

# An *in vitro* comparison of the effect of various surface treatments on the tensile bond strength of three different luting cement to zirconia copings

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## Abstract

**Aim:** The aim of this study was to evaluate and compare the tensile bond strength of zirconia copings subjected to three different surface treatment methods and cemented with three different luting agents.

**Materials and Methods:** Seventy-two extracted maxillary premolar teeth were prepared to receive zirconia copings milled using computer-aided design/computer-aided manufacturing technology, which were divided into 9 groups of 8 specimens each. Three surface treatment protocols such as hydrofluoric acid etch treatment, air abrasion with 110- $\mu$ m aluminum oxide ( $Al_2O_3$ ), and tribochemical silica coating (Rocatec) treatment were carried out, and copings were cemented with three luting agents such as resin-modified glass ionomer cement (RelyX luting 2), 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) resin cement (Panavia F 2.0) and 4-methacryloxyethyl trimellitic acid (4-META) resin cement (G-Cem). Tensile bond strength of the copings was tested in a universal testing machine. Zirconia copings fabricated on the prepared extracted tooth. After the three surface treatments and cementing the zirconia crowns with three luting agents tensile bond strength is tested. The mean and standard deviations (SD) were calculated for the nine groups using one-way ANOVA, followed by Tukey–Kramer *post hoc* using the SPSS software.

**Results:** The ANOVA test showed that the measured mean bond strength values were 4.22 MPa (tribochemicalsilica coating and MDP resin), 2.71 MPa (air abrasion and MDP resin), 2.61 MPa (tribochemical treatment with META), and 0.66 MPa (RelyX with air abrasion). According to the pairwise comparison of Tukey’s honestly significant difference test, significant differences were exhibited among all the groups ( $P < 0.05$ ).

**Conclusion:** Tribochemical silica coating in combination with 10-MDP and 4-META adhesive resins provided the maximum bonding for zirconia copings.

**Keywords:** 10-methacryloyloxydecyl dihydrogen phosphate, 4-methacryloxyethyl trimellitic acid, air abrasion, hydrofluoric acid, Panavia F 2.0, tribochemical surface treatment, zirconia

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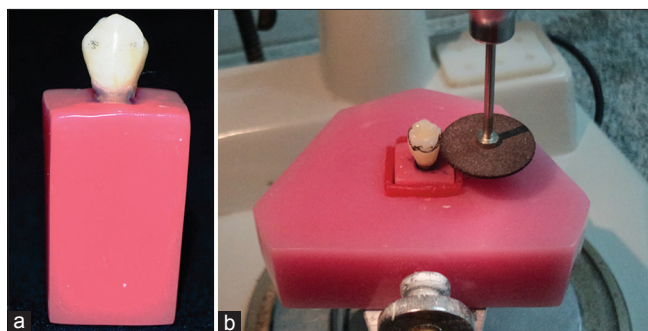
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## INTRODUCTION

The introduction of high strength and esthetically acceptable zirconia ( $ZrO_2$ ) also called as “ceramic steel” has the chemical and dimensional stability which makes it an excellent material for prosthetic rehabilitation.<sup>[1,2]</sup> Zirconia has exhibited superior wear resistance, but to achieve a strong and durable bond with the resin, in comparison with other ceramic materials, it requires alternative methods such as air abrasion and tribochemical surface treatment.<sup>[3-5]</sup> The newer resin-based adhesives with phosphate monomers such as 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) and 4-methacryloxyethyl trimellitic acid (4-META) have also exhibited improved bonding of the zirconia restorations.<sup>[6-8]</sup> This study focuses on the effect of three surface treatment methods on the bond strength of the zirconia copings using three luting agents, to zero in on the best surface treatment and luting agent combination for zirconia.

