

# Bonding Polycrystalline Zirconia With 10-MDP-containing Adhesives

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## Clinical Relevance

10-MDP-containing adhesives are important for bonding resin cements to zirconia. However, various concentrations of this component need further investigation to determine the most optimal amount responsible for improving the bonding to Y-TZP.

## SUMMARY

**Objective:** The objective of this study was to evaluate the influence of adhesives with different 10-MDP concentrations on the shear bond strength of a resin cement to zirconia.

**Methods and Materials:** Six experimental adhesives were prepared with the following

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composition: camphorquinone, 1,2-diaminobenzene, butylhydroxytoluene, diphenyliodonium hexafluorophosphate, 2-hydroxyethyl methacrylate triethylene glycol dimethacrylate, ethoxylated bisphenol A glycol dimethacrylate, urethane dimethacrylate, bisphenol A diglycidyl methacrylate, and ethanol. The 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) monomer was added at 0wt%, 3wt%, 6wt%, 9wt%, 12wt%, or 15wt%. Three commercially available adhesives were evaluated: Single Bond Universal, Single Bond 2, and Signum Zirconia Bond. Resin cement cylinders made with RelyX Ultimate were bonded to yttria-stabilized tetragonal zirconia polycrystal with one of the evaluated adhesives and were subjected to the shear bond strength evaluation. Failure modes were analyzed with a stereoscopic loupe. Statistical analyses were performed with one-way analysis of variance and the Tukey's Honestly Significant Difference test ( $\alpha=0.05$ ). Pearson's was used to correlate the percentage of 10-MDP in the experimental adhesives and shear bond strength.

**Results:** There were significant differences between adhesives ( $p<0.00001$ ). The highest shear bond strength values were obtained with the Signum Zirconia Bond and Single Bond Universal. Single Bond 2 showed the lowest values. There were no differences between experimental adhesives. All groups showed

**adhesives failures. A nonlinear correlation was found between bond strength and percentage of 10-MDP in experimental adhesives ( $r=0.872$ ).**

**Conclusions: The commercially available adhesives indicated for bonding to zirconia showed the highest bonding values.**

## INTRODUCTION

The clinical success of all-ceramic yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) indirect restorations not only depends on the correct knowledge and handling of the material itself but also the use of an adhesive system associated with resin-based cement to provide satisfactory bonding of the prosthetic work to the dental structures.<sup>1</sup> Y-TZP is widely recognized for its excellent mechanical, physical, and thermal properties; biocompatibility; high fracture toughness; hardness; and wear resistance.<sup>2</sup> Although Y-TZP ceramics present all these excellent properties, the effectiveness of adhesive cementation procedures is still a problem, because Y-TZP ceramics cannot be conditioned by the application of hydrofluoric acid and conventional silane coupling agents due to the absence of silica and a glass phase.

In search of solutions, different procedures to improve the bond of the resin cement to the inner surface of zirconia have been tested, such as surface preparation with erbium-doped and yttrium-aluminum-garnet laser (Er: YAG), grinding with diamond rotary instruments, selective infiltration etching, surface roughening by aluminum oxide blasting of different particle sizes before or after sintering, surface roughening by alumina-silica particles before silanization, and application of a liner.<sup>2-10</sup> All these methods seek to improve the mechanical and micromechanical interlocking through the increase in the roughness of the surface. However, some of these treatments have proved to be ineffective, and, in several cases, they may cause surface damage.<sup>11</sup>

A different approach to improve the bond strength to zirconia is to develop a chemical interaction between the surface and the applied resin cement.<sup>12</sup> For this task, research has focused on the use of primers that contain phosphate monomers that have an affinity for metal oxides with 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) being one of the most used monomers.<sup>13,14</sup> Although important for bonding to both dental substrates and zirconia, the maximum concentration of 10-MDP monomers that should be added to dental adhesives still needs

to be evaluated, as it has been suggested that the polymerization of camphorquinone (CQ)/amine-based adhesives may be negatively affected by the interaction between functional monomers such as 10-MDP and tertiary amines.<sup>15</sup> Therefore, it is important to evaluate how the incorporation of different concentrations of 10-MDP may improve or jeopardize the bonding of adhesive systems to zirconia.

