Study of the effectiveness of primers, plasma and their combination on bond strength of a resin cement to zirconia

Beatriz Curvello de Mendonça[1]
William Matthew Negreiros[2]
Gláucia Maria Bovi Ambrosano[3]
Marcelo Giannini[4]

1) Universidade de Campinas, Faculdade de Odontologia de Piracicaba (Piracicaba/SP, Brazil).
2) Universidade de Campinas, Programa de Pós-graduação em Materiais Dentários (Piracicaba/SP, Brazil).
3) Universidade de Campinas, Departamento de Odontologia Social, Biostatistics Division (Piracicaba/SP, Brazil).
4) Universidade de Campinas, Departamento de Odontologia Restauradora, Operative Dentistry Division (Piracicaba/SP, Brazil).

Abstract: The aim of this study was to evaluate the shear bond strength of a dual-cure resin cement (Panavia F, Kuraray) to two zirconia ceramics (Lava, 3M ESPE and Katana, Kuraray Noritake) with prior application a coupling agent (Z-Prime Plus, Bisco or Monobond Plus, Ivoclar Vivadent), argon plasma or a combination of coupling agent and plasma. Zirconia plates with 13-mm length, 5-mm width, and 1-mm thickness approximately were used. The zirconia surfaces were abraded with Al_2O_3 sandpaper (#600-grit) and treated in accordance with each experimental group (untreated, coupling agent and/or plasma). Cylindrical-shaped specimens were prepared on treated zirconia surface using silicone matrix with internal dimensions of 1.4-mm diameter and 1-mm height. The shear test was carried out in a testing machine after 24 hours and one year of water storage of specimens. Bond strength data values were expressed in MPa and values, analyzed by three-way ANOVA and Tukey's test (5%). Although the non-use of coupling agents and plasma showed higher bond strength values, the adhesion reduced after one year. The use of Z-Prime Plus with or without plasma application did not reduce the bond strength after one year, but the bond strengths were lower than those obtained with the use of resin cement alone without previous treatment of zirconia. Plasma did not increase the bond strength to zirconia. Thus, for the adhesion to zirconia, the use of resin cement only was enough to obtain adequate bond strength results. **Keywords:** Zirconium. Plasma gases. Shear strength.

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Contact address: Marcelo Giannini. Av. Limeira, 901, Areião – Piracicaba/SP - CEP: 13.414-903

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INTRODUCTION

The increase in the demand for indirect restorations made in pure ceramics has boosted the development of zirconia ceramics (Y-TZP), which have gained great prominence in Dentistry due to its favorable mechanical and aesthetic properties and because they offer a wide variety of clinical applications.[1] However, its molecular structure does not allow appropriate adhesion of resin cements[2] without a surface treatment.

Obtaining reliable adhesion to ceramics requires surface treatments based on physical retention, by sandblasting, and chemical bonding of the ceramic/substrate, through silanes and/or primers. Many studies have described that sandblasting with aluminum oxide particles can increase the roughness of the zirconia, which in turn increases its surface area and facilitates the micromechanical bonding of the resin to the zirconia surface.[3,4] After sandblasting, it is interesting to create some type of chemical bonding by using primers with phosphate monomers, such as 10-MDP (10-metacryloyloxydecyl dihydrogenphosphate). These monomers, like silanes, are bifunctional molecules that react at one end to zirconia oxides and, on the other, have groups that copolymerize with the resin matrix of the cements.[5]

When air abrasion with aluminum oxide particles is associated with a phosphate ester (10-MDP) monomer, adequate bond strength results have been obtained, especially when stored for a long period in water.[6] Air abrasion is also indicated to clean and activate the surface of the zirconia,[6] increasing its surface energy and consequently promoting a better wetting of the primers.[8] Subsequently, it is followed by the use of resin cement. Thus, for efficient adhesion, it is necessary to combine the micromechanical retention and the chemical bonding of the adhesive material (adhesive and resin cement) to the zirconia ceramic.[9]

