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Surface Roughness Assessment of the Sandblasted and Acid- Etched Zirconia Ceramic

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الخلاصة

الأهداف: تهدف الدراسة الى تأثير المعالجات السطحية الميكانيكية والكيميائية والتي تضم (اوكسيد الالمنيوم البلوري ١١٠ ميكرون) ومحلول حامض الستريك (٣٠٪) على خشونة سيراميك الزركونيا. **المواد وطرائق العمل**: تم تحضير ٣٠ عينه قرصية من مادة الزركونيا الملبدة جزئيا (ايماكس زركاد ام تي). قسمت العينات عشوائي الى ثلاث مجاميع رئيسية وفقًا لطرق المعالجة السطحية (ن=١٠). المجموعه ١ : سطح زركونيا بدون معالجة سطحية ، المجموعه ٢: في هذه المجموعة تم معالجة جميع العينات ميكانيكيا عن طريق استخدام ١١٠ ميكرون من جزيئات أكسيد الألمنيوم البلوري بينما المجموعة ٣ عولجت العينات باستخدام ٣٠٪ من محلول حامض الستريك لمدة ١٠ دقائق. تم قياس خشونة سطح لمادة الزركونيا بأستخدام جهاز(بروفايلوميتر) . تم تحليل جميع البيانات باستخدام تحليل التباين الأحادي عند مستوى معنوي ٥٪ . **النتائج** : استخدام جزيئات أكسيد الألمنيوم البلوري يزيد من خشونة الزركونيا بينما لم يظهر حمض الستريك أي تغيرات مهمة على خشونة الزركونيا مقارنة بالمجموعة الضابطة. **الاستنتاجات**: ضمن حدود هذه الدراسة المختبرية ، يعمل العلاج الميكانيكي لسطح الزركونيا باستخدام جزيئات أوكسيد الألمنيوم البلوري على زيادة خشونة سيراميك الزركونيا بينما لم يظهر حامض الستريك اي تاثيير .

ABSTRACT

Aims: To evaluate the effect of sandblasting with Al_2O_3 particle and 30% citric acid etching on the roughness of zirconia ceramic. **Materials and Methods**: Thirty disks were prepared from partial sintered zirconia (IPS e.max ZirCAD MT blank disk, Ivoclar Vivadent; Schaan, Liechtenstein). Specimens were assigned randomly into three groups based on surface treatment techniques (n=10). Group I zirconia surface was left without surface treatment (control). Group II specimens were subjected to sandblasting with 110 µm Aluminum oxide particles (Al_2O_3) while the group III surface was treated with citric acid solution 30% for 10 minutes. The surface roughness of the zirconia was investigated using a profilometer. Data were analyzed with one-way ANOVA and Duncan's tests at 5% level of significance. **Results**: Sandblasting with AL_2O_3 110µm significantly increased the surface roughness value of the zirconia surface. Chemical treatment with 30% citric acid did not exhibit any significant changes in comparison to the control group. **Conclusions**: Sandblasting with AL_2O_3 increased zirconia surface roughness but acid etching has no effect.

Key words: Acid etching, Roughness, Sandblasting, Zirconia ceramic.

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INTRODUCTION

Zirconia ceramic has been successfully used for dental prosthesis due to its superior structural properties such as esthetic, biocompatibility, and high mechanical properties [1, 2, 3]. Effective bonding of zirconia to tooth tissues is challenging when compared to silica-based ceramic material [4, 5]. This is due to the polycrystalline structure of zirconia make the material highly resistant to different surface treatments unlike glassy materials [6].

Different studies demonstrate the weak adhesion property of cement to zirconia of low surface roughness [4, 5, 6]. Thus continuous effort and techniques still emerge for increasing the surface roughness since the mean roughness values of untreated zirconia range between 0.2 and 0.98µm [7]. Many studies revealed that bond strength will increase when resin cement adheres to the rougher surface [8]. Some techniques and materials have been suggested in the literature to overcome the poor adhesion of resin cement to its surface [9].

The mechanical and chemical modifications of the zirconia surface are well-documented methods for surface modification to establishing a reliable bond [10]. Among the commonly used techniques and materials a surface grinding with diamond rotary instruments, air abrasion with alumina particles, tribochemical silica-coating (TSC), selective infiltration etching (SIE), laser, and acid etching solution [11, 12, 13]. Sandblasting with 50-125 µm AL_2O_3 particles was identified as an essential step in establishing a durable bond between the resin cement and the zirconia [6, 8]. Several authors reported that air abrasion with aluminum oxide (Al_2O_3) particles is needed before luting zirconia, even when applied novel primers and universal adhesives [14, 15, 16]. However, sandblasting may affect the mechanical characteristic of zirconia as it causes surface damage and phase transformation (tetragonal to monoclinic) by introducing flaws and reshaping the surface (17). To avoid such drawbacks and surface damage researchers have tried other techniques.

