The Effects of Acid Etching Time on Shear Bond Strength of 3Y-TZP and 5Y-TZP Zirconia to Composite Resin

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Abstract

To investigate the effects of different etching time with 9.5%HF on shear bond strength between 3Y-TZP and 5Y-TZP to composite resin.

3Y-TZP and 5YTZP specimens were randomly divided into control group and experimental groups including sandblast group and 9.5%HF immersion for 15 min, 30 min, 1 h, 2 h, 3 h, or 24 h. Three specimens from each group were examined for surface roughness by profilometer. Scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) were used to characterize the effects of such treatments. The flowable composite resin was apply on treated zirconia surface of control and experimental group. The shear bond strength of zirconia to flowable composite resin was measured with a universal testing machine at a crosshead speed 0.5 mm/min. Statistical analysis was performed with two-way ANOVA and Dunnett's test ($P < 0.05$).

There was no significant difference between control and HF-treated for 15 min to 1 h in 3Y-TZP and there was no significant difference between control and HF-treated for 15 min in 5Y-TZP ($P < 0.05$). However, the control group showed significantly lower shear bond strength than HF-treated over 2 h in 3Y-TZP and 30 min in 5Y-TZP.

HF can roughen surface of 3Y-TZP and 5Y-TZP and improve bond strength between zirconia and composite resin with the treatment time of at least 2 h in 3Y-TZP and 30 min in 5Y-TZP.

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Introduction

Fixed dental prostheses (FPDs) is the most advocated treatment to replace missing teeth due to caries or trauma[1]. For many decades, porcelain fused to metal (PFM) restorations has been the first choice of rehabilitation because of their esthetics, durability, and fit to the abutments[2]. Nowadays, as a result of the demand for natural esthetics and biocompatibility, metal-free prostheses including zirconia or zirconium oxide (ZrO_2) have become more preferable. Zirconia has been introduced in prosthetic dentistry for the fabrication of FPDs due to its exceptional mechanical properties[3-5], esthetic appearance, and biocompatibility[4].

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Zirconia is a polymorphic material occurring in three temperature-dependent forms: monoclinic, cubic, and tetragonal[6]. The transformation from the tetragonal to the monoclinic form increases in volume preventing the crack propagation[3, 6]. Zirconia was also modified to Yttria (Y_2O_3) tetragonal zirconia polycrystal (Y-TZP) to stabilize the tetragonal form at the room temperature and improve the physical properties[7]. Zirconia can currently be categorized into four generations according to mechanical and optical properties[8]. The 1st generation of zirconia is a 3 %mol yttria-stabilized tetragonal zirconia polycrystals (3Y-TZP). The 2nd generation is a 3 %mol yttria with reduced alumina content for improving the translucency. The 3rd generation is a 5 %mol yttria-stabilized zirconia polycrystals (5Y-TZP) with the higher translucency, however, inferior mechanical properties due to the greater the number of cubic crystals. The 4th generation is a 4 %mol yttria-stabilized tetragonal zirconia polycrystals (4Y-TZP).

Zirconia has poor bond strength with conventional cement because of their inertness. The surface treatments of zirconia were highly recommended for increasing surface roughness, surface energy and wettability to improve bond strength. The treatments were divided into mechanical and chemical surface treatments[4, 9]. Sandblasting with aluminum oxide (Al_2O_3) particles is the most common method for providing micro-mechanical interlocking for enhanced bonding of zirconia[4, 5, 10]. A combination of sandblasting and functional monomer such as 10-Methacryloyloxydecyl dihydrogen phosphate (MDP) is the most reliable method for improve bond strength of zirconia, especially for long-term success rate[4, 10-12]. However, the inertness of zirconia results in weak adhesion to the variety of substrates[4], thus bonding to zirconia has become an interesting topic in recent years. Different treatments of the zirconia, application of primers or adhesives, and various types of resin cements have been studied[13].

Hydrofluoric acid (HF) is a chemical compound commonly used for surface roughening of silica-based ceramics[5, 14]. Several studies have reported that zirconia-base ceramics are resistant to acid etching due to silica-free composition structure[5, 15-18]. Furthermore, many studies have reported that surface treatment with HF can change morphology of zirconia surface[19, 20]. Additionally, a previous study[14] has reported that acid etching with HF can change micro-morphological surface of zirconia when immersion in 9.5% HF at 25°C for 1, 2, 3, or 24 h, immersion in 9.5% HF at 80°C for 1, 3, 5, or 30 min, and immersion in 48% HF at 25°C for 30 or 60 min. Another study[21] have also found that 9.5%HF solution can increase roughness of zirconia surface after 15 mins of immersion, while 5%HF solution is not able to roughen zirconia surfaces.

Many studies recommend a combination of mechanical and chemical treatment which tends to produce higher bond strength such as sandblasting together with MDP-containing primer application[4, 11-13, 18]. Moreover, previous studies[22, 23] have reported that sandblasting with alumina particle with an addition of MDP-based adhesive system can improve shear bond strength (SBS) of veneering composite to zirconia. In addition, various universal bonding systems can be used for different dental materials including zirconia[24-26] such as Z-Prime Plus (Bisco, USA), Scotchbond Universal (3M ESPE, USA) and All Bond Universal (Bisco, USA). Surface treatment of zirconia with HF has remained controversial because of the absence of glassy phase in zirconia. Previous studies[14, 21] have shown that HF can roughen zirconia surface with longer treatment time and higher concentration of HF, or higher temperature. However, only few studies have determined the effects on bond strength of HF-treated zirconia and the difference between the effects on 3Y-TZP and 5Y-TZP has not been investigated yet. Thus, the aim of this study is to investigate the effects of different acid etching time with 9.5%HF on shear bond strength between 3Y-TZP and 5Y-TZP to composite resin.

