European Journal of Prosthodontics and Restorative Dentistry (2023) **31**, 161-168

Is Ammonium Hydrogen Difluoride a Solution for Zirconia Surface Conditioning?

Keywords

SEM
XRD
Acids
Zirconium
Hydrofluoric Acid

Authors

Esra Talay Çevlik *
(DDS, PhD)

Duygu Kürklü Arpaçay §
(DDS, PhD)

Bora Bağiş ‡
(DDS, PhD)

Address for Correspondence

Esra Talay Çevlik *
Email: esra.talay.cevlik@adu.edu.tr

* *Assistant Professor, Department of Prosthodontics, Faculty of Dentistry, Aydın Adnan Menderes University, Aydın, Turkey*

§ *Associate Professor, Department of Prosthodontics, Faculty of Dentistry, İzmir Democracy University, İzmir, Turkey*

‡ *Professor, Private Practice, İzmir, Turkey*

Received: 01.06.2022
Accepted: 29.07.2022

doi: 10.1922/EJPRD_2446Cevlik08

ABSTRACT

Introduction: This research investigated the topographical features and phase transformation of high-translucent monolithic zirconia after different surface conditioning methods. Methods: Zirconia slabs were divided into six groups according to surface treatment method. Group I: etched with hydrofluoric acid (HF); Group II: etched with an experimental acid solution (EAS); Group III: melt-etched with ammonium hydrogen difluoride (AHD); Group IV: air abrasion (AB); Group V: etched with EAS after air abrasion (AB+EAS); Group VI: melt-etched with AHD after air abrasion (AB+AHD). Surface topographies of specimens were documented by scanning electron microscopy (SEM). Tetragonal-to-monoclinic phase transformation was detected by X-ray diffraction and surface evaluation of zirconia specimens; surface roughness and contact angle measurements were performed. The data were statistically analyzed by the Kruskal-Wallis test and post hoc tests (P<0.05). Results: The acid-etched zirconia groups (Group I, II, and III) showed the lowest contact angle and surface roughness values (P<0.05), while the air abrasion groups (Group IV, V, VI) showed the highest. The SEM images also supported these results. Conclusion: Within the limitations of this in vitro study, treating the monolithic zirconia surfaces with EAS or AHD after air abrasion may be recommended to alter the zirconia surfaces.

INTRODUCTION

Yttria-stabilized zirconia polycrystalline (Y-TZP) has many advantages, including high strength, resistance to corrosion, good biological compatibility, and translucency, as a core material in bilayered restorations, as well as in monolithic restorations with lower dimensions.[1] Conversely, a major disadvantage of zirconia is its opaque whiteness that may affect the color of esthetic prostheses.[2] The esthetic appearance of Y-TZP-based restorations is generally achieved by veneering porcelains.[3] The advancement of translucent monolithic zirconia also allowed dentists to use them in some esthetic restorations.

Despite superior mechanical characteristics, zirconia can present a challenge in clinical application. One of the most frequent technical difficulties is the loss of tooth-supported crown retention.[4] The hardness of yttria-stabilized zirconia causes problems with surface enhancement and makes it difficult to ensure appropriate preparation for bonding between the luting cement and the prosthetic restoration.[5] To improve the bond strength between resin cement and zirconia, a variety of techniques have been investigated.[4] Increasing the surface area and surface energy of zirconia surfaces may be the most popular method for improving the bond strength between resin cement and zirconia.[6]

ejprd.org - Published by Dennis Barber Journals.

Several surface treatment techniques, the application of primers or adhesives, and various compositions of acid solutions[7-11] have been investigated in order to increase zirconia's surface and bond strength with resin-based cement. Scaminaci *et al.*[12] have summarized the main zirconia surface treatment methods in detail in their systematic review of current conditioning methods of zirconia.

Airborne aluminum particle abrasion increases bond strength with resin cement for zirconia oxide (ZrO_2)-based ceramics by enhancing surface roughness and eliminating contaminating substances.[13,14] However, surface treatments can generate surface microcracks by weakening the material and making it more susceptible to crack propagation. It is debatable which surface treatment approach is ideal for ZrO_2-based ceramic dental restorations prior to adhesive cementation. As a result, various surface treatments are still required to improve zirconia's adhesive bond strength without jeopardizing its mechanical qualities.[10]

The zirconia surface, which is generally resistant to chemical and mechanical effects, is ideal for testing the effects of various chemicals and acids in different combinations. Recently, several investigations emphasized the improved interfacial adhesion of resin-based materials to zirconia either by etching with potassium hydrogen difluoride (KHF_2) or ammonium hydrogen difluoride (NH_4HF_2).[15-17] Preconditioning could improve interfacial adhesion by increasing surface area; therefore, strong-acid solutions were also evaluated in the recently published studies.[10,14,18] Strong-acid solutions have been used to alter the surface of zirconia, but the results are controversial.[8,10,19] Goyatá *et al.*[8], concluded that a 48% concentration of hydrofluoric acid was more effective than nitric acid/hydrofluoric acid treatment in increasing the roughness of Y-TZP. Ansari *et al.*[10] showed that etching the zirconia surface with Zeta Etching Solution (ZES) for 60 minutes has changed the morphological properties and microstructure of the zirconia, causing the surface to be more irregular.