## MATERIALS AND METHODS

Seventy-two intact extracted human maxillary premolar teeth with approximately similar diameter were collected. The teeth after extraction were placed in 0.5% sodium hypochlorite solution for 5 min, cleaned using ultrasonic scaler, and then stored under water.<sup>[9]</sup> The roots of the selected teeth were notched for retention and embedded along their long axis with the cemento-enamel junction positioned 1 mm above the custom-made mounting base fabricated using autopolymerizing acrylic resin (DPI-RR Cold Cure, Mumbai), and the alignment of the tooth was verified using a surveyor [Figure 1a]. The mounted tooth base was then fixed in the custom-made template and positioned in the cast holder of the milling machine (Paraskop M, Model No: 26060, BEGO, Germany). The occlusal surface of each mounted tooth specimen was sectioned flat 4 mm from the cemento-enamel junction using carborundum discs (San-I Grinding Wheel Products Co., Ltd.) of dimension 25 mm × 0.6 mm × 1.8 mm [Figure 1b]. A constant length



**Figure 1:** (a) Custom-made base with embedded tooth (b) sectioning the occlusal surface of the tooth in the milling machine

of 4 mm was achieved by marking the desired level on the head of the diamond point and then orienting the mark with the flat prepared occlusal surface. To ensure a standardized preparation of the tooth specimens, a custom-made steel clamp was fabricated to hold the airtor handpiece (Pana Air Σ NSK) securely, to the surveying arm of the surveyor (Marathon 103-Saeyang Microtech) [Figure 2]. The high-speed handpiece (350,000–450,000 rpm) with a coarse diamond tapered rotary bur (S0-20; Lot D14J044600) with 3° taper was oriented parallel to the vertical axis of the tooth and manually rotated to establish a standardized angle of convergence of 6°. The axial reduction of 1–1.5 mm and axial length of approximately 4 mm with deep chamfer finish line were achieved. The preparation was carried out under water spray, and for each tooth specimen, a new diamond bur was used. The surface area of each preparation was measured using a metal caliper (API™ German Stainless) to calculate the bond strength.

The tray adhesive (3M ESPE VPS, Seefeld Germany) was applied to the intaglio surface of the custom trays fabricated using autopolymerizing acrylic resin. Impressions of the prepared tooth were made with polyvinyl siloxane putty and light body material (3M ESPE, Seefeld Germany) and poured with scan-able die material. The master dies were trimmed and scanned in dental wing scanner which sent the command to the computer-aided manufacturing (CAM) machine (CNC Milling Machine vhf CAM 4-02, Germany) for the milling of the zirconia copings from the presintered zirconia blanks (ZIECON, Jyoti ceramics Pvt. Ltd., Nashik, India). In each of the copings, a 2-mm diameter hole was drilled in the predesigned 6-mm projection of the abutments using a round rotary cutting instrument (018; 801 Predator Zirconia, Prima Dental, India) with copious water irrigation [Figure 3]. The prescribed area for the hole was measured and marked for all the samples



**Figure 2:** Custom clamp attached to surveyor for tooth preparation

before the drilling procedure. The hole allowed a wire to be used as an attachment from the zirconia coping engaging the hook of the custom-made jig in the universal testing machine (UTE-9302), which exerted the tensile forces to separate the zirconia copings from the abutments during the testing procedure. The zirconia copings were sintered and placed on the respective dies to evaluate the fit, and for proper alignment during the cementation procedure, a line was marked on the coping and the mounted teeth. The seventy-two zirconia copings milled were divided into 9 groups, containing 8 specimens each. The copings of Groups A1, A2, and A3 were etched with hydrofluoric acid (HF) and cemented with resin-modified glass-ionomer cement (GIC) (RelyX™ luting 2, 3M ESPE-3525, USA), 10-MDP self-adhesive resin (Panavia™ F 2.0, Kuraray Noritake Dental Inc., Japan), and 4-META adhesive resin (G-CEM Capsule luting, GC Corp., Tokyo, Japan), respectively. The copings of Groups B1, B2, and B3 were surface air abraded and cemented with RelyX luting 2, Panavia F 2.0, and G-CEM, respectively, and the copings of Groups C1, C2, and C3 were tribochemical silica coated and cemented with RelyX luting 2, Panavia F 2.0, and G-CEM, respectively.

### Surface treatment of copings

The copings of Groups A1, A2, and A3 were surface treated with 9.6% HF acid gel (Pulpdent Corporation, USA). The gel was dispensed using a prebent needle on the intaglio surfaces of the copings, left for 1 min, and rinsed with water [Figure 4]. The copings of Groups B1, B2, and B3 were air abraded with 110- $\mu\text{m}$   $\text{Al}_2\text{O}_3$  particles (Korox 110, Bego, Germany) from a distance of 10 mm for 10 s in a sandblaster (Korostar Z, Bego, Germany). The copings from Groups C1, C2, and C3 were first air abraded with 110- $\mu\text{m}$   $\text{Al}_2\text{O}_3$  particles (Rocatec Pre, 3M ESPE, Seefeld, Germany) followed by 30- $\mu\text{m}$  silica-coated  $\text{Al}_2\text{O}_3$  particles (Rocatec Plus, 3M ESPE, Seefeld, Germany) [Figure 5a] on the intaglio surface of zirconia coping from the distance of approximately 10 mm for a period of 15 s, followed by application of silane coupling agent (Monobond-S, Ivoclar Vivadent AG) and allowed for 5 min for the silane reaction [Figure 5b]. The prepared tooth was cleaned thoroughly with pumice slurry, rinsed with a water spray, and air dried before the cementation.

