ASC
Acta Stomatologica Cappadocia

Volume 3 / Issue 1 / June 2023

Research Article

Influence of Different Surface Conditioning Methods on Adhesion of Resin Cement to Zirconia

Tuba Yılmaz Savaş, DDS, PhD[1*], Abdulhaluk Savaş, DDS,[2]

[1]Selçuk University, Faculty of Dentistry, Prosthetic Dentistry, Konya, Turkey

[2]Necmettin Erbakan University, Faculty of Dentistry, Prosthetic Dentistry, Konya, Turkey

**Corresponding author: tuba-yilmaz@windowslive.com*

Yılmaz Savaş, Tuba. & Savaş, Abdulhaluk. "Influence of Different Surface Conditioning Methods on Adhesion of Resin Cement to Zirconia". *Acta Stomatologica Cappadocia.* 3;1 (June 2023): 5-15
DOI: https://doi.org/10.54995/ASC.3.1.1

Received: 29.05.2023; Accepted: 21.06.2023

Influence of Different Surface Conditioning Methods on Adhesion of Resin Cement to Zirconia

Tuba Yılmaz Savaş, Abdulhaluk Savaş

Abstract

Statement of the problem: The challenge of enhancing the bond between zirconia and resin remains, and the efforts continue to determine a simple method for achieving a durable bond.

Objective: This study aimed to evaluate the effect of different surface conditioning strategies on the bond strength between zirconia and resin cement.

Materials & Methods: Forty zirconia specimens (Lava Plus) were fabricated and divided into four groups: control (no surface treatment), primer application (Z-Prime Plus), experimental acid (H_2O:HF:H_2O_2) application, and the combination of the acid and primer application (n=10). A dual-cure resin cement (RelyX Ultimate) was applied over the zirconia specimens. Shear bond strength tests were conducted using a universal testing machine with a crosshead speed of 1 mm/min. The results were analyzed using one-way ANOVA followed by Tukey's post hoc test within the 95% confidence interval.

Results: The results of the one-way ANOVA showed that acid application alone (22.99 ± 2.49 MPa) and acid and primer application (25.24 ± 3.19 MPa) had significantly higher bond strength than the primer (18.87 ± 3.13 MPa) and control (14.91 ± 3.72 MPa) groups ($P<0.05$). However, there was no significant difference between the acid group and acid and primer combination group ($P=0.396$). The control group had the lowest bond strength in all test groups ($P<0.05$).

Conclusions: Experimental acid treatment alone or in combination with primer application could be effective in improving the bond strength.

Keywords: Acid Treatment, Hydrogen Peroxide, Hydrofluoric Acid, Primer, Bond Strength, Zirconia, Surface Conditioning

Introduction

Zirconia is a popular dental material used for the fabrication of crowns, bridges, and hybrid prostheses due to its high mechanical properties, esthetic appearance, and biocompatibility.1 However, zirconia's inherent hydrophobicity and chemical inertness can limit its adhesion to resin-based cements, which can compromise the survival and success of the restoration.[2] Therefore, various surface treatments have been developed to enhance the bonding between zirconia and resin cement, including mechanical and chemical methods such as sandblasting, tribochemical silica coating, silane coupling agents, primer application, laser treatment, and acid etching.[3-6]

Among these surface treatments, sandblasting with alumina or zirconia particles has been widely utilized to increase the surface roughness and promote micromechanical retention for the resin cement.[7] Sandblasting can also induce a phase transformation in the zirconia surface layer, converting the tetragonal phase to the monoclinic phase, which can improve the bonding strength.

However, excessive sandblasting can cause surface damage and compromise the mechanical characteristics of zirconia, ultimately resulting in decreased durability of the restoration.[8]

In addition to sandblasting, the use of primers has gained significant attention due to their ability to increase the surface energy of zirconia and provide micromechanical retention for the resin cement.[9] These primers typically contain functional monomers, such as 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) or 4-methacryloxyethyl trimellitic acid (4-META), which possess phosphate or carboxylic acid groups capable of forming chemical bonds with zirconia surfaces. Through the application of zirconia primers, a chemical interaction is established between the functional monomers and zirconia, facilitating the adhesion of resin cements.[10-12] Many studies showed that phosphate monomer containing primers significantly increased bond strengths between resin cements and zirconia ceramics.[13-17]

