








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Corresponding Author:

Taweesak Prasansuttiorn,
Department of Restorative
Dentistry and Periodontology,
Faculty of Dentistry, Chiang Mai
University, Chiang Mai 50200,
Thailand
E-mail: taweesak.p@cmu.ac.th,
dent.taweesak@gmail.com

Effect of Various Roughness Parameters on Surface Wettability of 3Y-TZP Ceramic

Ornnicha Thanatvarakorn¹, Taweesak Prasansuttiorn^{2,3}, Thanapat Sastraruji⁴,
Keiichi Hosaka⁵, Kittisak Sanon⁶, Junji Tagami^{6,7}

¹Faculty of Dentistry, Bangkokthonburi University, Thailand

²Department of Restorative Dentistry and Periodontology, Faculty of Dentistry, Chiang Mai University, Thailand

³Center of Excellence in Material Science and Technology, Chiang Mai University, Thailand

⁴Dental Research Center, Faculty of Dentistry, Chiang Mai University, Thailand

⁵Department of Regenerative Dental Medicine, Tokushima University, Graduate School of Biomedical Sciences, Japan

⁶Department of Operative Dentistry, Faculty of Dentistry, Chulalongkorn University, Thailand

⁷Department of Cariology and Operative Dentistry, Graduate of Medical and Dental Sciences, Institute of Science Tokyo, Japan

Abstract

Objectives: The surface wettability of zirconia has been reported to correlate with the roughness. However, most studies commonly used the arithmetical mean deviation of the roughness (Sa) to predict surface wettability. It is of interest whether other roughness parameters affect the wettability of zirconia. Thus, this study aimed to evaluate the effect of various roughness parameters on the surface wettability of 3Y-TZP.

Methods: Zirconia slabs were prepared and divided into 4 groups (n=10) based on the surface treatment as follows: no treatment (control), air abrasion (AB), etching with 48% hydrofluoric acid (HF), and etching with the mixture of 37% hydrochloric acid and 68% nitric acid (CN). The specimens were scanned using atomic force microscopy (AFM) to evaluate vertical roughness parameters (Sa, Ssk: the skewness, Sku: the kurtosis) and non-vertical roughness parameters (Rsm: the mean width of the profile elements, Sdq: the mean slope of the surface, Sdr: the rate of increase in the surface area). Wettability was determined by surface contact angle (θ_c) using the sessile drop technique. The data were analyzed statistically with one-way ANOVA and Tukey's test ($p < 0.05$).

Results: The group showing statistically higher Sdq and Sdr values than that of the control group ($p = 0.025$ and 0.019 , respectively) possessed significantly lower θ_c ($p = 0.000$), despite similar Sa, Ssk, Sku, and Rsm patterns ($p > 0.05$).

Conclusions: The Sdq and Sdr parameters could affect the surface wettability of 3Y-TZP. The higher slope of the profile and the higher surface area synergistically established better wettability of 3Y-TZP.

Keywords: surface area, surface roughness, surface slope, surface wettability, zirconia

Introduction

In adhesive dentistry, achieving a strong bond between the adhesive and the tooth surface is important for successful restorations. One of the critical factors influencing bonding efficacy is surface wettability, which can be evaluated by measuring the contact angle.⁽¹⁾ A low contact angle indicates good wettability, signifying intimate contact and enhanced mechanical interlocking between the adhesive and adherent.^(2,3) Therefore, surface wettability is a valuable parameter to predict the strength of the adhesive bond.^(4,5)