### Cementation of the copings

The copings of Groups A1, B1, and C1 were cemented with RelyX luting 2 using the clicker dispenser [Figure 6a]; base and catalyst were mixed in pad with a plastic spatula until uniform color was obtained. A thin layer of the cement was applied on the intaglio surface of the copings



Figure 3: Milled zirconia coping with a hole



Figure 4: Surface treatment with hydrofluoric acid



Figure 5: (a) Rocatec plus-silica-coated aluminum oxide, (b) silane coupling agent

and seated firmly over the prepared teeth using finger pressure for 5-min excess cement was removed. For the copings of Groups A2, B2, and C2 to be cemented with Panavia F 2.0 [Figure 6b], equal amounts of ED Primer

II A and B were mixed and applied to the prepared tooth for 30 s. The cement pastes were mixed using plastic spatula on a mixing pad for 20 s until uniform color was achieved. A thin layer of cement was applied to copings of and seated firmly using finger pressure for 5 min, excess cement was removed, and Oxyguard II was applied to cure the material in the margins for 3 min. The G-CEM capsule resin [Figure 7a] for the Groups A3, B3, and C3 was set in an amalgamator (SYG-200, Hangzhou Sifang Medical Apparatus Co. Ltd., China) and mixed for 10 s ( $\pm 4000$  rpm) [Figure 7b]. The capsule applicator (GC Asia Dental Pte Ltd., Singapore) [Figure 7c] extruded the mixed cement directly into the internal surface of the copings and seated with moderate finger pressure for 5 min and excess cement removed.

**Tensile bond strength test**

The cemented copings were subjected to tensile dislodgement with a crosshead speed of 0.5 mm/min in the universal testing machine (UTE 9302). The dislodgment values recorded in Newton (N) was converted to the tensile bond strength in MPa unit by dividing the surface area ( $\text{mm}^2$ ) of the prepared tooth which was calculated using the following formula,<sup>9,10</sup>

$$\text{Area} = \pi/4 d_1^2 + \pi h/2 (d_1 + d_2) + \pi/4 (d_3^2 - d_2^2)$$

Where,  $d_1$  – diameter at the top of the preparation,  $d_2$  – diameter at the base of the preparation,  $d_3$  – diameter of the base of the preparation plus 1-mm margins either side,  $h$  – axial height.

**RESULTS**

Data obtained were compiled on the MS Excel sheet. The mean and standard deviations (SD) were calculated

for the nine groups using one-way ANOVA, followed by Tukey–Kramer *post hoc* test to compare and identify the greater differences among the mean values. Statistical analysis was performed using the SPSS software, Version 23.0. (IBM Corp, Armonk, NY) at a significance level of 5% ( $P = 0.05$ ).

The mean and SD bond strength values of the nine groups were tabulated [Table 1]. The highest mean bond strength values among the Groups A1, A2, and A3 with HF acid surface treatment of zirconia copings was shown by G-Cem (A3 – 1.41 MPa). The highest mean bond strength values with air abrasion Groups B1, B2, and B3 were exhibited by Panavia F 2.0 (B2–2.71 MPa) followed by G-Cem (B3–1.33 MPa). The tribochemical surface treatment Groups C1, C2, and C3 revealed significantly higher mean bond strength values with Panavia F 2.0 (C2 – 4.22 MPa). The comparison between resin-modified GIC RelyX Groups A1, B1, and C1 showed higher mean bond strength values for tribochemical treatment (C1 – 1.11MPa). In the Groups A2, B2, and C2, where Panavia F2.0 (MDP) was used for cementing the zirconia copings, the mean bond strength values were significantly higher with tribochemical surface treatment (C2 – 4.22 MPa). In the