Hydrofluoric acid treatment is effective method for glass-based ceramics which removes the glassy matrix and creates micro retentive surface.[18,19] However, zirconia is resistant to conventional acid etching techniques, due its silica-free content.[20,21] There were studies that reporting the conventional hydrofluoric (HF) acid etching did not improve the resin bond strength to zirconia.[22-24] Therefore, using different acid mixtures, concentrations, or acid etching with heat treatment has come to the fore.[21,25-30] The acid solution is designed to create a roughened and active zirconia surface by removing the surface contaminants and inducing a mild chemical reaction that produces hydroxyl groups and surface defects.[30] This surface modification can improve the wettability and adhesion of zirconia to resin cement, leading to increased bond strength and long-term durability. A recent study reported that the use of 20% hydrogen peroxide for 2 hours on the zirconia surface has increased the surface roughness and wettability of the zirconia.[25] Many investigations have indicated that the mixture of sulfuric acid (H_2SO) and hydrogen peroxide (H_2O_2), has the potential to enhance the bonding ability between resin cement and zirconia.[31,32] Furthermore, the adoption of hot chemical etching,[28] which entails the use of different proportions of HF solutions and necessitates a heat treatment with an acidic solution, has exhibited encouraging results.[33]

However, the optimal surface treatment protocol for zirconia bonding remains controversial, and there is a lack of consensus in the literature regarding the most effective surface treatment approach. Therefore, the purpose of this study is to evaluate the effect of different surface conditioning methods, including primer, and the experimental H_2O:HF:H_2O2 acid application, on the bond strength between zirconia and resin cement. The null hypotheses of this in vitro study was the used surface conditioning methods would not have an effect on the shear bond strength between the resin cement and zirconia.

Materials & Methods

Forty zirconia discs (10 mm diameter x 2 mm thickness) were fabricated from pre-sintered zirconia blocks (Lava Plus, 3M ESPE, USA). The discs were sintered according to the manufacturer's instructions by using a sintering furnace to achieve a final density. All discs were ultrasonically cleaned in distilled water for 10 minutes and air-dried before use. The zirconia discs were randomly divided into four groups (n = 10) according to the surface treatment method:

Control group: No surface treatment was performed.

Primer group: The zirconia surfaces was coated with Z-Prime Plus (Bisco Inc., USA) for 60 seconds and air-dried for 5 seconds following the recommendation of the manufacturer.

Acid group: The zirconia surface was etched with an H_2O:HF:H_2O_2 in ratio of 20:1:1 (ACS Reagent, Sigma-Aldrich, UK) acid solution for 90 seconds, followed by rinsing with distilled water for 90 seconds and air-dried.

Acid + Primer Group: The zirconia surface was etched with an H_2O:HF:H_2O_2 in ratio of 20:1:1 (ACS Reagent, Sigma-Aldrich, UK) acid solution for 90 seconds, followed by rinsing with distilled water for 90 seconds and air-dried. After acid application, the primer (Z-Prime Plus, Bisco Inc., USA) was applied for 60 seconds and air-dried for 5 seconds following the recommendation of the manufacturer.

A cylindrical Teflon mold (2.8 mm diameter and 3 mm height) was placed at the center of each zirconia surface for the application of the resin cement. A dual-cure resin cement (RelyX Ultimate, 3M ESPE, USA) was applied by using the custom mold according to the manufacturer's instructions and the mold was removed after photopolymerization for 40 seconds. All the specimens were stored in distilled water at 37°C for 24 hours before testing.

The shear bond strength (SBS) test was performed using a universal testing machine (Mares, Mares Engineering, İstanbul, Turkey) at a crosshead speed of 1 mm/minute until failure occurred. The maximum load required to debond the resin cement from the zirconia was recorded in Newtons (N). The SBS was calculated in MPa using the formula: SBS = F/A, where F is the maximum load recorded, and A is the bonding area (πr^2).

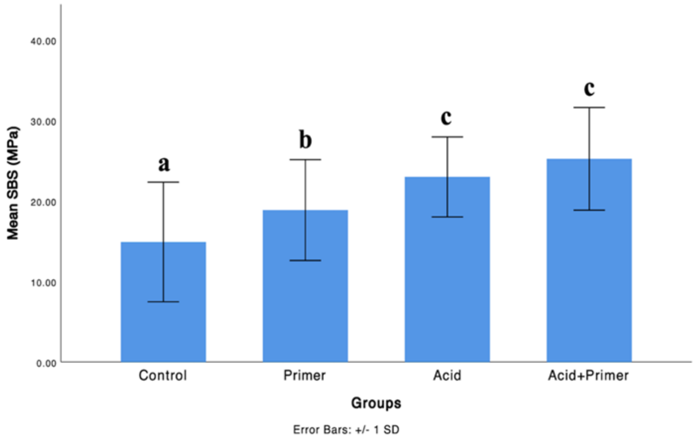
After the SBS test, debonded specimen surfaces were examined in order to analyze the failure types using a stereomicroscope (Olympus SZ 40, Olympus, Tokyo, Japan) at x40 magnification. To determine the failure mode in zirconia/cement interfaces, failure was classified as: Adhesive failure mode (failure between zirconia and resin cement), cohesive failure mode (failure within the resin cement), and mixed failure mode (combination of adhesive and cohesive failure modes.

