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Laboratory Research

Bond Strength of Resin Cements to a Zirconia Ceramic with Different Surface Treatments

AN Cavalcanti • RM Foxton • TF Watson

MT Oliveira • M Giannini • GM Marchi

Clinical Relevance

Air abrasion and the use of special functional monomers able to chemically bond to zirconium dioxide can improve bonding to a highly dense zirconia ceramic.

SUMMARY

This study evaluated the influence of surface treatments and metal primers on the bond strength of resin cements to a yttrium-stabilized tetragonal zirconia (Y-TZP) ceramic. Two-hun-dred and forty plates of Y-TZP ceramic were randomly assigned to 24 groups (n=10) according to the combination of surface treatment (none, air abrasion with Al2O3 particles, Er:YAG laser irradiation), metal primer (none, Alloy Primer, Metal Primer II or Metaltite) and resin cement (Calibra[Bis-GMA-based] or Panavia F2.0 [MDP-based]). Fragments of dentin with a cylindrical edge (0.8 mm in diameter) were fixed to ceramic surfaces with the resin cements. The micro-shear bond test was carried out at a 1 mm/minute speed until failure, and the ceramic surfaces were examined after debonding. Bond strengths were analyzed through three-way ANOVA/Tukey test with a 5% significance level. Changes in topography after surface treatments were evaluated with scanning electron microscopy. Surface treatments significantly modified the topography of the Y- TZP ceramic. Air abrasion resulted in increased bond strength for both resin cements. However, air abraded and laser irradiated specimens presented higher bond strength with the Bis-GMA- based resin cement than with the MDP-based cement. Both cements presented similar behavior on untreated surfaces.

- Andrea Nóbrega Cavalcanti, DDS, MS, PhD, Department of Restorative Dentistry, Piracicaba Dental School, UNICAMP, Piracicaba, Brazil
  Richard Mark Foxton, BDS, PhD, MFDS RCS (Ed), Department of Conservative Dentistry, King's College London Dental Institute, King's College London, London, UK
  Timothy Frederick Watson, BDS, PhD, FDS RCS (Eng), Biomaterials, Biomimetics & Biophotonics Research Group, King's College London Dental Institute, King's College London, London, UK
  Marcelo Tavares Oliveira, DDS, MS, PhD student, Department of Restorative Dentistry; Piracicaba Dental School, UNICAMP, Piracicaba, Brazil
  Marcelo Giannini, DDS, MS, PhD, Department of Restorative Dentistry, Piracicaba Dental School, UNICAMP, Piracicaba, Brazil
  Giselle Maria Marchi, DDS, MS, PhD, Department of Restorative Dentistry, Piracicaba Dental School, UNICAMP, Piracicaba, Brazil
- Reprint request: Av Limeira, 901, 13.414-903, Piracicaba, Brazil; e-mail: dea.cavalcanti@uol.com.br

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The three metal primers yielded a significant increase in bond strength, regardless of the surface treatment and resin cement. Adhesive failures were the most prevalent. Air abrasion with Al2O3 particles and the application of metal primers increased bond strength to Y-TZP surfaces for both resin cements.

INTRODUCTION

The evolution of y INTRODUCTION tetragonal zirconia (Y-TZP) materials has introduced a new class of dental ceramics to the market.1 Although Y-TZP has been used as a ceramic biomaterial in medical applications since the late 1960s, its use in dentistry is relatively recent and occurred following advances in CAD-CAM (com- puter-aided design/manufacturing) technology.1-4 These high-strength materials offer a wide variety of clinical applications, such as orthodontic brackets, posts, implant abutments and frameworks for crowns and bridges.[5-6]

