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Original Article

Investigation of the effect of different experimental acid solutions applied on the zirconia surface on the bond strength between zirconia and resin cement: A pilot study

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Abstract

Aim: The purpose of this study is to examine the effects of two different experimental acid solutions and sandblasting on the bond strength between zirconia ceramic core material and resin cement.
Methodology: Forty zirconia core materials were prepared and separated into four groups based on the surface treatment to be applied (n = 10) as follows: 1) Control Group: no surface treatment was performed; 2) Sandblasting Group: the specimen surfaces were sandblasted with 50 µm aluminum oxide particles from a distance of 10 mm at 3 bar pressure for 15 seconds; 3) Sol-1 Group: H_2O:HF:H_2O_2 solution was applied for 60 seconds, washed, and then dried; and 4) Sol-2 Group: H_2O:HF:HNO_3 solution was applied to the surfaces of the specimens for 60 seconds, washed, and then dried. All specimens were coated with a dual-cure resin cement with a 2.8-mm diameter. After 24 hours, the specimens were tested for shear bond strength (SBS) at 1 mm/min using a universal testing device. Statistical analysis was conducted using one-way analysis of variance (ANOVA) and Tukey HSD tests with a confidence interval of 95%.
Results: The difference among the groups was statistically significant ($p = 0.002$) based on the one-way ANOVA results. The Control Group had the lowest SBS (8.06 ± 4.52), whereas the Sol-1 Group had the highest SBS (17.10 ± 5.67). The Sol-1 and Sol-2 Groups demonstrated comparable bonding strength values ($p = 0.17$). The Sandblasting and Sol-1 Groups exhibited considerably stronger bond strength than the Control Group ($p = 0.05$).
Conclusion: The experimental acid solutions enhanced the bond strength between the resin cement and the zirconia ceramic at the sandblasting treatment level.

Keywords: Acid etching, bond strength, hydrogen peroxide, nitric acid, zirconia

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Introduction

The demand for zirconia ceramics has increased due to their high mechanical strength and biological compatibility, which can be widely used for various indications (1, 2). Although zirconia has higher mechanical properties, including durability, hardness, and fatigue resistance, most restorations cannot be cemented using conventional procedures because of insufficient bond strength (3–5). For a long-term and durable bond to be formed at the zirconia and cement interface, both chemical and micromechanical retention must be provided (6, 7).

Inadequate bonding with resin cement is among the most important disadvantages of zirconia ceramics compared with other all-ceramic systems (8). In contrast to glass ceramic systems, zirconia ceramics do not have a glass matrix (1, 2). Resin bonding to these materials is less stable and needs significantly different bonding techniques than resin bonding to ceramics containing silica (9). Surface treatment methods, such as hydrofluoric (HF) acid application, which is commonly used for simple micromechanical adhesion, cannot sufficiently roughen the zirconia surface (2). Therefore, several strategies have been devised to improve the bond strength of resin cement to zirconia. Various surface treatment methods, such as sandblasting, tribochemical silica coating, plasma spray method, laser etching, and primer application, are used to increase the bonding of resin cement (10–12). A combination of mechanical and chemical pretreatments seems to be essential for achieving long-lasting adhesion (13). Moreover, to obtain a durable bond between the zirconia and resin cement, special monomer containing resin cements are recommended (14).

Zirconia is commonly treated by sandblasting with aluminum oxide (Al_2O_3) particles. As a result, rough areas are formed on the zirconia surface, surface energy decreases, and wettability increases (15). Sandblasting may strengthen the bonding between the zirconia and the resin cement, but it also causes phase changes on the zirconia surface, thus weakening the restoration structure and leading to microcracks (16, 17).

Due to these drawbacks, attempts to strengthen the bonding of the resin cement continue to be enhanced. There have been reports stating that combining sulfuric acid with hydrogen peroxide (H_2O_2), known as the “Piranha solution” ($3H_2SO_4{:}1H_2O_2$), can enhance the bonding strength of resin cement to zirconia (18,19). Some studies utilizing hot chemical etching (20), which uses HF acid solutions in different ratios and requires a heat treatment with an acidic solution, have shown promising results (21). However, no research has been conducted to determine whether both $H_2O{:}HF{:}H_2O_2$ and $H_2O{:}HF{:}HNO_3$ solutions used for chemical etching can roughen the surface of dental zirconia or strengthen the bonding with resin cement. The objective of this in vitro investigation is to determine the impacts of two experimental acid solutions and sandblasting on the bond strength between zirconia ceramic core material and resin cement. The null hypothesis of this study is as follows: Different methods for surface conditioning would have no effect on the shear bond strength (SBS) between zirconia and resin cement.