One of the factors contributing to surface wettability is surface roughness. According to Wenzel's theory, wettability is increased according to increased surface roughness for hydrophilic materials, whose contact angles are less than 90°.⁽⁶⁾ Thus, surface roughening has been indicated as a part of zirconia surface pretreatments, not only for creating the micro-mechanical interlocking but also for facilitating the wettability of the adhesive.⁽⁷⁾ Numerical methods have been introduced to roughen zirconia surfaces, such as airborne particle abrasion and chemical etching. Air abrasion is a process involving the high-velocity impact of Al₂O₃ particles onto the ceramic surface.⁽⁸⁾ This treatment induces surface melting of the zirconia-based materials, resulting in a roughened surface with a correspondingly higher degree of wettability.⁽⁹⁾ Using acids like hydrofluoric acid and/or various strong acids was able to etch the zirconia surface and increase its roughness.⁽¹⁰⁻¹²⁾ High-concentration HF etching can corrode both intergranular and intragranular regions of the zirconia surface⁽¹³⁾, resulting in a roughened surface and increased wettability.⁽⁵⁾

Although several studies have supported the relationship between increasing roughness and improving the surface wettability of zirconia, some studies indicated that the increase in surface roughness did not improve the wettability of the zirconia surface directly.^(14,15) As a matter of fact, the roughening process alters the surface roughness in three dimensions⁽¹⁶⁾, other details of the roughness feature should be taken into consideration. Thus, measuring only one roughness parameter seems to be insufficient to entirely refer to the surface wettability. However, there is still limited research on the effect of the other roughness parameters of zirconia on its wettability.

Therefore, this study aimed to evaluate the effect of roughness parameters, including vertical and non-vertical

planes, on the surface wettability of 3Y-TZP. Various roughness patterns on 3Y-TZP were created by airborne particle abrasion and acid etching.

Materials and Methods

Specimen preparation

Forty zirconia slabs with the dimensions of 12x12x2.5 mm³ were prepared from partially sintered zirconia milling blanks (BruxZir® Shaded; Glidewell Laboratories, CA, USA) by using a low-speed diamond saw (IsoMet™ low-speed cutter; Buehler, IL, USA). The top and bottom surfaces of each slab were manually polished for 20 vertical strokes under running water using abrasive silicon carbide paper (2000 grit; DWetordry™, 3M). All the surfaces were rigorously rinsed with water and dried using delicate task wipers (KIMTECH™). Then, all the specimens were fully sintered in a zirconia sintering furnace according to the manufacturer's instructions at a holding temperature of 1580°C for 2.5 hours with a heating and cooling rate of 10°C/min. Due to the 20-25% shrinkage upon sintering, the final dimension of each specimen was approximately 10x10x2 mm³.

Regarding sample size calculation, G*Power was used with the following parameters: effect size=1.87; α =0.05; power=0.80; number of groups=4. The effect size was estimated using contact angle data collected in a pilot study.

The initial contact angle measurement was carried out to allocate the specimens into 4 groups (n=10) with similar mean contact angle values. The method of contact angle measurement is described below.

Surface treatment

To create various surface roughness on zirconia, specimens in each group were treated as follows: Group 1 (control): no surface treatment, Group 2 (AB): air abrasion using 50 μ m alumina particles at 2 bars pressure, applied 10 mm perpendicularly from the specimen's top surface for 10 s, followed by 10 min ultrasonic cleaning in deionized water, Group 3 (HF): etching with 48% HF, and Group 4 (CN): etching with 37% hydrochloric acid (HCl) mixed with 68% nitric acid (HNO₃) at a 4:1 ratio by volume. Materials used in this study are shown in Table 1. In group 3 and group 4, 50 μ L of designated acid was dropped on the specimen's top surface and wetted the zirconia surface for 10 min, followed by rinsing

with deionized water for 3 min at ambient temperature and humidity. Since the concentrated HF is an extremely hazardous acid, the etching procedure was performed within a chemical fume hood, while the operator was protected by a long-sleeve laboratory coat, goggles, a surgical mask, and double-layer gloves. The acid waste was collected as a hazardous waste in a compatible plastic container, neutralized with a base, and disposed of according to the Enhancement of Safety Practice of Research Laboratory in Thailand (ESPREL). All the specimens were then dried and stored in a closed plastic container for 4 months before the following investigations.