Materials and methods

The materials used in this study are listed in Table 1.

Specimen preparation

Fully-sintered 3Y-TZP and 5Y-TZP zirconia disks were prepared into square-shaped specimens (7×7×1 mm^3) and polished with sandpapers 100 400 and 600 grit size sequentially under a grinder polisher (MaPao™ 160E, Qingdao, China) and water-cooling, then ultrasonicated in distilled water for 10 min.

The specimens were randomly assigned into 8 groups for 3Y-TZP and 5Y-TZP including;

Group 1 was used as a control (C).

Group 2 was sandblasted (SB) with 50 μm Al_2O_3 particles at 0.38 MPa pressure and 10 mm distance for 20 s (sandblasting machine, Renfert basic classic, Hilzingen, Germany).

Group 3 was immersed in 9.5%HF for 15 min. (HF15m)

Group 4 was immersed in 9.5%HF for 30 min. (HF30m)

Group 5 was immersed in 9.5%HF for 1 h. (HF1h)

Group 6 was immersed in 9.5%HF for 2 h. (HF2h)

Group 7 was immersed in 9.5%HF for 3 h. (HF3h)

Group 8 was immersed in 9.5%HF for 24 h. (HF24h)

After the immersion, specimens were rinsed with deionized water for 1 min, and ultrasonicated in deionized water for 10 min.

Elemental composition analysis

A specimen was gold sputter coated and three surface areas were randomly selected for Energy dispersive X-Ray spectrometer (EDS) with SEM at 1,000× magnification for analyzed elemental composition.

Surface roughness

Three specimens were randomly selected from each group, surface roughness (Ra in µm) was measured using a profilometer (Mitutoyo Surftest SJ-310, Kanagawa, Japan) with a cut-off length 1.5 mm and a measuring length 4 mm at 5 different locations for each specimen. The average Ra values was then determined.

Surface topography

Two specimens were randomly selected for gold sputter coating for the scanning electron microscope (SEM) examination (Jeol JSM-IT300, Massachusetts, USA) at 10,000× magnification and 15 kV accelerating voltage.

Shear bond strength

Ten specimens were treated with Clearfil Ceramic Primer Plus, the primer was applied to each specimen using a micropipette (5 µl) and smeared into thin coat with a microbrush, left for 20 s, then air-dried for 10 s. The specimen surface was ensured that it was completely dried. Each specimen was placed with a perforated sticker (3 mm diameter) to define bonding area. The plastic tube with 3 mm inner diameter and 3 mm height was placed on intact zirconia surface, filled with flowable composite resin, and light-polymerized from 4 directions for 20 s (total 80 s) using a visible light curing source (EliparTM, 3M Deutschland GmbH, Neuss, Germany) at a light intensity > 600 mW/cm^2, and left for further polymerization. After 1 h, the stickers were removed carefully from specimens. Before testing, all specimens were stored in distilled water at 37°C for 24 h. The shear bond strength of zirconia to composite resin was tested by a universal testing machine (Instron 5566, Massachusetts, USA) at a crosshead speed of 0.5 mm/min according to the guidelines of ISO/TS 11405:2003. The load at failure was recorded and converted to shear bond strength expressed in Mega Pascals (MPa). After the bond test, failed specimens were classified mode of failure into 3 groups: adhesive failure between the zirconia and composite resin, cohesive failure within composite resin, or mixed cohesion and adhesion failure under a stereomicroscope with digital camera (Olympus SZX7 & SZ2-ILST led illuminator stand & E-330, Tokyo, Japan) at 20x magnification.

Statistical analysis

Means and standard deviations of the surface roughness and shear bond strength were calculated, and mean values were compared by two-way ANOVA and Dunnett's test at a significance level of P < 0.05. Statistical analysis was performed using SPSS version 26 (IBM SPSS Inc., Chicago, Illinois).

Results

Elemental composition analysis

The results of elemental composition analysis with EDS under SEM at 1,000× magnification are presented in Table 2. Sandblast group showed a higher alumina composition than the control, while HF-treated group showed a higher fluorine composition which increased according to longer immersion durations.

Surface roughness

The results of surface roughness analysis are presented in Table 2. Statistical analysis was performed using two-way ANOVA. The quality of variances was not assumed. Therefore, post-hoc test (Dunnett T3) was performed. There was no significant difference in surface roughness between the control and HF-treated groups except in HF-treated for 24 h (P < 0.05). The highest roughness was observed in sandblast group of 3Y-TZP (1.59±0.28) and 5Y-TZP (1.23±0.20). The 3Y-TZP treated with sandblasting showed significantly higher roughness than the 5Y-TZP group (P < 0.05). However, the 3Y-TZP exhibited significantly lower surface roughness than 5Y-TZP with HF-treated for 24 h. The interaction term in a two-way ANOVA revealed a statistically significant interaction between the effects of 3Y-TZP, 5Y-TZP and surface treatment methods on surface roughness (F=19.054, P<0.001).

