all groups (n=12)

| Group (N=12) | Mean±SD | Max | Min |
|--------------|------------|-------|-------|
| ZPP | 12.02±3.24 | 17.20 | 6.25 |
| MBP | 2.92±1.78 | 7.00 | 0.74 |
| PBR | 3.60±2.92 | 9.83 | 0.12 |
| ZPP+PBR | 22.29±8.86 | 34.49 | 11.24 |
| MBP+PBR | 15.75±2.81 | 19.61 | 9.97 |

Table 3. Comparison of shear bond strength (MPa) between the study groups

| Study groups | MD | 95%CI | | P-value |
|---------------------|--------|--------|-------|---------|
| | | LB | UB | |
| ZPP vs. MPB | 9.11 | 5.69 | 12.53 | <0.001 |
| ZPP vs. PBR | 8.43 | 4.58 | 12.28 | <0.001 |
| ZPP vs. ZPP+PBR | -10.27 | -18.79 | -1.75 | 0.02 |
| ZPP vs. MPB+PBR | -3.73 | -7.52 | 0.07 | 0.06 |
| MPB vs. PBR | -0.68 | -3.69 | 2.33 | 0.96 |
| MPB vs. ZPP+PBR | -19.37 | -27.71 | 11.04 | <0.001 |
| MPB vs. MPB+PBR | -12.83 | -15.76 | -9.91 | <0.001 |
| PBR vs. ZPP+PBR | -18.70 | -27.14 | 10.25 | <0.001 |
| PBR vs. MPB+PBR | -12.16 | -15.63 | -8.69 | <0.001 |
| ZPP+PBR vs. MPB+PBR | 6.54 | -1.89 | 14.97 | 0.17 |

MD: Mean Difference; CI: Confidence Interval; LB: Lower Bound; UB: Upper bound

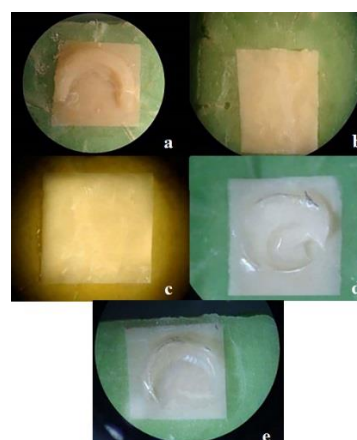
Pairwise comparisons showed that this difference was due to significant differences in SBS of ZPP compared with that of MBP and PBR ($P<0.05$). But the difference between ZPP and PBR groups was not significant ($P>0.05$). Evaluation of the effect of type of zirconia primer and use/no use of PBR showed that ZPP was superior to MBP, and use of PBR in combination with ZPP and MBP increased the SBS, and the effect of both factors on SBS was significant ($P<0.05$). However, the interaction effect of these two factors on SBS was not significant ($P>0.05$, Table 3).

Table 4 shows the frequency of modes of failure in the five groups. The five groups were significantly different in modes of failure ($P<0.001$). The frequency of adhesive failure in MBP and PBR groups was significantly different from that in MBP+PBR and ZPP+PBR groups. The frequency of cohesive failure in ZPP+PBR group was significantly different from that in MBP and PBR groups.

Table 4. Frequency of modes of failure in all groups

| Group | Adhesive N(%) | Cohesive N(%) | Mixed N(%) |
|---------|---------------|----------------------|------------|
| ZPP | 6(50) | 0 | 6(50) |
| MBP | 11(91.6) | 0 | 1(8.3) |
| PBR | 12(100) | 0 | 0 |
| ZPP+PBR | 0 | 7(58.3) ^v | 5(41.6) |
| MBP+PBR | 1(8.3) | 4(33.3) | 7(58.3) |

Figure 1 shows different modes of failure in different groups under a stereomicroscope at x20 magnification. SEM micrographs revealed mixed failure in both samples of ZPP group (Figure 2). Both samples randomly selected from MBP group showed adhesive failure (Figure 3). Both samples randomly selected from PBR group also showed adhesive failure (Figure 4). Of the two samples selected from ZPP+PBR group, one sample showed mixed (Figure 5a) and the other showed cohesive failure (Figure 5b). Mixed failure was noted in both samples randomly selected from MBP+PBR group (Figure 6).