Lee *et al.*[19] prepared a strongly acidic solution by mixing nitric acid, hydrofluoric acid and hydrogen peroxide to etch the zirconia surface and have stated that etching zirconia with this strongly acidic solution causes significant surface changes. Similar to the study of Lee *et al.*[19] , an experimental acid solution was prepared in the present study.

The purpose of the study was to investigate the topographical features and phase transformation in high-translucent monolithic zirconia after different etching treatments by scanning electron microscopy (SEM), X-ray diffraction (XRD), surface roughness evaluation, and contact angle measurements. The null hypothesis was that NH_4HF_2 and the experimental acid solution and their combinations with air abrasion would not affect the zirconia surface properties in terms of surface roughness.

METHODS

Y-TZP SLABS PREPARATION

A diamond-wafering blade and a dental-cutting machine (Mod Dental, Esetron Smart Robotechnologies, Ankara, Turkey) were used to cut and prepare the pre-sintered Y-TZP blocks (Sirona InCoris ZI, Sirona, Dental Systems GmbH, Bensheim, Germany) into slabs. The surfaces were then ground with silicon carbide paper of up to 600 grit. Specimens were sintered in a high-temperature furnace (Mihm-Vogt-VOGT GmbH & Co. KG, Blankenloch, Germany) at 1510°C for 2 h, according to the manufacturer's instructions. The fully sintered Y-TZP slabs have final measurements of 2 mm × 10 mm × 12 mm. The surfaces were cleaned in ethanol with ultrasonic treatment for 20 min, washed with distilled water, and finally air-dried before the surface treatments.

Y-TZP slabs were randomly divided into six groups according to the surface treatment etched with HF, Group I (HF); etched with EAS, Group II (EAS); etched with NH_4HF_2, Group III (AHD); air abraded with Al_2O_3, Group IV (AB); air abraded with Al_2O_3 then etched with the experimental acid solution, Group V (AB +EAS); air abraded with Al_2O_3 then etched with NH_4HF_2, Group VI (AB +AHD).

- Group I (HF): The specimens were etched with 10% HF (Attaque Gel, Biodinamica, Ibiporã, Paraná, Brazil) for 15 min. The gel was rinsed for 20 s with water and then dried with an oil-free air-water spray.
- Group II (EAS): The specimens were etched for 15 min with an experimental acid solution made up of 70% nitric acid (HNO_3) and 48% HF, with hydrogen peroxide (H_2O_2) added to make a 10% wt. mixed solution. Specimens were then washed with water spray for 15 s and air-dried for 15 s. The production and application of the experimental acid solution were devised based on the findings of a previous study on zirconia etching.[19]
- Group III (AHD): In an agate mortar, NH_4HF_2 (Solvay Chemical, Brussels, Belgium) crystals were ground to a fine powder and dispersed on one flat side of the zirconia slabs. All specimens were powder-coated with 70.0 ±15.0 mg NH_4HF_2. The powder-coated specimens were placed on a metal plate and heated in a preheated furnace (Programat® P310, Ivoclar Vivadent AG, Schaan, Liechtenstein) at 170°C for 10 min. (NH_4HF_2; melting point of 125°C). After 10 min, the plate with the specimens was removed from the oven and allowed to cool on the bench. To ensure no traces of the treatments remained on the inner surfaces, etched specimens were washed with water spray for 15 s and air-dried for 15 s.
- Group IV (AB): The specimens were air abraded with 50 µm aluminum oxide (Al_2O_3) particles (DynaFlex, Saint Ann, MO, USA) at 0.35 MPa pressure for 20 s according to instructions for pretreatment of the Sirona InCoris ZI

restoration before adhesive bonding. At a distance of 10 mm, the blasting nozzle tip was perpendicular to the zirconia surface.

- Group V (AB +EAS): The specimens were exposed to the Group IV (AB) treatment, followed by the Group II (EAS) treatment.
- Group VI (AB +AHD): The specimens were exposed to the Group IV (AB) treatment, followed by Group III (AHD) treatment.