Chemical acid etching solutions including hydrofluoric acid was investigated thoroughly because of its efficiency in ceramic etching [18]. Such an acidic solution can't be applied for a glass-free zirconia substrate therefore a substitute acidic solution for zirconia was invented by different studies but with a different outcome concerning surface changes induction [19]. A recent study evaluated hydrogen peroxide and citric acid solution of 30% applied for 10min. They claimed that surface roughness was possible with this type of protocol when observed under an atomic force microscope. [20] Xie *et al* [21] also evaluated acidic solution effects on mechanical properties with promising results. Accordingly, acid etching may be an alternative technique to substitute sandblasting for the creation of surface irregularities on zirconia ceramic. It appears that improving the surface roughness property of the prosthesis like zirconia is essential to achieve reliable cement interlock.

Surface roughness evaluation methods may include a micro-scale level or macro-scale application procedure. Accordingly different non-contact machines could be used for this purpose as a confocal laser microscope, SEM, or atomic absorption microscope. [22, 23] The profilometer is one of the simple, reliable, inexpensive, and practical widely used contact methods for assessment of zirconia surface irregularities after different treatments. [13, 24] Many researchers studied the surface roughness of pre-sintered zirconia due to the easy changes induced within the material structure. [24, 25] This study showed a good result to improve roughness due to less hardness associated with this stage of zirconia. [25] Good bonding of sandblasted pre-sintered zirconia was observed resulted from the higher micromechanical interlocking of the cement. Few studied evaluated the possible surface roughness of the sintered zirconia surface when treated with a citric acidic solution in comparison to those treated with aluminum particle abrasion. [14, 26]

As acidic etching may produce microscopical changes while those surfaces subjected to sandblasting may show macroscopical alteration. In this study the hypothesis to be tested that both acid etching and sandblasting would not modify the zirconia ceramic surface roughness. This study aimed to evaluate and compare the influence of sandblasting with aluminum oxide particle and 30% citric acid etching on the sintered surface roughness of zirconia ceramic.

MATERIAL AND METHODS

Specimen preparation

The study was approved by Research Ethics Committee board (University of Mosul, College of Dentistry, REC reference No. POP/S.14/6/20). Thirty disks (2x10X10 mm) of yttrium-oxide–stabilized zirconium blocks (IPS e.max ZirCAD MT blank disk, Ivoclar Vivadent; Schaan, Liechtenstein) were prepared by utilizing CAD/CAM system (Hint-ELs, Griesheim, Germany). After milling the specimens were carefully separated from the zirconia blank disc utilizing tungsten carbide burs. Margins of each specimen were adjusted with a football-shaped and fine fissure diamond burs to remove any excess. Specimens were placed on the firing tray and insert into the furnace for complete the sintering procedure (Programat S1 1600, Ivocalar Vivadent, Schaan, Liechtenstein). The sintering was performed for 2.0 h at 1500◦C with heating and cooling rates at 10◦C/min as manufacturing instruction. Specimens were ultrasonically cleaned for 15 minutes using ultrasonic cleaner (Shenzhen Langee Ultrasonic Electric Co., China).

Surface treatment methods:

Zirconia specimens were assigned randomly and equally into three groups according to the surface treatments (n=10). Group I received no treatment (NST), group II was subjected to aluminum oxide particles sandblasting treatment (SB) as follows. Specimens were fixed in a metallic tool at a distance of 10mm between the surface of the zirconia and the blasting tip (Figure 1). The surfaces were air abraded with 110 µm Aluminum oxide particles (Al_2O_3), using a sandblasting device (Rotaks Dent Dişcilik San Ve Tuc Ltd., İstanbul, Turkey). The sandblasting pen with the tip nozzle size of 3 mm was used in rotational movements and the operational pressure was constant (2.5 bar) for 15 s. Group III: the specimens were subjected to chemical etching with 30% citric acid solution (CAS) for 10 min using the micro brush. After that, the specimens were washed gently with distilled water for one minute and dried.