Zirconia materials differ from other high strength dental ceramics because of their distinct mechanism of stress-induced transformation toughening, which means that the material undergoes microstructural changes when submitted to stress.2,4 Y-TZP ceramics can actively resist crack propagation through transfor- mation from a tetragonal to a monoclinical phase at the tip of a crack, which is accompanied by a volume increase.2 The mechanical properties of Y-TZP materi- als, such as flexural and fracture resistance, are con- siderably higher than those of other dental ceramics.[4]
The flexural resistance of Y-TZP ceramics can reach val- ues from 700 to 1200 MPa.7-8 These values exceed the maximal occlusal loads during normal chewing.8 Y-TZP materials might also exhibit fracture resistance higher than 2,000 N, which is almost twice the value obtained for alumina-based materials and at least three times the value demonstrated by lithium disilicate-based ceramics.[9]

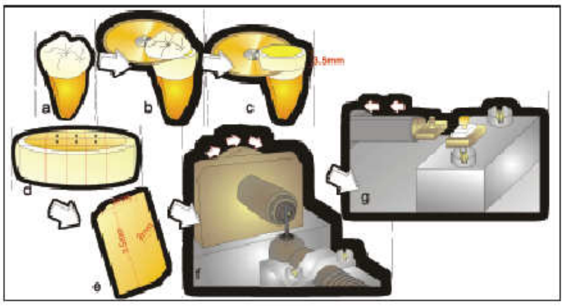
Although improved mechanical properties are impor- tant for the long-term performance of a ceramic materi- al, the clinical success of fixed ceramic prostheses seems to be strongly dependent on the cementation pro- cedure. There is a common thought that conventional methods of adhesive cementation, which include prior acid etching of the ceramic surface with hydrofluoric acid and further silanation, are not efficient for Y-TZP ceramics, because of their lack of silica and glass phase.1,3,10 Even though some Y-TZP manufacturers sug- gest the use of air abrasion or tribochemical coating prior to adhesive cementation, the effect of those sur- face treatments on the mechanical properties of Y-TZP materials is controversial, and both positive and nega- tive results have been described in the literature.[11-12]

Therefore, the most appropriate surface treatment for
Y-TZP ceramics still has to be determined. Some stud- ies have suggested the use of Er:YAG (erbium-doped yttrium aluminum garnet) laser to enhance the bond strength of adhesive materials to resin composites used for indirect restorations and lithia-based ceramics.[13-14]
However, the capacity of the Er:YAG laser to increase the roughness of Y-TZP ceramics for adhesive luting procedures has not been investigated.
There is some evidence that improved bonding to Y- TZP ceramics might be achieved using materials with a chemical affinity for metal oxides.1,5,10,15-16 Phosphate ester monomers, such as MDP (10-methacryloyloxyi- decyl-dihyidrogenphosphate), chemically react with zir- conium dioxide, promoting a water-resistant bond to densely sintered zirconia ceramic.10 Phosphate ester monomers can be present in both resin cements and adhesive systems. In addition, special adhesive monomers for improving bonding to metal alloys have been developed, and their effects on adhesive bonding to Y-TZP ceramics should be evaluated. These metal primers contain MDP and other monomers, including VBATDT (6-[4-vinylbenzyl-n-propyl]amino-1,3,5-tri- azine-2,4-dithione), MEPS (thiophosphoric methacrylate) and MTU-6 (6-methacryloyloxyhexyl-2-thiouracil-
5-carboxylate).[17-18]
Despite the increase in clinical use of zirconia ceram- ics, further evidence regarding the adhesive cementa- tion of Y-TZP restorations is necessary to establish the most reliable technique.19 There are still some possibil- ities for improving bonding to Y-TZP ceramics that need to be tested, including modern techniques for surface treatments and adhesive primer materials. Therefore, the current study evaluated the effect of different metal primers and surface treatment methods on the bond strength of two resin cements to a Y-TZP ceramic. The hypotheses tested were that surface treatments and metal primer application do not influence bond strengths to Y-TZP ceramic regardless of the type of resin cement used (MDP-based and Bis-GMA-based).

METHODS AND MATERIALS

Figure 1 presents the method of the current study.