Surface topography

The SEM images of 3Y-TZP experimental groups showed the different surface morphologies as shown in Figure 1. Control group surface (Figure 1a) exhibited the grooves and scratches caused by the polishing process on a smooth surface. While sandblast group (Figure 1b) clearly showed rough and irregular surface compared to the control group. The 3Y-

TZP surface after treated with 9.5% HF for 15 min (Figure 1c), showed small and shallow pits on a smooth surface. In addition, with longer times, the images of 3Y-TZP treated for 30 min, 1 h, 2 h, and 3 h (Figure 1d-g) portrayed a rougher surface with erosion of superficial surface in irregular shape and expansion of the pits which increased according to longer immersion durations. Especially in HF-treated for 24 h, the SEM image (Figure 1h) showed fine grains and large inter-grain space that created porosity on surface. Similarly, SEM images of 5Y-TZP surfaces in Figure 2 showed the scratches on a smooth surface of control group. (Figure 2a) The 5Y-TZP surface after treated with 9.5% HF for 15 min (Figure 2c), showed irregular grain structure. Additionally, 5Y-TZP HF-treated for 30 min, 1 h, 2 h, 3 h, or 24 h (Figure 2d-h) exhibited increased roughness with dislodgment of superficial structure and expansion of the inter-grain space with the longer immersion time. Furthermore, immersion for 3 h (Figure 2g) showed irregular sharp surface and immersion for 24 h (Figure 2h) showed large and deep holes on irregular sharp surfaces which are different from 3Y-TZP.

Shear bond strength (SBS)

The results of SBS analysis of 3Y-TZP and 5Y-TZP are presented in Table 3. The lowest SBS was observed in control group of 3Y-TZP (4.39±1.04) and 5Y-TZP (3.88±0.53). SBS analysis of 3Y-TZP indicated that there was no significant difference between control and HF-treated for 15 min to 1 h. Also, there was no significant difference between control and HF-treated for 15 min in 5Y-TZP ($P < 0.05$). Interestingly, the control group showed significantly lower shear bond strength than HF-treated over 2 h in 3Y-TZP and 30 min in 5Y-TZP. However, there was no statistically significant interaction between the effects of 3Y-TZP, 5Y-TZP and surface treatment on SBS ($F=1.637$, $P=0.13$).

Mode of failure

The percent of mode of failure are presented in Table 3. The failure mode of the HF-treated group majority was in the main adhesive failure. However, the sandblast group of 5Y-TZP exhibited both adhesive and mixed failure mode equally. Figure 3 showed the images of mode of failure after shear bond testing under stereomicroscope at 20x magnification (Figure 3a, 3b) and under SEM at 25x magnification (Figure 3c, 3d). Figure 3a, 3c showed adhesive failure at the interface between zirconia (Zr) and composite resin. Figure 3b, 3d showed mixed type with most part of adhesive failure and a small part of cohesive failure within the composite resin (CR). Figure 3e taken at 1,000x magnification showed that the composite resin has been completely removed from the zirconia surface after the shear bond strength test. Lastly, Figure 3f showed combination of adhesive failure and cohesive failure within the composite resin. Some residual composite resin bonding on the zirconia surface was also observed after the shear bond strength test.

Discussion

HF is an inorganic acid practical of etching glass and removing oxides from metals[27]. HF is typically used at 4% to 10% concentration, which considered safe for dental procedures, including intraoral repair of restorations[28]. The glassy matrix is selectively eliminated by reaction of HF with SiO_2 to form silicon fluorides, then the crystalline phase was exposed resulting in surface roughness[27, 29, 30], so etching procedures are used to increases surface area and wettability accessible for mechanical interlocking[5,31,32]. However, HF etching in zirconia has been controversial because of the high crystalline phase in zirconia.

Previous studies[14,21] have reported that acid etching with HF can change micro-morphological and increase roughness of zirconia surface with difference HF concentration, etching time, and temperature. Similarly, this study found that surface roughness (Ra) of both 3Y-TZP and 5Y-TZP was not significantly different between control and HF-treated groups except in HF-treated for 24 h group. Moreover, the highest roughness was observed in sandblast group. Additionally, there was an interaction between the two independent variables; type of zirconia (3Y-TZP, 5Y-TZP) and surface treatment methods on the dependent variable; surface roughness. In sandblast group, 3Y-TZP showed significantly higher surface roughness than the 5Y-TZP group. On the contrary, 3Y-TZP showed significantly lower roughness than the 5Y-TZP in 24h HF-treated group. The sandblast technique was found to result in an increase of surface roughness due to phase transformation from tetragonal to monoclinic[4,9]. Therefore, 3Y-TZP with more tetragonal component exhibited increased roughness when compared to 5Y-TZP.

The SEM images revealed that sandblast group showed roughen surface of 3Y-TZP and 5Y-TZP corresponding to the increased surface roughness (Ra) value when compared to the control group. The SEM images of HF-treated groups also captured rougher surface with irregular shape of eroded superficial grains and increased inter-grain space creating porosities on zirconia surface with longer treatment time, especially at 24 h. Furthermore, larger porosity and more dislodgement on 5Y-TZP surface was observed when compared to 3Y-TZP according to the higher surface roughness of 5Y-TZP; it is possible that 5Y-TZP has lower acid resistance compared to 3Y-TZP. Also, the SEM images of HF-treated for 24 h of 5Y-TZP showed large and deep porosity surface. However, there was no significant difference in the SBS between the sandblast and 24 h HF-treated. This is likely because the high viscosity of composite resin cannot infiltrate into the porosity of the etched zirconia surface[14].

