Effect of Different Surface Treatments on Surface Roughness and Vickers Micro-Hardness of Monolithic Zirconia

Afrah Radhy Hashim, B.Sc. and Nidhal Sahib Mansoor, MSc.

1, 2Department of Prosthetic Dental Technology, College of Health and Medical Technology, Middle Technical University, Baghdad, Iraq.

Corresponding Author: Afrah Radhy Hashim
E-mail: hashmafrah1@gmail.com

Received April 11, 2021.
Accepted for publication on April 16, 2021.
Published April 30, 2021.

Abstract
Background Zirconia is highly material-dependent so that many surface treatments may positively or negatively affect the surface as well as the mechanical properties
Objectives The objective of this study was to evaluate the effect of different surface treatments on surface roughness and Vickers micro-hardness number (VHN) of monolithic zirconia (m z).
Material and Methods 24 monolithic zirconia discs (10 mm in diameter, and 2 mm in thickness) were created by using (STL) file and then milled, sintered, and flattened on the surface with 600, 800, 1200 grit aluminum oxide paper. They were then divided into 3 groups (n=8) according to the surface treatment: Glaze (G), polishing kit (PK), and diamond polishing paste (DPP). Surface roughness (Ra) and Vickers hardness (VHN) measuring after the surface treatments were performed.
Results The surface treatment seems to significantly affect the roughness and (VHN) of monolithic zirconia. All specimens polished with a polishing kit showed significantly different Ra values than the glazed specimens. The lowest Ra values were observed with the use of a polishing kit, while the largest Ra values were observed in the glazed group. The largest (VHN) was shown in a polishing kit group and the lowest (VHN) value was obtained with the glazing group. The SEM observations demonstrated that the polishing techniques affected the roughness of monolithic zirconia. Conclusions polishing kit can be used as an alternative to glazing for monolithic zirconia. This may help in reducing the number of appointments of the patients and time-consuming.

Keywords: CAD-CAM technology; monolithic zirconia; surface roughness; Vickers micro-hardness; SEM.
Introduction
Dental ceramics are widely used as a restorative material in a variety of clinical conditions (Qassadi et al, 2021). Patients are increasingly requesting metal-free solutions as their understanding of esthetics and biocompatibility increases (Zghair and Kanaan, 2019). Dental ceramics have good mechanical properties and can be used to create esthetic, biocompatible, and long-lasting fixed dental prostheses (FDPs) for implants or teeth (Scherrer et al, 2020). Zirconia which is a subclass of ceramics is becoming increasingly common in daily dental practice due to its excellent mechanical properties. Transformation toughening and the ability to delay crack propagation and increase fracture resistance are two essential properties of zirconia (Janyavula et al, 2013). Some selected examples are all-ceramic crowns, bridge restorations, laminates, implant abutments, inlays, and Onlay (Ji et al, 2019). Zirconia veneered with feldspathic porcelain and monolithic zirconia are the two main types of zirconia restorations used (Azeez and Salih, 2019). More recently monolithic zirconia restorations manufactured exclusively by the CAD-CAM technology. They have many advantages such as high flexural strength, need lower conservative dental preparation; reduce antagonist wear, exhibit satisfactory aesthetics, require less laboratory time and fewer dental sessions; and because they are monolithic; and they do not have the problematic effect of chipping (Kontonasaki et al, 2019). To enhance esthetic properties, some monolithic or contoured crowns are do not need to be veneered but only glazed and stained superficially during fabrication (Amaya-Pajares et al, 2016, Hmaidouch et al, 2014). The CAD/CAM technology has been used to enable the production of highly accurate monolithic crowns with a more homogeneous structure and fewer imperfection voids than their porcelain-veneered prosthesis (Furtado de Mendonça et al, 2019). This technology guided dental practice toward more standardized dental restorations with lower costs, time, and requirements. Smooth surfaces are known to be critical for the restoration’s aesthetics and long-term success (Preis et al, 2015a). Surface finishing methods include polishing with an adjustment kit, polishing with an adjustment kit plus diamond polishing paste, and glazing. While glazing is a common method for restoring the restoration’s high-gloss surface, glazed layers are said to wear out within six months of installation. The polishing technique, on the other hand, adds no coating to the monolithic zirconia restoration’s surface (Siam et al, 2021, Mai et al, 2019). Various polishing kits are available with the aim of removing flaws and creating smooth surfaces (Incesu and Yanikoglu, 2020). Glazing appears to strengthen the restoration, but this seems debatable (Rashid, 2014). Qualitative and quantitative measurements have been used in studies on the surface characteristics of ceramics (Al Hamad et al, 2019). The average roughness (Ra) parameter defines overall roughness and is considered as the most commonly used measuring scheme in surface roughness research (Walia et al, 2019). The surface roughness of restorative materials may be affected by causing superficial staining, gingival inflammation, and secondary caries, which eventually affect the clinical performance of restorations (Hamed et al, 2020). In terms of roughness, the use of a zirconia polishing kit may be a cost-effective and time-saving alternative option to the re-glazing procedure (Preis et al, 2015b). Increased surface roughness can increase the material’s abrasiveness on the opposing dentition (Vrochari et al, 2017). Polished zirconia has also been documented to cause less wear of opposing enamel than enamel opposing itself (Fontolliet et al, 2020). In addition to sur-
face roughness, other factors could affect the overall success of the restorations such as the Vickers hardness number (VHN) of the restorations. Since the zirconia surface is in direct contact with the antagonist’s tooth due to the absence of veneering porcelain, the hardness of monolithic zirconia is essential (Candido et al., 2018). Vickers hardness testing is one of the most effective methods for determining a material’s hardness. Vickers hardness parameters are achieved by pressing a pyramidal diamond indenter into a sample’s surface (Tanaka et al., 2020). There were no studies that reported the effect of polishing and glazing on (VHN) of monolithic zirconia therefore this study aimed to assess the surface roughness and micro-hardness (VHN) of monolithic zirconia after different surface treatment and alongside their effectiveness. The null hypothesis was that the surface treatment protocol does not influence surface roughness and micro-hardness of monolithic zirconia, whatever the case of the polishing systems used.

