



## Evaluation of the Effect of Different Zirconia Etching Solutions on Shear Bond Strength. A Concise Review

Noor Raed<sup>1</sup>, Ahmed Sleibi<sup>1</sup>, and Abdul Rahman M Saleh<sup>2,3</sup>

<sup>1</sup> Department of Conservative Dentistry, College of Dentistry, Mustansiriyah University, Baghdad, Iraq

<sup>2</sup> Department of Clinical Sciences, College of Dentistry, Ajman University, Ajman, United Arab Emirates

<sup>3</sup> Center of Medical and Bio-allied Health Sciences Research, Ajman University, Ajman, United Arab Emirates

Correspondence: Ahmed Sleibi

Email: [sleibi1975@uomustansiriyah.edu.iq](mailto:sleibi1975@uomustansiriyah.edu.iq)

Received: 15 October 2023; Accepted: 21 December 2024; Published: 30 December 2024

### Abstract

**Aim of the study:** 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.

**Material and method:** 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 (Kim et al., 2022). 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 (Yu et al., 2021). Zirconia surface roughness would be elevated by changing hydrofluoric acid concentration and temperature, this change exerts a favorable influence on the bonding conditions (Kim et al., 2022). 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).

**Table 1:** Included studies.

Author (year)	Zirconia generation	Surface treatment groups	Etching solution composition
Sadid-Zadeh et al., 2021 (Sadid-Zadeh et al., 2021)	(3YSZ)	Group I :APA 50-µm Al2O3 Zirconia etching solution (ZES) for 2 hours Group II :APA + ZES Group III: ZES + APA	hydrofluoric acid (HF), hydrochloric acid (HCl), sulfuric acid (H2SO4), nitric acid (HNO3), and phosphoric acid (H3PO4)
Ansari et al., 2018 (Ansari et al., 2018)	(3YSZ)  (5YSZ)	Group I: APA 50-µm Al2O3 Group II: APA 50-µm Al2O3 + ZES for 60 minutes in an ultrasonic bath Group III: APA 50-µm Al2O3 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( Yu, Oh et al. 2021)	(3YSZ)	Group I: tribochemical silica abrasion (control) Group II: etcheing with 5% HF solution for 10 min Group III: etcheing with 10% HF solution for 10 min Group IV: etcheing with 20% HF solution for 10 min 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 et al., 2020 (Colombo M et al., 2020)		Group I : No treatment Group II : APA 50-µm Al2O3 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 et al., 2020 (Kim et al., 2020)	3 to 8 mol% yttrium oxide (Y-TZP)	Group I : not etched (control) Group II :etching with 5% HF solutions for 10 min Group III: etching with 10% HF solutions for 10 min Group IV: etcheing with 20% HF solution for 10 min Group V: etcheing with 40% HF solution for 10 min ( 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 et al., 2019 (Yu et al., 2019)		Group I : not etched (control) Group II :etching with 37% phosphate etchant for 10 min (20°C–25°C) Group III: etching with 4% porcelain etchant for 10 min (20°C–25°C) 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 %), 59% PA (vol %) and 1% HCl (vol %)

**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).

**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 (Seo et al., 2022)	Abstract only
Resin bond strength to zirconia: effects of surface treatments and resin cements.	Carvalho, Rippe et al. 2019 (Carvalho, Rippe et al., 2019)	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 (Saade J et al. 2020)	The study investigated other etching methods (e.g. Laser etching)
Chemical durability of high translucent dental zirconia	Seiji Ban, 2020 (Seiji Ban, 2020)	review discussed 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 (Xia et al., 2019)	Chinese language
--	--	------------------

**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, (Borges et al., 2003) creating micro-pits on the ceramic surface (Janda et al., 2003). 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 (Thamrongananskul et al., 2004). 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).

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 (Kim et al., 2020). 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 (Yu et al., 2021).

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<sub>3</sub>) (Colombo et al., 2020).

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 (Yu et al., 2020). (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 3Y and 5Y stabilized zirconia (Ansari et al., 2018).

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  $\mu\text{m}$   $\text{Al}_2\text{O}_3$  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 (Sadid-Zadeh et al., 2021).

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 (Sales et al., 2022).

### 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.

### Supplementary Material

None.

### Funding

This research received no external funding.

### Data Availability Statement

Data are available from the authors upon reasonable request.

### Conflict of interest

The authors reported that they have no conflicts of interest.

### Acknowledgments

The authors would like to thank Mustansiriya University ([www.uomustansiriyah.edu.iq](http://www.uomustansiriyah.edu.iq)), Baghdad, Iraq, for their support in the present work.

