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New zirconia primer improves bond strength of resin-based cements

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ABSTRACT

Objective. Various chemical interactions can be used to develop ceramic–resin bonding and specific approaches are available for zirconia ceramics. This study evaluated the effect of a new experimental primer, a mixture of organophosphate and carboxylic acid monomers, on the zirconia-to-resin shear bond strength (SBS).

Methods. Forty Y-TZP blocks ($15 \times 4 \times 2$ mm) were embedded in an acrylic resin base, polished, Al_2O_3 -sandblasted and randomly divided into eight groups. Three different resin-based luting agents (BisGem, Duo-Link, Panavia F) were used to build 2.4 mm-diameter cylinders ($n = 15$) onto the zirconia surface with and without the new experimental zirconia primer. The new primer was also tested with Z100 restorative composite resin cylinders. In addition, Panavia was used with its own primer (Clearfil Ceramic Primer). SBS testing was carried out after 24 h of storage in water. Scanning electron microscopy (SEM) was used to evaluate the zirconia surface topography and failure mode.

Results. According to ANOVA and Tukey test ($\alpha = 0.05$), the association of the experimental primer with the restorative composite resin Z100 yielded the highest SBS (29.35 MPa) followed by DuoLink with the new primer (26.68 MPa). The groups that did not receive the experimental primer presented the lowest SBS values (from 5.95 to 9.79 MPa). The failure mode was adhesive for the non-primed specimens and predominantly mixed in the primed groups.

Significance. The use of the new zirconia experimental primer based on organophosphate/carboxylic acid monomers increased the bond strength of different resin-based luting agents including Z100 restorative material.

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1. Introduction

There has been a significant growth of interest for zirconium-oxide (zirconia) ceramics in recent times, with a major impact in the field of prosthodontics and implant dentistry. Due to its mechanical properties [1–3], biocompatibility, and optical

properties, zirconia has been elected as a metal-free alternative. “Transformation toughening” is a distinctive capability of Yttrium stabilized tetragonal zirconia polycrystals (Y-TZP), through which it can resist crack propagation by transforming from a tetragonal to a monoclinic phase [1,3]. One of the major limitations regarding the use of zirconia is the difficulty to adhere to this material. Zirconia FDPs and full-veneer crowns

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Table 1 – Experimental groups.

Groups	Primer			Luting material			
	No primer	Experim. primer	Clearfil C. primer	BisCem	DuoLink	Panavia F	Z100
NP-BisCem	■			■			
NP-DuoLink	■				■		
NP-Panavia	■					■	
ZrP-BisCem		■		■			
ZrP-DuoLink		■			■		
ZrP-Panavia		■				■	
ZrP-Z100		■					■
CCP-Panavia			■			■	

have been successfully used when cemented with conventional cements, such as glass ionomer [4]. A reliable bond to zirconia, however, needs to be achieved when retention of the restoration relies primarily on adhesion, as in partial veneers and resin-bonded FPDs.

The search for a predictable bond to zirconia started in the 80s where the bond strength of ceramic brackets was evaluated by Urano [5]. The main challenge resides in the fact that highly crystalline ceramics resist conventional etching techniques (absence of a glassy phase) [6–9]. Classic surface roughening methods (airborne-particle abrasion) can only produce a mild coarsening of the zirconia surface [6] and reliable resin bond strength may not be always achieved [8,10]. As a result, a large body of literature has been produced and innovative adhesive strategies combining new surface roughening procedures [11,12], laser treatments [13,14], and chemical bonding have been developed [10,15–20].