**Fig. 1.** Modes of failure under a stereomicroscope at x20 magnification: (a) mixed failure in ZPP; (b) adhesive failure in MB; (c) adhesive failure in PBR; (d) cohesive failure in ZPP+PBR; (e) mixed failure in MBP+PBR

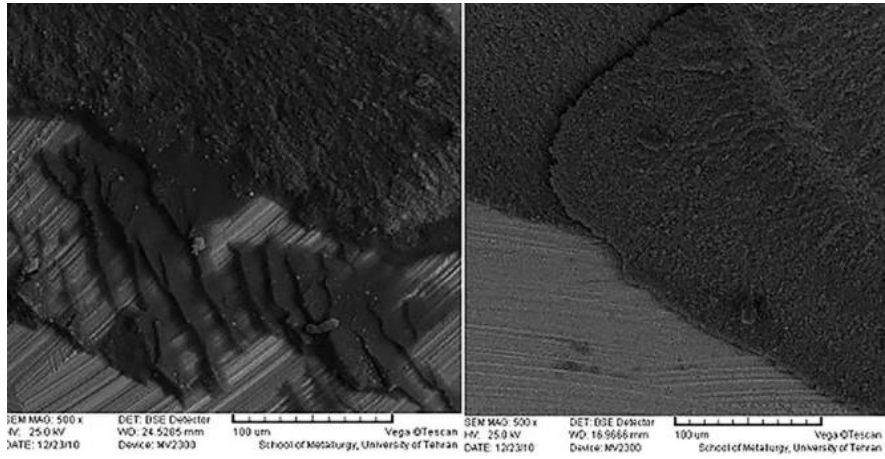


Fig. 2. Mixed failure in both samples randomly selected from ZPP group

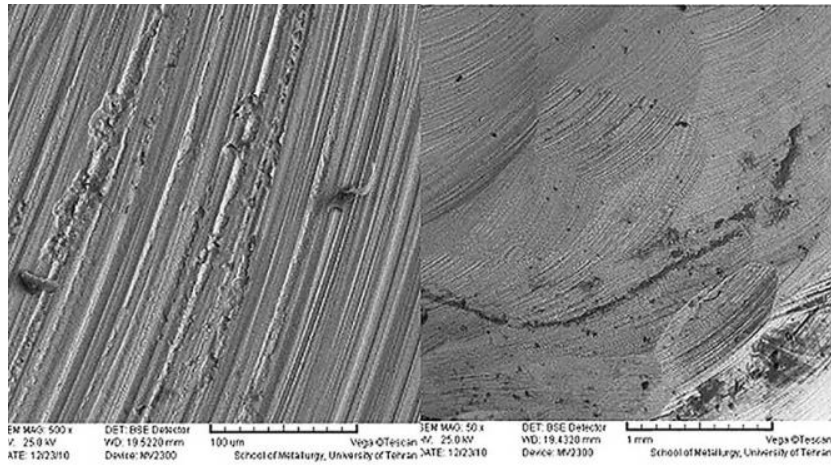


Fig. 3. Adhesive failure in both samples randomly selected from MBP group

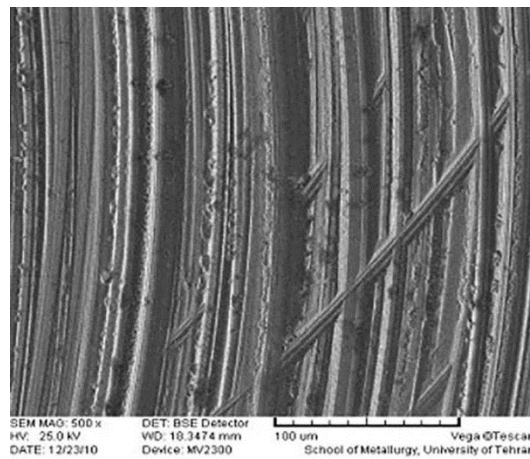


Fig. 4. Adhesive failure in both samples randomly selected from PBR group

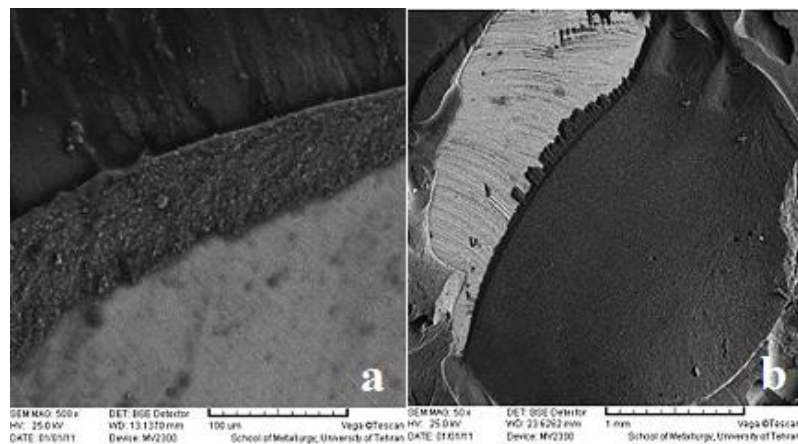


Fig. 5. Mixed failure (a) and cohesive failure (b) in samples randomly selected from ZPP+PBR group

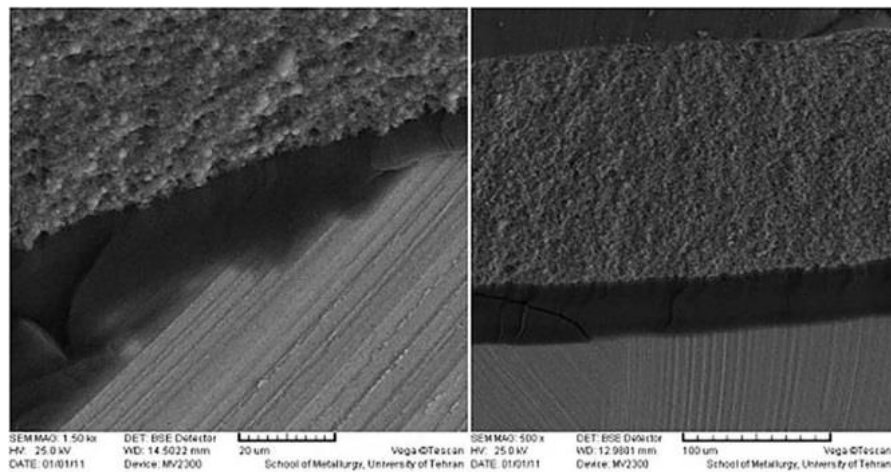


Fig. 6. Mixed failure in both samples randomly selected from MBP+PBR group

DISCUSSION

In the recent years, high demand for all-ceramic restorations has resulted in production of ceramic materials with unique properties. Zirconia is a ceramic material made of crystallized zirconium oxide, which has optimal biocompatibility, high strength and durability, and acceptable esthetics. These favorable properties have resulted in extensive clinical use of zirconia in fabrication of intracanal posts, abutments, implants and Maryland bridges [23,24]. However, despite the afore-mentioned advantages, zirconia ceramic restorations have drawbacks as well such as fracture or chipping of the veneering. The core-veneering interface is among the

weakest parts of all-ceramic restorations [6]. Evidence shows that single crown restorations have adequate resistance against occlusal forces; however, fixed long-span restorations are at higher risk of chipping and crack formation [25,26]. In such cases, intraoral restoration repair is a better option than replacement for both the patient and clinician, and can save time and cost.

In the recent years, researchers have proposed methods to increase the repair bond strength of resin to these restorations. Sandblasting is a suggested technique for this purpose. However, it only increases the bond strength mechanically. Moreover, increasing the sandblasting pressure causes some

unwanted complications. Furthermore, methods such as etching are not effective for zirconia restorations. Ongoing research to find a material that can chemically bond to zirconia resulted in development of zirconia primers. Zirconia primers have other applications as well, and can be used for bonding to metal and alumina, and repair of intraoral restorations. This study assessed the efficacy of zirconia primers for intraoral repair of zirconia restorations. Two commonly used types of zirconia primers namely ZPP and MBP were evaluated in this study. We used shear test for measurement of bond strength, which is the most commonly used test for assessment of resin-ceramic bond strength [27,28].

