

# Bond strength between zirconia Y-TZP and resin cements: evaluation of surface treatment, cure mode, and failure types

## Abstract

**Purpose:** The aim of this study was to evaluate the effect of the type of curing and surface treatment with universal adhesive containing 10-methacryloyloxydecyl dihydrogen phosphate (MDP) and sandblasting with aluminum oxide particles (50  $\mu$ m) on the bond strength between two resin cements (RelyX Ultimate and U200) and yttria-stabilized zirconia (Y-TZP).

**Materials and methods:** Zirconia cylinders (3.5×16×7mm) were divided into two groups according to the type of cement, for dual and chemical curing. Each group was divided into four subgroups (n=10) according to the surface treatment: adhesive and sandblasting; adhesive only; sandblasting only; and control (no treatment). After cementation, specimens underwent shear bond strength testing in a universal testing machine. A stereomicroscope was used to analyze the failure pattern. Data were statistically analyzed considering  $p < 0.05$ .

**Results:** For both dual-cure cements, the highest values were found for the adhesive and sandblasting treatment group. The results for dual curing were statistically different from the results for chemical curing. Adhesive failure prevailed with both cements.

**Conclusion:** The use of adhesive with MDP can be a favorable alternative to improve the bond strength between resin cements and zirconia Y-TZP. Sandblasting with aluminum oxide particles is not a key factor in improving bond strength.

**Keywords:** yttria stabilized tetragonal zirconia, resin cements, aluminum oxide

Volume 8 Issue 5 - 2017

Bruno Gustavo da Silva Casado,<sup>1</sup> Rafaela de Souza Leao,<sup>1</sup> Joel Ferreira Santiago Junior,<sup>2</sup> Paulo Afonso Silveira Francisconi,<sup>3</sup> Virgilio Bernardino Ferraz Jardim,<sup>1</sup> Edla Layanne Florencio de Carvalho,<sup>1</sup> Sandra Lucia Dantas de Moraes,<sup>1</sup> Marcos Antonio Japiassu Resende Montes<sup>1</sup>

<sup>1</sup>Graduate Program in Dentistry, Faculty of Dentistry, Universidade de Pernambuco, Brazil

<sup>2</sup>Graduate Program in Dentistry, Universidade do Sagrado Coracao, Bauru, Brazil

<sup>3</sup>Graduate Program in Dentistry, Faculty of Dentistry, Universidade de Sao Paulo, Brazil

**Correspondence:** Bruno Gustavo da Silva Casado, Faculty of Dentistry, University of Pernambuco FOP-UPE, Av. General Newton Cavalcanti, 1650, Tabatinga, Camaragibe-PE, 54.756-220, Brazil, Email [brunocasado@hotmail.com](mailto:brunocasado@hotmail.com)

**Received:** October 12, 2017 | **Published:** October 25, 2017

**Abbreviations:** Y-TZP, yttria-stabilized tetragonal zirconia polycrystalline;  $Al_2O_3$ , aluminum oxide; MDP, phosphate ester monomer 10-methacryloyloxydecyl dihydrogen phosphate.

## Introduction

A number of ceramic materials have been developed with improved mechanical properties to meet the increasing demand for metal-free restorations. Yttria-stabilized tetragonal zirconia polycrystalline (Y-TZP) has been used for extensive rehabilitation and in complex restorations because of its high flexural strength, fracture resistance, and chemical durability compared with other all-ceramic materials.<sup>1-3</sup> Surface stability makes cement adhesion to this material difficult, and the establishment of a lasting chemical and mechanical bond to zirconia is a challenge. Various surface treatments to increase the adhesive properties of zirconia-based ceramic restorations have been recommended. Some of these are based on physical entrapment through sandblasting and others are based on chemical bonding using silanes and/or primers.<sup>4</sup> Surface treatment with aluminum oxide ( $Al_2O_3$ ) blasting results in surface roughness from particle friction during the sandblasting process, increasing the contact area of the cement with the zirconia surface.<sup>5</sup> After blasting, a chemical bond should be created via primer, adhesive, or cement containing phosphate monomers. The use of materials containing phosphate ester monomer 10-methacryloyloxydecyl dihydrogen phosphate (MDP) is key to obtaining satisfactory adhesion of the resin cement to zirconia, because MDP directly connects to metal oxides.<sup>3-8</sup> In addition to restoration pretreatment, other factors may affect the indirect adhesive cementation of ceramic restorations, such as the type of cement used

and the curing process. Some studies have shown different results for bond strength for dual-cure adhesive cements when curing is solely chemical and when it is done after photopolymerization. Cement thickness and room temperature can also influence the bond strength between dual-cure adhesive resin cements and zirconia ceramics.<sup>9</sup> The aim of this study was to evaluate the effect of the curing process and surface treatments with a universal adhesive containing MDP and blasting with aluminum oxide particles on the shear bond strength of two resin cements and zirconia Y-TZP.