It is well known that light-cured resin-based materials such as adhesives commonly contain CQ and tertiary amines that allow free-radical polymerization triggered by the irradiation with visible light. On the other hand, it has been suggested that the inclusion of a third component to the CQ/amine photoinitiating system such as iodonium salts could improve the photo-activation induced by visible light sources.<sup>16</sup>

10-MDP monomers are required to obtain bonding with acid resistant polycrystalline ceramic materials such as zirconia. It has been suggested that these monomers can interact with metal oxides, enabling chemical bonding of ceramic oxides with or without an additional coupling agent.<sup>17</sup> Thus, the purpose of the present study was to evaluate the influence of different 10-MDP concentrations on the shear bond strength of a resin cement to zirconia. For this purpose, 10-MDP was added to six experimental adhesives and compared with three commercially available materials. The null hypotheses evaluated were (1) the concentration of 10-MDP monomers in the experimental adhesives would not influence the shear bond strength; and (2) there would be no difference between the experimental and commercially available adhesives.

## METHODS AND MATERIALS

Experimental adhesives contained of a mixture of 2-hydroxyethyl methacrylate (HEMA), triethylene glycol dimethacrylate (TEGDMA), ethoxylated bisphenol A glycol dimethacrylate (Bis-EMA), urethane dimethacrylate (UDMA), and bisphenol A diglycidyl methacrylate (Bis-GMA). Ethanol (10wt%), CQ (0.50wt%), 1,2-diaminobenzene (DABE) (1.00wt%), iodonium salt diphenyliodonium hexafluorophosphate (DPIHP) (0.45wt%), and butylhydroxytoluene (BHT) (0.20wt%) were added. Five concentrations of 10-MDP were added to this basic adhesive: 3wt%, 6wt%, 9wt%, 12wt%, or 15wt%. Materials were used without further purification. As controls, three commercially available adhesive systems were evaluated: Single Bond 2 (not indicated for bonding to zirconia), Single Bond Universal

Table 1: *Materials and Composition*

Adhesive (manufacturer)	Composition	Instructions for use
Adper Single Bond 2 (3M ESPE, Sumaré, SP, Brazil)	BisGMA, HEMA, dimethacrylates, silica nanofiller, ethanol, water, photoinitiator system and methacrylate functional copolymer of polyacrylic and polyitaconic acids	(a) Apply two to three consecutive coats of adhesive to the ceramic surface for 15 seconds with gentle agitation using a fully saturated applicator; (b) gently air thin for five seconds to evaporate solvents; (c) light cure for 10 seconds
Single Bond Universal (3M ESPE, Seefeld, Germany)	MDP phosphate monomer, dimethacrylate resins, HEMA, Vitrebond copolymer, filler, ethanol, water, initiators, silane	(a) Clean the surface with alcohol and dry it with compressed air; (b) apply with a microbrush to the surface for 20 seconds; (c) apply compressed free oil air for five seconds; (d) light cure for 10 seconds
Signum Zirconia Bond (Heraeus Kulzer, Hanau, Germany)	Signum zirconia bond I <sup>a</sup> : Acetone, 10-MDP, acetic acid. Signum zirconia bond II <sup>a</sup> : methyl methacrylate, diphenyl(2,4,6-trimethylbenzoyl)phosphine oxide. MMA, initiators	(a) Clean the surface with alcohol and dry it with compressed air; (b) Signum Zirconia bond I is dispensed and applied with a suitable brush to the entire surface and air dried for five seconds; (c) Signum Zirconia bond II is applied and light cured for 40 seconds

<sup>a</sup> Obtained from the manufacturer's safety data sheet.

(indicated for bonding to zirconia), and Signum Zirconia Bond (indicated for bonding to zirconia). Table 1 describes the composition, manufacturers, and instructions for the use of these adhesives.

Polycrystalline ceramic blocks (IPS e.max ZirCAD, Ivoclar Vivadent, Schaan, Liechtenstein) were cut into 2-mm-thick slices with a cutting machine (Isomet 1000 Low Speed, Buehler, Lake Bluff, IL, USA) using a diamond disc (15LC diamond no. 11-4254, Buehler) at a speed of 275 rpm under constant water irrigation. Four ceramic slices were used in each group. The slices were polished with sequential sandpaper discs (grit sizes, #800, #1000, and #1200; K2000 Polishing Paper, Exact, Nordestedt, Schleswing-Holstein, Germany) using a metallographic polishing machine (Exact, Nordestedt) to standardize the ceramic surfaces. Forty slices were prepared.