Contact angle (Θ_c) measurement

Contact angle measurement was done by using the sessile drop technique. A 3 μ L droplet of deionized water, whose conductivity was 12.9 $M\Omega/cm^2$, was placed on top of each specimen surface and left for 1 min before the measurement using a contact angle meter (KINO/SL200KS, CAST3 system, Boston, USA). All measurements were taken at ambient temperature and humidity, which were approximately 23.6°C and 53%, respectively. The measurement was performed three times on each specimen by three locations of water dropping, which were at the center, 1 mm above the center, and 1 mm below the center of the specimen surface. Then, the average value was used as the representative contact angle of that specimen. The contact angle data were statistically analyzed using SPSS Statistics 20.0 software (IBM; Chicago, IL, USA) with one-way ANOVA and Tukey's test ($p < 0.05$). The normality assumption required for the application of the method was verified using the Shapiro-Wilk normality test ($p > 0.05$), while the homogeneity of variance was verified using Levene's test ($p > 0.05$).

Surface characterization using atomic force microscopy (AFM)

The area of 5 x 5 μm^2 at the center of each specimen was scanned using AFM (PARK XE7, Park Systems,

Gyeonggi, South Korea) in non-contact mode with a scan rate of 0.5 Hz to obtain three-dimensional surface images. Six surface roughness parameters, which were Sa, Ssk, Sku, Rsm, Sdq, and Sdr were obtained using analysis software (XEI 4.3, Park Systems, Gyeonggi, South Korea). The Sa, Ssk, Sku, Rsm, Sdq, and Sdr data were preliminarily tested for normality and homogeneity using the Shapiro-Wilk test and Levene's test, respectively. Accordingly, the data were statistically analyzed using one-way ANOVA, where Tukey's post-hoc tests were further used to detect statistically significant differences at $\alpha = 0.05$.

The definition and interpretation of each parameter are described as follows^(17,18):

1) Sa refers to the arithmetical mean deviation of the roughness evaluated over the calculated 3D surface representing the mean of the average height difference for the average plane.

2) Ssk refers to the Skewness of the 3D surface texture, representing the degree of symmetry of the surface heights about the mean plane. A positive Ssk (> 0) indicates the predominance of peaks, while a negative Ssk (< 0) indicates valley structures.

3) Sku refers to the Kurtosis of the 3D surface texture, presenting inordinately high peaks/deep valleys if $Sku > 3$, whereas $Sku < 3$ indicates flat peaks and valleys. $Sku = 3$ signifies that surface heights are normally distributed.

4) Rsm refers to the mean width, representing the mean width profile elements within the sampling length. It is used to evaluate the horizontal size of parallel grooves and grains rather than height parameters.

5) Sdq refers to the root mean square gradient, representing the steepness of the surface by indicating the mean magnitude of the local gradients (slope) of the surface. The surface is more steeply inclined as the value of the parameter Sdq becomes larger.

6) Sdr refers to the developed interfacial area ratio. This signifies the rate of increase in the surface area.

Table 1: Materials used in this study.

Agents	Manufacturer	Batch number	Composition
HF (Emsure [®] Hydrofluoric acid)	Merck, Darmstadt, Hesse, Germany	B1004344747	48% w/w HF
HCl (Emsure [®] Hydrochloric acid)	Merck, Darmstadt, Hesse, Germany	K45311117505	37% w/w HCl
HNO ₃ (Gammaco [™] Nitric acid)	Gammaco, Bang Kruai, Nonthaburi, Thailand	3095045	68% w/w HNO ₃

Results

The mean and standard deviation of Sa, Ssk, Sku, Rsm, Sdq, and Sdr parameters, including contact angle, are shown in Table 2. All the surface treatments showed no statistically significant difference in Sa, Ssk, Sku, and Rsm compared with the control group ($p>0.05$). The Sdq and Sdr of the HF group showed significantly higher values than those of the control group ($p=0.025$ and 0.019 , respectively).

