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# Evaluation of the effect of different zirconia etching solutions on shear bond strength. A concise review

**Noor Raed[1] , Ahmed Sleibi[2], Abdul Rahman M Saleh[3]**

[1]MSc researcher, Department of Conservative Dentistry, College of Dentistry, Mustansiriyah University, Baghdad, Iraq, Email: noor_alimam@yahoo.com.

[2]Department of Conservative dentistry, Faculty of Dentistry, Mustansiriyah University, Baghdad, Iraq. Email: sleibi1975@uomustansiriyah.edu.iq, ORCID: https://orcid.org/0000-0002-7747-7785.

[3]Department of Clinical Sciences, College of Dentistry, Ajman University, Ajman, United Arab Emirates; Center of Medical and Bio-allied Health Sciences Research, Ajman University, Ajman, United Arab Emirates, Email: rm.saleh@ajman.ac.ae, ORCID: https://orcid.org/0000-0002-9014-8695.

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## Abstract

**Aim:** Different surface treatments were developed to enhance zirconia-resin cement bond strength. Conventional etching with hydrofluoric acid is not effective in enhancing bond strength, however, recent emerging acid etching formulations are thought to enhance zirconia-resin cement. The purpose of the current review is to clarify and summarize zirconia acid etching solutions that were tried to be used as a surface treatment, and to show its capability to enhance zirconia-resin cement bond strength.

**Materials and methods:** PubMed and Google Scholar were searched for studies that investigated (zirconia etching) and (shear bond strength), and (zirconia etching solution), 11 studies were found, 6 of which matched the study criteria and were included in this review.

**Conclusion:** highly corrosive acids in high temperature and concentrations could enhance the shear bond strength of zirconia to resin cement.

**Keywords:** Zirconia; Etching solutions; Shear bond; resin cement; hydrofluoric acid.

## Introduction:

Resin types of cement have the best fracture resistance, margin seal, and retention as compared to zinc phosphate or modified glass-ionomer cements.[1] However, zirconia polycrystalline structure is resistant to conventional silica-based ceramics bonding strategies; acid etching, and silane application. Consequently, zirconia surface treatments gained wide attention in the literature, since huge efforts have been made to enhance zirconia bonding to resin cement However, several recent zirconia surface treatment studies investigated the use of hydrofluoric acid (HF) solutions of various concentrations and temperatures, these studies reported that elevated porosity and roughness of the surface could improve the bonding strength.[2] Zirconia surface roughness would be elevated by changing hydrofluoric acid concentration and temperature, this change exerts a favorable influence on the bonding conditions.[1] In addition, the influence of various formulations of different acids on shear bond strength was also investigated in number of studies. However, no review summarizes all these studies in detail concisely, most present reviews discuss all mechanical, and chemical surface treatments as a whole. The purpose of the current review is to clarify and summarize zirconia acid etching solutions that were tried to be used as a surface treatment, and that were aimed to enhance zirconia-resin cement bond strength.

## Materials and method:

A Pub-med search of (zirconia etching) AND (shear bond strength) AND (zirconia etching solution), showed 12 studies, 5 of which were excluded, the remaining 7 studies were included in the current review, articles with English language only were selected.

### Inclusion criteria:

In the current review, only shear bond studies of zirconia-resin cement were included, wherein any formulation of zirconia etching solution/gel in any concentration or time was investigated. However, studies of other etching methods (e.g. Laser etching), were not included. Only recent studies published in the last five years were included (2018-2023) (Table 1).

### Exclusion criteria:

Tests other than SBS test, (e.g: tensile bond test, roughness tests).

Studies found as (Abstract only).

Studies of other etching methods (e.g. Laser etching).

Studies before 2018, (Table 2).