After surface standardization, the ceramic slices were sintered (INFIRE Oven HTC Speed, Sirona Dental Systems, Long Island City, NY, USA) according to the recommendations of the ceramic manufacturer. The slices were then randomly divided into nine groups and embedded in acrylic resin. The surfaces were polished with sequential sandpaper discs (grit sizes #800 to #1200; K2000 Polishing Paper, Exact) to remove any acrylic resin that might have covered the samples. Samples were washed under copious deionized water cooling and dried with oil-free air with a pressure of 40 psi, at a standardized distance of 15 cm and an angle of 45°. Experimental adhesives were applied as follows: all the ceramic surfaces were cleaned and dried; adhesives were applied with a microbrush and remained untouched for 20 seconds; oil-free air with

pressure of 40 psi was applied at 90° from a distance of 15 cm for five seconds to evaporate solvents; and adhesives were light cured for 10 seconds with an LED device (VALO Cordless, Ultradent Products, South Jordan, UT, USA) operating at an irradiance of 1100 mW/cm<sup>2</sup>. Commercially available adhesives were applied as per manufacturers' instructions and were light cured with the same device.

Surgical catheters with an internal diameter of 1.40 mm and a height of 1 mm were used for the resin cement cylinders (RelyX Ultimate, 3M ESPE, St. Paul, MN, USA). Four cylinders per ceramic surface were prepared, resulting in 16 for each group. The cement was mixed according to the manufacturer's recommendations, inserted into the catheters, and light cured for 20 seconds with the VALO curing device with irradiance of 1100 mW/cm<sup>2</sup>. After light curing, the surgical catheters were kept untouched for 10 minutes before being removed with #11 scalpel blades (Embramed, Jurubatuba, SP, Brazil) to expose the resin cement cylinders. Samples lost during removal of the catheters were counted and considered as 0 MPa in the statistical analysis.

The samples were stored in deionized water for 24 hours at 37°C. After storage, the samples were subjected to shear bond strength evaluation using a 0.2-mm wire-loop positioned as close as possible of the adhesive interface (Figure 1) and a universal testing machine (Instron 3342, Illinois Tool Works, Norwood, MA, USA) with a 500-N load cell operated at a crosshead speed of 0.5 mm/min.

Data were analyzed with one-way analysis of variance and the Tukey Honestly Significant Differ-

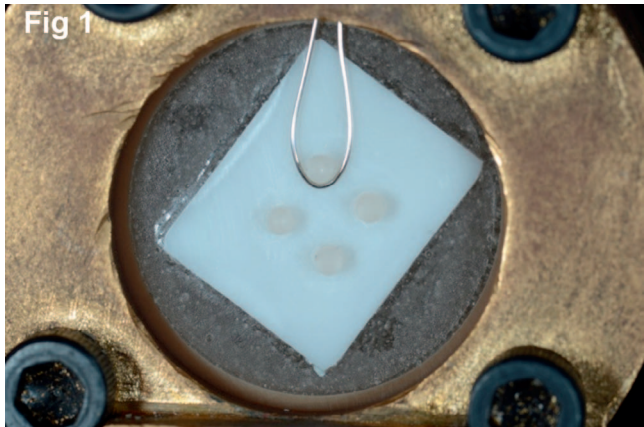


Figure 1. Example of samples adapted to the universal testing machine before the shear bond strength evaluation.

ence test. A global significance level of 5% was adopted. Pearson correlation analysis was used to determine whether there was a correlation between the percentages of 10-MDP in the experimental adhesives and shear bond strength. The failure modes were evaluated with a stereoscopic loupe.

RESULTS

Mean values, number of lost samples for each group, and SDs for the shear bond strength are shown in Table 2. There were significant differences between groups ( $F=6.5741$ ;  $p<0.00001$ ). The highest shear

Table 2: Mean Values and SDs for Shear Bond Strength		
Adhesive	Shear bond strength, MPa	Sample losses (% of 16 samples)
Single Bond 2	6.06 ± 5.78 A	4 (25%)
Single Bond Universal	14.02 ± 6.5 BC	2 (12.5%)
Signum Zirconia Bond	20.86 ± 6.11 c	0 (0%)
0% 10-MDP	7.89 ± 8.38 AB	6 (37.5%)
3% 10-MDP	9.34 ± 6.75 AB	2 (12.5%)
6% 10-MDP	12.56 ± 7 AB	1 (6.25%)
9% 10-MDP	13.43 ± 6.62 AB	3 (18.75%)
12% 10-MDP	10.7 ± 6.08 AB	2 (12.5%)
15% 10-MDP	11.81 ± 4.2 AB	0 (0%)
Different letters represent statistically significant differences ( $p<0.05$ ).		

bond strength values were obtained with the Signum Zirconia Bond and Single Bond Universal. There were no differences between the experimental adhesives. Single Bond 2 showed the lowest values. The failure mode was adhesive for all specimens. A nonlinear correlation was found between bond strength and percentage of 10-MDP in the experimental adhesives ( $r=0.872$ ; Figure 2).