This study report that HF can roughen zirconia surface which was increased according to the treatment duration, although the surface roughness of HF-treated zirconia was significantly lower compared to the sandblast group. Interestingly, there was no significant difference in SBS between the sandblast and HF-treated with immersion times more than 2h in 3Y-TZP and 30 min in 5Y-TZP. However, Ra is not the only factor determining SBS as the surface topography also plays an important role as well[33]. The SEM images in our study revealed the difference of the surface morphology between sandblast and HF-treated groups. HF-treated groups showed the pits and porosity due to acid corrosion, while sandblast groups showed the irregular surface due to loss of surface material and phase transition. Thus, the difference of surface morphology may explain the comparable SBS of HF-treated and sandblast groups despite the lower Ra caused by HF treatment. Moreover, after immersion in HF for a long time, the slow phase transformation in 3Y-TZP and 5Y-TZP can be induced from tetragonal to monoclinic structure with moist conditions due to Low Temperature Degradation (LTD) which reduces strength and creates microcracking[2, 14]. On the other hand, recent studies[34-36] reported that 5Y-TZP is more resistant to LTD compared to 3Y-TZP as their studies evaluated LTD using water or water vapor with or without applied stress. However, there are no reports about acid resistance ability of 3Y-TZP compared to 5Y-TZP.

In our study, the lowest SBS was observed in the control group which was only treated with primer. According to previous studies[4, 37], using primer alone without mechanical pretreatments is inadequate to improve bond strength between zirconia and composite resin. Similarly, another study[23] investigated short-term bond strength between zirconia and indirect composite resin after various sandblasting processes and bonding using an agent containing 10-MDP. They found that sandblasting with alumina particles resulted in significantly higher SBS compared to the control group which was polished without sandblasting. On the other hand, the study[12] reported that no significant difference was observed in the SBS between sandblast and non-sandblast groups treated with primers in short-term. However, after 10,000 thermocycles, zirconia treated with both sandblasting and primer showed significantly higher SBS compared to primer treatment without sandblasting. In our study, MDP-containing primers were applied on all samples. Therefore, our results suggest that mechanical treatment may potentially have major effects on SBS of zirconia. Nevertheless, sandblasting without primer application can result in higher initial bond strength to zirconia which is definitely reduced over time[12, 38].

Furthermore, the results of this study found there was no significant interaction on SBS between 3Y-TZP, 5Y-TZP and surface treatment including control group, sandblast group, and HF-treated groups. Similarly, another studies show no significant difference in SBS between 3Y-TZP and 5Y-TZP group[34, 39, 40]. They suggest that sandblasting with alumina particles and MDP-containing primer application can improve bonding efficacy of both 3Y-TZP and 5Y-TZP. The difference between 3Y-TZP and 5Y-TZP is %mol of Yttria added to stabilize zirconia with cubic-tetragonal microstructure which makes 5Y-TZP more resistant to hydrothermal aging, while the mechanical strength is reduced[8, 41].

The failure mode analysis in this study shows that adhesive type was mostly observed in control and HF-treated group debonded at zirconia and composite resin interface due to inertness of zirconia. Furthermore, sandblast group of 5Y-TZP exhibited both mixed type and adhesive failure equally. However, stereomicroscope and SEM showed mixed failure including the adhesive interface failure mostly as well as some cohesive failure in resin. Similarly, recent studies[42-44] investigated bond strength of 3Y-TZP and 5Y-TZP specimens were treated with sandblasting and different cement bonding method, they reported the major of all specimens failed were classified as adhesive failure at the zirconia side. The other specimens showed mixed failures. Furthermore, another study[45] show zirconia specimens that were etched with 9.5% hydrofluoric acid gel for 1 min, all failures were completely adhesive failure at the interface between zirconia and indirect composite.

The results of our study shows that HF can roughen surface and improve SBS of zirconia to composite resin with a long immersion time (more than 30 min). To date, sandblasting has remained a standard and simple procedure to increase the bond strength between dental materials in clinic and laboratory. However, our study suggests that HF may potentially be used as an alternative method to sandblasting as HF treatmeant was able to induce irregular morphologic change in surface roughness of zirconia.

Conclusions

Hydrofluoric acid can roughen surface of both 3Y-TZP and 5Y-TZP and improve bond strength between zirconia and composite resin with the treatment of at least 2 h in 3Y-TZP and 30 min in 5Y-TZP. Moreover, sandblasting with alumina particles results in the highest surface roughness (Ra) and shear bond strength of 3Y-TZP and 5Y-TZP to composite resin. However, using primers alone is insufficient to achieve a strong bond without mechanical pretreatments.

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Declaration of Interest

The authors report no conflict of interest.