## Materials and methods

The materials used in this study are described in Table 1. Thirty (30) Cercon CAD/CAM DENTSPLY system Y-TZP cylinders were prepared. Each cylinder was 3.5mm in diameter, 16mm high, with a 7-mm base curve; the area to be used as the cementing surface was 3.5-mm in diameter.

## Division of groups

Specimens were grouped according to the curing method of the resin cement (chemical or dual) and by the type of surface treatment used (Table 2).

## Standardization of the cementing surface

All zirconia cylinders had their surface planed and polished with 320, 600, and 1200 grit wet sandpaper in a metallographic polisher for 10s for each grit. Then, the cylinders were cleaned in an ultrasonic vat with 92.81NPM ethanol for 10min. After cleaning, they were blotted dry for 1min and then blown with dry, oil-free air for 10s.

**Table 1** Materials used in the study

Product/Batch number	Manufacturer	Composition
Zirconia (Y-TZP)	Cercon CAD/CAM DENTSPLY	-
RelyX U200 (batch no. 591080)	3M ESPE (Seefeld, Germany)	Multifunctional phosphoric acid methacrylates, dimethacrylates, acetate, initiator/stabilizer, powdered glass, silica, substituted pyrimidine, calcium hydroxide, peroxide compound, pigments
RelyX Ultimate (1st cement batch no. 1511700365, 2nd batch no. 150550033)	3M ESPE (Seefeld, Germany)	Multifunctional phosphoric acid methacrylates, powdered glass, silane-treated silica, dimethacrylates, calcium hydroxide, initiator/stabilizer, substituted pyrimidine, propionic acid, pigments
Single Bond Universal Adhesive (1st adhesive batch no. 577056, 2nd batch no. 571695)	3M ESPE (Sumaré, Brazil)	Bis-GMA, ethyl alcohol, methacrylates, camphor quinone, acrylic copolymer, itaconic acid
Aluminum oxide (batch no. 33086)	Bio-Art (São Carlos/SP, Brazil)	50µm aluminum oxide particles

**Table 2** Division of study groups, according to surface treatment, cement and cure

	Control group (without blasting/ without adhesive)	Group without blasting/with adhesive	Group with blasting/without adhesive	Group with blasting/with adhesive
U200 chemical cure	10	10	10	10
Ultimate chemical cure	10	10	10	10
U200 dual cure	10	10	10	10
Ultimate dual cure	10	10	10	10

## Sandblasting

A microblaster (Bio-Art, Sumaré/SP, Brazil) with 50-µm aluminum oxide particles blasted at a distance of 10mm for 10s under 2.8 bar pressure was used. Zirconia surfaces that were not to be sandblasted were protected with Teflon tape. After the procedure, the cylinders were cleaned in an ultrasonic vat for 3min with 96% alcohol.

## Adhesive system application

The adhesive system (Single Bond Universal Adhesive) was applied with the aid of a disposable applicator, following the manufacturer's recommendations.

## Cylinder cementation

Two resin cements were used: RelyX U200 and RelyX Ultimate, both from 3M ESPE. In order to standardize this procedure, a bipartite Teflon matrix and a standard positioning device were used. The cylinder was placed between the bipartite matrixes to provide a standardized thickness of cement of 3 mm and then placed in the device. The cements were handled according to the manufacturer's recommendations, placed on the zirconia surface with a resin spatula, and kept under manual pressure by a single operator with polyester tape and a transparent acrylic device until polymerization time

was completed. For the chemical cure groups, a 6-minute period after cement manipulation was observed, as recommended by the manufacturer. For the dual-cure groups, a photopolymerizer (Radii Cal, SDI) was used with radiation between 480 and 500mW/cm<sup>2</sup> for 40s, in addition to the 6 min recommended for chemical curing.

## Shearing test

After cylinder cementation, bond strength was measured in a universal testing machine (Kratos K2000MP, São Paulo, Brazil) by means of the tensile test to assess shear bond strength using a 200 kg force load cell at a speed of 0.55mm/min until breakage of the zirconia/resin cement set. Bond strength values were converted to megapascals (MPa).