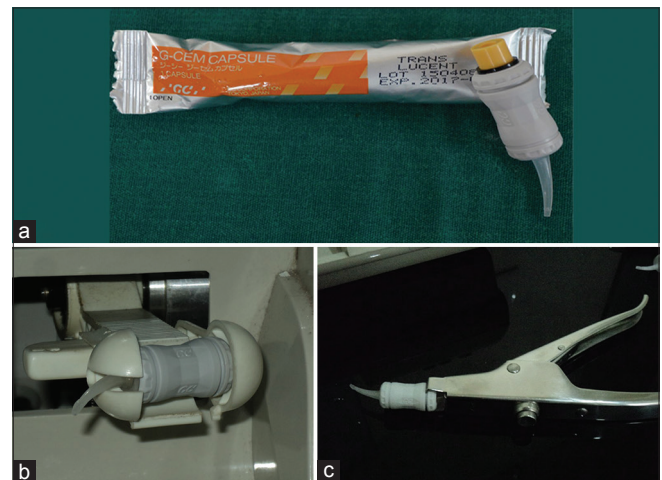
**Table 1: The mean, standard deviation, and significance of Pearson correlation ratio**

Groups	Mean (MPa)	SD	F	P significance
A1	1.03	0.517	40.802	<0.001***
A2	1.26	0.209		
A3	1.41	0.338		
B1	0.66	0.138		
B2	2.71	0.478		
B3	1.33	0.443		
C1	1.11	0.220		
C2	4.22	0.698		
C3	2.61	0.935		

$P < 0.05$ , \*\*\*Highly significant. SD: Standard deviation



**Figure 6:** (a) Resin-modified glass ionomer cement, (b) methacryloyloxydecyl dihydrogen phosphate monomer-containing self-adhesive



**Figure 7:** (a) Methacryloyloxyethyl trimellitic acid-containing adhesive resin, (b) amalgamator, (c) capsule applicator

G-Cem (META) Groups A3, B3, and C3, the highest mean bond strength value was exhibited by tribochemical surface treatment (C3 – 2.61 MPa).

The overall highest mean bond strength values were exhibited by tribochemical silica treatment cemented with Panavia F 2.0 (C2 – 4.22 MPa), followed by air-abraded zirconia copings cemented with Panavia F 2.0 (B2 – 2.71 MPa), tribochemical silica coating of zirconia cemented with G-Cem (C3 – 2.61 MPa), and air-abraded zirconia copings cemented with G-Cem (B3 – 1.33 MPa), and HF acid-etching and resin-modified GIC gave the least bond strength values.

The pairwise comparison of bond strength values with Tukey's honestly significant difference test among air abrasion Groups B1, B2, and B3 and tribochemical surface treatment Groups C1, C2, and C3 revealed significant mean differences in the bond strength values ( $P < 0.05$ ) with all the three luting cement, and HF acid surface treatment Groups A1, A2, and A3 showed no significant differences irrespective of the luting cement used. Tukey's *post hoc* test for MDP-containing Panavia F 2.0 Groups A2, B2, and C2 and META-containing G-CEM Groups A3, B3, and C3 revealed highly significant differences whereas comparison between Groups A1, B1, and C1 resin-modified glass ionomer RelyX luting showed no significant differences regardless of the surface treatment method used [Table 2].

## DISCUSSION

Zirconia has shown good fracture resistance and strength *in vitro*, but their clinical use requires a reliable bonding with the luting agent.<sup>[5]</sup> In the present study, the tensile bond strength of zirconia copings was determined after subjecting it to three surface treatment protocols, namely HF acid treatment, air abrasion, and tribochemical surface treatment and cemented with three different luting agents such as resin-modified GIC (RelyX luting 2), 10-MDP monomer-containing cement (Panavia F 2.0) and 4-META monomer-containing cement (G-Cem resin). The selection of the suitable luting agent is an important factor as the long-term clinical success of fixed prostheses and also depends on the type of the dental cement used. The dental cement must seal the interface between the tooth and restoration, provide a barrier against microleakage, and bond them together by mechanical and chemical methods or in combination of the both.<sup>[11]</sup>

In the current study, the highest bond strength was achieved by the tribochemical surface treatment of zirconia with all the three luting agents used, consistent with the