Forty recently extracted non-carious human third molars were used to obtain cylindrical dentin specimens. After extraction, the teeth were gathered after approval by the Commission for Ethics of Piracicaba Dental School (#108/2006) and stored in 0.1% thymol solution for less than six months. Dentin slices 3.5-mm thick were obtained by removing the root portion and the occlusal enamel of each tooth. The dentin slices were polished with silicon carbide papers under water to remove remnants of enamel over the dentin surface and to standardize the smear layer. The slices were sectioned in the “x” and “y” axis, which resulted in six



sticks with a 2 x 2 x 3.5-mm dimension. The sticks were placed in a specimen-former device and trimmed with fine diamond burs (FG507, Kerr Diamond Blu White Burs, Kerr Corporation, Orange, CA, USA) in a high-speed handpiece with constant water-cooling. After trimming, the fragments had a cylindrical extremity 1.3 mm high and 0.8 mm in diameter. Two- hundred and forty dentin specimens were obtained in this manner and stored in distilled water at 37°C.

Two-hundred and forty plates with a dimension of 5 x 3 x 0.75 mm were obtained from a 94% ZrO_2 stabilized by 5% Y_2O_3 ceramic (Cercon Smart Ceramics, Degudent, Hanau, Germany). The ceramic plates and the trimmed dentin specimens were randomly allocated into 24 groups (n=10) according to the combination of surface treatment, metal primer and resin cement.

Three groups with 80 Y-TZP plates were submitted to one of the following surface treatments: none (control), air abrasion or laser irradiation. In the air-abraded and laser-irradiated groups, the superficial area to be further treated (1.76 mm^2) was delimitated with adhesive tape. Surface treatments were performed as follow:

- *Surface treatment control*: Specimens were only ultrasonically cleaned with 96% isopropanol for three minutes.
- *Air abrasion*: Air abrasion was performed with 53 µm aluminum oxide particles (Aquacut, Medivance Instruments Ltd, London, UK) at a 2.5 bar pressure for 15 seconds at a distance of 10 mm perpendicular to the surface. The adhesive tape was then removed and the plates were ultrasonically cleaned with 96% iso- propanol for three minutes.
- *Er:YAG laser irradiation*: The surfaces were coated with graphite prior to laser irradiation to increase energy absorption. The laser equipment used was an Er:YAG laser (OPUS 20 Er:YAG/CO_2 Dental Laser Surgical System, Sharplan Medical Systems, Yokneam, Israel) emitting a 2.94 µm wavelength. A 1,000-µm-diameter straight-type contact probe was used perpendicular to the surface. The surfaces were irradiated for five seconds using a fine water spray. The pulse repetition rate was set at 10 pps and the energy intensity was set at 200 mJ. After irradiation, the adhesive tape was removed and the surfaces were ultrasonically cleaned in 96% isopropanol for three minutes.

The 80 ceramic plates from each surface treatment group were divided into four subgroups (n=20): no coating (control), Alloy Primer (VBATDT/MDP-based [Kuraray Medical Inc, Okayama, Japan]), Metal Primer II (MEPS-based [GC Corporation, Tokyo, Japan]) or Metaltite (MTU-6-based [Tokuyama Dental Corporation, Tokyo, Japan]). The respective metal primer was applied in a thin coat and left undisturbed for 60 seconds.

Following the respective surface treatment and metal primer application, the 20 plates were divided into two groups (n=10) according to the resin cement used: MDP-based resin cement (Panavia F2.0, Kuraray Medical Inc) or Bis-GMA-based resin cement (Calibra, Dentsply Caulk, Milford, DE, USA). The dentin specimens were cemented onto the ceramic plates with the following recommendations.

- *Panavia F2.0*: Equal amounts of adhesive system solutions A&B (ED Primer II) were mixed and applied to the dentin cylinder. After 30 seconds, the adhesive layer was gently air dried. Equal amounts of paste A&B of the resin cement were dispensed and mixed for 20 seconds. This mixture was applied to the dentin surface, which was seated onto the ceramic plate. Excess cement was removed with a dental explorer. The margins were light cured for 20 seconds per surface, and oxygen-blocking gel (Oxyguard II) was applied for three minutes, then washed with air-water spray.
- *Calibra*: The dentin surface was etched with 35% phosphoric acid for 15 seconds, rinsed with water for the same amount of time and dried with absorbent papers. One drop of the adhesive system (Prime & Bond NT, Dentsply Caulk) and another of the self-cure activator were placed into a clean plastic mixing well. The contents were mixed for two seconds with a brush tip. The mixed adhesive/activator was applied to thoroughly wet the dentin surface. After 20 seconds, the excess solvent was removed by gently air drying for five seconds. The mixed adhesive/activator was light cured for 10 seconds.