For contact angle, all surface treatments caused a statistically significant decrease in contact angle, compared to the control group ($p=0.000$). The HF group revealed the lowest contact angle, which was statistically lower than that of the AB and CN groups ($p=0.001$ and 0.025 , respectively).

3DAFM images of representative specimens from each group were shown in Figure 1. The control group showed a smooth surface with well-defined grain boundaries. The HF group exhibited unclear grain boundaries with

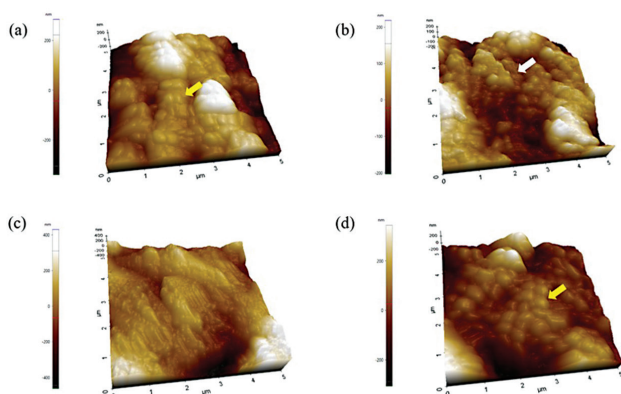


Figure 1: Representative 3D AFM images of the (a) control; (b) HF; (c) AB; and (d) CN groups showed zirconia surfaces with different morphology. The grain boundaries (yellow arrows) were clearly observed in (a) and (d), whereas they were absent in (c), whose surface was non-uniform. The nano-scale surface irregularities (white arrow) were mainly observed in (b).

nano-scale surface irregularities on the zirconia surface. In contrast, the AB group did not show the characteristic of zirconia grains, but rather the non-uniform surface with crevices. Meanwhile, the surface morphology of the CN group regarding grain boundaries rarely changed from the control group.

Discussion

From the results of this study, it was shown that the HF group possessed the statistically highest Sdq and Sdr values, concomitant with the lowest contact angle among the studied groups. Meanwhile, AB and CN groups exhibited a higher trend of Sdq and Sdr values than that of the control group, and their contact angles were significantly lower compared to the control group.

Surface wettability is the property of a material contributed by the factors of surface topography and surface chemistry.⁽¹⁹⁾ To solely investigate the influence of surface topography on the wetting behavior, the variation in surface chemistry should be eliminated. Therefore, all the specimens in this study were intentionally stored in closed containers for 4 months. This method was proposed to mask the specimens' surfaces with airborne hydrocarbon, after which the underneath surface chemistry could be shielded.⁽²⁰⁾ Given that the chemical properties of the zirconia surfaces in all groups were consistent, the measured contact angle is supposed to be solely reflected from the surface topography. However, several studies demonstrated the decreased wettability of the specimens that underwent long-term storage in the air.^(21,22) It was explained that the airborne hydrocarbon absorbed on the specimen's surface decreased surface energy, thus water wettability decreased.^(22,23) Therefore, the measured contact angles in this study were unavoidably higher than those expected under realistic conditions.

Table 2: Mean and standard deviations of surface roughness parameters (Sa, Ssk, Sku, Rsm, Sdq, Sdr) and surface contact angle (θ_c) of each group (n=10).