Material	Composition	Manufacturer	Lot no.
Katana Zirconia - ML - UTML	Zirconium oxide	Kuraray Noritake Dental Inc., Japan	- DWLFF - DWLFC
Hydrofluoric acid solution	Hydrofluoric acid in water	QRëC, New Zealand	202281-0524
Clearfil Ceramic Primer Plus	Ethanol, prehydrolyzed γ -MPTMS, 10-MDP (10-methacryloyloxydecyl dihydrogen phosphate)	Kuraray Noritake Dental Inc., Japan	1C0071
Filtek Supreme Flowable Restorative	BisGMA, TEGDMA and Procrylat resins	3M ESPE, USA	NF10830

Table 1. Materials used in this study.

Group	Elemental composition						Surface roughness (Mean ± SD)	
	%Zr		%Al		%F			
	3Y	5Y	3Y	5Y	3Y	5Y	3Y	5Y
C	98.80	99.75	0.25	0.25	0.95	0.00	0.34 ± 0.04abc	0.36 ± 0.05bc
SB	93.64	94.55	6.36	5.45	0.00	0.00	1.59 ± 0.28g	1.23 ± 0.20f
HF15m	98.55	99.21	0.24	0.00	1.22	0.79	0.29 ± 0.05a	0.35 ± 0.05abc
HF30m	95.84	93.10	0.25	0.13	3.91	6.77	0.39 ± 0.06bc	0.39 ± 0.04bc
HF1h	85.48	89.61	0.23	0.27	14.29	10.12	0.40 ± 0.05bc	0.40 ± 0.05c
HF2h	86.78	79.84	0.18	0.19	13.04	19.97	0.33 ± 0.06ab	0.33 ± 0.03ab
HF3h	85.54	75.95	0.30	0.46	14.16	23.59	0.34 ± 0.04abc	0.36 ± 0.04bc
HF24h	70.37	73.44	0.23	0.14	29.40	26.42	0.56 ± 0.06d	0.75 ± 0.09e

Table 2. Energy dispersive X-Ray spectrometer (EDS) analysis (%wt), mean and SD of surface roughness (Ra).
Note: Different letters indicate significant differences by Dunnett's Test ($P < 0.05$).

Group	Shear bond strength (Mean ± SD)		%Mode of failure					
			Adhesive		Cohesion		Mixed	
	3Y	5Y	3Y	5Y	3Y	5Y	3Y	5Y
C	4.39 ± 1.04ab	3.88 ± 0.53a	100	100	0	0	0	0
SB	8.41 ± 1.13d	9.43 ± 2.95d	80	50	0	0	20	50
HF15m	5.94 ± 1.15bcd	5.09 ± 1.30abc	100	100	0	0	0	0
HF30m	7.15 ± 1.76bcd	7.46 ± 1.88cd	100	90	0	0	0	10
HF1h	6.40 ± 0.98bcd	7.63 ± 1.14d	80	90	0	0	20	10
HF2h	7.19 ± 1.15cd	7.01 ± 1.92bcd	90	80	0	0	10	20
HF3h	7.56 ± 0.57d	7.24 ± 1.41cd	90	90	0	0	10	10
HF24h	8.48 ± 1.72d	7.15 ± 2.19bcd	80	80	0	0	20	20

Table 3. Mean and SD of shear bond strength (MPa) and percent of mode of failure (n=10).
Note: Different letters indicate significant differences by Dunnett's Test ($P < 0.05$).

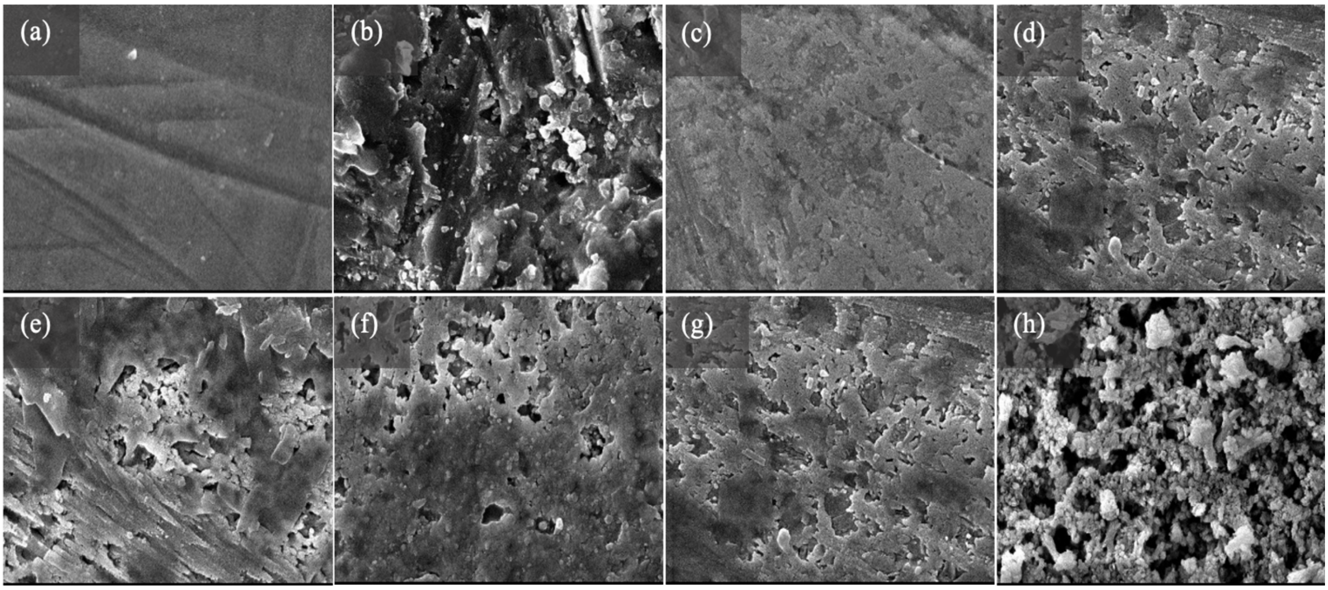

Figure 1. SEM images of 3Y-TZP surfaces at 10,000× magnification: control (a), sandblast (b), after immersion in 9.5% HF for 15 min (c), 30 min (d), 1h (e), 2h (f), 3h (g) and 24h (h).