## Types of failure

After the specimens broke, the cementing surface was analyzed under a stereomicroscope (Dino-Lite AM7013MZT, São Paulo, Brazil) at × 20 magnifications by a single trained evaluator. Failures were classified as adhesive, cohesive, or mixed. Adhesive failure occurred when more than 75% of the zirconia surface was visible. A cohesive failure occurred when more than 75% of the zirconia was covered with resin cement. The remaining cases were classified as mixed failures.

## Statistical analysis

Data were tabulated in an Excel spreadsheet, and the three-way (ANOVA) F test and equal and unequal variance Student t tests were performed. Verification of the hypothesis of normality and equal variances was carried out by Levene's F test and the Shapiro-Wilk test, respectively. Statistical Package for the Social Sciences (SPSS) software version 21.0 was used for the statistical analyses.

## Results

For the dual-cure samples (Table 3) in which U200 cement was used, the highest strength average was obtained for adhesive and sandblasting group (7.76). However, averages ranged from 1.86 to 5.57 for the other treatments, with no significant differences between the adhesive only and blasting only groups ( $p>0.05$ ). For the Ultimate cement samples, the highest average also occurred in the adhesive and sandblasting group (6.31), but there were no statistically significant

differences compared with the adhesive-only group (5.68). The groups without adhesive were statistically different compared with those that used the system. Samples subjected to chemical curing only with U200 cement (Table 4) showed no statistically significant differences between the surface treatments studied, except for the control group, which had the lowest average (0.80). As for Ultimate cement, the adhesive and sandblasting group achieved the best bond strength values (2.04), showing statistically significant differences from the other groups. When the bond strength averages for chemical and dual-cure samples were compared, the average for photoactivation initiated samples was higher than that of the chemically activated samples (Table 5). These differences were statistically significant for both cements in all surface treatments studied, even in the absence of treatment ( $p<0.05$ ). Considering the specimen failure mode, the adhesive failure prevailed for both Relyx Ultimate (57.2%) and for Relyx U200 (76.25%) cements.

**Table 3** Cement strength statistics (tensile strength) according to surface treatment for dual-cure samples

Cement	Statistics	Group				P Value <sup>1</sup>
		With adhesive/with blasting	With adhesive/without blasting	Without adhesive/with blasting	Without adhesive/without blasting	
U200	Mean	7.76A	5.57B	4.35B	1.86C	<0.001*
	Standard deviation	1.11	1.72	0.78	0.65	
	Coefficient of variation	14.3	30.88	17.93	34.95	
Ultimate	Mean	6.31A	5.68A	2.93B	2.30B	<0.001*
	Standard deviation	1.31	1.56	0.5	0.67	
	Coefficient of variation	20.76	27.46	17.06	29.13	
p value <sup>2</sup>	0.015*	0.884	<0.001*	0.148		

\*Significant difference at 5.0% level. Superscript uppercase letters indicate a significant difference between the corresponding groups

<sup>1</sup>F test (ANOVA) with Tamhane's T<sup>2</sup> pairwise comparisons

<sup>2</sup>Student t test with equal variance

**Table 4** Strength statistics (tensile strength) by cement according to subgroup in the chemically activated group

Cement	Statistics	Subgroup				P Value <sup>1</sup>
		With adhesive/with blasting	With adhesive/without blasting	Without adhesive/with blasting	Without adhesive/without blasting	
U200	Mean	1.41 A	1.57A	1.44 A	0.80 B	<0.001*
	Standard deviation	0.36	0.47	0.34	0.26	
	Coefficient of variation	25.53	29.94	23.61	32.5	
Ultimate	Mean	2.04 A	1.47 B	1.21B	1.03B	<0.001*
	Standard deviation	0.54	0.42	0.34	0.44	
	Coefficient of variation	26.47	28.57	28.1	42.72	
p value <sup>2</sup>		0.007*	0.652	0.152	0.172	

\*Significant differences at 5.0% level

<sup>1</sup>F (ANOVA) test with Tukey comparisons

<sup>2</sup>Student t test with unequal variance

**Table 5** Comparison between chemical and dual curing

Cement	Adhesive	Blasting	Dual cure, Mean±SD (CV)	Chemical Cure, Mean±SD (CV)	p Value
U200	With	With	7.76±1.11 (14.30)	1.41±0.36 (25.53)	<0.0011,*
		Without	5.57±1.72 (30.88)	1.57±0.47 (29.94)	<0.0011,*
	Without	With	4.35±0.78 (17.93)	1.44±0.34 (23.61)	<0.0011,*
		Without	1.86±0.65 (34.95)	0.80±0.26 (32.50)	<0.0012,*
Ultimate	With	With	6.31±1.31 (20.76)	2.04±0.54 (26.47)	<0.0011,*
		Without	5.68±1.56 (27.46)	1.47±0.42 (28.57)	<0.0011,*
	Without	With	2.93±0.50 (17.06)	1.21±0.34 (28.10)	<0.0012,*
		Without	2.30±0.67 (29.13)	1.03±0.44 (42.72)	<0.0012,*