The results of this study demonstrated that ZPP zirconia primer yielded a significantly higher bond strength to zirconia than MBP and PBR. The bond strength provided by ZPP was more than three times the value provided by MBP and PBR. Thus, ZPP seems to be superior to MBP and PBR. This difference in bond strength is probably due to the chemical composition of the two primers since MBP contains silane and phosphoric acid while ZPP contains MDP phosphate monomer. In MBP, presence of silane in combination with phosphoric acid increases the risk of hydrolysis of silane before its clinical application, and subsequent reduction of bond strength. However, ZPP has a different composition and contains MDP phosphate monomer.

Considering the recent introduction of zirconia primers, comprehensive studies about their properties are not available. Magne et al. [29] polished the samples, sandblasted them and applied ZPP and Clearfil Ceramic Primer (Kuraray). They reported that ZPP yielded a higher bond strength around 29 MPa. They explained the reason to be the different compositions of zirconia primers since Clearfil Ceramic Primer similar to MBP contains acid in combination with silane. Aboushelib et al. [7,30] used selective infiltrated etching (which can only be used in vitro and cannot be applied clinically) along with four different types of zirconia primers to enhance bonding to zirconia. The zirconia primer used in their

study was two-bottle silane, which was activated by addition of carboxylic acid and yielded 31 to 40 MPa immediate bond strength and 15 MPa bond strength after 90 days of water storage. They recommended the application of zirconia primers.

Our study also evaluated the effect of using PBR in combination with ZPP and MBP zirconia primers. This has not been evaluated in any previous study. The manufacturers of zirconia primers have not recommended the application of PBR either. An interesting finding of our study was that the application of PBR along with zirconia primer significantly increased the SBS.

This increase in SBS was around two folds for ZPP and four folds for MBP. Application of PBR along with MBP increased its bond strength close to the level of bond strength provided by ZPP. The bond strength of MBP was not significantly different from that of no-primer control group, and was minimal (3.86 MPa versus 3.59 MPa); whereas, the difference in bond strength of ZPP and the control group was significant, and ZPP provided a bond strength 3.5 times stronger than that of no-primer control group. However, after the use of PBR, the bond strength in both groups significantly increased compared with the control group.

Thus, use of PBR along with zirconia primer is recommended. Further studies are required to assess the chemical interactions of these materials and the mechanism behind this increase in bond strength. Evaluation of the mode of failure under a stereomicroscope and SEM revealed significant differences in this respect among the groups. The mode of failure of samples was compatible with the results of SBS test such that in the no-primer control group with the lowest bond strength, adhesive failure had 100% frequency and the zirconia surface was free from any adhesive. The ZPP+PBR group that had the highest SBS showed no adhesive failure (0%) and fractures were cohesive (within the composite) or mixed (within the composite and at the bonding interface) with relatively equal frequency values (58% and 41%, respectively). The higher percentage of cohesive failure indicates the high bond

strength provided by the zirconia primer and PBR, resulting in fracture within the composite, which is clinically acceptable and preferred. Intraoral repair can be performed by further application of composite. In MBP+PBR group, mixed failure had the highest frequency (58%) while cohesive failure had 33% frequency, which indicates that the bonding interface was weaker than that in the ZPP group. Future studies are required to assess the chemical interactions between PBR and zirconia primers. Also, the durability of composite-zirconia bonding with/without the application of PBR and zirconia primer should be evaluated. The effect of PBR along with zirconia primer on cement-zirconia bonding should be investigated as well.

CONCLUSION

Use of ZPP zirconia primer significantly increased the bond strength of composite to zirconia ceramic compared with MBP. Application of PBR with both zirconia primers increased the bond strength of composite to zirconia ceramic.

CONFLICT OF INTEREST STATEMENT

None declared.

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