Although these methods have satisfactory results in relation to Y-TZP[10] zirconia, abrasion with aluminum oxide particles can create defects in zirconia, making its longevity less reliable.[11] Thus, avoiding sandblasting with aluminum oxide may be advantageous for the elimination of deleterious effects on zirconia.[12] Due to this problem, a possible alternative is the use of plasma generated from argon gas. Plasma treatment using argon, an inert gas, has been widely used to modify biomaterials surface. When electronically activated in a plasma state, it consists of several energetically and chemically reactive species, including high energy electrons, electronically excited neutrons, and free radicals. The reactive species produced by argon plasma can chemically modify the surface of the substrate without affecting the properties of the material body.[13] Thus, the application of the plasma increases the surface energy of the treated substrate, favoring the molecular chemical interactions and, consequently, has the capacity of promoting better adhesion results between zirconia and the resin cement.[14]

In view of the above, this study aimed at investigating a protocol that has not yet been described in the literature, which consists of applying argon plasma over the adhesive previously applied to the zirconia. The hypothesis is that the application of plasma to the zirconia surface, which is little reactive, could promote better chemical adhesion. Thus, this study aimed at evaluating the effect of primers and of argon gas plasma application on these primers as a potentiator of adhesion between zirconia and resin cement, by changing the zirconia surface energy and promoting better chemical interaction between zirconia and the primers.

MATERIAL AND METHODS

Material

Table 1 shows the materials used in this project. The experimental groups of this study were formed according to the materials (two zirconia ceramics: Lava and Katana), specimens storage time in water (24 hours and 1 year) and zirconia surface treatment (six types), which were:

- Application of resin cement without zirconia treatment (Control).
- Application of plasma to zirconia + resin cement.
- Application of Monobond Plus + resin cement.
- Application of Z-Prime Plus + resin cement.
- Application of Monobond Plus + plasma + resin cement.
- Application of Z-Prime Plus + plasma + resin cement.

Table 1: Composition and manufacturers of the materials tested in this study.

MATERIAL	COMPOSITION	MANUFACTURER
Katana (zirconia)	94.4% zirconium oxide, 5.4% yttrium (Y_2O_3), 0.2% other	Kuraray Noritake Dental Inc., Japan
LAVA (zirconia)	99% zirconium oxide	3M ESPE, USA
Monobond Plus (primer)	Alcoholic solution of silane methacrylate, phosphoric acid methacrylate and sulphide methacrylate	Ivoclar Vivadent, Liechtenstein
Z-Prime Plus (primer)	10-MDP (<10%); Ethanol (<80%)	Bisco Inc., USA
Panavia F (resin cement)	Paste A: 10-MDP; hydrophobic aromatic dimethacrylate; hydrophobic dimethacrylate; dimethacrylatealiphatic hydrophil; silanized colloidal silica; camphorquinone; initiators Paste B: hydrophobic dimethacrylate; hydrophobic aliphatic methacrylate; hydrophilic dimethacrylate; silanized barium glass; catalysts; accelerators; pigments	Kuraray Noritake Dental Inc., Japan

Preparation of specimens and application of resin cements

Sixty Katana plates (Kuraray Noritake) and sixty Lava plates (3M ESPE) with dimensions of 13 mm long, 5 mm wide, and 1 mm thick were used. These plates were obtained from ingots of industrial manufacturing, sectioned with a diamond disk (Buehler) and sintered in the laboratory to reach the measurements necessary for the study, and included in epoxy resin. The specimens had their surfaces standardized with #600-grit sandpaper until a flat surface was obtained. Then the plates were sandblasted with aluminum oxide for 20 seconds, followed by cleaning in ultrasonic bath with distilled water for 5 minutes. The plates were randomly divided into 12 groups ($n = 10$) as previously described (two zirconia and six treatments).