Considering the specimen failure pattern, adhesive failure prevailed for both RelyX Ultimate (57.2%) and for RelyX U200 (76.25%) cements  
\*Significant differences at 5.0% level

<sup>1</sup>Student t test with equal variance

<sup>2</sup>Student t test with unequal variance

## Discussion

Dental material manufacturers provide protocols for product use to ensure satisfactory and safe clinical performance. However, studies are being done in search of alternatives to optimize the results of these materials. This study evaluated zirconia Y-TZP surface treatments, including those recommended by RelyX U200 and RelyX Ultimate cement manufacturers, by alternating the use of universal adhesive and aluminum oxide particle blasting. RelyX U200 cement dual-cure analyses showed variations when comparing the surface treatment recommended by the manufacturer (the use of aluminum oxide particle blasting) with the other treatments. The addition of a Single Bond Universal Adhesive layer showed favorable bond strength results, even though it is a self-adhesive cement, particularly when associated with aluminum oxide particle sandblasting. These results are in agreement with those of other studies which show that the combination of two types of surface treatment is positive in zirconia bonding.<sup>10-12</sup> The use of a universal adhesive system for RelyX Ultimate cement proved to be essential; the values for the groups in which adhesives were not used were much lower than those for the groups with adhesives ( $p<0.001$ ). These results were expected because of the characteristics of the cement and the way the product has been marketed whereby the cement is not sold separately from the adhesive. Blasting, on the other hand, was not a key factor in the bond strength results for RelyX Ultimate cement, in as much as there were no statistical differences ( $p<0.001$ ) between the groups that used this treatment and those that did not. When the performance of the two cements was compared for the standard protocol indicated by the manufacturer (RelyX Ultimate, adhesive + sandblasting; RelyX U200, sandblasting), the RelyX Ultimate cement showed better results. This fact can be justified by the use of a universal adhesive that includes MDP in its composition, a monomer that was developed to achieve direct bifunctional adhesion with metal oxides and Bis-GMA matrix, thus establishing a more effective chemical adhesion between surfaces. The good performance with PEM is highlighted when the results for U200 cement for the blasting-only and adhesive-only groups are compared. The lack of statistical differences shows that blasting can be replaced by adhesive containing MDP without loss of zirconia bonding strength. This can facilitate clinical practice by eliminating the need for additional equipment (microblaster),

thus decreasing costs and working time for zirconia P-YTZ-based indirect restorations. Moreover, zirconia-related failures eventually occur from the action of mechanical wear, including blasting itself as a cause of structural damage, as shown in a study by Chintapalli RK et al.<sup>13</sup> This does not mean that sandblasting should be banned, as the same author states a year later that, when performed with care and control, blasting is beneficial.<sup>14</sup> However, replacing the blasting process with the use of adhesive containing MDP appears to be a more common practice in clinical routine.<sup>15-17</sup> A recent systematic review by Ozcan & Bernasconi<sup>18</sup> recommends caution when using self-adhesive cements, because of lower bond strength results compared with cements containing MDP. Thus, the results of the present study are in agreement with others,<sup>2</sup> which report that the use of MDP gave higher bonding values. Therefore, the use of systems containing MDP appears to be an effective and safe alternative for zirconia surface treatment. Another relevant analysis for zirconia-based restorations is the resin cement curing method used, because the area at innermost cementing line does not get the same intensity of light because of the opaque structure of zirconia. By comparing chemically activated dual-cure groups, samples subjected to an initial photoactivation showed better results than chemically activated samples for all surface treatments and for both cements. This is in agreement with previous studies which have concluded that the type of polymerization may affect the mechanical properties and that photopolymerization of dual-cure cement provides superior mechanical properties compared with chemical cure only cements, because the mechanism of chemical polymerization of dual-cure resin cements is slower.<sup>9-20</sup> Despite the significant statistical differences between the curing processes, this result are believed to be influenced by the time elapsed from cementation until the analysis. The predominance of adhesive failure at the initial stage of cementation was observed for most samples in this study. This warns us of the difficulties with zirconia bonding, even when MDP-containing adhesive blasting is applied, inasmuch as this failure is associated with lower bond strength values. This difficulty should stimulate new studies in the area.

