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Research Article

Does the Duration of Acid Treatment Affect the Bond Strength Between Resin Cement and Zirconia?

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Does the duration of acid treatment affect the bond strength between resin cement and zirconia?

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Abstract

Statement of the problem: The bonding between the resin cement and zirconia still needs to be improved and the effect of the experimental acid application duration on the resin bond strength with zirconia is unknown.

Objective: This study aimed to investigate the effect of different acid application times on the shear bond strength between zirconia and dual-cure resin cement.

Materials & Methods: A total of 50 zirconia disks were prepared in 2 mm thickness and randomly divided into five treatment groups: control (no treatment was applied), 30 seconds HF:H2O:H2O2 acid application group, 60 seconds HF:H2O:H2O2 acid application group, 90 seconds HF:H2O:H2O2 acid application group, and 120 seconds HF:H2O:H2O2 acid application group. Then a dual-cure resin cement was applied over the 3 mm diameter and 3 mm thickness of the zirconia specimens. The specimens were immersed in distilled water and maintained at a temperature of 37°C for a duration of 24 hours. The shear bond strength tests were performed using a universal testing machine with a force of 1 mm/min until the fracture occurs, and the data were analyzed using one-way ANOVA and Tukey's post-hoc test.

Results: The highest mean shear bond strength was found in the 90 seconds acid application group, which was significantly higher than the control and 30 seconds acid application groups ($P<0.05$). However, the 60- and 120 seconds acid application groups showed similar shear bond strength values with the control, 30- and 90 seconds acid application groups ($P>0.05$).

Conclusions: The use of a 60-, 90- and 120 seconds acid application can improve the bond strength between zirconia and resin cement. However, caution should be taken when using this acid due to its potential toxicity and hazards.

Keywords: Zirconia, Resin Cement, Shear Bond Strength, Hydrofluoric Acid, Hydrogen Peroxide, Surface Treatment.

Introduction

Zirconia is a popular material in dentistry due to its excellent mechanical and esthetic properties.[1] However, bonding zirconia to teeth or other materials can be challenging due to its high surface energy and chemical inertness.[2] Several surface treatment methods have been established to improve the adhesive capacity between zirconia and resin cement. These include sandblasting, tribochemical silica coating, laser treatment, and chemical etching.[3] Sandblasting is mostly preferred method that can be used for zirconia surface treatment,[4] but it has been reported to have several disadvantages, such as phase transformation, surface roughness variations, and microcracking.[5]

One of the commonly used methods for ceramic surface treatment is acid etching, which involves applying an acid solution to create micromechanical retention and increase surface energy.[6] Acid etching is a process where acid is applied to a porcelain or ceramic surface to create pores to enhance the bond strength of an adhesive or a coating to the surface.[7] The process of acid etching the bonding layer of glass-ceramic restorations is widely regarded as the most efficacious treatment option for achieving an efficient bond with composite cement.6 Zirconia is a specific ceramic material that exhibits resistance to etching with hydrofluoric acid (HF) under normal conditions due to its non-silica-based composition.[8] Therefore, other acid mixtures have been developed for zirconia surface treatment, including sulfuric acid, phosphoric acid, and the hydrochloric acid mixture.[9-12]

Several investigations have demonstrated encouraging outcomes[3] using hot chemical etching,[13] which involves the utilization of HF acid solutions in varying proportions and necessitates a heat treatment with an acidic solution. According to certain reports, a mixture of hydrogen peroxide (H_2O_2) with sulfuric acid, commonly referred to as the "Piranha solution" has the potential to increase the adhesive potency of resin cement to zirconia.[14, 15]

The application time of the acid solution is a crucial factor that can affect the efficacy of the surface treatment.[12,16] The optimal application time for zirconia surface treatment with the hydrofluoric acid, hydrogen peroxide and water combined acid mixture ($HF:H_2O:H_2O_2$) is not well established. Several studies have investigated the effect of different acid application

times on the shear bond strength between zirconia and resin cement, but the results have been inconsistent.[17,18] Therefore, this in-vitro study aimed to evaluate the effect of different duration of the acid treatment on the shear bond strength between the zirconia and resin cement. The null hypothesis of this study was that the acid application duration would not affect the bond strength of the resin cement to the zirconia.

Materials & Methods

A total of 50 zirconia specimens (thickness: 2 mm) were prepared from pre-sintered zirconia blocks (IPS e.max ZirCAD, Ivoclar Vivadent, Schaan, Liechtenstein). The specimens were then sintered according to the manufacturer's instructions. The zirconia specimens were then randomly assigned to 5 experimental groups (n=10 per group): a control group (no surface treatment), and 4 experimental groups subjected to an experimental acid solution. The experimental acid solution was composed of hydrofluoric acid (HF), hydrogen peroxide (H_2O_2), and distilled water in a ratio of 20:1:1 (ACS Reagent, Sigma-Aldrich, UK). The experimental groups were subjected to the acid solution for different application times: 30 seconds, 60 seconds, 90 seconds, and 120 seconds with a nylon brush. The specimens were then rinsed with distilled water for 30 seconds, 60 seconds, 90 seconds, and 120 seconds according to the duration of acid treatment in each group, and dried with air.

A dual-curing resin cement (SpeedCem Plus, Ivoclar Vivadent, Schaan, Liechtenstein) was applied onto the zirconia surface using a custom Teflon mold in 3 mm diameter and 3 mm thickness. The cement was then light-cured for 20 seconds using a LED curing unit with a light intensity of 1200 mW/cm2. The mold was carefully removed, and the specimens were stored in distilled water at 37°C for 24 hours.