SURFACE TOPOGRAPHY AND CHARACTERIZATION

SEM ANALYSIS

The specimens were sputter-coated with a layer of gold-palladium (Sputter Coater 108 Auto, Cressington Scientific Instruments Ltd., Watford, England). After all surface treatments, the zirconia surfaces (n = 1) were examined using a scanning electron microscope (SEM-ZEISS LS-10, England, UK) at magnifications of 1000X and 5000X.

XRD ANALYSIS

XRD analysis was performed (n = 1) to identify the crystalline phases of the surface-treated specimens using an X-ray diffractometer (Bruker D8 Advance, Bruker AXS, Karlsruhe, Germany) operating at 40 mA. Data were collected with a step-size of 0.02° over a 2θ range of 20-40° and analyzed using computer software (Diffrac Eva, Bruker AXS, Karlsruhe, Germany) and the index of the International Center for Diffraction Data (ICDD, Newton Square, PA, USA).

SURFACE ROUGHNESS MEASUREMENTS

The surface roughness (R_a) of specimens from each group was measured after the surface treatment process. The measurements were done three times and the mean values were recorded. Results were expressed as the arithmetical mean deviation (R_a) of the roughness profile measured using a contact profilometer with a cutoff value of 0.25 mm and a transverse length of 5 mm, based on international standards.[20]

CONTACT ANGLE MEASUREMENTS

Three zirconia specimens from each treatment group were sprayed with 4 μL of distilled water droplets. All contact angle measurements were taken at room temperature using a goniometer (DataPhysics OCA-Series, Filderstadt, Germany). The average contact angle of 120 measurements from the left and another 120 measurements from the right side of the droplets was calculated using the software accompanying the goniometer.

STATISTICAL ANALYSIS

The Kruskal-Wallis test (IBM SPSS Statistics 24, IBM Corp. Armonk, NY, USA) followed by the Tamhane test were performed as a non-parametric test to evaluate the differences among the groups. The statistical significance level was set at α = .05.

RESULTS

SEM images (*Figure 1A-F*) revealed the morphological appearance of zirconia specimens exposed to different surface treatments. With air abrasion, large grooves and voids were observed on the surface of zirconia slabs. Group III (AHD), which was powder-coated with NH_4HF_2 and then heated at 170°C for 10 min, demonstrated notched and roughened irregularities on the zirconia surface. From a microscopic perspective, EAS, AHD and AB+AHD caused greater surface roughness of zirconia than the other treatment methods. Zirconia presented a more irregular surface following treatment with EAS (Group II) or AB+EAS (Group V) than with HF (Group I) treatment.

XRD was used to detect the phases present after surface treatment and whether the surface treatment procedures caused a phase transformation. The X-ray diffractograms differed among the groups (*Figure 2*). With respect to the relative amounts of the monoclinic phase in the surface-treated specimens, the highest ratio was observed after AB+EAS (21%), followed by AB +AHD (13%), AB (12%), AHD (8%), EAS (4%), and HF (3%). In the XRD pattern, the main peak of the monoclinic phase appeared at 2θ of 29°-32°, and 31° for the tetragonal phase.

The median, minimum, and maximum surface roughness (Ra in μm) values of the groups after the designated treatment protocol were given in Table 1. There were substantial statistical differences between groups, with Group IV (AB), Group V (AB +EAS), and Group VI (AB +AHD) having the highest surface roughness.

The water contact angles of the six test groups were given in Figure 3. Group I (HF) and Group II (EAS) exhibited the lowest contact angle values while Group IV (AB) had the highest contact angle followed by Group V (AB+EAS), Group VI (AB+AHD) and Group III (AHD), respectively. It can be observed that all experimental groups had a contact angle of less than 90°, characterizing hydrophilic materials.

DISCUSSION

ZrO_2-based ceramics are gaining popularity due to their superior mechanical properties, but there are still some clinical challenges to overcome, such as the problems of adhesion to resin materials. Compared to glass-ceramics, etching these materials is difficult. Increasing the surface roughness increases the surface energy and thus the wettability of ZrO_2-based ceramics and their interfacial adhesion with resin materials.[9] Air abrasion, creates micro-cavities and fissures that facilitate micromechanical retention for adhesive resin but can have detrimental effects on the materials.