All statistical analyses were performed using SPSS version 25.0 (IBM Corp., USA). Normality was checked with Shapiro-Wilk tests ($P>0.05$). The data were analyzed using one-way analysis of variance (ANOVA) and Tukey's post-hoc test to compare the mean SBS values between the groups ($\alpha = 0.05$).

Results

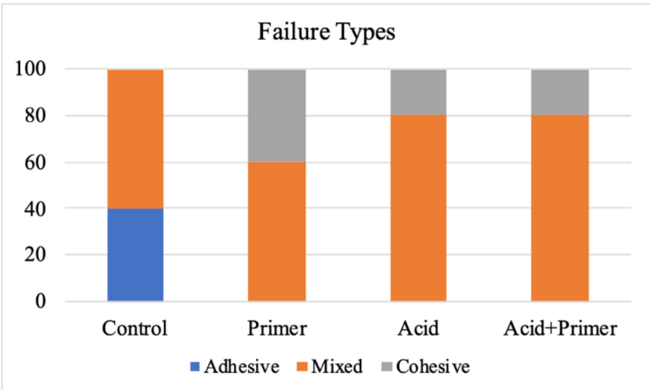
Mean SBS values and standard deviations (MPa) was shown in Figure 1.

Figure 1. Mean SBS values and standard deviations (MPa). Different letters (a, b, c) indicate significant differences between the groups (P<0.05).



One-way ANOVA revealed significant differences in the SBS values among the test groups (F=20.879, df=3, P<0.001). Tukey's post-hoc test revealed that the acid group (mean SBS = 22.99 ± 2.49 MPa) and acid and primer group (mean SBS = 25.24 ± 3.19 MPa) showed significantly higher bond strength than the primer group (mean SBS = 18.87 ± 3.13 MPa) and control group (mean SBS = 14.91 ± 3.72 MPa). There was no significant difference in the bond strength between the acid and primer group and the acid group (P=0.396). Surface conditioning significantly improved the SBS values and the control group showed significantly lower mean SBS value than all tested groups (P<0.05). The primer group showed significantly lower bond strength than the acid group and acid and primer group (P<0.05). Failure mode types and percentages were presented in Figure 2.

Figure 2. Failure type percentages (%) of the test groups.



Failure mode analyses revealed that the most of the specimens showed the mixed type of failure. Adhesive failures were seen only in the control group. Cohesive failures were seen in all the groups except the control group.

Discussion

In this study, the effect of different surface treatments on the bond strength between zirconia and resin cement was evaluated using a shear bond strength (SBS) test. The results showed that the primer, acid, and acid and primer application had significantly higher bond strength than the control group. Therefore, the null hypotheses of this in vitro study was rejected.

As a result of the application of containing MDP primers, phosphate monomers form chemical bonds with the zirconia surface.[34,35] The available data support the use of primers containing adhesive monomers like MDP, which create long-term stable resin connections to zirconia.[34,36] The results indicate that the bond strength exhibited by the control group was significantly lower than that of all the groups, indicating that surface treatment is necessary to achieve a strong bond between zirconia and resin cement.[37] The use of a primer, can help to improve wettability and chemical bonding by providing functional groups for chemical bonding with the resin cement.[37] Similarly, Z-Prime Plus primer application significantly improved the adhesion of resin cement to zirconia in this study.

Experimental acid used in this study demonstrated significantly higher bond strength than control and primer application group. This finding is consistent with prior research that has demonstrated that acid etching of zirconia can improve the bond strength with resin cement by creating a micromechanical interlocking between the cement and the zirconia surface.[21,27,28,30] The use of an experimental $H_2O:HF:H_2O_2$ acid application in this study may have contributed to the improved bond strength by increasing the surface roughness and enhance the micromechanical interlocking between the zirconia and resin cement.[21,25,28-30] It is worth noting that the acid and primer combination group did not show a significant improvement in bond strength compared to the acid group alone. This suggests that the acid treatment may have been sufficient to improve the bonding between the zirconia and resin cement, and that the addition of a primer did not provide any additional benefit in terms of bond strength. However, it is possible that the use of a primer could provide other benefits, such as improved marginal adaptation or reduced microleakage, which were not evaluated in this study.