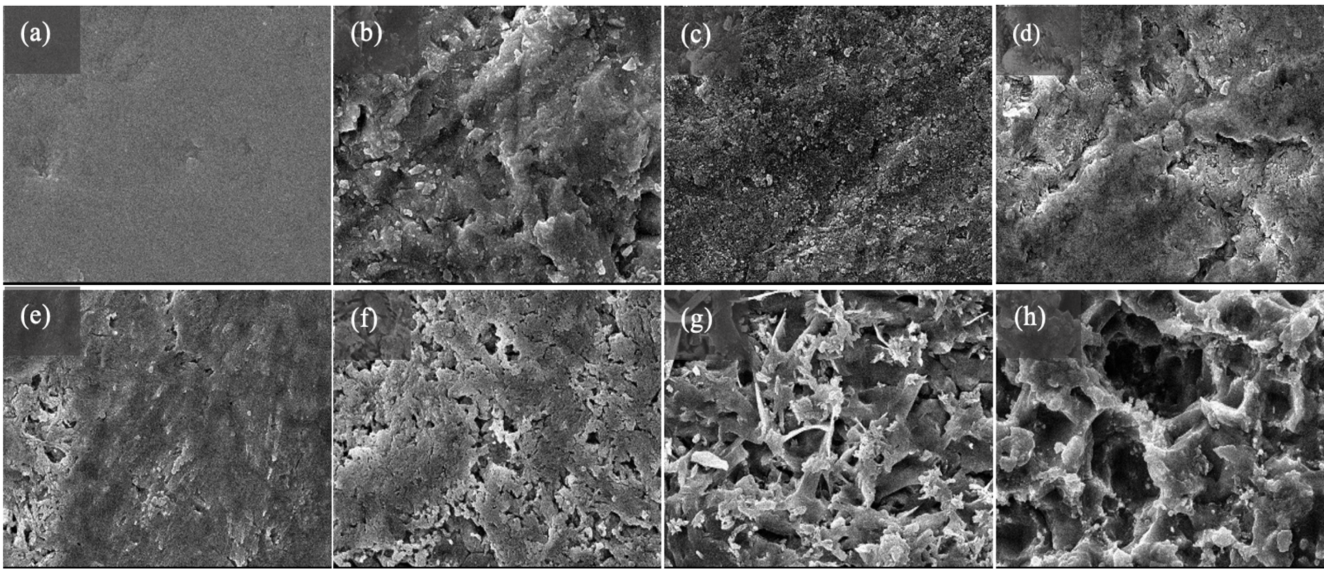

Figure 2. SEM images of 5Y-TZP surfaces at 10,000× magnification: control (a), sandblast (b), after immersion in 9.5% HF for 15 min (c), 30 min (d), 1h (e), 2h (f), 3h (g) and 24h (h).