A single coat of mixed adhesive/activator was applied to the ceramic surface and air dried for five seconds. Equal amounts of the base and catalyst paste of the resin cement (regular viscosity) were mixed for 20 seconds. A uniform thin layer of cement was applied onto the dentin surface and the cylinder was seated onto the ceramic plate. Gross excess cement was removed from the marginal areas using the dental explorer. All marginal areas were light cured for 20 seconds from each direction, for a total of 80 seconds of light expo- sure.

During the restorative procedures, the light output of the light-curing unit (Optilux VCL 401, Demetron Research Co, Danbury, CT, USA) was measured with a radiometer (Demetron Research Co) and was greater than 660mW/cm^2. The specimens were stored in distilled water at 37°C for 24 hours. Then, excess resin cement and adhesive were removed using razor blades under an optical microscope (45x, SDZ-PL, Kyowa Optical Co, Tokyo, Japan).

Each ceramic plate with its dentin cylinder was fixed to a microshear device adapted to a miniature load- testing machine (SMAC LAL95, SMAC Europe, Horshan, Sussek, UK) with cyanoacrylate glue (SuperGlue Loctite, Henkel Loctite, Hertfordshire, UK). A thin wire (0.2-mm thick) was looped around the dentin cylindrical extremity, and the shear force was applied at a crosshead speed of 1 mm/minute until debonding. KgF values were converted in MPa. After debonding, the fractured surfaces were evaluated with an optical microscope (100x magnification) to classify the failure modes into one of the following categories:

(A) adhesive failure at the interface between the ceramic and resin-luting agent or between the resin- luting agent and the dentin interface; (C) cohesive failure within the ceramic, within the resin-luting agent or within the dentin only and (M) adhesive and cohesive failure at the same site or a mixed failure.

Bond strength data were statistically analyzed by three-way ANOVA, with the main factors resin cement, surface treatment and metal primer. All possible inter- actions were included in the model. Multiple pairwise comparisons were done with the Tukey test. Statistical analysis was carried out in SAS 9.1 (SAS Institute, Cary, NC, USA) with a significance level of 5%.

Six additional Y-TZP plates were examined using scanning electron microscopy to evaluate changes in the ceramic topography after the surface treatments. The ceramic plates (n=2 for each surface treatment group) were treated and cleaned as described previously. They were then placed in a metallic stub, keeping the treated surface face-up. The surfaces were gold- sputter coated (E5100, Polaron Equipment Ltd, Hertfordshire, UK) and viewed in a scanning electron microscope (SEM/Hitachi S-3500N, Hitachi High- Technologies, Tokyo, Japan). The microscope operated at an accelerating voltage of 15kV, with a working distance of between 15 and 27 mm.

RESULTS

Table 1 shows the bond strength results. A significant statistical interaction between the resin cements and surface treatments was detected (p=0.004). Therefore, the association between these two main factors was similar in the four levels of the factor metal primer. A significant difference was also noted in the factor metal primer (p<0.01), and the three solutions increased the bond strength to a similar extent, regardless of the resin cement and surface treatment.

In the surface treated groups (air abrasion and Er:YAG laser irradiation), the Bis-GMA-based resin cement had higher bond strength than the MDP-based cement. Both materials presented similar bond strengths when no surface treatment was used. With the groups cemented with the MDP-based material, air abrasion resulted in significantly higher bond strengths, while laser irradiation and the absence of surface treatment presented similar results. In the groups luted with the Bis-GMA-based material, there was a significant difference among all surface treatments; air abrasion promoted the highest mean bond strength followed by Er:YAG laser irradiation and no surface treatment.