The next step in the specimen production was the preparation of the zirconia plates for the application of the resin cement. Two cylindrical silicon matrices were positioned on treated or untreated (control) surfaces. The resin cement was then handled according to the manufacturer's recommendations and applied inside the matrices with a # 5 exploratory probe (Hu-Friedy). The resin cement was light-activated for 20 seconds with a Valo light-curing unit (1000 mW/cm^2, Ultradent Products Inc.) and the matrices were removed, exposing the two resin cement cylinders (1.4-mm diameter, 1-mm height), with a bonding area of 1.53 mm^2 on the zirconia surface. The zirconia samples with the two cylinders of resin cement attached to its surface were stored in deionized water at 37°C for 24 hours. One cement cylinder was tested after this time and the other, after one year of storage in water, which was replaced every 15 days.

Shear testing

Each resin cement cylinder was positioned on the test device attached to the universal test machine (EZ-Test, Shimadzu). The loading was applied at the base of the resin cement cylinder with a steel wire (0.7-mm diameter) at a 0.5 mm/min speed, until failure.

The value of each specimen and the bond strength data in kilogram-force (KgF) were converted into MegaPascal (MPa), tabulated and submitted to statistical analysis. The analysis of the results for bond strength values was conducted using the three-way analysis of variance (ANOVA), with factorial design of 2 x 6 x 2 (zirconia X treatment X evaluation time) using the Minitab Statistical software (Minitab Inc., State College). With the identification of at least one significant statistical difference between the groups, the Tukey test was applied ($\alpha = 0.05$).

RESULTS

Table 2 shows data for the bond strength of resin cement to zirconia plates, according to each experimental group. There was no statistical difference in the bond strength means when both zirconias (Lava and Katana) were compared, regardless of the use of argon plasma and the evaluation time ($p < 0.05$). Regarding the test after 24 hours, the Monobond Plus primer produced higher bond strength for Katana zirconia, when compared to the use of Z-Prime Plus without the use of plasma ($p > 0.05$), but did not differ from the control group ($p < 0.05$). Plasma application in combination with the use of the Monobond Plus primer decreased the bond strength for both zirconias ($p > 0.05$). The application of only the argon gas plasma (without primers) in the zirconia did not show statistical difference in comparison with the control group ($p < 0.05$).

After one year of storage of the specimens, the Z-Prime Plus Primer produced lower bond strength for Lava zirconia, when compared to the use of Monobond Plus ($p > 0.05$). The use of argon

Table 2: Mean of bond strength (standard deviation) between resin cement and zirconia as a function of plasma used, treatments and evaluation times (in MPa).

TIME	PLASMA	ZIRCONIA	WITHOUT PRIMER	MONOBOND PLUS	Z-PRIME
24 hours	Without	Lava	$^{\$}$24.2 (6.3) Aa	$^{\$*}$22.2 (11.9) Aa	3.7 (2.4) Ba
		Katana	$^{\$}$16.9 (7.2) ABa	$^{\$*}$21.0 (10.7) Aa	9.5 (6.3) Ba
	With	Lava	$^{\$}$15.0 (10.3) Aa	6.7 (5.5) Aa	5.2 (4.9) Aa
		Katana	$^{\$}$16.4 (10.7) Aa	4.9 (4.5) Ba	6.2 (3.2) ABa
1 year	Without	Lava	8.6 (6.2) ABa	*15.2 (8.9) Aa	2.8 (3.1) Ba
		Katana	10.3 (7.6) Aa	6.0 (7.5) Aa	6.4 (5.7) Aa
	With	Lava	6.5 (5.6) Aa	2.5 (2.5) Aa	2.8 (2.7) Aa
		Katana	8.6 (9.0) Aa	3.3 (3.0) Aa	4.4 (2.9) Aa

Means followed by distinct letters (upper case – row, comparing treatments for the same zirconia, within the same evaluation time, with or without plasma application) (lower case – column, comparing the zirconia within each time, with or without plasma) differ from each other ($p \leq 0.05$). * Differ from the plasma group in the same conditions of treatments ($p < 0.05$). $\$$ Differ from the time 24 hours in the same conditions of treatments ($p < 0.05$).

gas plasma over primers previously applied to zirconia did not show statistical difference in comparison with the control group ($p<0.05$).