## Discussion:

The application of hydrofluoric acid treatment is a widely employed technique for the treatment of ceramics composed of silica-based materials, in such a way that it removes or reacts with the glassy matrix that contains silica exposing the crystalline phase, which is responsible for the surface roughness. Specifically, the reaction of hydrofluoric acid with silicon dioxide yields the water-soluble silicic derivatives that could be flushed away,[3] creating micro-pits on the ceramic surface.[4] This reaction is also responsible for the increased ceramic surface's wettability and surface energy that provide a deeper penetration of resin cement tags, resulting in a stronger ceramic-cement bond.[5] Zirconia have a polycrystalline nature with almost no silica particles, this polycrystalline nature causes zirconia resistance to the conventional silica-based ceramics bonding strategies; such as hydrofluoric acid etching and silane application (Sadid-Zadeh et al., 2021).[6]

A study utilizing hydrofluoric acid concluded that application of a 20% hydrofluoric acid (HF) solution at temperatures ranging from 70 to 80 °C, or a 40% hydrofluoric solution at both room temperature and temperatures ranging from 70 to 80 °C, has been seen to be an effective method for acid-etching the surface of zirconia. This process leads to the generation of a highly roughened surface with irregularities. However, the author recommended a 40% hydrofluoric solution at room temperature to enhance the bond strength of zirconia resin cement.[7] In this respect Yu, Oh *et al.* (2021), reported that surface treatment of 3% yttria-stabilized zirconia (3YSZ) with high concentration HF solution, might be considered as an alternative method for a reliable long-term bond of zirconia to MDP-containing resin cement[2]

On the contrary, a study showed that the bond strength of air-borne particle abrasion (APA) surface treatment was superior in (Zirconia Copran Monolith HT; KAT 2—Zirconia Katana ML) or equal in (Zirconia Metoxit Z-CAD HTL) to untreated zirconia, and the hot etching solution hydrochloric acid- ferric chloride (800 mL HCl and 5 mL $FeCl_3$).[8]

A different study investigated zirconia bond strength after it was subjected to SE solution (Smart Etching, Yesbiogold Inc., Seoul, Korea), and concluded that the shear bond strength of zirconia-resin cement was enhanced when SE solution was utilized at a temperature of (70-80°C), the author believed that the elevated temperature increased the molecular activity of the etching solution, which resulted in a faster etching procedure.[9] (Table 1 listed SE solution composition).

Ansari et al., (2018), reported that etching with (Zeta etching solution, Eunjin Chemical Co.); a solution consisting of hydrochloric acid, nitric acid, phosphoric acid, and sulfuric acid, on high translucent zirconia 5% yttria-stabilized zirconia (5YSZ), significantly raised the SBS of zirconia-resin cement ($p$<.05). However, bond strength of zirconia (3YSZ) did not rise significantly ($p$>.05), as compared to APA. The author attributed that to differences in the composition of the two zirconia materials, as the phase structure of (5YSZ) is characterized by more cubic phase than that present in the (3YSZ). In addition, the author mentioned that using of Zeta etching solution didn't compromise the hardness of both 3Yand 5Y stabilized zirconia.[10]

Sadid et al., (2021), concluded that the use of (Zircos-E solution) on (3YSZ) provided a bond strength value similar to that provided by 50 µm $Al_2O_3$ APA ($p$>.05); however, Zircos-E solution surface treatment had a hazardous nature, as it consisted of high corrosive acid formulations including hydrofluoric acid, hydrochloric acid, sulfuric acid, nitric acid, and phosphoric acid.[6]

Sales et al., (2022) reported that using Zircos-E solution on (3YSZ) and on (5YSZ) enhanced its bond strength to resin cement, as compared to other surface treatment methods.[11]

**Conclusion:**

In conclusion, zirconia surface treatment by various acids of different temperatures and concentrations has been investigated in an attempt to increase zirconia bond strength. Within the limitation of this review, the acidic surface treatment seems to be promising for enhancing the shear bond strength between zirconia and resin cement.

**Conflict of interest**

The authors reported that they have no conflicts of interest.