Table 2 describes the distribution of failure modes in the groups cemented with MDP-based and Bis-GMA- based resin cements, respectively. Adhesive failures were most prevalent in the 24 experimental groups, with an average of 78% adhesive failure between the ceramic and resin-luting agent or between the resin- luting agent and dentin interface and 22% mixed failures. No cohesive failure of the substrates (ceramic, resin cement or dentin) was observed.

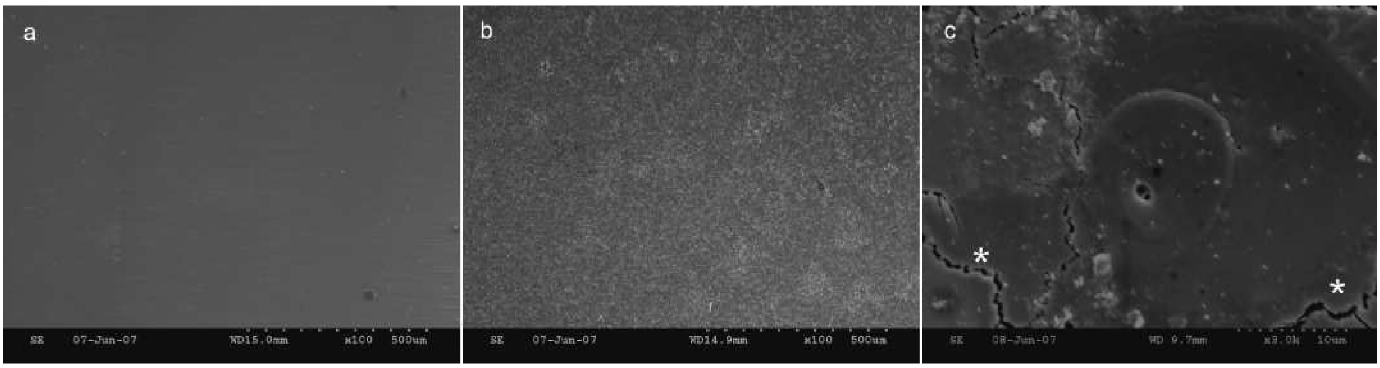


Figure 2: SEM images (original magnification 500x) of ceramic surfaces. Figure 2a: Y-TZP surface untreated (control; Figure 2b: Y-TZP surface treated air-abrasion with 53 µm Al2O3 particles; Figure 2c: Y-TZP surface irradiated with the Er:YAG laser, asterisks(*) indicate cracks on the surfaces.

Table 1: *Mean (standard-deviation) of the Microshear Bond Strength in MPa*

Metal Primer	Resin Cement	Surface Treatment: None	Surface Treatment: Air Abrasion	Surface Treatment: Er:YAG Laser	
None	Panavia F2.0	17.0 (3.5) Ab n=10	22.3 (5.2) Ba n=10	15.8 (3.5) Bb n=8	♣
	Calibra	16.6 (2.1) Ac n=9	24.3 (3.0) Aa n=7	3.0 (4.1) Ab n=8	
Alloy Primer	Panavia F2.0	20.4 (4.6) Ab n=9	24.2 (2.5) Ba n=8	19.6 (1.5) Bb n=10	♦
	Calibra	21.0 (3.3) Ac n=9	26.6 (2.4) Aa n=10	20.7 (6.4) Ab n=7	
Metal Primer II	Panavia F2.0	21.8 (3.0) Ab n=9	26.1 (3.9) Ba n=9	19.8 (3.2) Bb n=9	♦
	Calibra	21.60 (3.67) Ac n=8	27.99 (4.48) Aa n=9	22.72 (5.28) Ab n=8	
Metaltite	Panavia F2.0	20.6 (5.2) Ab n=7	24.3 (3.9) Ba n=9	19.3 (3.2) Bb n=9	♦
	Calibra	20.7 (3.4) Ac n=7	26.5 (3.5) Aa n=8	23.0 (3.0) Ab n=8	

Coefficient of variation = 15.75%

Same letters are not statistically different (three-way ANOVA/Tukey test, α=0.05). Upper case letters compare resin cements within surface treatment/metal primer. Lower case letters compare surface treatments within resin cement/metal primer. Symbols (♣ ♦) represent differences between metal primers.

