Ceramic Repair: Influence of Chemical and Mechanical Surface Conditioning on Adhesion to Zirconia

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Abstract

Aims: To evaluate the influence of mechanical surface treatment and chemical primer application on the composite shear bond strength to zirconia.

Methods: Eighty blocks of Lava Frame Zirconia were randomly assigned to the experimental groups, according to the several possible combinations between mechanical (untreated/aluminum oxide sandblast) and chemical treatment (no primer/Monobond Plus/Experimental Zirkon-Primer/Z-Prime Plus) (n=10). Adhesive system and composite resin were then applied, and samples were thermal cycled (5ºC-55ºC, 500 cycles). Shear bond strength tests and failure mode analysis were performed. SBS data were analyzed by two-way ANOVA, followed by Student-Newman-Keuls post-hoc tests, and failure mode by Kruskal-Wallis and Mann-Whitney tests ($p<0.05$).

Results: Shear bond strength mean values ranged from 6.9 to 23.2 MPa. Surface treatment with aluminum oxide sandblasting allowed a statistically significant increase ($p<0.05$) in bond strength values. The group treated with Z-Prime Plus achieved ($p<0.05$) higher bond strength results than the other chemical treatments.

Conclusions: The surface mechanical treatment almost doubled the bond strength values. Z-Primer Plus have enhanced bond strength values of composite to zirconia.

Key Words: Zirconia, Shear Strength, Dental restoration repair, Primer, Air abrasion

Introduction

Zirconia has been increasingly used in dentistry due to its exceptional biocompatibility, mechanical properties, esthetic and the high demand for metal-free [1-3]. Although, it can be use as single restorative material, it is mainly used as a core material, veneered by a stratified feldspathic ceramic [4,5]. Zirconia core rarely fractures [6,7], but the veneering feldspathic ceramic has shown a high incidence of chipping and delamination exposing the underlying zirconia [8], when compared to conventional metal-ceramic restorations [9].

The location and extent of the fracture will determine either the restoration´s repair or replacement. The lower cost involved and the possibility of being performed in just one appointment, are some of the advantages of repair [10]. To perform ceramic restorations repair, some systems, using composite resins, were developed [11].

With minor differences in the literature, the protocol for bonding composite resins to feldspathic ceramic advocates the use of sandblasting, with aluminum oxide, or hydrofluoric acid conditioning to create micro-retentions, followed by silane application, to chemically interact with silica [11-13].

Since zirconia cannot be effectively conditioned with hydrofluoric acid and has no silica in its structure, the feldspathic ceramic adhesion protocol is not efficient [14]. New methods of surface conditioning have been proposed to increase bond strength between composite resins and zirconia, such as silica coating techniques and zirconia primers [14-21]. These new developed primers usually contain phosphate ester, carboxylic acid and other organic acidic monomers that may react with hydroxyl groups presented on zirconia surface, allowing the contact of the composite resin with zirconia [16,22-24].

Aims

The aims of this *in-vitro* study were to assess the effect of a mechanical surface treatment technique and three different priming agents on the shear bond strength (SBS) of a composite resin to zirconia. The null hypotheses to test were that 1) mechanical treatment with 50 μm Al_2O_3 does not increase the SBS of resin composite to zirconia, and 2) chemical treatment with zirconia primers does not increase the SBS of resin composite to zirconia.

Methods

The sample size (n=10) was estimated with a power analysis to provide statistical significance alpha=0.05 at an 80% power.

Eighty blocks of pre-sintered high-purity zirconium-oxide ceramic (Lava Frame Zirconia, 3 M ESPE, Seefeld, Germany) sized 12x12x6 mm were prepared with an Isomet 1000 precision saw (Buehler Ltd, Lake Bluff, Illinois, USA), and polished with silicon carbide paper strips grit-220, 400 and 600, under running water for 5 seconds.

After sintered, the zirconia blocks were randomly assigned to two surface treatment groups. One group was submitted to sandblasted with 50 μm aluminum oxide particles for 5 seconds at a pressure of 40 psi and a distance of 10 mm, and the other had no surface treatment. Specimens were then immersed and ultrasonically washed for 1 minute in distilled water.

Each group was further divided into four subgroups, according to the primer application: A) control group (no primer), B) Monobond Plus (Ivoclar Vivadent AG, Schaan, Liechtenstein), C) Z-Prime Plus (Bisco, Schaumburg, Illinois, USA), D) Experimental Zirkon-Primer #1043073 (Voco, Cuxhaven, Germany).

One layer of the selected primer was applied to a

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standardized 3 mm diameter area, left undisturbed for 60 seconds and air dried for 5 seconds.