DISCUSSION

The present study evaluated the shear bond strength of a resin cement to zirconia after the application of 10-MDP-containing experimental and commercially available adhesives. Although a nonlinear correla-

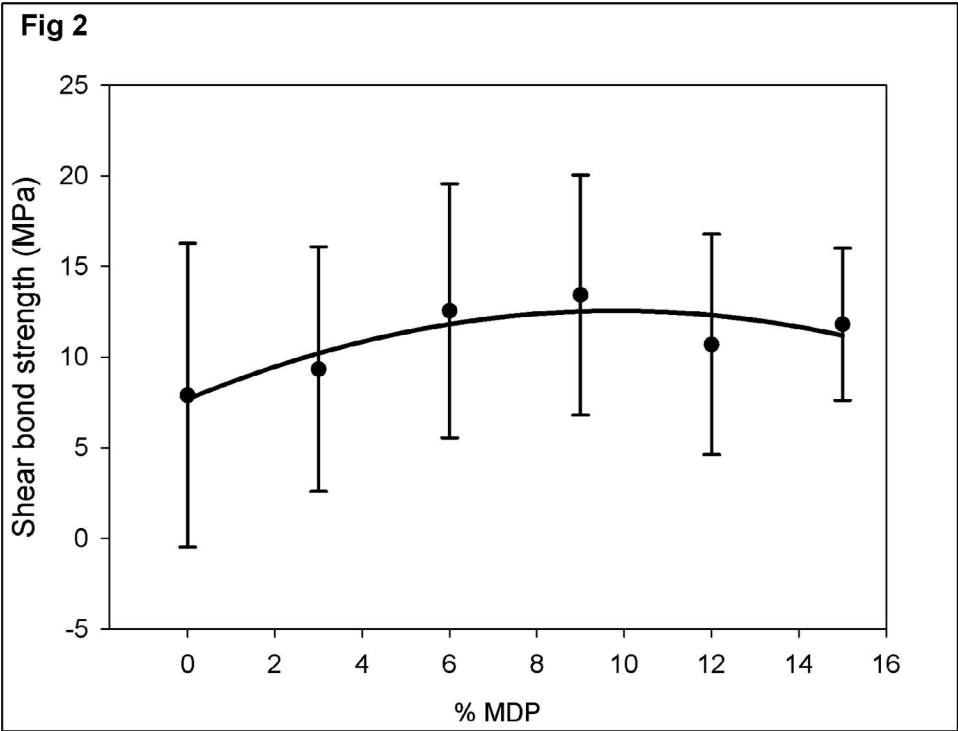


Figure 2. Pearson correlation analysis. A nonlinear correlation was found between bond strength and percentage of 10-MDP in the experimental adhesives ( $y = -0.0506x^2 + 0.9927x + 7.6836$ ;  $r=0.872$ ).

tion was found between bond strength and percentage of 10-MDP added to the experimental adhesives (Figure 2), in this study, the first null hypothesis was accepted because no statistically significant difference was found between experimental adhesives. The second null hypothesis was rejected, because there were differences between experimental and commercially available adhesives.

In the present study, no ceramic pretreatment was conducted. Several pretreatments have been suggested and studied in the literature to improve the adhesion between Y-TZP and resin cements. As a standard method, the aluminum oxide sandblasting technique has been described.<sup>10,18</sup> Different advantages of this technique include increasing surface roughness, wettability, and surface energy of the ceramics, which improve the micromechanical retention.<sup>18</sup> On the other hand, Y-TZP ceramics may suffer a phase transformation from tetragonal to monoclinic, which may be detrimental for the durability and mechanical properties of this ceramic.<sup>10</sup> As the present study was designed to address the bond strength of different adhesive systems without the interference of surface-related characteristics such as roughness, no sandblasting or any other mechanical surface treatment was conducted. Additionally, to better understand the influence of the different adhesives evaluated, after they were embedded in acrylic resin, Y-TZP discs were polished with #1200 sandpaper. However, because Y-TZP ceramic restorations are obtained by milling technologies, higher bond strength values would be expected due to the increased roughness.