A recent study reported that the experimental 20% H_2O_2 acid application for 2 hours may cause a phase transformation in zirconia, leading to increased surface roughness and higher chemical reactivity.[25] The experimental acid application duration in this study was 90 seconds which was quite low compared to the aforementioned study and the phase transformation did not evaluated in this study. Another study used 48% HF acid soliton at 100°C for 25 minutes reported that HF acid significantly improved the resin bond strength without inducing a phase transformation.[28]

The findings of this study suggest that surface treatment of zirconia is essential to achieve a durable bond between the zirconia and resin cement. Acid treatment alone or in combination with primer application could be effective in improving the bond strength. These results are consistent with previous studies that have demonstrated the effectiveness of acid treatment in improving the bond strength between zirconia and resin cement.[21,27,28,30]

However, the use of an experimental H_2O:HF:H_2O_2 acid application may require further evaluation in terms of its long-term clinical performance and potential side effects. Besides, in order to work with HF, it is imperative to have access to a workspace that is both isolated and well-ventilated. Although HF is not classified as a highly potent acid, its ability to penetrate cellular membranes and interfere with metabolic processes results in cytotoxicity.[21]

It is worth noting that the current study only evaluated the bond strength after 24 hours. Lack of aging procedures, absence of surface roughness measurements, and phase transformation evaluations are the limitations of this study. Future studies should evaluate the long-term bond strength and durability of different surface treatments. Furthermore, the effect of thermocycling and aging on the bond strength should also be investigated.[38]

Conclusion

Within the limitations of this in vitro study, this study demonstrated that the H_2O:HF:H_2O_2 acid treatment alone or in combination with primer application could significantly increase the bond strength of resin cement to zirconia. The use of an experimental H_2O:HF:H_2O_2 acid application may have potential for enhancing the bond strength, although further investigation is required.

References

1.Denry I, Kelly JR. State of the art of zirconia for dental applications. Dent Mater 2008;24:299-307.

2. Scaminaci Russo D, Cinelli F, Sarti C, Giachetti L. Adhesion to zirconia: A systematic review of current conditioning methods and bonding materials. Dent J 2019;7:74.

3.Akın H, Ozkurt Z, Kırmalı O, Kazazoglu E, et al. Shear bond strength of resin cement to zirconia ceramic after aluminum oxide sandblasting and various laser treatments. Photomed Laser Surg 2011;29:797-802.

4.Cheung J, Botelho MG. Zirconia surface treatments for resin bonding. J Adhes Dent 2015; 17:551-8.

5.Moon J-E, Kim S-H, Lee J-B, Han J-S, et al. Effects of airborne-particle abrasion protocol choice on the surface characteristics of monolithic zirconia materials and the shear bond strength of resin cement. Ceram Int 2016;42:1552-62.

6.Şanlı S, Çömlekoğlu MD, Çömlekoğlu E, Sonugelen M, et al. Influence of surface treatment on the resin-bonding of zirconia. Dent Mater 2015;31:657-68.

7.Oh GJ, Yoon JH, Vu VT, Ji MK, et al. Surface characteristics of bioactive glass-infiltrated zirconia with different hydrofluoric acid etching conditions. J Nanosci Nanotechnol 2017;17:1645-648.

8.Kosmač T, Oblak C, Jevnikar P, Funduk N, et al. The effect of surface grinding and sandblasting on flexural strength and reliability of Y-TZP zirconia ceramic. Dent Mater 1999;15:426-33.

9.Júnior VVBF, Dantas DCB, Bresciani E, Huhtala MFRL. Evaluation of the bond strength and characteristics of zirconia after different surface treatments. J Prosthet Dent 2018;120:955-9.

10.Inokoshi M, De Munck J, Minakuchi S, Van Meerbeek B. Meta-analysis of bonding effectiveness to zirconia ceramics. J Dent Res 2014;93:329-34.

11.Özcan M, Bernasconi M. Adhesion to zirconia used for dental restorations: a systematic review and meta-analysis. J Adhes Dent 2015;17:7-26.

12.Thammajaruk P, Inokoshi M, Chong S, Guazzato M. Bonding of composite cements to zirconia: A systematic review and meta-analysis of in vitro studies. J Mech Behav Biomed Mater 2018;80:258-68.

13.Chen L, Suh BI. Bonding of resin materials to all-ceramics: A review. Curr Res Dent 2012;3:7-17

14.Cura C, Özcan M, Isik G, Saracoglu A. Comparison of alternative adhesive cementation concepts for zirconia ceramic: glaze layer vs zirconia primer. J Adhes Dent 2012;14:75.