### References

1. Kim, S.H., Cho, S.C., Lee, M.H., Kim, H.J. and Oh, N.S. (2022) 'Effect of 9% hydrofluoric acid gel hot-etching surface treatment on shear bond strength of resin cements to zirconia ceramics', *Medicina*, 58(10), p. 1469. Available at: <https://doi.org/10.3390/medicina58101469>.
2. Yu, M.K., Oh, E.J., Lim, M.J. and Lee, K.W. (2021) 'Change of phase transformation and bond strength of Y-TZP with various hydrofluoric acid etching', *Restorative Dentistry & Endodontics*, 46(4), p. e54. Available at: <https://doi.org/10.5395/rde.2021.46.e54>.
3. Borges, G.A., Sophr, A.M., De Goes, M.F., Sobrinho, L.C. and Chan, D.C. (2003) 'Effect of etching and airborne particle abrasion on the microstructure of different dental ceramics', *The Journal of Prosthetic Dentistry*, 89, pp. 479–488. Available at: [https://doi.org/10.1016/S0022-3913\(02\)52704-9](https://doi.org/10.1016/S0022-3913(02)52704-9).
4. Janda, R., Roulet, J.F., Wulf, M. and Tiller, H.J. (2003) 'A new adhesive technology for all-ceramics', *Dental Materials*, 19, pp. 567–

573. Available at: [https://doi.org/10.1016/S0109-5641\(02\)00106-9](https://doi.org/10.1016/S0109-5641(02)00106-9).
5. Thamrongananskul, N., Busabok, C., Poolthong, S., Uo, M. and Tagami, J. (2014) 'Dental zirconia can be etched by hydrofluoric acid', *Dental Materials Journal*, 33, pp. 79–85. Available at: <https://doi.org/10.4012/dmj.2013-243>.
  6. Sadid-Zadeh, R., Strazzella, A., Li, R. and Makwoka, S. (2021) 'Effect of zirconia etching solution on the shear bond strength between zirconia and resin cement', *The Journal of Prosthetic Dentistry*, 126, pp. 693–697. Available at: <https://doi.org/10.1016/j.prosdent.2020.09.016>.
  7. Kim, H.E., Lim, M.J., Yu, M.K. and Lee, K.W. (2020) 'Changes in bond strength and topography for Y-TZP etched with hydrofluoric acid depending on concentration and temperature conditions', *Medicina*, 56(11), p. 568. Available at: <https://doi.org/10.3390/medicina56110568>.
  8. Colombo, M., Gallo, S., Padovan, S., Chiesa, M., Poggio, C. and Scribante, A. (2020) 'Influence of different surface pretreatments on shear bond strength of an adhesive resin cement to various zirconia ceramics', *Materials*, 13, p. 652. Available at: <https://doi.org/10.3390/ma13030652>.
  9. Yu, M.K., Lim, M.J., Na, N.R. and Lee, K.W. (2020) 'Effect of hydrofluoric acid-based etchant at an elevated temperature on the bond strength and surface topography of Y-TZP ceramics', *Restorative Dentistry & Endodontics*, 45, p. e6. Available at: <https://doi.org/10.5395/rde.2020.45.e6>.
  10. Ansari, S., Jahedmanesh, N., Cascione, D., Zafarnia, P., Shah, K.C., Wu, B.M. and Moshaverinia, A. (2018) 'Effects of an etching solution on the adhesive properties and surface microhardness of zirconia dental ceramics', *The Journal of Prosthetic Dentistry*, 120, pp. 447–453. Available at: <https://doi.org/10.1016/j.prosdent.2017.11.026>.
  11. Sales, A., Rodrigues, S.J., Mahesh, M., Ginjupalli, K., Shetty, T., Pai, U.Y., et al. (2022) 'Effect of different surface treatments on the micro-shear bond strength and surface', *International Journal of Dentistry*, 2022, pp. 1–12. Available at: <https://doi.org/10.1155/2022/1546802>.
  12. Seo, S.H., Kim, J.E., Nam, N.E. and Moon, H.S. (2022) 'Effect of air abrasion, acid etching, and aging on the shear bond strength with resin cement to 3Y-TZP zirconia', *Journal of the Mechanical Behavior of Biomedical Materials*, 134, p. 105348. Available at: <https://doi.org/10.1016/j.jmbbm.2022.105348>.
  13. Carvalho, R.F., Rippe, M.P., Melo, R.M., Bottino, M.A. and Souza, R.O. (2019) 'Resin bond strength to zirconia: effects of surface treatments and resin cements', *General Dentistry*, 67, pp. 71–77.
  14. Saade, J., Skienhe, H., Ounsi, H.F., Matinlinna, J.P. and Salameh, Z. (2020) 'Evaluation of the effect of different surface treatments, aging and enzymatic degradation on zirconia-resin micro-shear bond strength', *Clinical, Cosmetic and Investigational Dentistry*, 2020(12), pp. 1–8. Available at: <https://doi.org/10.2147/CCIDE.S219705>.
  15. Ban, S. (2020) 'Chemical durability of high translucent dental zirconia', *Dental Materials Journal*, 39, pp. 12–23. Available at: <https://doi.org/10.4012/dmj.2019-109>.
  16. Xia, Y., Xu, F., Chen, L., Zhou, X., Cao, Y. and Tong, H. (2019) 'Effect of different surface zirconium oxide treatments on binding strength between zirconia and veneering ceramics', *Zhong nan da xue xue bao. Yi xue ban = Journal of Central South University. Medical Sciences*, 44, pp. 53–58.