**Table 2: Pairwise comparison using Tukey's honestly significant difference test (*post hoc*)**

Group	Compared with	Mean difference (I-J)	Significance		
A1	A2	-0.228	0.992		
	A3	-0.375	0.856		
	B1	0.373	0.859		
	B2	-1.676*	0.000		
	B3	-0.301	0.954		
	C1	-0.075	1.000		
	C2	-3.183*	0.000		
A2	C3	-1.576*	0.000		
	A3	-0.146	1.000		
	B1	0.602	0.306		
	B2	-1.447*	0.000		
	B3	-0.072	1.000		
	C1	0.153	0.999		
	C2	-2.955*	0.000		
A3	C3	-1.347*	0.000		
	B1	0.748	0.091		
	B2	-1.301*	0.000		
	B3	0.073	1.000		
	C1	0.300	0.955		
	C2	-2.808*	0.000		
	C3	-1.201*	0.000		
B1	B2	-2.050*	0.000		
	B3	-0.675	0.176		
	C1	-0.448	0.693		
	C2	-3.557*	0.000		
	C3	-1.950*	0.000		
	B2	B3	1.375*	0.000	
		C1	1.601*	0.000	
C2		-1.507*	0.000		
C3		0.100	1.000		
B3		C1	0.226	0.992	
		C2	-2.882*	0.000	
		C3	-1.275*	0.000	
	C1	C2	-3.108*	0.000	
		C3	-1.501*	0.000	
		C2	C3	1.607*	0.000

\*The mean difference is significant at the 0.05 level

earlier reports of Senyilmaz *et al.*, who concluded that pretreatment of a zirconia surface with tribochemical treatment improves the bond strength of resin cement.<sup>[12]</sup> Tribochemical surface treatment includes formation of a thin SiO<sub>2</sub> film by high-speed impaction of silica-modified alumina particles on the zirconia creating a rough layer with increased surface area and surface energy.<sup>[13]</sup> The silane (-Si-O-CH<sub>3</sub>) in the coupling agent reacts with water to form siloxane (-Si-O-Si-O-) network, which bonds with the silica-coated surface of zirconia.<sup>[7,8,14]</sup> The 10-MDP resin monomer contains a phosphoric acid group [-P(=O)(OH)<sub>2</sub>], which penetrates the silane coupling layer and selectively adsorb the hydroxyl groups of the zirconia oxide surface by producing a "hybrid" layer.<sup>[15,16]</sup> Thus, the strong covalent bond formed results in roughening and activation of zirconia improving the surface wettability and thereby improving resin bond strength with low percentage of adhesive failure from zirconia surface.<sup>[15,17]</sup> Ernst *et al.* reported a 50% increase in the median retentive strength of zirconia with the use of tribochemical surface treatment.<sup>[18]</sup>

The Panavia F2.0 exhibited significantly higher bond strength values with tribochemical coating compared to the META-containing cement (G-CEM) and resin-modified GIC (RelyX) consistent with earlier reports of Atsu *et al.* and Bottino *et al.*<sup>[5,12,19]</sup> Yang *et al.* reported that 10-MDP-containing luting system was the most suitable to bond zirconia surfaces compared to self-adhesive or conventional resin cement, and it did not require any pretreatment on the ceramic surface before luting.<sup>[15]</sup> Takeuchi *et al.* also reported that a combination treatment using MDP monomer and tribochemical treatment roughened and activated the zirconia surface.<sup>[17]</sup> In contrast, de Oyagüe *et al.* found that silica coating promoted a chemical bonding at the ceramic resin cement interface but did not result in a frank surface modification.<sup>[20]</sup>

Air abrasion of the zirconia also showed significantly higher bond strength when cemented with Panavia F 2.0 in agreement with the reports of Friederich and Kern, who suggested that phosphate monomer-containing composite resin significantly improved the bond strength of air-abraded ceramic,<sup>[21]</sup> and Phark *et al.* reported that air abrasion increased the bond strength of the zirconia regardless of the abrasive particle size.<sup>[10]</sup> Wolfart *et al.* suggested that air abrasion with 110- $\mu\text{m}$   $\text{Al}_2\text{O}_3$  activates the zirconia surface by increasing the surface area and roughness, and the use of a MDP-containing resin cement is necessary to achieve durable bond to zirconia ceramics.<sup>[16]</sup> It also removes any surface contamination and improves the wetting of adhesives for chemical bonding as reported by Komine *et al.* and Stawarczyk *et al.*<sup>[22,23]</sup> Air abrasion can be used an alternative to tribochemical surface treatment to bond zirconia with MDP-containing resin cement.