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**Table 1**: Included studies.

| Author (year) | Zirconia generation | Surface treatment groups | Etching solution composition |
|---|---|---|---|
| Sadid-Zadeh et al., 2021[6] | (3YSZ) | Group I :APA 50-µm Al2O3<br>Zirconia etching solution (ZES) for 2 hours<br>Group II :APA + ZES<br>Group III: ZES + APA | hydrofluoric acid (HF), hydrochloric acid (HCl), sulfuric acid (H2SO4), nitric acid (HNO3), and phosphoric acid (H3PO4) |
| Ansari et al., 2018[10] | (3YSZ)<br><br>(5YSZ) | Group I: APA 50-µm Al2O3<br>Group II: APA 50-µm Al2O3 + ZES for 60 minutes in an ultrasonic bath<br>Group III: APA 50-µm Al2O3<br>Group IV: APA 50-µm Al2O3 + ZES for 60 minutes in an ultrasonic bath | HF, hydrochloric acid (HCl), sulfuric acid (H2SO4), nitric acid (HNO3), and phosphoric acid (H3PO4) |
| (Yu, Oh et al. 2021)[2] | (3YSZ) | Group I: tribochemical silica abrasion (control)<br>Group II: etcheing with 5% HF solution for 10 min<br>Group III: etcheing with 10% HF solution for 10 min<br>Group IV: etcheing with 20% HF solution for 10 min<br>Group V: etcheing with 40% HF solution for 10 min | HF solutions of 5%, 10%, 20%, 40% were prepared from 40% HF solution (MKBH5499V, Sigma-Aldrich Co., St. Louis, MO, USA) using distilled water and an electronic scale. |
| Colombo M etal., 2020[8] | | Group I : No treatment<br>Group II : APA 50-µm Al2O3<br>Group III : Hot etching | HCl–FeCl3 (800 mL of hydrochloric acid HCl and 5 mL of ferric chloride FeCl3) for 10 min at a temperature of 100 °C |
| Kim etal., 2020[7] | 3 to 8 mol% yttrium oxide (Y-TZP) | Group I : not etched (control)<br>Group II :etching with 5% HF solutions for 10 min<br>Group III: etching with 10% HF solutions for 10 min<br>Group IV: etcheing with 20% HF solution for 10 min<br>Group V: etcheing with 40% HF solution for 10 min<br>( The etched groups were divided into: at room temperature and at 70-80 C | HF solutions (5%, 10%, 20%, and 40%) for 10 min |
| Yu etal., 2019[9] | | Group I : not etched (control)<br>Group II :etching with 37% phosphate etchant for 10 min (20°C–25°C)<br>Group III: etching with 4% porcelain etchant for 10 min (20°C–25°C)<br>Group IV: etcheing with SE solution for 10 min (70°C–80°C) in a water bath 20% HF solution | The SE etching (Smart Etching,Yesbiogold Inc., Seoul, Korea) consisted of 40% HA (vol %),<br>59% PA (vol %) and 1% HCl (vol %) |

**Table 2**: Excluded studies.

| Title | Author (year) | Reason of exclusion |
|---|---|---|
| Effect of air abrasion, acid etching, and aging on the shear bond strength with resin cement to 3Y-TZP zirconia | Seo et al., 2022[12] | Abstract only |
| Resin bond strength to zirconia: effects of surface treatments and resin cements. | Carvalho, Rippe et al. 2019[13] | Abstract only |
| Evaluation Of The Effect Of Different Surface Treatments, Aging And Enzymatic Degradation On Zirconia-Resin Micro-Shear Bond Strength | Saade J et al. 2020[14] | The study investigated other etching methods (e.g. Laser eching) |
| Chemical durability of high translucent dental zirconia | Seiji Ban, 2020[15] | review disscussed low temperature degradation (LTD), discoloration, and erosion of high translucent dental zirconia |
| Effect of different surface zirconium oxide treatments on binding strength between zirconia and veneering ceramics | Xia et al., 2019[16] | Chinese language |