The restorative procedure was the same in every specimen. First, the fluid resin from Adpter Scotchbond Multi-Purpose system (3M ESPE Dental Products, St. Paul, Minnesota, USA) was applied and light cured for 10 seconds. Then, 2 mm increments of restorative composite resin Tetric Evoceram (Ivoclar Vivadent AG, Schaan, Liechtenstein) were applied and light cured for 20 seconds each. Materials were light cured using an Ortholux LED Curing Light (3M Unitek, Puchheim, Germany), with 800 mW/cm^2.

After 24 hours storage period in an incubator at 37°C and 100% humidity, all specimens were thermal cycled (500 cycles at 5ºC and 55ºC), and included in a single plane lap shear bond strength device [25]. Tests were performed in a universal machine (Instron 4502, Instron Ltd., Bucks, England) with a crosshead speed of 0.5 mm/min and a load cell of 1 kN.

Failure mode was determined using a stereomicroscope (EMZ-8TR, Meiji Techno Co., Saitama, Japan) with a 20x magnification and classified, by two independent observers as: adhesive, when the failure occurred at the adhesive interface, or mixed, when the combination of cohesive in composite and adhesive failure occurred [17,26,27].

The results were statistically analyzed using IBM SPSS Statistics 20 (SPSS Inc., Chicago, Illinois, USA). After assessing normality and homoscedasticity with Kolmogorov-Smirnov and Levene's tests, shear bond strength data was analyzed using two-way ANOVA, followed by Student-Newman-Keuls post-hoc tests. Kruskal-Wallis and Mann-Whitney tests were used to analyze failure mode data. A statistical significance of 0.05 was set for both statistical analyses.

Results

Shear bond strength (SBS) values and failure mode distribution are presented in *Table* 1. The bond strength mean values ranged from 6.9 MPa, in the Experimental Zirkon-Primer group with no surface treatment, to 23.2 MPa, in the Z-Prime Plus sandblasted group.

Two-way ANOVA showed that both mechanical ($p< 0.001$) and chemical surface treatment ($p=0.001$) significantly influenced SBS values (*Table* 2). No interaction was detected between these two factors ($p=0.515$). Aluminum oxide sandblasting achieved higher SBS values than no mechanical surface treatment (*Figure* 1). Regarding the chemical treatments Z-Prime Plus achieved higher ($p<0.05$) SBS values than all the other groups (*Figure* 2). No statistical differences were found between the control, Monobond Plus and Experimental Zirkon-Primer #1043073.

Mann-Whitney test revealed a statistically significant ($p=0.002$) influence of the surface treatment on the failure mode. Sandblasting predominantly led to mixed mode, while no mechanical surface treatment resulted in mainly adhesive failures. Kruskal-Wallis test showed no statistical differences among the zirconia primers ($p=0.141$).

Discussion

The direct extrapolation of *in vitro* studies results to the clinical performance of restorative materials should be carefully made. However, achieving high bond strength values is important for the clinical success of any restoration [23,28].

As expected, and in agreement with previous reports, ceramic surface pre-treatment with aluminum oxide sandblasting yielded higher SBS values than those obtained in the non-sandblasted group [29-31]. Ceramic sandblasting increases surface roughness, area and energy, improving the adhesive forces achieved [15,29,30,32]. This improvement has also been justified by the cleaning effect and chemical activation of the surface [27]. Despite concerns that the impact of aluminum oxide particles on the ceramic surface may generate fracture lines that lead to a decrease in cohesive strength, some researchers report that a transformation from tetragonal to monoclinic phase on Yttrium stabilized tetragonal zirconia polycrystals due to sandblasting counteracts the effect of these flaws on the material strength [14,33].

Even though ultrasonic cleaning is not reproducible in a

Table 1. *Descriptive statistics of shear bond strength (SBS) and failure mode (n=10).*

Mechanical treatment	**Chemical treatment**	**SBS (MPa)**	**Failure mode [n (%)]**	
		Mean ± SD	Adhesive	Mix
No treatment	No primer	7.2 ± 3.5	7 (70%)	3 (30%)
	Monobond® Plus	7.7 ± 3.6	7 (70%)	3 (30%)
	Z-Prime™ Plus	11.6 ± 6.7	4 (40%)	6 (60%)
	Experimental Zirkon-Primer	6.9 ± 2.8	6 (60%)	4 (40%)
50 µm Al_2O_3 Sandblasting	No primer	13.7 ± 4.2	4 (40%)	6 (60%)
	Monobond® Plus	15.3 ± 6.4	4 (40%)	6 (60%)
	Z-Prime™ Plus	23.2 ± 4.1	1 (10%)	9 (90%)
	Experimental Zirkon-Primer	16.3 ± 10.7	1 (10%)	9 (90%)

Table 2. *Two-way ANOVA.*

Source	**df**	**Sum of squares**	**Mean square**	**F**	**p***
Mechanical treatment	1	1539.8	1539.8	45.9	<0.001
Chemical treatment	3	595.4	198.4	5.9	0.001
Mechanical * chemical treatment	3	77.5	25.8	0.8	0.515
Error	72	2416.9	33.6		
Total	80	17589.8			

*p<0.05 indicates statistically significant differences.