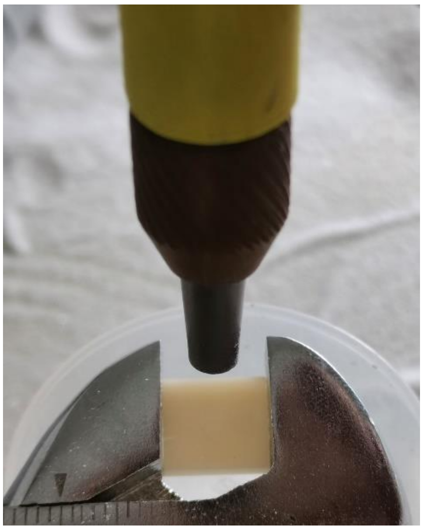

Figure (1): Specimen was fixed in a metallic tool for sandblasting procedure. Surface roughness evaluation

The surface roughness (Ra, µm) of each specimen was recorded with a profilometer device (Taylor-hobson, tylasurf 10/ Leicester, England) (Figure 2). Surface Roughness was determined as the arithmetic means of all distance of the roughness profile to the zero lines. The Ra value expressed the average roughness value for a surface that has been traced by the profilometer. The height Ra-value indicates a rough surface whilst a low value represents a smooth surface. Each specimen was measured ten times at different locations, and the average of these measurements was used to obtain the Ra value of each specimen (Figure 3). The traveling distance of this profilometer device was 1.25 mm across the treated zirconia specimens. The Ra-value was evaluated with 25x vertical magnification and 100x horizontal magnification. Before measurement, the instrument was calibrated against a reference block for which the Ra value was 2.54 µm. The data for Ra (µm) were statistically analyzed using one-way ANOVA test and Duncan's Multiple Range Test for post hoc comparison. All the analysis was performed at 5% level of significance.

Figure (2): Profilometer device used in this study.

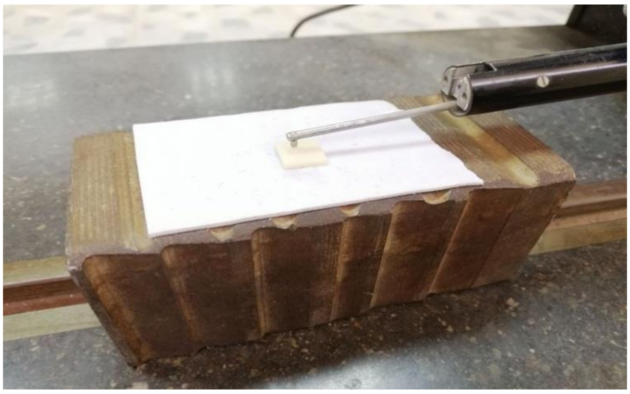

Figure (3): Specimen under stylus head during zirconia surface roughness measurement.

RESULTS

The means and standard deviation (SD) of zirconia (Ra) surface roughness values following different surface treatment techniques were illustrated in Table (1). One-way ANOVA showed that there was a significant difference between experimental groups ($p < 0.05$) (Table 2). Duncan's Multiple Range Test revealed that the mean Ra value of the sandblasted group was significantly higher compared to other groups (the control and citric acid- treated group) ($p < 0.05$). Figure (4) illustrated that there was no significant difference between the citric acid group and the control group (no surface treatment) ($p > 0.05$).

Table (1): Descriptive statistics of experimental groups surface roughness (Ra).

Surface Treatment	Mean Ra (µm)	Std. Deviation
NST	0.78	0.12
SB	1.94	0.39
CAS	0.86	0.30

NST: No Surface Treatment, SB: Sandblasting, CAS: Citric Acid Solution.

Table (2): One way (ANOVA) showing the effect of different surface treatments on Ra value of the zirconia

	Sum of Squares	Df	Mean Square	F	Sig.
Between Groups	8.335	2	4.168	48.238	.000
Within Groups	2.333	27	.086		
Total	10.668	29			

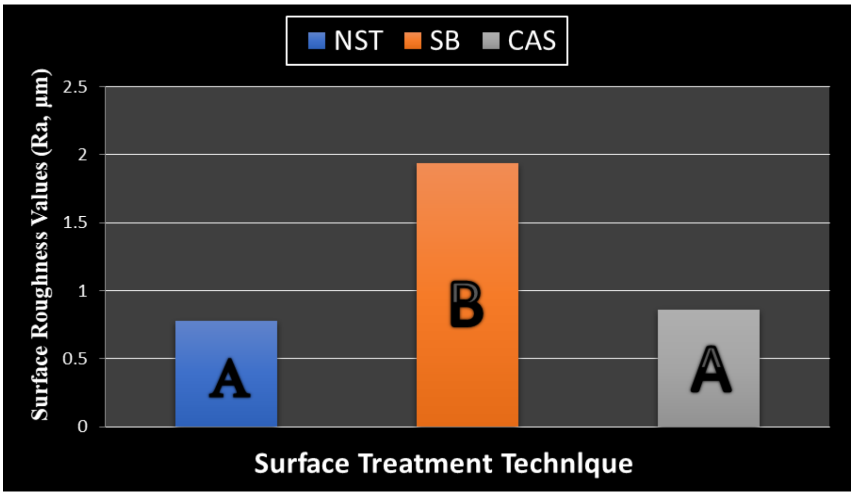

Figure (4): Column graph shows Duncan's Multiple Range Test for different groups.