To understand the effect of etching materials, EAS and AHD were applied alone and in combination with AB to the translucent monolithic zirconia specimens in this study. According to the results, the null hypothesis was rejected. Some changes occurred in the surface characteristics of zirconia after

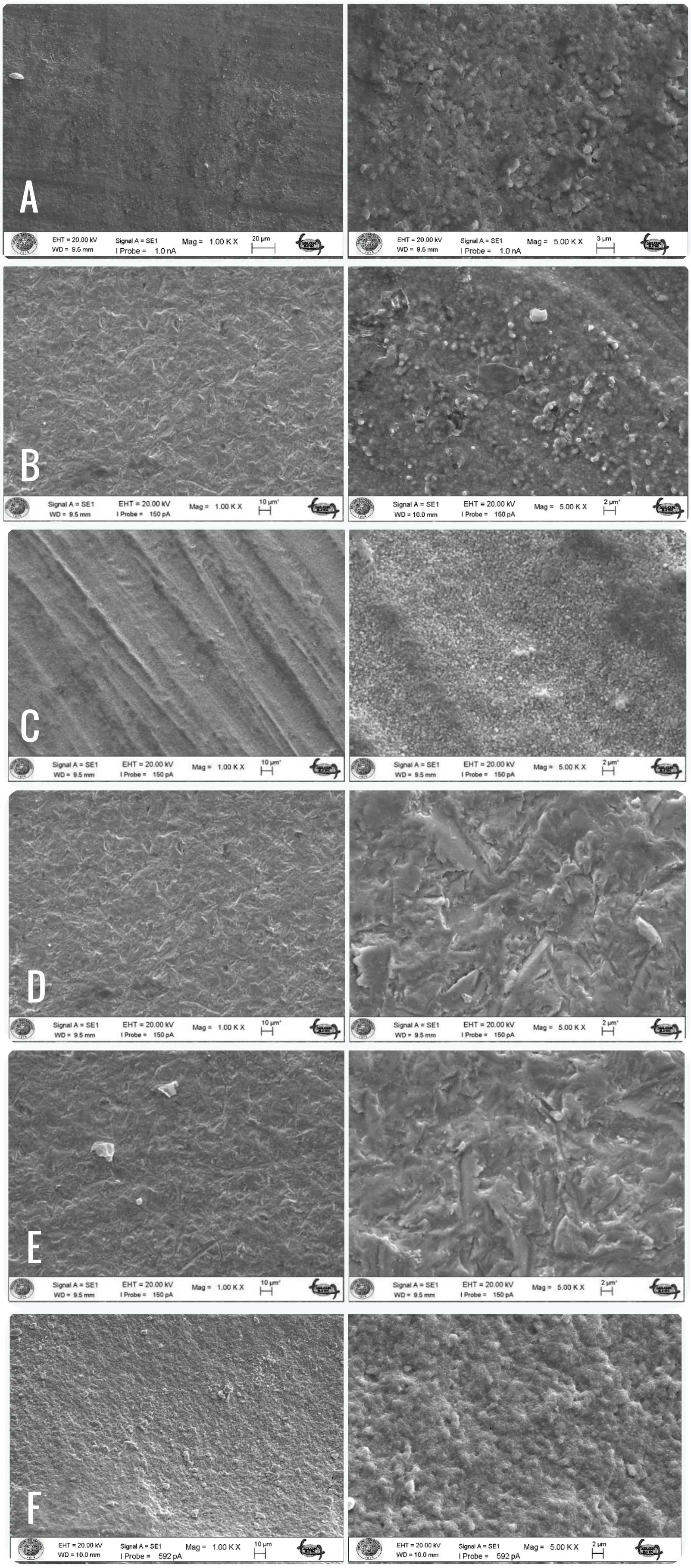

Figure 1: SEM images of the zirconia specimens after different surface treatments. (A) HF-etched, Group I: HF; (B) EAS-etched, Group II: EAS; (C) AHD powder-etched, Group III: AHD; (D) Air abraded, Group IV: AB; (E) EAS-etched after air abrading, Group V: AB +EAS; (F) AHD powder-etched after air abrading, Group VI: AB +AHD.