## Conclusion

Within the limitations of this in vitro study, it can be concluded that:

- a. Dual curing significantly improves the bond strength between resin cement and zirconia Y-TZP.
- b. The use of a MDP-containing adhesive system can be a favorable alternative to improve the bond strength between resin cements and zirconia Y-TZP.
- c. Zirconia Y-TZP blasting with aluminum oxide particles (50µm) is not a key factor in bond strength improvement.

## Funding details

None.

## Acknowledgments

This study was supported by a social demand scholarship to master's students, granted by Commission for the Improvement of Higher Education Personnel (CAPES).

## Conflicts of interest

There were no conflicts of interest.

## References

1. Qeblawi DM, Munoz CA, Brewer JD, et al. The effect of zirconia surface treatment on flexural strength and shear bond strength to a resin cement. *J Prosthet Dent*. 2010;103(4):210–220.
2. Xie H, Li Q, Zhang F, et al. Comparison of resin bonding improvements to zirconia between one-bottle universal adhesives and tribochemical silica coating, which is better? *Dent Mater*. 2016;32(3):403–411.
3. Yun J, Ha S, Lee J, et al. Effect of sandblasting and various metal primers on the shear bond strength of resin cement to Y-TZP ceramic. *Dent Mater*. 2010;26(7):650–658.
4. Souza G, Hennig D, Aggarwal A, et al. The use of MDP-based materials for bonding to zirconia. *J Prosthet Dent*. 2014;112(4):895–902.
5. Kern M, Barloï A, Yang B. Surface conditioning influences zirconia ceramic bonding. *J Dent Res*. 2009;88(9):817–822.
6. Attia A, Lehmann F, Kern M. Influence of surface conditioning and cleaning methods on resin bonding to zirconia ceramic. *Dent Mater*. 2011;27(3):207–213.
7. Kawai JLN, Youmaru ASH, Shynia A, et al. Effects of tree luting agents and cyclic impact loading on shear bond strength to zirconia with tribochemical treatment. *J Dent Sciences*. 2012;7–2:118–124.
8. Kim JM, Kim YK, Kim KH, et al. Shear bond strengths of various luting cements to zirconia ceramic: Surface chemical aspects. *J Dent*. 2012;39(11):795–803.
9. Luhrs AK, Pongprieska P, De Munck J, et al. Curing mode affects bond strength of adhesively luted composite CAD/CAM restorations to dentin. *Dent Mater*. 2014;30(3):281–291.
10. Inokoshi M, De Munck J, Minakuchi S, et al. Meta-analysis of bonding effectiveness to zirconia ceramics. *J Dent res*. 2014;93(4):329–334.
11. Carvalho RF, Louzada F, Monteiro E, et al. Bond strength to zirconia ceramic/resin cement: Influence of an alternative surface treatment. *Dent Mater*. 2012;(29–1):e19.
12. Castro HL, Bottino MA, Della Bona A. Influence of resin cement and ceramic treatment on bond strength. *Dent Mater*. 2011;(27–1):e5–e6.
13. Chintapalli RK, Marro FG, Jimenez-Pique E, et al. Phase transformation and subsurface damage in 3Y-TZP after sandblasting. *Dent Mater*. 2012;29(5):566–572.
14. Chintapalli RK, Rodriguez AM, Marro FG, et al. Effect of sandblasting and residual stress on strength of zirconia for restorative dentistry applications. *J Mech Behav Biomed Mater*. 2014;29:126–137.
15. Ahn J, Yi Y, Lee Y, et al. Shear bond strength of MDP-containing self-adhesive resin cement and Y-TZP ceramics: Effect of phosphate monomer-containing primers. *BioMed Research International*. 2015;(11):1–6.
16. Tanis MÇ, Akçaboy C. Effects of different surface treatment methods and MDP monomer on resin cementation of zirconia ceramics an *in vitro* study. *J Lasers Med Sci*. 2015;6(4):174–181.
17. Xie H, Tay FR, Zhang F, et al. Coupling of 10-methacryloyloxydecylidihydrogenphosphate to tetragonal zirconia: Effect of pH reaction conditions on coordinate bonding. *Dent mater*. 2015;(31):e218–e225.
18. Ozcan M, Bernasconi M. Adhesion to zirconia used for dental restorations: A systematic review and meta-analysis. *J Adhes Dent*. 2015;17(1):7–26.
19. Jongsma LA, Kleverlaan CJ, Pallav P, et al. Influence of polymerization mode and C-factor on cohesive strength of dual-cured resin cements. *Dent Mater*. 2012;28(7):722–728.
20. Pereira SG, Fulgêncio R, Nunes TG, et al. Effect of curing protocol on the polymerization of dual-cured resin cements. *Dent Mater*. 2012;26(7):710–718.