Shear bond strength was measured using a universal testing machine (Marestek, Mares Engineering, İstanbul, Turkey) with a 1 mm/min crosshead speed until failure. The load at failure was recorded in Newtons (N), and the SBS was calculated in MPa by dividing the load by the bonding area (πr2).

A software program (SPSS Statistics v26, IBM Corp., USA) was used for statistical analyses. The data's normality and homogeneity were checked with Kolmogorov-Smirnov and Levene tests, respectively. The data were then analyzed using one-way analysis of variance (ANOVA) and Tukey's post-hoc test (α=0.05).

Results

Table 1 presents the means and standard deviations of the test groups. According to the one-way ANOVA results, there were significant differences among the SBS of the groups (F=5.281, df=4, P=0.001). The highest bond strength was found in the 90 seconds acid application group, which is significantly higher than the control and 30 seconds acid application group (P<0.05). The 30 seconds, 60 seconds, and 120 seconds acid application groups showed similar bond strength values with the control group (P>0.05)(Table 1).

Table 1. Means, standard deviations, and 95% confidence intervals of the test groups.

	n	Mean	Std. Deviation	95% Confidence Interval for Mean	
				Lower Bound	Upper Bound
Control	10	15.19[a]	4.53	11.95	18.43
30 seconds	10	12.22[a]	3.96	9.39	15.05
60 seconds	10	17.47[ab]	5.64	13.43	21.50
90 seconds	10	21.74[b]	6.01	17.44	26.04
120 seconds	10	17.01[ab]	3.25	14.69	19.34

Different superscript letters indicate a significant difference among the groups (P<0.05).

Discussion

The present study aimed to investigate the effect of different acid application times on the shear bond strength between zirconia and resin cement. The results of the one-way ANOVA revealed that the bond strength of the 90 seconds acid application group was significantly higher than the control and 30-seconds acid application groups, while the other groups showed similar bond strength values with the control group. Therefore, the null hypothesis of this study was rejected.

In the current study, an experimental acid solution composed of $HF:H_2O:H_2O_2$ was used for the surface treatment of zirconia. This acid solution might be effective in creating micro retentive surface features on zirconia, which could improve the mechanical interlocking of the resin cement.[13] The applied acid enhanced the bond strengths of the resin cement to the zirconia, especially the 90 seconds application time led to a significant increase in bond strength compared with the control group. These findings are consistent with previous studies that have reported the effectiveness of acid etching on zirconia surfaces to improve the bond strength of resin

cements.[10,13,17] This finding is remarkable because zirconia is known to be resistant to acids as zirconia does not have a glassy phase and therefore cannot be etched with hydrofluoric acid, which is commonly used for surface treatment of glass-based ceramics.[16]

It is important to note that over-etching of zirconia can lead to surface damage and decreased bond strength.[10] The phenomenon of over-etching has been observed to have adverse effects on the adhesion process and structural strength of materials. This is attributed to the formation of large and deep pores, which impede the infiltration of luting agents into the substrate. Such outcomes are deemed unsatisfactory and could compromise the overall quality of the material.[19,20] In the current study, the acid application time was varied from 30 seconds to 120 seconds. The results showed that the 90 seconds acid application group had the highest bond strength, while the 30 seconds, 60 seconds, and 120 seconds groups showed similar bond strength values with the control group. The 120 seconds group, on the other hand, did not show a significant increase in bond strength compared to the 90 seconds group, suggesting that over-etching may have occurred. A similar result was reported by a recent study that used an etching solution composed of 25%HF, 16% sulfuric acid, H_2O_2, methyl alcohol, and purified water within the time intervals of 0, 3, 5, 10, 20, and 30 minutes. The authors reported that the shear bond strength between the resin cement and zirconia increased with the acid application; however, decreased significantly after 30 minutes of acid application.[17]

Another surface treatment method that is commonly used for zirconia is sandblasting. However, sandblasting can lead to microcracks and surface flaws, which can negatively affect the bond strength.[20,21] In contrast, acid treatment may be advantageous as it requires less equipment and is easier to apply than sandblasting. However, although the effect of the acid application used in this study on the zirconia surface has not been analyzed, it is thought that etching on the zirconia surface may both increase the surface roughness and provide micromechanical bonding and improve bonding by increasing the surface energy.[17]

It is important to note that while the $HF:H_2O:H_2O_2$ acid has been shown to improve the bond strength between zirconia and resin cement in this study, it might be potentially toxic and corrosive.[14] The use of this acid in dental applications requires careful consideration and proper handling to ensure the safety of both the patient and the clinician. Therefore, the use of proper personal protective equipment, such as gloves, masks, and protective eyewear, is crucial when working with this kind of acid.[14] Additionally, proper disposal methods must be followed to prevent environmental contamination.

Lack of aging and in vitro test conditions were the limitations of this study. Further studies are needed to fully understand the effects of this acid on the biocompatibility and long-term clinical outcomes of zirconia restorations.

Conclusion

Within the limitations of this in vitro study, the results of this study suggest that an acid solution composed of $HF:H_2O:H_2O_2$ can be an effective surface treatment method for zirconia to improve the bond strength of resin cements. However, over-etching should be avoided, and an acid application time of 90 seconds appears to be optimal for achieving the highest bond strength. Future studies could investigate the long-term durability of the bond strength using this acid application method.

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