Surface treatment	Sa (μm)	Ssk	Sku	Rsm (μm)	Sdq (rad)	Sdr (%)	θ_c ($^\circ$)
Control	0.091 (0.01) ^a	0.120 (0.35) ^a	3.315 (0.86) ^a	2.016 (0.31) ^a	0.526 (0.09) ^a	11.425 (2.99) ^a	104.368 (2.26) ^a
HF	0.097 (0.02) ^a	0.168 (0.37) ^a	3.791 (0.73) ^a	1.933 (0.30) ^a	0.649 (0.09) ^b	15.714 (3.20) ^b	89.210 (3.68) ^b
AB	0.100 (0.02) ^a	-0.062 (0.29) ^a	3.359 (1.03) ^a	2.125 (0.72) ^a	0.567 (0.10) ^{ab}	12.702 (3.49) ^{ab}	95.838 (2.97) ^c
CN	0.093 (0.01) ^a	-0.048 (0.35) ^a	3.129 (0.52) ^a	1.816 (0.27) ^a	0.559 (0.09) ^{ab}	12.733 (2.67) ^{ab}	93.830 (4.50) ^c

Different superscript letters indicate statistically significant differences between surface treatments ($p<0.05$).

To achieve zirconia surfaces with various micro- and nano-scale roughness, airborne-particle abrasion, etching with HF, and etching with a mixture of HCl and HNO₃ were employed. Air abrasion is a procedure used to treat ceramic surfaces by impacting the target surface with Al₂O₃ particles at high velocity.⁽⁸⁾ After airborne-particle abrasion, the zirconia surface was roughened, and the typical 3Y-TZP morphology was no longer present as the well-defined grain boundaries were not observed.⁽²⁴⁾ This treatment produced a coarse surface with grooves and sharp edges.⁽²⁵⁾ On the other hand, HF is an inorganic acid of hydrogen fluoride. In the presence of water, it dissociates into its constituent ions according to the reaction $\text{HF} + \text{H}_2\text{O} = \text{F}^- + \text{H}_3\text{O}^+$.⁽²⁶⁾ The reaction of zirconia with HF proceeds according to equation $\text{ZrO}_2 + 6\text{HF} = \text{ZrF}_6\text{H}_2 + 2\text{H}_2\text{O}$, leading to the corrosion of zirconia grains, of which the atoms around the grain boundaries are more chemically reactive.⁽²⁶⁾ Thus, the dissolution occurs more rapidly at the grain boundaries, causing the dislodgement of the grains and the formation of irregular grooves at the boundary regions.⁽¹²⁾ As a result, the HF-etched zirconia manifested as a rough surface with both micro- and nano-scale roughness.^(5,27,28) In contrast, the HCl/HNO₃ mixture exhibited minimal etching of the zirconia substrate despite its robust oxidative capabilities and ability to dissolve common metallic oxides and hydroxides.⁽²⁹⁾

All the treatments employed in this study could not significantly increase average surface roughness (Sa) nor change the vertical topography (Ssk, Sku) of 3Y-TZP. For air abrasion, previous numerical studies showed a significant increase in average surface roughness in both 2D (Ra)^(30,31) and 3D (Sa)^(9,25) measurements under similar conditions of air abrasion. However, the measurement in this study was unable to detect a significant change in surface roughness arithmetically, which is in agreement with the previous study stating that the hardness of sintered zirconia was sufficiently high to resist mechanical abrasion.⁽³²⁾ Nevertheless, the polished pattern was no longer seen on the surfaces of the specimens in the AB group using SEM (data not shown), indicating that the zirconia surfaces were actually abraded. Such discrepancies in the results might derive from the roughness measuring methods and protocols⁽³³⁾, scan rate, condition of the scanning tip, and external factors/noise⁽³⁴⁾, which influence the image resolution and roughness data. Similarly, the HF group did not exhibit a significant

change in these vertical roughness parameters from the control group, which was inconsistent with the previous study.⁽⁵⁾ Apart from the above-mentioned factors, a previous study evaluated 2D parameters, which are considered line roughness parameters and are not sensitive to individual peaks or valleys. A possible anisotropy of the surface can have a strong influence on the measurement values, rendering 2D and 3D parameters incomparable.⁽³⁵⁾