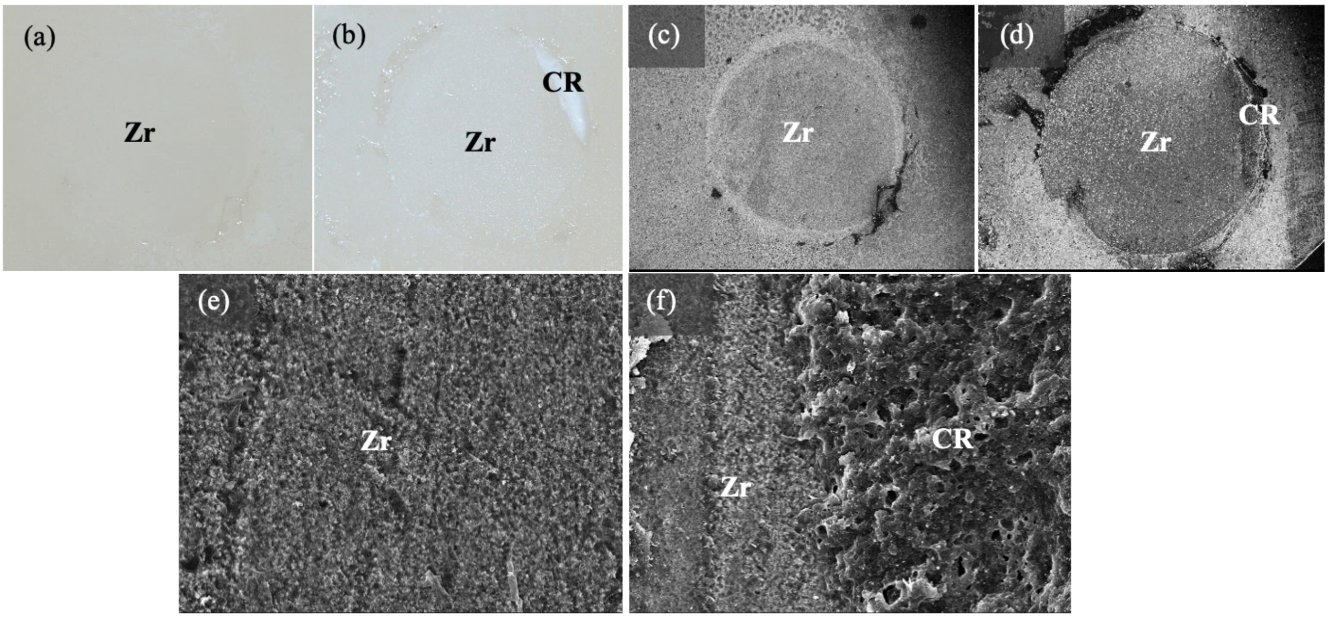

Figure 3. Stereomicroscope images of mode of failure after shear bond testing at 20x magnification locating both zirconia (Zr) and composite resin (CR) area: adhesive (a) and mixed (b) failure mode. SEM images at 25x magnification showing both adhesive (c) and mixed (d) failure mode. SEM images at 1,000x magnification showing zirconia surface (e) and zirconia surface with residual composite resin (f).

References

1. Arunraj D, Gnanam P, Chander GN. Prosthodontic rehabilitation of a patient with missing teeth and loss of vertical dimension using telescopic overdentures. Contemp Clin Dent. 2021;12(1):67-72.
2. Miyazaki T, Nakamura T, Matsumura H, Ban S, Kobayashi T. Current status of zirconia restoration. J Prosthodont Res. 2013;57(4):236-61.
3. Bona AD, Pecho OE, Alessandretti R. Zirconia as a dental biomaterial. Materials. 2015;8(8):4978-91.
4. Khan AA, Kheraif AA, Jamaluddin S, Elsharawy M, Divakar DD. Recent trends in surface treatment methods for bonding composite cement to zirconia: a review. J Adhes Dent. 2017;19(1):7-19.
5. Thompson JY, Stoner BR, Piascik JR, Smith R. Adhesion/cementation to zirconia and other non-silicate ceramics: where are we now? Dent Mater. 2011;27(1):71-82.
6. Denry I, Kelly JR. State of the art of zirconia for dental applications. Dent Mater. 2008;24(3):299-307.
7. Kwon SJ, Lawson NC, McLaren EE, Nejat AH, Burgess JO.