DISCUSSION

This study used a resin cement containing the 10-MDP monomer (Panavia F), which is able to produce adequate adhesion to zirconia, even in the absence of sandblasting, plasma application or primers. For this reason, little influence of these treatments was identified. The chemical bonding occurs between the phosphate grouping of the cement and the oxides of the restorative material.[5]

According to previous studies, it is recommended to pretreat the zirconia surface with primers containing 10-MDP, such as Monobond Plus and Z-Prime Plus, as they increase the bond strength of the resin cement to the prosthetic restorative material. The use of primers and cements both containing the 10-MDP monomer in its composition supposedly, according to the literature, should further increase the adhesion of the resin cement to zirconia ceramics.[15]

In addition, in the literature there are studies that have proved that plasma actually influences the adhesion of the resin cement to the zirconia, and that the aluminum oxide sandblasting or the treatment with primers do not enhance this adhesion when the plasma is used previously. On the other hand, when the plasma is not applied, these other surface treatments are important to achieving adequate adhesion (sandblasting and primer application of 10-MDP).[14]

In this study, plasma application was not able to increase bond strength, neither with sandblasting nor with prior application of primers. The use of plasma tends to reduce the angle of contact and thus increase the adhesion of materials applied to the plasma-treated surface. Since zirconia is partially stabilized by yttrium (Y-TZP), it contains important metal oxides for the plasma to act favorably, resulting in quantitative/qualitative changes in the chemical bonds with those metal oxides and the 10-MDP monomer.[13] However, in this study, the protocol used did not produce favorable results compared to the other types of performed treatments.

Sandblasting with aluminum oxide has been the most appropriate procedure to obtain adequate adhesion in cases of zirconia prosthesis cementation, and it was performed in all samples of this study. Sandblasted surface presents micro-roughness that retains the resin-based material and promotes a better micro-mechanical interaction between the resin cement and the zirconia. Considering that professionals are more familiar and have easier access to sandblasting with aluminum oxide abrasive particles, this protocol has been widely used for the surface treatment of acid-resistant ceramics, such as zirconia. In addition, aluminum oxide sandblasting is also effective for cleaning the ceramic surface contaminated with saliva and other residues.[16]

Some studies have shown that the same surface energy found on zirconia surfaces sandblasted with aluminum oxide was found in zirconia treated with plasma application for 10 seconds.[14] Thus, the application of plasma increases the adhesion to zirconia in the same way as the sandblasting, but does not alter its crystalline structure, as the latter does.[17] The combination of these two types of treatments together, and their advantages over adhesion should be further studied.

A study showed that the combination of silicatization, silanization and cementation with materials containing the 10-MDP monomer provides high values of bond strength for “acid-resistant ceramics”, so it is not necessary to apply plasma or primers to increase these values. In addition, the sandblasting of the part tends to improve even more the results, since it creates micro-roughnesses that facilitate the wetting of the surface.[18]

Thus, although the bond strength results reported in the literature are not directly comparable —due to differences in the experimental methodologies used—, it can be concluded that the previous zirconia treatment with aluminum oxide blasting presented favorable results to zirconia bond strength to the resin cement, and that the protocols chosen for use with the argon gas plasma and primers (Monobond Plus and Z-Prime Plus) were not suitable for improving bond strength. Therefore, it is necessary to carry out more detailed studies, to define the most suitable protocol to be used in the cementation of zirconia parts.

CONCLUSION

The present results suggest that:

- The bond strength produced by the application of the resin cement directly into the zirconia without the use of primers, with or without argon gas plasma, presented reduction after one year, for both zirconia.
- Plasma application only, without primer application, did not increase the bond strength of resin cement to zirconia (Katana and Lava), for both tests: after 24 hour and after one year of storage.
- The combination of primer and argon gas plasma application reduced the bond strength.
- The use of Monobond Plus without plasma application was the primer that promoted higher bond strength of resin cement to zirconia, in the two evaluation times.