Regarding the non-vertical roughness parameters, the average distance between peaks (Rsm) and the average slope of the surface (Sdq) were evaluated. Rsm values were shown to insignificantly differ among groups, whereas Sdq values were shown to be statistically higher in the HF group than the control group. The higher Sdq value of the HF group, indicating the steeper slopes, might derive from the dislodgement of zirconia grains along valley walls, where the acid was accumulated during the 10-min etching time. It is postulated that the steeper slopes of HF-treated zirconia form deeper and more acute-angle valleys. These morphologies might facilitate more water wettability and contribute to the significant decrease in the contact angle of the HF group. Firstly, the deeper valleys can contain more water, causing the contact angle to be lower. Secondly, the more acute angle creates a narrower groove at the bottom, which facilitates more water accumulation due to capillary action.⁽³⁶⁾ Also, this phenomenon is evident in the dip coating process, in which the frame is dipped into the liquid. It can be seen that the film of liquid is formed a little thicker at the corners of the frames, where the more acute the angle of the frame, the more liquid accumulation.⁽³⁷⁾ However, a too steep slope is not advantageous, since the water wettability could be hindered by the air pocket formation in the cavities. Saying that the Sdq of a completely level surface is 0, and a plane with gradient components of 45 degrees has an Sdq value of 1, the average inclination of slopes on zirconia surfaces in the present study could be estimated to be lower than 45 degrees (Sdq ranges from 0.526 to 0.649). These relatively small slopes can promote the complete penetration of water into surface cavities without underneath an air pocket formation.⁽³⁸⁾ Thus, it can be suggested that, within the optimal range of steepness, the steeper slope of the surface is preferable to promote water wettability according to the capillary effect.

Additionally, the HF group possesses a significantly higher surface area, which was determined by the Sdr

parameter, than the control group. The increased surface area is speculated to be acquired from the nano-scale surface irregularities on zirconia grains, as shown by AFM images. It was explained that such nano-scale roughness could suck water rapidly due to capillary action, resulting in a decreased water contact angle.⁽³⁹⁾ Moreover, the larger surface area offers a larger wetting area, as well as higher surface energies derived from the increased surface atoms.⁽⁴⁾ Presumably, a higher surface area is also an essential prerequisite to enhance surface wettability.

AB and CN groups have similar roughness in all studied parameters; thus, their contact angles are undoubtedly statistically equivalent. However, their contact angles are significantly lower than the control, despite the insignificant differences in Sdq and Sdr values from the control group. It is noticeable that both the Sdq and Sdr values of AB and CN groups are in the upper range of those found in the control group. Therefore, it could be suspected that Sdq and Sdr contribute synergistically to reducing the contact angle.

The limitation of this study was that the surface treatments by air abrasion and acid etching altered the roughness of 3Y-TZP randomly. Although only the roughness parameters showing significant differences were used for interpreting the effect on the contact angle, the remaining parameters might play a part in surface wettability to some extent. To confirm the results of this study, roughening the zirconia surface with a more controllable method, such as a laser, would lead to the more desirable roughened surfaces, which can ensure the influence of each roughness parameter individually. Furthermore, the type of wetting fluid, regarding chemical properties and charged components, is one of the important factors influencing the resulting wettability behavior.⁽⁴⁰⁾ The sessile drop technique using resin adhesive in place of water revealed a significantly lower adhesive contact angle on the assintered zirconia surface.^(41,42) To obtain a more clinically relevant situation, further study utilizing a 10-MDP-containing primer as the wetting fluid should be pursued.

Conclusions

Within the limitations of this study, it could be concluded that the vertical roughness parameters (Sa, Ssk, Sku) alone are insufficient predictors of surface wettability. Instead, the non-vertical roughness parameters, such

as the slope of the profiles (Sdq), including the surface area (Sdr), potentially influence the wettability. The surface with higher slopes and surface area manifested as nano-scale surface irregularities is favorable in promoting water wettability on the zirconia surface. These findings can be used as a goal for zirconia surface pretreatment, of which the wettability of primer and resin cement could be maximized.

Acknowledgments

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Conflict of Interest

The authors declare no conflict of interest.

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