**Materials and Methods**

A total of twenty-four highly translucent zirconia discs (UPCERA, FDA, HT, 89 ×16 mm, China) were prepared based on those of previous studies (Ravella and Krishnan, 2014, Mohammed et al., 2015). The dimensions were 10mm in diameter and 2 mm in thickness. Using a specialized 3D modeling software program (Sketchup 3D design software, V 2016) the STL file of the designed specimen is exported to the CAD/CAM device. The specimen’s drawings are then translated into a 3D template to be milled using a CAD/CAM unit. Monolithic zirconia specimens were milled automatically from zirconia blocks using a milling machine (imes-icore, 5-axes, COR TEC 250i dry, Germany). Then according to the manufacturer’s recommendations, the discs were sintered at (1530 °C) for 8 h in a sintering oven (VITA, Germany). For surface standardization, all specimen to be glazed and polished was initially polished with 600, 800, 1200 grit Waterproof aluminum oxide papers (AL-Alamain Ghalib, KSA) (Campos et al., 2021). The procedure takes place in sequence and each was polished for 15 seconds at a speed of 300 rpm by using a polishing machine (DAP-5, STRUERS, Denmark) underwater cooling system (Yener et al., 2015). Specimens were randomly divided into 3 groups according to the proposed surface treatment (n=8).

**Group 1:** polishing with a three-step polishing kit (PK), the polishing kit (PK) specimens were polished with 3-step extra oral diamond wheels polishing kit (NHT, HIGH TECHNOLOGY, Republic of South Korea) as in Figure (1. a) in the first step a regular grit polisher was used, then fine grit polisher for initial shine, and finally ultrafine grit high shine polisher was used for 30 seconds. Each step was in a sweeping motion with forwarding and backward direction.

**Group 2:** polishing with two-step rubber and diamond paste (DPP), at first, polishing with rubber polisher for 30 seconds (NHT, HIGH TECHNOLOGY, and Republic of South Korea), then polishing with a small quantity of polishing diamond paste (Renfert Polish, all-in-one, Germany) using Robinson bristle brush, Figure (1.b). This was accomplished in a single direction using the handpiece (MARATHON-3, Republic of South Korea) for 1 min without water spray. To ensure uniformity, both polishing techniques were applied by one investigator using light finger pressure.

**Group 3:** Glazing (G), two coats of glazing material (powder & liquid) (VITA AKZENT® Plus, GLAZE LT, Germany) in a creamy consistency was applied on specimens and sintered in (Program at P 300, Ivoclar, Vi-
vadent, Germany) porcelain furnace at 900 °C according to the manufacturer’s instructions. One single operator performed all specimens. The dimensions of the specimens were checked with electronic digital calipers (LOUISWARE, China). Before surface roughness and the VHN measurement all specimens, were cleaned in the ultrasonic machine (MESTRA ® CALYPSO, Spain) using distilled water for 10 min, then dried with absorbent paper (Somacal et al, 2019).

**Surface roughness measurement**
Quantitative evaluation of surface roughness was performed numerically in micrometers (μm) for all specimens by using a contact stylus profilometry (Pocket Surf®, U.S.A.) with maximum Stylus Force (1500 mgf /15.0 m N), cut-off value (0.8 mm), and speed of 5.08 mm/s at three different locations on one side of the specimen. The mean surface roughness (Ra) was then calculated.