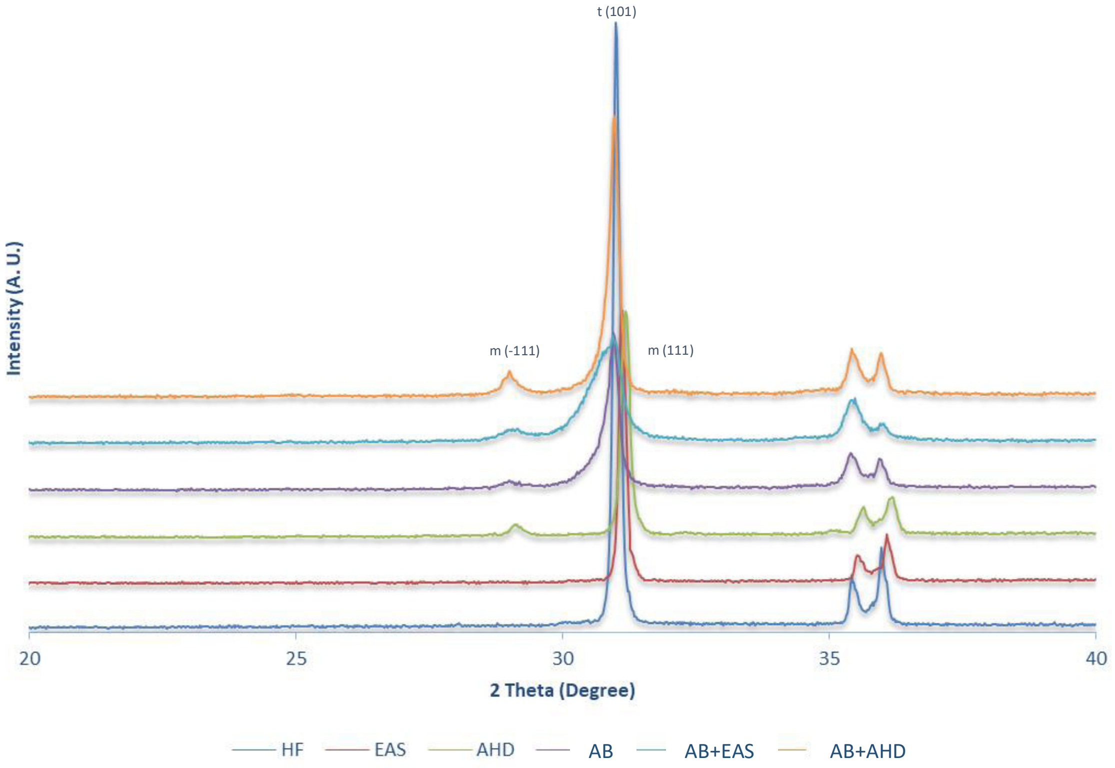

Figure 2: A demonstration of XRD patterns of the specimens.

Table 1. The median, minimum, and maximum surface roughness (Ra in µm) values of the groups.

Surface Treatment Methods	Median	Minimum	Maximum
HF	0.24^{b}	0.22	0.26
EAS	0.27^{b}	0.22	0.28
AHD	0.25^{b}	0.23	0.29
AB	0.55^{a}	0.54	0.58
AB+EAS	0.62^{a}	0.56	0.65
AB+AHD	0.51^{c}	0.49	0.52

The same superscript lowercase letters in the same column denote groups that were not significantly different (Tamhane test, $P > 0.05$).

the surface treatments. AB was more effective than HF, EAS, or AHD to increase the roughness of zirconia specimens, but there were still promising results, with higher surface roughness values for EAS and AHD when used after AB than alone. The SEM evaluations supported these results, but the contact angle values of acid-etched zirconia groups were lower than the air abraded or the air abraded then etched (combination) groups.

It is critical to establish safe and standardized zirconia adhesive cementation protocols to properly complete the conservative/prosthetic treatment plan, especially when the preparation is not retentive or when the mechanical characteristics of the tooth-prosthesis complex are important.[12] In recent years, various surface treatments of zirconia materials have been studied for their effects on surface characteristics and the bonding to different types of cement. The zirconia surface is impervious to mechanical and chemical factors; these properties make the material suitable for undertaking fundamental research on the application of different combinations of chemicals (e.g., acids) and alternative treatment methods.[5] Nonetheless, the best zirconia surface pretreatment is still debatable.[9]

Particle deposition techniques are commonly used to alter the surfaces of the materials. The abrasives most commonly used are Al_2O_3 or silicon dioxide (SiO_2) particles varying from 30 to 250 mm.[21] Airborne-particle abrasion (APA) with Al_2O_3 is the preferred treatment method for high-strength ceramic