Table 2: *Percentage of the Failure Modes in Each Experimental Group*

Resin Cement	Metal Primer	Surface Treatment					
		None		Air Abrasion		Er:YAG Laser	
		Adhesive	Mixed	Adhesive	Mixed	Adhesive	Mixed
Panavia F2.0	None	70	30	71	29	78	22
	Alloy Primer	75	25	75	25	71	29
	Metal Primer II	70	30	67	33	75	25
	Metaltite	78	22	70	30	86	14
Calibra	None	80	20	100	0	100	0
	Alloy Primer	60	40	75	25	100	0
	Metal Primer II	75	25	86	14	88	13
	Metaltite	70	30	83	17	71	29

SEM images show morphologic differences among the Y-TZP plates after surface treatments (Figure 2). Air abrasion with 50 µm Al2O3 particles (Figure 2b) created a rougher surface compared with the control surface and Er:YAG laser irradiation (Figures 2a and 2c). Er:YAG laser irradiation originated a smooth surface, with some perceivable cracks (Figure 2c/asterisks).

DISCUSSION

Previous studies ha DISCUSSION he bond strength of adhesive restorative materials to Y-TZP ceramics. Shear and tensile bond strength tests were the methods most frequently used in those investigations.1,3,5-6,10,15-16,19-21 In both methods, the specimens had only one adhesive interface to be tested, that is, between the ceramic and resin-based materials.

However, in the clinical situation, both interfaces between the ceramic and adhesive restorative material and between the adhesive restorative material and tooth structure are present.

Thus, the performance of the complex tooth structure—restorative material—Y- TZP ceramic unit—must be investigated.

During preliminary investigations for the current study, Y-TZP plates were luted to dentin surfaces and the specimens were sectioned for the microtensile bond strength test. The incidence of premature failures during sectioning was very high (almost 100%), yielding unreliable results. The long period necessary to section the densely sintered Y-TZP ceramics seems to weaken the adhesive interface, resulting in the premature failures. To overcome this limitation and to test both interfaces, a modification of the shear bond strength test was per- formed in the current study. The microshear test was conducted using cylindrically-trimmed dentin specimens luted onto Y-TZP plates (Figure 1), instead of the conventional micro-shear method.[22]

The surface treatments investigated in the current study resulted in significantly different bond-strengths.

Moreover, SEM images demonstrated considerable qualitative differences in the surface topography of Y-TZP plates after the surface treatments. Images showed that air abrasion appears to be a more efficient method to modify zirconia surfaces compared with laser irradiation, which uses the parameters set for this study.

This finding can be directly related to bond strength results, which showed that both resin cements yielded higher bond strengths after air abrasion.

Some investigations also indicated that superior bonding to zirconia is obtained when surfaces are air abraded.5-6 Air-abraded surfaces might present an increased surface area, which favors wettability. However, some authors have stated that the micro- porosities created by surface treatments may act as crack initiators, weakening ceramic materials.[12,20]

Thus, the effect of those alterations on the durability of Y-TZP restorations should be investigated in long-term clinical trials to determine whether the higher retention of air-abraded surfaces compensate for the changes in mechanical properties.