**Micro-hardness measurement (VHN)**
Micro-hardness was tested on mirror-polished disk-shaped specimens using a Digital tester (Digital Micro Vickers Hardness Tester TH714, China). The mean hardness was measured using 3 Vickers indentations per specimen with loads of 9.8 N applied for 15 seconds (Mohammed and Yassen, 2019, Ozdogan and Yesil Duymus, 2020). Every three readings were averaged and the hardness value (VHN) was then calculated.

**Qualitative SEM evaluation**
One specimen from each group was randomly selected for more extensive qualitative analysis with a scanning electron microscopy (SEM), (Vega, TESCAN, Czech Republic). The scanning took place after coating the specimens with gold in a vacuum sputter coater (MTI CORPORATION, USA). Examination under the microscope was performed at a scale of 50 μm and magnification of (1000 x).

**Statistical analysis**
Analyses of this study were carried out using SPSS statistical software. Levine’s test showed heterogeneity of variance. Therefore, the means of each group were compared using one-way ANOVA, (post hoc LSD test for surface roughness), and (post hoc Tukey test for VHN). These to assess each pair of means were substantially different. A p-value of ≤0.05 was considered statistically significant.

**Results**
The study data were analyzed statistically. The surface roughness values depending on the surface treatments were yielded the highest Ra value of 0.85 (±0.38) μm for the Glazing, but the lowest Ra value of 0.29 (±0.15) μm was yielded for the (Polishing Kit), Figure (2). The results of one-way ANOVA demonstrated that surface treatment influenced the variations in surface roughness and VHN values. Statistical analysis in Table (1) showed the presence of significant differences between PK and G with a p-value of (.001), and between DPP and PK with a p-value of (0.013). The most interesting finding of this study was that there were no significant differences noticed between the DPP group and G group with a p-value of (0.320). In Figure (3), different (VHN) was found for the different surface treatments. The highest Vickers (VHN) value for monolithic zirconia was achieved in a group (PK) of 1364.20 (±65.38), while the lowest (VHN) mean value among all the groups was in the group (G) 801.54 (±55.78). In Table (2), a statistically significant difference was shown between G and DPP with a P-value of (.000), and between G and PK with a p-value of (.000). On the other hand, DPP and PK there were non-significantly
different from each other with a p-value of (0.851). The most significant findings found in this study that there was a strong relationship between the Ra and the VHN i.e. as the Ra decrease 0.29 (±0.15) µm, the VHN will increase 1364.20 (±65.38), and vice versa as the Ra increase 0.85 (±0.38) µm, the VHN will decrease 801.54 (±55.78). According to SEM analysis, and as in Figure (4), polishing and glazing might alter the topographic pattern of monolithic zirconia. SEM observations of the glazed specimen (G) showed irregular and rather slightly granular surface features, Figure (4. a). Whereas group DPP had many uneven regions, Figure (4.b). While after the polishing steps, the surfaces were progressively smoothed, but some shallow, parallel, grooves following the direction of bur movement could not be eliminated after polishing with ultrafine grit polisher group PK, Figure (4. c).

**Table (1): Statistical analysis (ANOVA, post hoc LSD) to compare the difference in average surface roughness values between different surface treatments.**

<table>
<thead>
<tr>
<th>(G) Groups</th>
<th>(G) Groups</th>
<th>Mean Difference (I-J)</th>
<th>p-value</th>
<th>Sig.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Monolithic-Diamond Paste (DPP)</td>
<td>Monolithic-Diamond Paste (DPP)</td>
<td>0.269</td>
<td>.269</td>
<td>NS</td>
</tr>
<tr>
<td>Monolithic-Polishing Kit (PK)</td>
<td>Monolithic-Polishing Kit (PK)</td>
<td>0.569†</td>
<td>.001</td>
<td>$</td>
</tr>
<tr>
<td>Monolithic-Diamond Paste (DPP)</td>
<td>Monolithic-Polishing Kit (PK)</td>
<td>0.409†</td>
<td>.013</td>
<td>$</td>
</tr>
</tbody>
</table>

**Table (2): Statistical analysis (ANOVA, post hoc- Tukey test) to compare the difference in Vickers micro-hardness number (VHN) values between different surface treatments.**

<table>
<thead>
<tr>
<th>(G) Groups</th>
<th>(G) Groups</th>
<th>Mean Difference (I-J)</th>
<th>p-value</th>
<th>Sig.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Monolithic-Glazing (G)</td>
<td>Monolithic-Diamond Paste (DPP)</td>
<td>-628.2791†</td>
<td>.000</td>
<td>$</td>
</tr>
<tr>
<td>Monolithic-Polishing Kit (PK)</td>
<td>Monolithic-Diamond Paste (DPP)</td>
<td>-662.6442†</td>
<td>.000</td>
<td>$</td>
</tr>
<tr>
<td>Monolithic-Diamond Paste (DPP)</td>
<td>Monolithic-Glazing (G)</td>
<td>-34.2769</td>
<td>0.861</td>
<td>NS</td>
</tr>
</tbody>
</table>