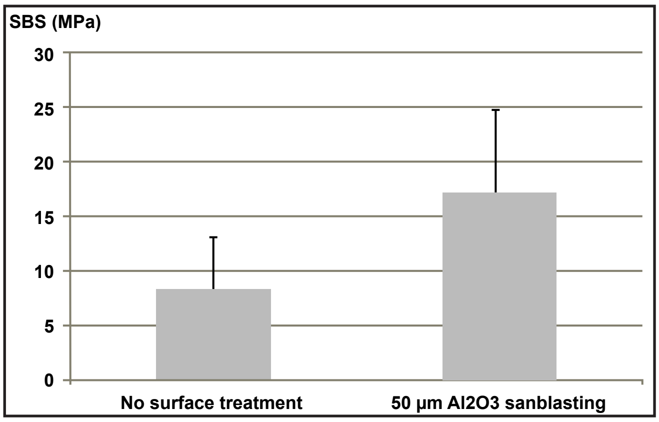

Figure 1. *Influence of mechanical surface treatment on shear bond strength between zirconia and composite resin – a significant difference was found between the two groups (p<0.001).*

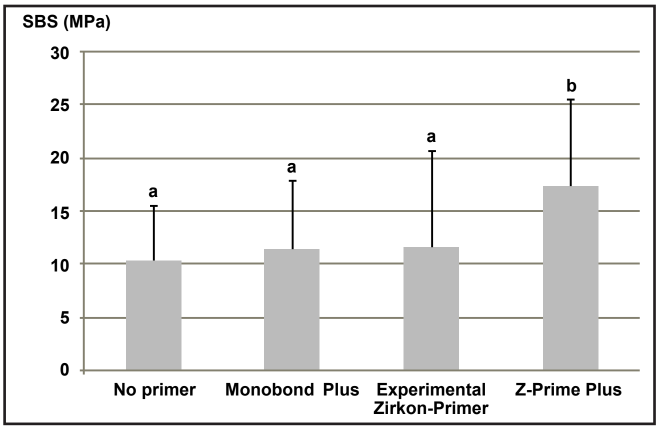

Figure 2. *Influence of chemical treatment on shear bond strength between zirconia and composite resin – means with similar superscript letters were not statistically different (p ≥ 0.05).*

clinical scenario, the method was used in this study, in order to guarantee a clean surface of the sample.

Thermal cycling was performed, according to the ISO 11405 specifications for testing adhesion to tooth structure. This procedure could be controversial and although it has been stated that 500 cycles do not stand for a sufficient amount of aging [34,35], all specimens were treated in the same way.

The use of zirconia primers has also been described in literature as an effective method to increase bond strength between zirconia and composite resins [16,36,37]. However, in the present study only Z-Prime Plus application resulted in higher SBS than the group without primer. Z-Prime Plus main components are organophosphate and carboxylic acid monomers. Organophosphates monomers have two functional groups: a phosphoric acid group that can bond to zirconia surface oxides and, usually, a methacrylate group to copolymerize with organic monomers of the composite [16]. Monobond Plus consists of an alcohol solution of silane methacrylate, phosphoric acid methacrylate and methacrylate sulphide. Although, this primer also is composed of phosphate-based monomers, the SBS results were lower than obtained with Z-Primer Plus. These differences can perhaps be explained by the fact that the specimens were subjected to thermocycling, since a previous study suggested that the bond to zirconia established by the Z-Primer Plus was more stable than the one promoted by the Monobond Plus, probably due to the carboxylic acid that would lead to a chemical attachment to zirconia [38]. As for the Experimental Zirkon-Primer, information regarding the chemical composition provided by the manufacturer is very scarce, which makes it difficult to speculate about why the SBS was not increased. In this particular case, the primer is sold as part of an adhesive system that also has an adhesive. The primer contains organic acids and silane. The composition of the adhesive is Bis-GMA, HEMA, TEGDMA, BHT and acetone. According to the manufacturer's instructions, this adhesive must be applied to the surface of zirconia previously prepared with the primer. However, the complete ceramic repair systems were not used as per manufacturer´s instructions since the aim of the present study was to determine the primer effects and compatibility with a fluid resin from a different adhesive system. Some lack of chemical compatibility between materials can justify the poor results achieved, or as may have happened with the Monobond Plus, the bond was not stable enough.