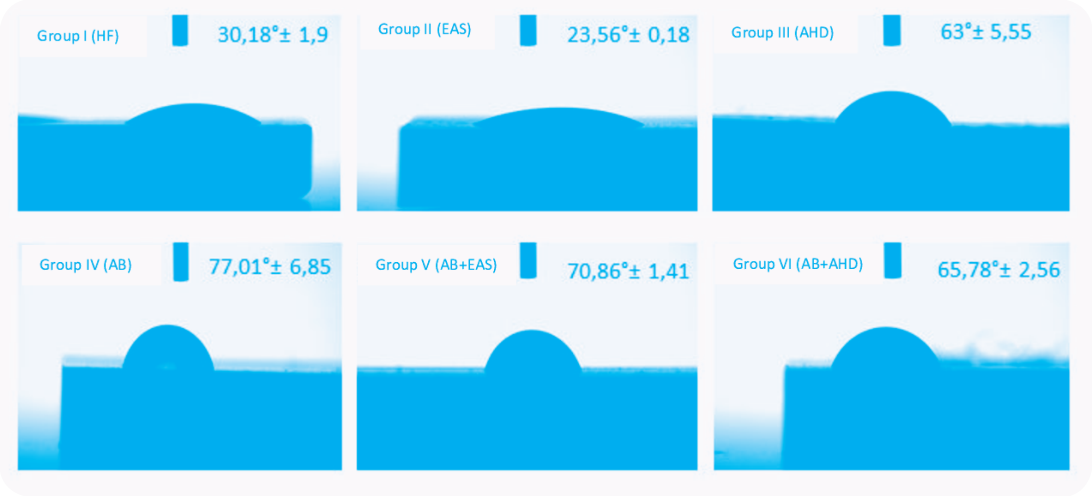

Figure 3: Representative images of water contact angle measurements of zirconia specimens exposed to different surface treatments.

materials. However, APA can cause surface defects, cracks, and damage. As a result, zirconia's mechanical properties may be affected.[22] It is recommended that APA be carried out using appropriate pressure, distance from the source, and particle size parameters.[12]

Zirconia is resistant to chemical etching due to its chemistry and high crystalline concentration. Chemical surface-conditioning with HF has been shown to be ineffective for zirconia, but the concentration and temperature of HF can create micromorphological changes on zirconia surfaces.[23] By prolonging the immersion duration (up to 24 h)[23] or raising the temperature (80°C) for up to 30 min,[5] HF (9.5%) solution has been utilized to etch zirconia surfaces.

Therefore to find an ideal chemical solution, previous studies have examined different acid solutions or combinations with different concentrations to alter the zirconia surfaces.[5] One approach uses piranha solution, a powerful oxidant that hydroxylates most surfaces, rendering them highly hydrophilic. This mixture of sulfuric acid (H_2SO_4 96%) and H_2O_2 (30%) is useful in conditioning zirconia surfaces and increasing retention when paired with other surface treatments.[7,24,25] Zandparsa *et al.*[26] found that the piranha-etched group of zirconia specimens had significantly lower bond strength values than the APA group. One possible reason was that the etching solution hydroxylated and rapidly cleaned the surface without causing undercuts, which are critical in forming micromechanical interlocking with the cement.[26]

It has been suggested that the Zircos-E etching system (ZSAT: Zirconia Surface Architecturing Technique, M&C Dental Co., Eunjin Chemical Co., Seoul, Korea), a mixture of HNO_3 and HF solutions that can be applied at room temperature, could increase the shear-bond strength of resin cements to zirconia.[14] Zeta Etching Solution (Eunjin Chemical Co.) is a relatively new zirconia etching solution that contains HF, hydrochloric acid (HCl), H_2SO_4, HNO_3, and phosphoric acid (H_3PO_4). Researchers found that etching with Zeta Etching Solution for 30 min noticeably improved the shear bond strength of highly translucent Prettau Anterior zirconia restorations.[10]

Casucci *et al.*[9] devised an alternative experimental mixture solution for surface roughening of ZrO_2 ceramics. The hot (100°C) solution of 37% HCl and ferric chloride ($FeCl_3$) increased the mean arithmetic profile deviation (Ramean) roughness parameter.[9] Etching with a highly corrosive acid to chemically dissolve particles on the zirconia surface may be desirable because it allows for a more objective application and more consistent results than AB.[19]

A rough surface of zirconia grains may also be created by melt-etching, which advances the micromechanical retention of coupling agents and luting cement.[27] Recently, Ruyter *et al.*[16] published a melt-etching method for treating zirconia surfaces to improve resin cement adhesion strength. Melt-etching of the fluoride compounds KHF_2 or NH_4HF_2 on Y-TZP surfaces resulted in a rough-etched surface suitable for good and durable adhesion to a zirconia or dentin abutment.[16] KHF_2 and NH_4HF_2 are difluorides with symmetric hydrogen bonds with linear anions. The dissolution of ZrO_2 in molten NH_4HF_2 is responsible for the etching efficiency. Melt-etching may have also resulted in a fluoride surface with different surface energy than the mechanical treatments and a higher affinity for the resin cement.[16] Kvam *et al.*[17] observed significantly higher bond strength values with the melt-etch method compared to the air-abraided and ground groups.

Akazawa *et al.*[15] evaluated the effect of etching with KHF_2 and NH_4HF_2 on the bond strength of a self-polymerizing methyl methacrylate resin bonded to zirconia. According to the results, KHF2 and NH_4HF_2 exhibited higher bond strength and surface free energy (as measured by the contact angle) but lower surface roughness compared to the alumina-blasted group.[15] In general, when the liquid has a smaller contact angle with the solid, the surface free energy of the solid is larger, and thus the solid has better wettability for bonding; however, the surface roughness and the surface free energy are not correlated in ceramic bond strength.[15] In the current study, the NH_4HF_2 group (Group III, AHD) exhibited lower roughness and contact angle values than all three air-abraded groups. Although the roughness values were minimal, these results suggest that the effect of the different surface treatments on the wettability may, in turn, influence the bonding ability.