Chemical bonding to zirconia ceramics involves the use of various couplers. The use of a silane coupling agent in combination with aluminum oxide sandblasting presents low bond strength [6,8,15], which is expected given the absence of silica in the substrate. On the other hand, tribochemical silica coating allows high-strength alumina-based and zirconia-based ceramics to be chemically more reactive to resin through silane coupling agents yielding increased resin bond strength values [8,9,19,21,22]. The association of airborne-particle abrasion (aluminum oxide or silica-coating) and primer/luting agents containing phosphate ester monomer 10-methacryloyloxydecyl dihydrogenphosphate (MDP) [10,15,16,19,20] and zirconate coupler agent [17,18] has also been suggested. The presence of other acidic monomers such as 4-methacryloxyethyl trimellitic anhydride (4-META), and thiophosphoric acid methacrylate (MEPS) permits additional chemical bond with zirconium/metal oxides [23]. A new approach is to use a mixture of organophosphate and carboxylic acid monomers in form of a light-polymerizable priming agent.

The purpose of this study was to evaluate the effect of a Z-Prime Plus (Bisco, Schaumburg, IL) (organophosphate/carboxylic acid monomers) on the zirconia SBS to different types of resin-based luting agents which include: DuoLink (Bisco), a conventional dual-cured resin-based luting cement; Panavia F 2.0 (Kuraray), a MDP containing dual-cured resin cement; BisCem (Bisco), a self-adhesive resin cement which contains phosphate monomer other than MDP; and Z100 (3M-ESPE), a restorative composite resin with higher filler content (85 wt%). The null-hypothesis considered was that the

use of the new zirconia primer would not influence the SBS of Y-TZP to resin-based luting agents.

2. Materials and methods

Forty blocks (15 × 4 × 2 mm) of high-purity zirconium-oxide ceramic (LAVA; 3M-ESPE, Saint Paul, MN) were produced. The specimens were embedded in an acrylic resin base (Palapress; Heraeus Kulzer, Hanau, Germany) and polished with 400-, 600-, 800- and 1500-grit silicon carbide paper under water cooling. All groups received airborne-particle abrasion with 50 µm aluminum oxide (Al₂O₃) particles (RondoFlex 2013; Kavo, Biberach, Germany) under a pressure of 46–55 psi using a fine airborne-particle-abrasion unit (Kavo RONDOFlex Plus 360; Kavo) for 15 s at a distance of 10 mm perpendicularly to the surface. The blocks were randomly assigned to eight groups according to primer and resin luting agent used (Table 1). The characteristics of the materials are presented in Table 2.

The surface of each block was cleaned with 40% phosphoric acid (K-Etchant Gel; Kuraray, Kurashiki, Okayama, Japan) for 5 s, water rinsed for 15 s, and immersed in an ultrasonic bath for 2 min. Fifteen blocks did not receive further surface treatment (No Primer – NP). Following air-drying, 20 blocks received two even coats of Experimental Zirconia Primer (ZrP) (Bisco Inc., Schaumburg, IL) that were applied onto the surface, gently air-dried for 10 s and light polymerized for 20 s at 800 mW/cm² (Rembrandt Allegro LED Curing Light; Den-Mat, Santa Maria, CA). MDP-containing primer (Clearfil Ceramic Primer – CCP) was applied onto the surface of the five remaining blocks and left to dry for 5 min. Four different luting materials were used in this study and applied according to their manufacturer's recommendations (Table 2). For Panavia F 2.0 (Kuraray), equal amounts of pre-heated A and B pastes were mixed for 20 s and inserted in a plastic mold (Ultradent Jig; Ultradent Products, South Jordan, UT) with a bubble-free application system (Composite-Gun and no. 1916 Tubes and plugs; KerrHawe SA, Bioggio, Switzerland). The resin cement cylinder was light polymerized for 20 s. The cement surface was then protected with an oxygen barrier (Oxiguard II; Kuraray) for 3 min. BisCem and Duo-Link resin cements were applied into the plastic mold (Ultradent Jig) using the auto-mixing tips provided in their respective kit and light polymerized for 40 s. The restorative composite resin Z100 (3M-ESPE) was inserted into the plastic mold (Ultradent Jig) in two increments of approximately 1.8 mm using a spatula and each increment was light polymerized for 20 s. Three resin

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