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References

1. Wolfart M, Lehmann F, Wolfart S, Kern M. Durability of the resin bond strength to zirconia ceramic after using different surface conditioning methods. Dent Mater. 2007 Jan;23(1):45-50.
2. Piascik JR, Swift EJ, Thompson JY, Grego S, Stoner BR. Surface modification for enhanced silanation of zirconia ceramics. Dent Mater. 2009 Sept;25(9):1116-21.
3. Yang B, Wolfart S, Scharnberg M, Ludwig K, Adelung R, Kern M. Influence of contamination on zirconia ceramic bonding. J Dent Res. 2007 Aug;86(8):749-53.
4. Yang B, Lange-Jansen HC, Scharnberg M, Wolfart S, Ludwig K, Adelung R, et al. Influence of saliva contamination on zirconia ceramic bonding. Dent Mater. 2008 Apr;24(4):508-13.
5. Andreiuolo R, Gonçalves SA, Dias KRHC. A zircônia na Odontologia restauradora. Rev Bras Odontol. 2011 Jan-June;68(1):49-53.
6. Tanaka R, Fujishima A, Shibata Y, Manabe A, Miyazaki T. Cooperation of phosphate monomer and silica modification on zirconia. J Dent Res. 2008 July;87(7):666-70.
7. Valandro LF, Della Bona A, Antonio Bottino M, Neisser MP. The effect of ceramic surface treatment on bonding to densely sintered alumina ceramic. J Prosthet Dent. 2005 Mar;93(3):253-9.
8. Ozcan M, Nijhuis H, Valandro LF. Effect of various surface conditioning methods on the adhesion of dual-cure resin cement with MDP functional monomer to zirconia after thermal aging. Dent Mater J. 2008 Feb;27(1):99-104.
9. Yoshida K, Yamashita M, Atsuta M. Zirconate coupling agent for bonding resin luting cement to pure zirconium. Am J Dent. 2004 Aug;17(4):249-52.
10. Wegner SM, Wolfart S, Kern M. Long-term resin bond strength to zirconia ceramic. J Adhes Dent. 2000 Summer;2(2):139-47.
11. Zhang Y, Lawn BR, Malament KA, Van Thompson P, Rekow ED. Damage accumulation and fatigue life of particle-abraded ceramics. Int J Prosthodont. 2006 Sept-Oct;9(5):442-8.
12. Guess PC, Zhang Y, Kim JW, Rekow ED, Thompson VP. Damage and reliability of Y-TZP after cementation surface treatment. J Dent Res. 2010 Jun;89(6):592-6.
13. Colombo LMPC, Matafora FL, Moro AFV. Condicionamento de superfícies na Odontologia com plasma de argônio: uma revisão de literatura. Rev Bras Odontol. 2014 Jun;71(1):85-8.
14. Valverde GB, Coelho PG, Janal MN, Lorenzoni FC, Carvalho RM, Thompson VP, et al. Surface characterization and bonding of Y-TZP following non-thermal plasma treatment. J Dent. 2013 Jan;41(1):51-9.
15. Zhao L, Jian Y-T, Wang X-D, Zhao K. Bond strength of primer/cement systems to zirconia subjected to artificial aging. J Prosthet Dent. 2016 June;14:1-7.
16. Mosele JC, Borba M. Efeito do jateamento de partículas na resistência de união e comportamento mecânico de cerâmicas à base de zircônia: revisão. Cerâmica. 2014 Abr-Jun;60(354):179-86.
17. Ito Y, Okawa T, Fukumoto T, Tsurumi A, Tatsuta M, Fujii T, et al. Influence of atmospheric pressure low-temperature plasma treatment on the shear bond strength between zirconia and resin cement. J Prosthodont Res. 2016 Oct;60(4):289-93.
18. Soares EDS, Silva JVP, Neppelenbroek KH, Jorge JH, Urban VM. Tratamento de superficie de cerâmica pura para cimentação com cimentos resinosos. Rev Odontol UNESP. 2009;38(3):154-6.