15.Azimian F, Klosa K, Kern M. Evaluation of a new universal primer for ceramics and alloys. J Adhes Dent 2012;14:275.

16. Seabra B, Arantes-Oliveira S, Portugal J. Influence of multimode universal adhesives and zirconia primer application techniques on zirconia repair. J Prosthet Dent 2014;112:182-7.
17. Attia A, Kern M. Long-term resin bonding to zirconia ceramic with a new universal primer. J Prosthet Dent 2011;106:319-27.
18. Borges GA, Sophr AM, De Goes MF, Sobrinho LC, et al. Effect of etching and airborne particle abrasion on the microstructure of different dental ceramics. J Prosthet Dent 2003;89:479-88.
19. Chen J, Matsumura H, Atsuta M. Effect of etchant, etching period, and silane priming on bond strength to porcelain of composite resin. Oper Dent 1998;23:250-7.
20. Thompson JY, Stoner BR, Piascik JR, Smith R. Adhesion/cementation to zirconia and other non-silicate ceramics: where are we now? Dent Mater 2011;27:71-82.
21. Kim S-H, Cho S-C, Lee M-H, Kim H-J, et al. Effect of 9% hydrofluoric acid gel hot-etching surface treatment on shear bond strength of resin cements to zirconia ceramics. Medicina 2022;58:1469.
22. de Menezes FCH, Borges GA, Valentino TA, de Menezes Oliveira MAH, T et al. Effect of surface treatment and storage on the bond strength of different ceramic systems. Braz J Oral Sci 2009;8:119-23.
23. Derand P, Derand T. Bond strength of luting cements to zirconium oxide ceramics. Int J Prosthodont 2000;13.
24. Bona AD, Borba M, Benetti P, Cecchetti D. Effect of surface treatments on the bond strength of a zirconia-reinforced ceramic to composite resin. Braz Oral Res 2007;21:10-5.
25. Tuna T, Wein M, Altmann B, Steinberg T, et al. Effect of hydrogen peroxide on the surface and attractiveness of various zirconia implant materials on human osteoblasts: an in vitro study. Materials 2023;16:961.
26. Flamant Q, Marro FG, Rovira JJR, Anglada M. Hydrofluoric acid etching of dental zirconia. Part 1: etching mechanism and surface characterization. J Eur Ceram Soc 2016;36:121-34.
27. Lee Y, Oh KC, Kim N-H, Moon H-S. Evaluation of zirconia surfaces after strong-acid etching and its effects on the shear bond strength of dental resin cement. Int J Dent 2019;2019.
28. Liu D, Tsoi JK-H, Matinlinna JP, Wong HM. Effects of some chemical surface modifications on resin zirconia adhesion. J Mech Behav Biomed Mater 2015;46:23-30.

29.Smielak B, Klimek L. Effect of hydrofluoric acid concentration and etching duration on select surface roughness parameters for zirconia. J Prosthet Dent. 2015;113:596-602.

30.Sriamporn T, Thamrongananskul N, Busabok C, Poolthong S, et al. Dental zirconia can be etched by hydrofluoric acid. Dent Mater J 2014;33:79-85.

31.Lohbauer U, Zipperle M, Rischka K, Petschelt A, et al. Hydroxylation of dental zirconia surfaces: characterization and bonding potential. J Biomed Mater Res B Appl Biomater 2008;87:461-7.

32.El-Ashkar ASF, Nabil O. Dealing with the Internal and External Surfaces of Zirconia Restorations; between Past and Present Trends. A Review of Literature. Acta Sci Dent Sci 2022;6.

33.Xie H, Chen C, Dai W, Chen G, et al. In vitro short-term bonding performance of zirconia treated with hot acid etching and primer conditioning etching and primer conditioning. Dent Mater J 2013;32:928-38.

34.Yue X, Hou X, Gao J, Bao P, et al. Effects of MDP-based primers on shear bond strength between resin cement and zirconia. Exp Ther Med 2019;17:3564-72.

35. Kitayama S, Nikaido T, Takahashi R, Zhu L, et al. Effect of primer treatment on bonding of resin cements to zirconia ceramic. Dental Mater 2010;26:426-32.

36.Chen L, Suh BI, Brown D, Chen X. Bonding of primed zirconia ceramics: evidence of chemical bonding and improved bond strengths. Am J Dent 2012;25:103-8.

37.Tanış MÇ, Akay C, Karakış D. Resin cementation of zirconia ceramics with different bonding agents. Biotechnol Biotechnol Equip 2015;29:363-7.

38.Lüthy H, Loeffel O, Hammerle CH. Effect of thermocycling on bond strength of luting cements to zirconia ceramic. Dent Mater 2006;22:195-200.