**Figure (1): Polishing rotary instruments, a: Three-step polishing kit (step 1: Blue Wheel, step 2: Red Wheel, and step: 3 Green Wheel); and b: Two step polishing with diamond paste (step 1: rubber polisher, and step 2: Robinson bristle brush).**
Results of the study led to the rejection of the null hypotheses considering that the Ra and VHN were not affected by the surface treatment. Monolithic zirconia polished surfaces were found to be smoother than glazed surfaces, (Hmaidouch et al, 2014). The present study results were confirmed by a study established by Preis et al, in 2015. They found that both two-step and three-step systems showed good results and may be recommended for re-glazing of the ground surface (Preis et al, 2015a). This may in agreement with the present study findings. This study results confirmed a study performed by SILVIA et al., who reported that polished zirconia was less rough than glazed zirconia, (Amaya-Pajares et al, 2016). Monolithic zirconia’s excellent polishing performance may be due to its homogeneous and fine microstructure, which can be seen in SEM images. Polisher used with polishing paste DPP demonstrated intermediate surface roughness between PK and G. The polishing kit provides a smoother surface than polisher used with diamond paste possibly because of the lower diamond content and higher silicon carbide content of polisher used with paste as compared to this of polishing kit. This was probably in agreement with the previous study reported by Khaled et al., as they found that the final polishing with diamond paste did not show a significant reduction in surface roughness of monolithic zirconia, (Al Hamad et al, 2019). Since the tongue can detect roughness changes greater than 0.3 μm, an extremely rough restoration surface can hurt patient comfort. In this study, the G, and DPP were above this value, while PK was equal to this value therefore, PK seems to be the most appropriate polishing technique for monolithic zirconia. Opposing enamel in occlusal contact areas has a surface roughness of 0.64 μm, which is a clinically valuable reference for the
surface roughness of restoration, (Incesu and Yaniyokoglu, 2020). Since the surface roughness of PK of 0.29 (±0.15) μm was below the valuable reference. So, PK may cause less wear to the opposing enamel as compared to DPP and G. These findings could in agreement with (Janyavula et al, 2013), who reported that polished zirconia is wear-friendly to the opposing tooth and glazed zirconia causes more material and antagonist wear than polished zirconia. Also, these findings may agree with (Vrochari et al, 2017) who reported that Ra might be clinically relevant as it is within the critical threshold of 0.2µm. Glazing does not minimize surface roughness as effectively as polishing; this may be because the coating layer is not thick enough to effectively complete the ceramic surface micro-cracks and grooves, as reported by (Azeez and Salih, 2019). Yet, there were no studies that identified the effect of polishing and glazing procedure on the VHN of monolithic zirconia therefore, no further comparisons can be made with the results reported in earlier studies. Finally, since sintering conditions affect particle size, they have an impact on the final product’s stability and mechanical properties. Analysis of the different zirconia surface treatments by scanning electron microscopy (SEM) provided valuable information about the resulting topography. Photomicrographs of glazed rough surface as in Figure (4. a) showed the irregular surface with a sharp granular surface, with the small island of unblended surfaces indicating high roughness. Also, a non-homogenous surface is presented for the DPP photomicrograph, Figure (4. b). Despite the presence of several narrow pits and scratches, the final super-polishing with diamond paste revealed a more homogeneous texture compared to G. Polished surfaces appeared to be smooth as in Figure (4. c). With only fine ridges and grooves visible in photomicrographs, the polished surfaces also had a regular morphology and shallow scratches. Based on the findings of this research, polishing monolithic zirconia with a polishing kit providing lower roughness values and the highest VHN values compared to those promoted by polishing with diamond paste and glazing. Only one type of monolithic zirconia was investigated as well as the intraoral environment was not simulated in terms of variables like temperature and humidity, which was a limitation of this study.

Conclusion
Under the limitations of the present in vitro study, the following conclusions were drawn. Polishing with zirconia polishing kit showed excellent enhancement in reduction of surface roughness and increase in VHN as compared to glazing and polishing paste groups. And the significant difference was found in the surface roughness and VHN of the monolithic zirconia subjected to the different surface treatments. As well as this study concluded that there was a reverse relationship between surface roughness and VHN i.e. decrease in surface roughness leads to an increase in micro-hardness.

Conflict of interest
We are the author’s (Afrah Radhy Hashim, and Assist. Prof. Nidhal Sahib Mansoor) state that the manuscript for this paper is original, and it has not been published previously, and it is part of the MSc. dissertation and is not under consideration for publication elsewhere, and that the final version has been seen and approved by all authors.

References


mance. Dentistry journal, 7, 90. https://doi.org/10.3390/dj7030090