Another point to be addressed is the fact that in the present study the light activation of the resin cement was conducted directly, without the influence of a ceramic material between the light source and the resin cement. Although brands of Y-TZP materials may present different degrees of translucency, the light attenuation caused by the ceramics may play a role on the properties of the resin cement.<sup>19-21</sup> Although the interposition of ceramic materials between the light source and the resin cement has the effect of decreasing the degree of conversion and the hardness of the resin cement,<sup>19</sup> it has been suggested that the thickness of the interposing ceramic material influences the light transmittance, and as ceramic thickness increases, longer irradiation periods are required.<sup>21</sup> Besides the composition of the adhesive and the microstructure and thickness of the interposing ceramic material, it is known that the bonding between the ceramics and the dental substrate also depends on the proper

curing of the resin cement, and factors such as type of resin cement, characteristics of the light curing device, and light activation protocol could play a major role. For this reason, the results of the present study could be different not only if the ceramics were interposed but also if other resin cements and curing protocols were evaluated.

Regarding the shear bond strength method, some points should also be discussed. As described in the literature, two experimental designs, shear and tensile tests both at the micro- or macro-scale, are commonly used in this type of study.<sup>22</sup> In general, the shear bond strength test is used because it is simpler than tensile tests. On the other hand, shear tests have a nonhomogeneous stress distribution at the adhesive interface.<sup>23</sup> For this reason, absolute numerical results obtained from one study cannot be directly compared to another. It should be noted that, despite the described limitations regarding the bond strength evaluations, different materials are similarly ranked, as correlations between different methods have been found.<sup>24,25</sup>

In the present study, the shear bond strength evaluation was conducted on 24-hour water-stored samples. Although a nonlinear correlation was found between bond strength and percentage of 10-MDP in the experimental adhesives (Figure 1), it was shown that the concentration of 10-MDP monomers on the experimental adhesives did not influence the bond strength of the evaluated resin cement, whereas the two commercially available adhesives indicated for bonding to zirconia that also contain 10-MDP showed a better performance. These results are in agreement with others studies.<sup>17,26,27</sup> It is, however, important to note that, although no long-term storage or aging was conducted in the present study, the differences between materials containing different 10-MDP concentrations could be demonstrated if a long-term evaluation of the bond strength was conducted. Other studies confirm that active parts of 10-MDP react with the zirconia surface, but this is vulnerable to instability after aging.<sup>1,28</sup>

For a better interpretation of this study, it is important to understand how the 10-MDP monomer works. Chemically, this monomer bonds to oxide metals and tooth substrates. It has an amphiphilic structure with the vinyl group as the hydrophobic half and the phosphate group as the hydrophilic half.<sup>29</sup> The 10-MDP monomer is an effective bonding agent between the resin cement, zirconia, and other metal oxide materials.<sup>30</sup> Authors such as Yoshida and others<sup>17</sup> evaluated the interaction of hydroxyl groups of the phosphate half with the hydroxyl groups on the

zirconia surface through van der Waals forces or hydrogen bonds. Because of these studied properties, many commercial products such as adhesives, primers, and resin cements have incorporated 10-MDP in their composition, seeking to improve the bonding properties to Y-TZP ceramic materials.<sup>31</sup>

Signum Zirconia Bond is a 10-MDP-containing material especially designed for the purpose of bonding resin cements to Y-TZP ceramics. This bonding material is delivered by two different bottles. MDP monomers, acetic acid, and acetone are present in bottle 1, whereas bottle 2 contains diphenyl phosphine oxide and methyl methacrylate. In the present study, this material has shown increased shear bond strength. Other studies have also shown similar results.<sup>18,32</sup> According to Ural and others,<sup>33</sup> the key factor of this material could be the methyl methacrylate, which establishes primary bonds with the methacrylate present in the resin cement and improves the bond strength in that way. Thus, a further study with more focus on MDP and methyl methacrylate interaction is suggested. Although important for bonding resin cements to zirconia, the 10-MDP concentration alone is not the only factor to be considered.

## CONCLUSIONS

According to the results of the present study, it may be concluded that the commercially available adhesives indicated for bonding to Y-TZP showed the highest bonding values, and the concentration of 10-MDP on experimental adhesives was not significant.

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## Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Bauru School of Dentistry, University of São Paulo, Brazil.

## Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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