Tribochemical coating is an efficient strategy to roughen glass-infiltrated-based ceramics, but it seems less effective for zirconia ceramics.[6] Electrical discharge machining is a novel technology for eroding material using electrical impulses in a dielectric liquid. However, surface cracks can be identified via SEM analysis.[28] In the SEM images of the present study, similar surface irregularities and shallow pits were found in the surfaces of HF and EAS groups. Large grooves and voids were observed on the surface of AB, AB+EAS and AB+AHD groups. Roughened irregularities on the zirconia surface were observed in the AHD and AB+AHD groups. AHD is a material used to make ground glass. Therefore, it is thought that AHD roughened the zirconia surface and AHD and AB+AHD groups seem roughened over other groups.

Surface treatment procedures, such as Al_2O_3-air abrasion, increase the residual compressive stress on the surface, which triggers the phase transformation of $t\text{-}ZrO_2 \rightarrow m\text{-}ZrO_2$. The residual compressive stress may affect the mechanical properties of the material.[29] As a result, evaluating phase transformation in surface treatment studies is critical. According to the XRD results of the present study, it was observed that the relative amount of monoclinic phase content change was more in the AB+EAS, AB+AHD and AB groups than in others. This result may be related to the fact that air abrasion treatment may cause more phase transformation than other surface treatment procedures.

A range of adhesion techniques was discovered in the literature, including various zirconia treatment approaches. However, a standardized adhesive cementation process that produces definitive and reliable outcomes is yet to be found. Therefore, making clinical suggestions is challenging. This study aimed to evaluate the topographical features of high-translucent monolithic zirconia after different surface conditioning methods. Further studies related to adhesion to zirconia are recommended to take the study one step further. One of the limitations of this study is that NH_4HF_2 decomposes during melting, whereas KHF_2 is probably relatively stable up to and above the melting point. Only NH_4HF_2 was utilized in this study.

CONCLUSIONS

Within the constraints of this study, it is conceivable to conclude that AHD and EAS, as novel surface conditioning methods, affected the zirconia ceramic surfaces. These findings were supported by SEM analysis. Evaluating the effect of new conditioning methods on the mechanical properties of materials is imperative, particularly over an extended period. Additionally, bond-strength studies will allow improving predictions about the clinical efficacy of these surface treatments. In the context of the current study, AHD and EAS offer promising findings that will need to be confirmed by additional *in vitro* and *in vivo* research.

ACKNOWLEDGMENTS

The authors would like to thank Prof. Dr. Arife Alev Karagözler and her Research Assistant Doctor Rukiye Yavaşer at the Department of Chemistry, Faculty of Arts and Sciences, Aydın Adnan Menderes University, Turkey, for their kind assistance with preparing the experimental-acid solution. The authors would also like to thank Yorglass Satinated Glass Manisa Plant for supplying the NH_4HF_2 used in this study. No financial support was received either directly or indirectly by the authors. All authors declare that there are no competing interests. This study was self-funded.