The 4-META-containing G-CEM resin cement also showed relatively higher bond strength values when combined with tribochemical surface treatment. The mechanical interlocking of carbonyl group [ $-\text{C}(=\text{O})\text{OH}$ ] of the carboxylic acid monomer (4-META) with tribochemical coating of the zirconia oxide increased the adhesion as reported by Lin *et al.*<sup>[1,7]</sup>

The HF acid etching showed the least bonding of zirconia to resin suggesting that the crystalline phase or low amount of glass phase of the zirconia cannot be etched with clinically acceptable acid concentrations as reported by Senyilmaz *et al.* and Komine *et al.*<sup>[12,22]</sup> In contrast, Chen *et al.* reported that hot acid etching with  $\text{H}_2\text{SO}_4/(\text{NH}_4)_2\text{SO}_4$ , HF/ $\text{HNO}_3$ , has shown to have improved the initial bond strength of Y-TZP to Bis-GMA-based resin cement compared to alumina sandblasting which could be used as an alternative in the further studies.<sup>[8]</sup>

The resin-modified GIC (RelyX) performed poorly in comparison to the resin cement regardless of the surface treatment methods used, consistent with the reports by Ergin and Gemalaz, who demonstrated that bond strength also depends on the convergence angle of the prepared tooth.<sup>[6,24]</sup>

The frictional resistance between the prepared tooth and crown is also an important factor, compared to pronounced taper of approximately  $10^\circ$ , using a low taper angle of approximately  $6^\circ$  might have resulted in frictional retention of crown regardless of the type of luting cement used.<sup>[18,25]</sup> Another major factor to be considered is the flat occlusal reduction carried out in the current study, in oppose to the normal anatomical preparation. The bond strength values were significantly higher when a tooth was prepared anatomically as compared to a flat occlusal surface.<sup>[26]</sup> Although the flat occlusal reduction was a common finding, decreased the frictional retention, hence providing more definite bond strength values of luting agents and the surface treatments used, and also provides a standardized surface area to all the specimens in comparison to varying anatomical levels of different teeth, the flat reduction causes more or less tooth reduction and clinically not recommended.<sup>[26]</sup>

Also, the fact that finger pressure was used to cement the copings without the use of a standardized device also might have influenced the overall values, but in a clinical situation, the cementation pressure is manually controlled.

The method used to measure the surface area of the prepared teeth would have significantly influenced the overall result. Some studies have used methods such as correlating the weight of 0.1-mm tin foil wrapped around the preparation or scanning the prepared abutments with a Cerec 3D camera, and their bonded areas were estimated with the Cerec 3 volume program.<sup>[18,23]</sup> In the current study, the bonded area was calculated using the formula for a truncated cone to which area of the flat occlusal surface was added as done by Palacios *et al.* and Karimipour-Saryazdi *et al.*, this also would have influenced the bond strength values and makes data comparison with other studies difficult.<sup>[9,27]</sup>

It may also be noted that the inherent roughness of zirconia ceramics may be adequate to provide the necessary micromechanical interlocking of the luting agents.<sup>[27]</sup> There is more scope for research to examine the effect of various other surface treatments, long-term storage, thermomechanical cycling, various other zirconium oxide systems, and luting cement. Newer methods such as selective infiltration etching, plasma spraying (hexamethyldisiloxane) on the zirconium oxide

surface, surface treatments using erbium-doped yttrium aluminum garnet or CO<sub>2</sub> laser, and vapor deposition of silicon tetrachloride are also suggested.<sup>[28]</sup> In addition, long-term prospective, randomized clinical trials are needed to evaluate the benefits of certain clinical procedures and the newer adhesive methods for zirconia restoration.

## CONCLUSION

Within the limitations of this *in vitro* study, the following conclusions were drawn:

To ensure the utmost retention of the zirconia copings, they can be surface treated with tribochemical silica and cemented with MDP-containing cement. Air abrasion of the copings and META-containing adhesive resin are the other superior alternatives. HF acid etching does not contribute to the surface treatment of zirconia copings; also, the resin-modified GIC did not improve the retention of the copings which are best to be avoided.

## Clinical implications

The long-term success of the zirconia restorations is based on their chemical bonding to the tooth which can be ensured with the selection of the suitable surface treatments and adhesive cement combination.

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## Conflicts of interest

There are no conflicts of interest.

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