Regarding failure mode statistical differences were found only between the two mechanical surface treatments tested with higher number of mixed failures in the sandblasted group. This result matches with the higher mean SBS values observed in this group. As in other studies higher SBS values seem to be related to a predominance of mixed failure modes [20,39].

Techniques and materials used in this study should be tested in more demanding laboratory conditions to closer simulate clinical environment.

Conclusions

Within the limitations of the present study, both null hypotheses have to be rejected, as 1) sandblasting the zirconia surface with 50 μm Al_2O_3 particles, and 2) the chemical treatment with Z-Prime Plus increased the shear bond strength of the resin composite to zirconia.

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References

1. Zarone F, Russo S, Sorrentino R. From porcelain-fused-to-metal to zirconia: clinical and experimental considerations. *Dental Materials*. 2011; **27**: 83-96.
2. Scarano A, DiCarlo F, Quaranta M, Piattelli A. Bone response to zirconia ceramic implants: an experimental study in rabbits. *Journal of Oral Implantology*. 2003; **29**: 8-12.
3. Scarano A, Piattelli M, Caputi S, Favero G, Piattelli A. Bacterial adhesion on commercially pure titanium and zirconium oxide disks: an in vivo human study. *Journal of Periodontology*. 2004; **75**: 292–296.
4. Della Bona A, Kelly J. The clinical success of all-ceramic restorations. *Journal of the American Dental Association*. 2008; **139**: 8S-13S.

5. Saito A, Komine F, Blatz M, Matsumura H. A comparison of bond strength of layered veneering porcelains to zirconia and metal. *Journal of Prosthetic Dentistry*. 2010; **104**: 247-257.

6. Heintze SD, Rousson V. Survival of zirconia- and metal-supported fixed dental prostheses: a systematic review. *International Journal of Prosthodontics*. 2010; **23**: 493–502.

7. Triwatana P, Nagaviroj N, Tulapornchai C. Clinical performance and failures of zirconia-based fixed partial dentures: a review literature. *Journal of Advanced Prosthodontics*. 2012; **4**: 76-83.

8. Sailer I, Pjetursson BE, Zwahlen M, Hämmerle CHF. A systematic review of the survival and complication rates of all-ceramic and metal-ceramic reconstructions after an observation period of at least 3 years. Part II: fixed dental prostheses. *Clinical Oral Implants Research*. 2007; **18**: 86-96.

9. Quinn JB, Sundar V, Parry EE, Quinn GD. Comparison of edge chipping resistance of PFM and veneered zirconia specimens. *Dental Materials*. 2010; **26**: 13–20.

10. Bagis B, Ustaomer S, Lassila LVJ, Vallittu PK. Provisional repair of a zirconia fixed partial denture with fibre-reinforced restorative composite: a clinical report. *Journal of Canadian Dental Association*. 2009; **75**: 133–137.

11. Attia A. Influence of surface treatment and cyclic loading on the durability of repaired all-ceramic crowns. *Journal of Applied Oral Sciences*. 2010; **18**: 194–200.

12. Yoshida K, Tsuo Y, Atsuta M. Bonding of dual-cured resin cement to zirconia ceramic using phosphate acid ester monomer and zirconate coupler. *Journal of Biomedical Materials Research*. 2006; **77**: 28–33.

13. dos Santos JG, Fonseca RG, Adabo GL, dos Santos Cruz CA. Shear bond strength of metal-ceramic repair systems. *Journal of Prosthetic Dentistry*. 2006; **96**: 165–173.

14. Thompson JY, Stoner BR, Piascik JR, Smith R. Adhesion/cementation to zirconia and other non-silicate ceramics: Where are we now? *Dental Materials*. 2011; **27**: 71–82.

15. Piascik JR, Swift EJ, Thompson JY, Grego S, Stoner BR. Surface modification for enhanced silanation of zirconia ceramics. *Dental Materials*. 2009; **25**: 1116–1121.

16. Magne P, Paranhos MPG, Burnett LH Jr. New zirconia primer improves bond strength of resin-based cements. *Dental Materials*. 2010; **26**: 345–352.

17. 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. *Dental Materials Journal*. 2008; **27**: 99–104.

18. Lehmann F, Kern M. Durability of resin bonding to zirconia ceramic using different primers. *Journal of Adhesive Dentistry*. 2009; **11**: 479–483.