REFERENCES

1. Keshvad, A. and Hakimaneh, S.M.R. Microtensile bond strength of a resin cement to silica-based and Y-TZP ceramics using different surface treatments. *J Prosthodont* 2018; **27**:67-74.
2. Kim, G-H., Park, S-W., Lee K., *et al.* Shear bond strength between resin cement and colored zirconia made with metal chlorides. *J Prosthet Dent* 2015; **113**: 603-608.
3. Luo, X.P. and Zhang, L. Effect of veneering techniques on color and translucency of Y-TZP. *J Prosthodont* 2010; **19**:465-470.
4. Ishii, R., Tsujimoto, A., Takamizawa, T., *et al.* Influence of surface treatment of contaminated zirconia on surface free energy and resin cement bonding. *Dent Mater J* 2015: 2014-2066.
5. Smielak, B. and Klimek, L. Effect of hydrofluoric acid concentration and etching duration on select surface roughness parameters for zirconia. *J Prosthet Dent* 2015; **113**:596-602.
6. Kern, M. and Wegner, S.M. Bonding to zirconia ceramic: adhesion methods and their durability. *Dent Mater* 1998; **14**:64-71.
7. Lohbauer, U., Zipperle, M., Rischka, K., *et al.* Hydroxylation of dental zirconia surfaces: characterization and bonding potential. *J Biomed Mater Res B Appl Biomater* 2008; **87**:461-467.
8. Goyata, F., Galvao, Y., Simoes, T.R., *et al.* Effect of surface treatments with acid solutions on the surface roughness of an Yttrium-Tetragonal Zirconia Polycrystal. *J Clin Exp Dent* 2018; **10**:e367-e370.
9. Casucci, A., Osorio, E., Osorio, R., *et al.* Influence of different surface treatments on surface zirconia frameworks. *J Dent* 2009; **37**:891-897.
10. Ansari, S., Jahedmanesh, N., Cascione, D., *et al.* Effects of an etching solution on the adhesive properties and surface microhardness of zirconia dental ceramics. *J Prosthet Dent* 2018; **120**:447-453.
11. Giti, R. and Zarkari, R. The effect of a zirconia primer on the shear bond strength of Y-TZP ceramic to three different core materials by using a self-adhesive resin cement. *J Indian Prosthodont Soc* 2019; **19**:134.
12. Scaminaci Russo, D., Cinelli, F., Sarti, C., *et al.* Adhesion to zirconia: a systematic review of current conditioning methods and bonding materials. *Dent J* 2019; **7**:74.
13. Kern, M., Barloi, A. and Yang, B. Surface conditioning influences zirconia ceramic bonding. *J Dent Res* 2009; **88**:817-822.
14. Cho, J.H., Kim, S.J., Shim, J.S., *et al.* Effect of zirconia surface treatment using nitric acid-hydrofluoric acid on the shear bond strengths of resin cements. *J Adv Prosthodont* 2017; **9**:77.

15. Akazawa, N., Koizumi, H., Nogawa, H., *et al.* Effect of etching with potassium hydrogen difluoride and ammonium hydrogen difluoride on bonding of a tri-n-butylborane initiated resin to zirconia. *Dent Mater J* 2019; **38**:540-546.
16. Ruyter, E.I., Vajeeston, N., Knarvang, T., *et al.* A novel etching technique for surface treatment of zirconia ceramics to improve adhesion of resin-based luting cements. *Acta biomater Odontol Scand* 2017; **3**:36-46.
17. Kvam, K., Irkayek, A., Vangaeva, E., *et al.* Comparison of sandblasted, ground and melt-etched zirconia crowns regarding adhesion strength to resin cement. *Biomater Investig Dent* 2019; **6**:1-5.
18. Her, S-B., Kim, K.H., Park, S.E., *et al.* The effect of zirconia surface architecturing technique on the zirconia/veneer interfacial bond strength. *J Adv Prosthodont* 2018; **10**:259.
19. Lee, Y., Oh, K.C., Kim, N.H., *et al.* 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**:3564275.
20. ISO E. 4287–*Geometrical Product Specifications (GPS)–Surface Texture: Profile Method–Terms, Definitions and Surface Texture Parameters. International Organization for Standardization*: Geneva, Switzerland 1997.
21. Ozcan, M. and Bernasconi, M. Adhesion to zirconia used for dental restorations: a systematic review and meta-analysis. *J Adhes Dent* 2015; **17**:7-26.
22. Zhang, Y., Lawn, B.R., Rekow, E.D., *et al.* Effect of sandblasting on the long-term performance of dental ceramics. *J Biomed Mater Res B Appl Biomater* 2004; **71**:381-386.
23. Thamronganskul, N., Busabok, C., Poolthong, S., *et al.* Dental zirconia can be etched by hydrofluoric acid. *Dent Mater J* 2014; **33**:79-85.
24. Sirghi, L., Kylián, O., Gilliland, D., *et al.* Cleaning and hydrophilization of atomic force microscopy silicon probes. *J Phys Chem B* 2006; **110**:25975-25981.
25. Yoshida, K., Tsuo, Y. and Atsuta, M. Bonding of dual-cured resin cement to zirconia ceramic using phosphate acid ester monomer and zirconate coupler. *J Biomed Mater Res B Appl Biomater* 2006; **77**:28-33.
26. Zandparsa, R., Talua, N.A., Finkelman, M.D., *et al.* An *in vitro* comparison of shear bond strength of zirconia to enamel using different surface treatments. *J Prosthodont* 2014; **23**:117-123.
27. Sagen, M.A., Kvam, K., Ruyter, E.I., *et al.* Debonding mechanism of zirconia and lithium disilicate resin cemented to dentin. *Acta biomater Odontol Scand* 2019; **5**:22-29.
28. Matinlinna, J.P., Heikkinen, T., Özcan, M., *et al.* Evaluation of resin adhesion to zirconia ceramic using some organosilanes. *Dent Mater* 2006; **22**:824-831.
29. Inokoshi, M., Vanmeensel, K., Zhang, F., *et al.* Aging resistance of surface-treated dental zirconia. *Dent Mater* 2015; **31**:182-194.