Materials and Methods

Forty zirconia plates with 2-mm thickness were cut from pre-sintered zirconia blocks (IPS e.max ZirCAD MO; Ivoclar Vivadent AG, Schaan, Liechtenstein). The specimens were then sintered following the manufacturer’s instructions. The sintered specimens were ground with 600-grit silicon carbide abrasive papers to ensure surface standardization and subsequently cleaned in distilled water in an ultrasonic machine (BioSonic UC50, Whaledent, Langenau, Germany). Thereafter, the zirconia specimens were randomly split into four groups based on the surface treatment techniques used (n = 10) as follows:

- **Control Group:** No surface treatment was applied to the specimens
- **Sandblasting Group**: Airborne-particle abrasion with 50 µm Al_2O_3 particles (Korox 50, Bego Dental, Germany) from a distance of 10 mm at 3 bar pressure for 15 seconds
- **Sol-1 Group:** $H_2O{:}HF{:}H_2O_2$ solution (ACS Reagent, Sigma-Aldrich, UK) in 20:1:1 ratio was applied with a brush for 60 seconds, rinsed in distilled water for 60 seconds, and then air-dried
- **Sol-2 Group:** $H_2O{:}HF{:}HNO_3$ solution (ACS Reagent, Sigma-Aldrich, UK) in 50:1:1 was applied with a brush for 60 seconds, rinsed in distilled water for 60 seconds, and then air-dried.

A custom-made Teflon mold with an internal diameter of 2.8 mm and a thickness of 3 mm was placed on the zirconia surface. The resin cement (Nova Resin, Imicryl, Turkey) was applied using the mold and light-polymerized for 40 seconds (Bluephase C8, Ivoclar Vivadent AG). The specimens were kept in 37 °C distilled water for 24 hours after the resin cement application. The SBS tests were conducted using a universal testing device (Marestek, Mares Engineering, Istanbul, Turkey) with a knife-edged tip and a crosshead speed of 1 mm/min until failure occurred. The SBS values were converted to MPa by dividing the SBS value (N) by the bonded surface area (mm2).

The failure modes of the specimens were determined with a stereomicroscope (Olympus SZ 40, SZ-PT, Olympus Corp., Tokyo, Japan) under 20× magnification. The failure modes were classified as adhesive (no resin remnant on the zirconia surface), cohesive (failure within the resin cement), and mixed (combination of adhesive and cohesive failures on the zirconia surface).

Statistical analysis

Statistical analyses were performed using SPSS V26(IBM SPSS Inc., Armonk, NY, USA). Normality was checked with the Kolmogorov-Smirnov test. The data were analyzed using one-way analysis of variance (ANOVA), followed by Tukey's HSD test with a 95% confidence interval.

Results

The means and standard deviations of the SBS values for each group are presented in Table 1. The one-way ANOVA test showed a significant difference among the groups (F = 188.025, df = 3, $p = 0.002$). The mean SBS values of the sandblasting and Sol-1 group significantly increased compared with the control group ($p < 0.05$). However, there was no significant difference between the Sol-2 and control groups ($p > 0.05$).

The percentage distribution of the fracture modes is presented in Table 2. Adhesive and mix failures were predominantly observed in the control, sandblasting, and Sol-1 groups, whereas only adhesive failure was predominantly observed in the Sol-1 group.

Table 1. Means, standard deviations, and 95% confidence intervals of the shear bond strengths (MPa) of the groups.

Group	Mean	SD	95% Confidence Interval	
			Lower Bound	Upper Bound
Control	8.07^{a}	4.53	4.83	11.30
Sandblasting	16.97^{b}	4.58	13.59	20.14
Sol-1	17.10^{b}	5.68	13.04	21.16
Sol-2	11.90^{ab}	6.95	6.93	16.87

SD: Standard deviation. MPa: Shear bond strength.
Sol-1 group: Group in which the H_2O:HF:H_2O_2 solution was used.
Sol-2 group: Group in which the H_2O:HF:HNO_3 solution was used. Different lowercase superscript letters indicate a significant difference for the groups in a column ($p < 0.05$).

Table 2. Percentage distribution of the failure modes of the groups.

Group	Adhesive failure	Cohesive failure	Mixed failure
Control	60%	0	40%
Sandblasting	50%	0	50%
Sol-1	40%	0	60%
Sol-2	80%	0	20%

Sol-1 group: Group in which the H_2O:HF:H_2O_2 solution was used.
Sol-2 group: Group in which the H_2O:HF:HNO_3 was used.