19. Blatz MB, Phark JH, Ozer F, MAnte FK, Saleh N, et al. *In vitro* comparative bond strength of contemporary self-adhesive resin cements to zirconium oxide ceramic with and without air-particle abrasion. *Clinical Oral Investigations*. 2010; **14**: 187–192.

20. Attia A, Lehmann F, Kern M. Influence of surface conditioning and cleaning methods on resin bonding to zirconia ceramic. *Dental Materials*. 2011; **27**: 207–213.

21. Perdigão J, Fernandes SD, Pinto AM, Oliveira FA. Effect of artificial aging and surface treatment on bond strengths to dental zirconia. *Operative Dentistry*. 2013; **38**: 168-176.

22. Tsuo Y, Yoshida K, Atsuta M. Effects of alumina-blasting and adhesive primers on bonding between resin luting agent and zirconia ceramics. *Dental Materials*. 2006; **25**: 669–674.

23. Ural C, Külünk T, Külünk S, Kurt M, Baba S. Determination of resin bond strength to zirconia ceramic surface using different primers. Acta Odontologica Scandinavica. 2011; **69**: 48–53.

24. Aboushelib M, Matinlinna J, Salameh Z, Ounsi H. Innovations in bonding to zirconia-based materials: Part I. *Dental Materials*. 2008; **24**: 1268-1272.

25. Watanabe L, Marshall G, Marshall S. Variables influence on shear bond strength testing to dentin. In: Advanced adhesive dentistry - 3rd International Kuraray Symposium: Granada International Symposium, Kuraray Co, ltd. 1999; 75–90.

26. Wegner SM, Gerdes W, Kern M. Effect of different artificial aging conditions on ceramic-composite bond strength. *International Journal of Prosthodontics*. 2002; **15**: 267–272.

27. Wolfart M, Lehmann F, Wolfart S, Kern M. Durability of the resin bond strength to zirconia ceramic after using different surface conditioning methods. *Dental Materials*. 2007; **23**: 45–50.

28. de Souza GMD, Silva NRFA, Paulillo LAMS, de Goes MF, Rekow ED, et al. Bond strength to high-crystalline content zirconia after different surface treatments. *Journal of Biomedical Materials Research*. 2010; **93**: 318–323.

29. Bona Della A, Borba M, Benetti P, Cecchetti D. Effect of surface treatments on the bond strength of a zirconia-reinforced ceramic to composite resin. *Brazilian Oral Research*. 2007; **21**: 10–15.

30. Hummel M, Kern M. Durability of the resin bond strength to the alumina ceramic Procera. *Dental Materials*. 2004; **20**: 498–508.

31. Kern M, Barloi A, Yang B. Surface Conditioning Influences Zirconia Ceramic Bonding. *Journal of Dental Research*. 2009; **88**: 817–822.

32. Cavalcanti AN, Foxton RM, Watson TF, Oliveira MT, Giannini M, et al. Bond Strength of Resin Cements to a Zirconia Ceramic with Different Surface Treatments. *Operative Dentistry*. 2009; **34**: 280-287.

33. Kosmac T, Oblak C, Jevnikar P, Funduk N, Marion L. The effect of surface grinding and sandblasting on flexural strength and reliability of Y-TZP zirconia ceramic. *Dental Materials*. 1999; **15**: 426–433.

34. Gale MS, Darvell BW. Thermal cycling procedures for laboratory testing of dental restorations. *Journal of Dentistry*. 1999; **27**: 89-99.

35. De Munck J, Van Landuyt K, Peumans M, Poitevin A, Lambrechts P, et al. A critical review of the durability of adhesion to tooth tissue: methods and results. *Journal of Dental Research*. 2005; **84**: 118-132.

36. Kitayama S, Nikaido T, Takahashi R, Zhu L, Ikeda M, et al. Effect of primer treatment on bonding of resin cements to zirconia ceramic. *Dental Materials*. 2010; **26**: 426–432.

37. Matinlinna JP, Lassila LV. Enhanced resin-composite bonding to zirconia framework after pretreatment with selected silane monomers. *Dental Materials*. 2011; **27**: 273–280.

38. Piascik JR, Swift EJ, Braswell K, Stoner BR. Surface fluorination of zirconia: Adhesive bond strength comparison to commercial primers. *Dental Materials*. 2012; **28**: 604–608.

39. Valandro LF, Ozcan M, Amaral R, Vanderlei A, Bottino MA. Effect of testing methods on the bond strength of resin to zirconia-alumina ceramic: microtensile versus shear test. *Dental Materials Journal*. 2008; **27**: 849–855.