In the current study, irradiation of Y-TZP surfaces with Er:YAG laser was proposed as a surface treatment method. The Er:YAG laser has the ability to remove particles by micro explosions and by vaporization, a process called ablation. During laser treatment, local temperature changes due to heating and cooling phases create internal tensions that can damage the material.14 The mechanical properties of Y-TZP ceramics can be negatively affected by changes in temperature, which can induce phase transformation.[11]

Therefore, in the current study, a lower power setting for the Er:YAG laser was selected in accordance with the results of a preliminary investigation (200mJ) and the surfaces were irradiated with constant water cooling. Bond strength results indicated that laser irradiation was not as effective in improving bond strength as air abrasion for both resin cements. When the MDP- based resin cement was used, laser treated and untreated surfaces presented similar results. The manufacturer of the MDP-based material does not recommend the application of a layer adhesive system on the ceramic surface, which is in contrast to the instructions of the Bis-GMA-based resin cement. Since the MDP-based resin cement was applied directly to the laser irradiated ceramic, both the slight surface modification provided by the Er:YAG laser and the lack of contact between the resin cement and surface might have contributed to bond strengths that were similar to the untreated surfaces. On the other hand, when the Bis-GMA-based material was used, the bond strengths of the laser irradiated surfaces were superior to those of the untreated surfaces, thus, the better wettability of the adhesive system applied to the Y-TZP surface prior to the resin cement might have compensated for the limited surface modification promoted by the laser irradiation.

Metal primers were developed as an alternative approach to promote a durable bond to noble metal alloys.18,23 The VBATDT monomer, a thionethiol tautomer, was the first product introduced as a coupling agent between methacrylate-based monomers and noble metal alloys.[24]

The coupling mechanism of this monomer has been assigned to the transformation of thione to thiol groups on the noble metal surface, subsequently, primary bond formation and to the copolymerization of vinyl groups with the methacrylate- based resin monomer.23

The metal primers investigated in the current study are vinyl-thione coupling agents with different functional monomers. In addition to their different compositions, the bond strength results indicated a similar performance for the three systems, thus significantly strengthening the bond to the Y-TZP ceramic for both resin cements. This finding might suggest that vinylthione coupling agents also present a chemical affinity to zirconia surfaces. Previously, it was stated that the application of a MDP-containing bonding/silane coupling agent is the key factor for a reliable resin bond to a Y-TZP ceramic and is not influenced by the resin-luting agent.3 The bonding/silane agent improves the wettability of the air-abraded zirconia surface. Although a different ceramic material was investigated in the current study, the effect of the metal primers might have been similar, which is increasing the surface wettability of resin cements.

In contrast with the results of some previous studies, which have shown that the chemical affinity between MDP-based material and Y-TZP ceramic creates a strong bond, which is able to resist thermal aging and water storage,1,5,10 in the current study, the MDP-based resin cement did not present higher bond strength to Y- TZP surfaces compared with the Bis-GMA-based material. Both cements showed similar results only on untreated surfaces, while, in the air-abraded and laser- irradiated surfaces, the Bis-GMA-based resin cement showed significantly stronger bonding. One of the components of the adhesive system used, together with the Bis-GMA based resin cement, is a monophosphate monomer (PENTA). The chemical bond between the phosphate groups and zirconium dioxide was previous- ly suggested1,5,15 and might be a reason for the comparable bond between both resin cements on untreated surfaces. Nevertheless, other investigations indicated that the bonding of Bis-GMA composites to zirconia is not resistant to long-term storage and thermal aging.1,10 In the current study, only the immediate bond strength (measured 24 hours after the polymerization of the resin cements) was tested. Since a water expo- sure period of only 24 hours is insufficient to permit diffusion of water into the adhesive interface, it should be considered that different results might have been found if the specimens were submitted to an aging protocol. Therefore, further in vitro studies with longer storage periods should be performed to determine the long-term durability of the bond.

The failure mode results indicated that, regardless of the experimental group, most failures of the complex tooth structure—resin cement—Y-TZP ceramic—were adhesive, which left the zirconia plates free of remnants of adhesive materials. This finding might suggest that, even when higher bond strength to zirconia is obtained, this bond is not as strong as the adhesion between dentin and restorative material.

CONCLUSSIONS

The hypotheses tested rejected, since surface treatments and metal primer application have significantly influenced the bond strength to a Y-TZP ceramic.

It could be concluded that metal primers and air abrasion with 50 µm Al2O3 particles can have a synergic effect on dentin bonding to Y-TZP ceramics. Bis-GMA based resin cement presented a stronger immediate bond on treated surfaces than MDP-based material.

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