Discussion

This study assessed the effects of two experimental acid solutions and sandblasting on the shear bond strength between zirconia and resin cement. The results showed that the experimental acid solutions and the sandblasting procedure significantly increased the SBS in comparison with the control group. Therefore, the hypothesis is rejected.

Sandblasting is often employed in the process of zirconia surface roughening, maintaining micromechanical bonding and thus increasing the activated zirconia surface and the wettability. For this purpose, Al_2O_3 particles are used for different parameters, such as the particle size, application duration, and pressure (22). Sandblasting with Al_2O_3 particles under low pressure (2-4 bar) is recommended in literature (23). Furthermore, a particle size of 50 μm is recommended to prevent phase transformation and a decrease in the mechanical properties of zirconia (24, 25). Therefore, in this study, 50 μm of Al_2O_3 particles was used at a 3-bar pressure. The sandblasting application significantly enhanced the micromechanical bonding strength of the resin cement to zirconia, as it did in previous studies (7, 26, 27).

The bond strength values obtained in the acid etch groups in this study were comparable to those in the sandblasting group and higher than those in the control group. Cho et al. (28) used the HNO_3:HF solution at a 1:1 ratio over a zirconia surface for 1 to 3 h and reported improved bonding strength of the resin cement. Al-Zordk et al. (29) used a piranha acid solution and hot acid etching with HNO_3:HF at 1:1 ratios for 25 min. The shear bond strength was highest in the HNO_3:HF solution, but the piranha solution showed the lowest bond strength. Liu et al. (20) used 69% HNO_3 and 48% HF acid solution at a ratio of 1:1 at 100°C for 25 min and reported that the increased roughness improved the dissolving rate of zirconia grains in the presence of acid. Similarly, Qeblawi et al. (16) reported that the use of 5% to 9.5% HF partially dissolved the vitreous component of the zirconia ceramic and increased the surface roughness, which strengthened the bond with the resin cement. In the present study, H_2O:HF:HNO_3 (at the ratio of 50:1:1) and H_2O:HF:H_2O_2 (at the ratio of 20:1:1) solutions were applied to the zirconia surface for only 1 min, which is considerably shorter than the duration of the acid application in literature. Moreover, no additional heat treatment was used in the present study. However, the experimental acids used in this study significantly improved the bond strength compared to the control group. This increase may also be attributed to the hydroxylation on the surface, and the acid solutions might have contributed to the micromechanical retention by increasing the micro-roughness of the zirconia (30). The objective of the hydroxylation was to enhance the bond strength between zirconia and the resin cements by increasing the concentration of OH- groups on the zirconia surface (18, 19). Nevertheless, these applications may not be suitable for clinical use mainly because they contain hazardous acids that might be toxic (18). They may be suitable only for laboratory usage with proper safety protocols, including adequate ventilation and personal protective equipment (18).

Resin cement is favored for the cementation of zirconia restorations due to its higher retention quality, increased fracture resistance, and excellent marginal sealing (31). The addition of monomers that contain 4-methacryloyloxyethyl trimellitate anhydride (4-META), which exhibit chemical reactivity with oxide layers (32), to resin cement enhances the zirconia adhesive bond (32, 33). Therefore, in this study, a resin cement that contained 4-META was used. However, since no acceptable bond occurred in the control group, which had no surface treatment, it exhibited much lower SBS values than the sandblasting and acid-applied groups. Although it is emphasized that the monomer added to the resin cement increased the bonding strength, this result may indicate that an adequate bond with zirconia cannot be obtained without additional surface treatment.

Artificial aging methods are used in laboratory conditions to evaluate the effectiveness of restorative systems and to predict their behaviour in the oral environment. The most important limitation of this study is the absence of a thermocycling process. The application of the SBS test by imitating the thermal variables in the oral environment can provide more clinically useful data. Further studies are needed to understand the effect of the experimental acids used in this study on the mechanical properties of zirconia. Furthermore, the adverse effects and toxicity of these acids should be considered.

Conclusions

Within the limitations of this study, the following conclusions are made:

1. The lowest bond strength was found in the control group, which did not undergo any surface treatment.
2. The experimental acid solutions used increased the bond strength between resin cement and zirconia ceramic at the sandblasting level. However, the toxicity should be considered during the use of these acids, proper safety protocols should be applied, and contact with the vital tissues should be avoided.

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