Comparison of the mechanical properties of translucent zirconia and lithium disilicate. J Prosthet Dent 2018;120(1):132-7.
8. Guth JF, Stawarczyk B, Edelhoff D, Liebermann A. Zirconia and its novel compositions: What do clinicians need to know? Quintessence Int. 2019;50(7):512-20.
9. Choosuk S, Piangsuk T. The Effects of Clinical Procedures on Strength of Dental Zirconia: A Literature Review. J Int Dent Med Res. 2023;16(3):1317-22.
10. Tzanakakis EGC, Tzoutzas IG, Koidis PT. Is there a potential for durable adhesion to zirconia restorations? A systematic review. J Prosthet Dent. 2016;115(1):9-19.
11. Inokoshi M, Poitevin A, Munck JD, Minakuchi S, Meerbeek BV. Bonding effectiveness to different chemically pre-treated dental zirconia. Clin Oral Investig. 2014;18(7):1803-12.
12. Tsuo Y, Yoshida K, Atsuta M. Effects of alumina-blasting and adhesive primers on bonding between resin luting agent and zirconia ceramics. Dent Mater J. 2006;25(4):669-74.
13. Russo DS, Cinelli F, Sarti C, Giachetti L. Adhesion to zirconia: A systematic review of current conditioning methods and bonding materials. Dent J. 2019;7(3):74-92.
14. Thamronganankul N, Busabok C, Poolthong S, Uo M, Tagami J. Dental zirconia can be etched by hydrofluoric acid. Dent Mater J. 2014;33(1):79-85.
15. Borges GA, Sophr AM, Goes MFD, Sobrinho LC, Chan DC. Effect of etching and airborne particle abrasion on the microstructure of different dental ceramics. J Prosthet Dent. 2003;89(5):479-88.
16. Jevnikar P, Krnel K, Kocjan A, Funduk N, Kosmac T. The effect of nano-structured alumina coating on resin-bond strength to zirconia ceramics. Dent Mater. 2010;26(7):688-96.
17. Yun J, Ha S, Lee J, Kim S. Effect of sandblasting and various metal primers on the shear bond strength of resin cement to Y-TZP ceramic. Dent Mater. 2010;26(7):650-8.
18. Pardo NP, Araya PL, Pardo MP. Effect of different surface treatments on the bonds strength of a resin cement in zirconia frameworks. J Int Dent Med Res. 2016;9(1):193-7.
19. Menezes FCH, Borges GA, Valentino TA, Oliveira MAHM, Turssi CP, Sobrinho LC. Effect of surface treatment and storage on the bond strength of different ceramic systems. Braz J Oral Sci. 2009;8(3):119-23.
20. Bona AD, Borba M, Benetti P, Cecchetti D. Effect of surface treatments on the bond strength of a zirconia-reinforced ceramic to composite resin. Braz Oral Res. 2007;21(1):10-5.
21. Smielak B, Klimek L. Effect of hydrofluoric acid concentration and etching duration on select surface roughness parameters for zirconia. J Prosthet Dent. 2015;113(6):596-602.
22. Bunz O, Kalz P, Benz CI, Naumova EA, Arnold WH, Piwowarczyk A. Cold atmospheric plasma improves shear bond strength of veneering composite to zirconia. Dent J. 2021;9(6):59-67.
23. Su N, Yue L, Liao Y, Liu W, Zhang H, Li X, et al. The effect of various sandblasting conditions on surface changes of dental zirconia and shear bond strength between zirconia core and indirect composite resin. J Adv Prosthodont. 2015;7(3):214-23.
24. Passia N, Mitsias M, Lehmann F, Kern M. Bond strength of a new generation of universal bonding systems to zirconia ceramic. J Mech Behav Biomed Mater. 2016;62:268-74.
25. Santos RA, Lima EA, Mendonça LS, Oliveira JE, Rizuto AV, Tavares ÁFAS, et al. Can universal adhesive systems bond to zirconia? J Esthet Restor Dent. 2019;31(6):589-94.
26. Azimian F, Klosa K, Kern M. Evaluation of a new universal primer for ceramics and alloys. J Adhes Dent. 2012;14(3):275-82.
27. Genuino HC, Opembe NN, Njagi EC, McClain S, Suib SL. A review of hydrofluoric acid and its use in the car wash industry. J Ind Eng Chem. 2012;18(5):1529-39.
28. Alex G. Preparing porcelain surfaces for optimal bonding. Compend Contin Educ Dent. 2008;29(6):324-35.
29. Blatz MB, Sadan A, Kern M. Resin-ceramic bonding: a review of the literature. J Prosthet Dent. 2003;89(3):268-74.
30. Canay S, Hersek N, Ertan A. Effect of different acid treatments on a porcelain surface 1. J Oral Rehabil. 2001;28(1):95-101.
31. Chen J, Matsumura H, Atsuta M. Effect of different etching periods on the bond strength of a composite resin to a machinable porcelain. J Dent. 1998;26(1):53-8.
32. Yen T, Blackman RB, Baez RJ. Effect of acid etching on the flexural strength of a feldspathic porcelain and a castable glass ceramic. J Prosthet Dent. 1993;70(3):224-33.
33. Goodhand MN, Walton K, Blunt L, Lung HW, Miller RJ, Marsden R. The limitations of using “Ra” to describe surface roughness. J Turbomach Trans ASME. 2016;138(10):101003-8.
34. Alammar A, Blatz MB. The resin bond to high-translucent zirconia—A systematic review. J Esthet Dent. 2022;34(1):117-35.
35. Zhang F, Inokoshi M, Batuk M, Hadermann J, Naert I, Van Meerbeek B, et al. Strength, toughness and aging stability of highly-translucent Y-TZP ceramics for dental restorations. Dent Mater. 2016;32(12):e327-e37.
36. Spies BC, Zhang F, Wesemann C, Li M, Rosentritt M. Reliability and aging behavior of three different zirconia grades used for monolithic four-unit fixed dental prostheses. Dent Mater. 2020;36(11):e329-e39.
37. Cheung GC, Botelho MG, Matinlinna JP. Effect of surface treatments of zirconia ceramics on the bond strength to resin cement. J Adhes Dent. 2014;16(1):49-56.
38. Mattiello RDL, Coelho TMK, Insaurralde E, Coelho AAK, Terra GP, Kasuya AVB, et al. A review of surface treatment methods to improve the adhesive cementation of zirconia-based ceramics. Int Sch Res Notices. 2013;2013:1-10.
39. Yagawa S, Komine F, Fushiki R, Kubochi K, Kimura F, Matsumura H. Effect of priming agents on shear bond strengths of resin-based luting agents to a translucent zirconia material. J Prosthodont Res. 2018;62(2):204-9.
40. Shimizu H, Inokoshi M, Takagaki T, Uo M, Minakuchi S. Bonding efficacy of 4-META/MMA-TBB resin to surface-treated highly translucent dental zirconia. J Adhes Dent. 2018;20(5):453-9.
41. Jerman E, Lumkemann N, Eichberger M, Zoller C, Nothelfer S, Kienle A, et al. Evaluation of translucency, Marten’s hardness, biaxial flexural strength and fracture toughness of 3Y-TZP, 4Y-TZP and 5Y-TZP materials. Dent Mater. 2021;37(2):212-22.
42. Behr M, Proff P, Kolbeck C, Langrieger S, Kunze J, Handel G, et al. The bond strength of the resin-to-zirconia interface using different bonding concepts. J Mech Behav Biomed Mater. 2011;4(1):2-8.
43. Seabra B, Oliveira SA, Portugal J. Influence of multimode universal adhesives and zirconia primer application techniques on zirconia repair. J Prosthet Dent. 2014;112(2):182-7.
44. Zhang X, Liang W, Jiang F, Wang Z, Zhao J, Zhou C, et al. Effects of air-abrasion pressure on mechanical and bonding properties of translucent zirconia. Clin Oral Investig. 2021;25:1979-88.
45. Komine F, Fushiki R, Koizuka M, Taguchi K, Kamio S, Matsumura H. Effect of surface treatment on bond strength between an indirect composite material and a zirconia framework. J oral sci. 2012;54(1):39-46.