DISCUSSION

The durable bonding between zirconia and resin cement is a key factor for the clinical success of fixed prosthesis. [27] To achieve this goal reliable surface roughness is essential to obtain micromechanical retention between cement and zirconia ceramic. [6, 12] This has stimulated researchers to analyze the influence of various surface treatments on surface roughness [24]. One of the drawbacks of the non-contact method is that shiny surfaces are sometimes hard to measure due to the scattering effect of the reflected light which may cause incorrect readings. [28] Therefore, a contact method with a profilometer was utilized in the present study. The improvement in surface roughness indicates a larger surface area, which is essential for increasing the connection between the resin cement and the indirect restoration. [29] This study found that the applied treatment techniques had a significant influence on the roughness of the zirconia ceramic. The null hypothesis tested in this study that there are no differences in roughness between different treatment techniques was rejected because the investigated treatment techniques showed statistically significant differences in Ra values. Sandblasting with aluminum oxide particles with a cross-section of 50-125 µm is considered as the main zirconia surface treatment before the cementation procedure due to the results achieved. [6] We select 110 µm of AL_2O_3 particles at 0.25 MPa for the present study, as it has been used as a suitable value to achieve sufficient bond strength while avoiding excess damage in many previous studies [30, 31]. The airborne-particle abrasion has been used not only for the creation of rougher surface but also to clean the surface, and removes impurities. Also, by this procedure surface will be modified thus increase surface energy and wettability of luting material. [17] These changes will permit the resin cement to enter into these micro-retentions and generate a stronger micromechanical interlock. [21] There was a relationship between retention and roughness as an increase in roughness corresponds to an increase in retention. [13, 24, 32] The finding of this study was in agreement with several studies that concluded that sandblasting with aluminum oxide particles was the most effective surface treatment method in enhancing the surface roughness of zirconia. [24, 32, 33] On the contrary, some studies demonstrate the negative influence of air abrasion on the structure of zirconium dioxide therefore, another method by an acid etch was tested in this study. [34, 35] The surface treatment method (acid etching) that chemically dissolving particles on the zirconia surface by applying a strong acid, which may be advantageous because it allows a more objective application and yields more consistent results than sandblasting. [36] So the zirconia surface was treated with 30% of the citric acid solution for ten minutes as an experimental method for inducing surface changes on the non-glass ceramic prosthesis. One previous experiment showed changes within the zirconia surface after their exposure to such protocol and improve the bond strength of resin cement thus more micromechanical retention of the luting resin cement potential [20]. In this study, the mean Ra value of the citric acid-treated group not significantly different from the control group. This means that the citric acid solution doesn't induce a remarkable roughness of zirconia within the profilometric level. Although the citric acid solution can create a change to the surface roughness of ceramics due to its chelating effect [36] but the Ra value less than the sandblasted group. Thus the result of this study may similar to those studies that utilized hydrofluoric acid etching in concentration ranged 4-10%. These studies found that the application of acid solution with low concentration for a short duration to a zirconia surface does not cause any morphologic changes in its structure and does not increase surface roughness. [37-39] On the other hand, Sriamporn *et al* [39] demonstrate increasing application time, concentration, and temperature of low concentration hydrofluoric acid may create higher surface roughness. Searching for a safe and effective technique to create surface roughness like using a stronger acid other than hydrofluoric will be of great help. Xie *et al* [21] revealed that when zirconia specimens immersed in 20% of citric acid solution at ambient temperature produce no significant changes in mechanical properties and surface roughness. The unclear efficiency of citric acid solution to enhance surface roughness of zirconia may be due to low concentration, low temperature (at ambient temperature), and short duration of application of citric acid. Despite of these findings, some investigators concluded that acid etching produced nano-irregular pattern zirconia but without bond improvement and claimed that this because the etched-zirconia surface didn't allow the high viscosity of the resin cement to penetrate these nano-porosities. [40, 41] Thus according to this study result sandblasting produce an obvious increase in surface roughness within profilometric level but the 30% acetic acid etching for ten minutes could not.

CONCLUSIONS

Within the limitation of this study mechanical surface treatment of zirconia via sandblasting with AL_2O_3 (110µm) has created rougher zirconia surfaces than did acid etching with 30 % of citric acid solution.

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