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Evaluation Of Wear and Surface Roughness of Cubic Zirconia and Gradient Zirconia under Simulated Oral Conditions: in-Vitro Study

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ABSTRACT

Background: The nature of the oral environment is very complex, promoting wear and surface roughness of dental restorations, consequently altering function and esthetics. Therefore, wear and surface roughness of the recently introduced gradient zirconia are under investigation. ***Aim:*** to evaluate wear and surface roughness of cubic and gradient zirconia under simulated oral conditions ***Materials and methods:*** Twenty-four disc-shaped samples (10mm X 2mm) were designed and milled using CAD/CAM. They were divided into two equal groups (n=12) according to the material type: **Group C**: Cubic zirconia (BruxZir Esthetic) and **Group G**: Gradient zirconia (IPS e.max ZirCAD Prime). Finishing and polishing of all samples were done then baseline weight values were obtained by an electronic balance followed by baseline surface roughness measurement using a 3D non-contact profilometer. ROBOTA chewing simulator was used to apply a 5kg (49N) load for 75000 cycles, representing six months of clinical function, with a vertical movement of 1mm and a horizontal movement of 3mm. Weight values were re-measured as an indication of weight loss. Further measurement of Surface roughness was obtained to evaluate the surface changes. ***Results:*** Mean and standard deviation values (± SD) of weight loss and surface roughness before and after wear for group C and group G using independent t-test revealed a statistical significant difference for each group as $P<0.05$. While no statistical significant difference was found in weight loss and surface roughness before and after wear for intergroup comparison ***Conclusion:*** Both materials showed a similar wear and surface roughness behavior.

Keywords: Chewing simulator, wear, surface roughness, gradient zirconia.

INTRODUCTION

Zirconium oxide ZrO_2 caught the attention as a strong alternative to conventional ceramics. Moreover, the (CAD-CAM) technology facilitated manufacturing full anatomical prostheses by milling Zirconia blanks.[1]

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Zirconia is characterized by its high mechanical and biocompatible properties. The transformation toughening mechanism possessed by zirconia increased its toughening by hindering crack propagation. Despite the excellent mechanical properties of 3 mol % yttria-stabilized zirconia (3Y-TZP), it has an opaque color and lacks translucency.[2] Therefore, a glass-ceramic layer was needed to mask the opacity and enhance aesthetics. Yet, the layering technique is subjected to fractures, de-lamination, and chipping of the ceramic veneer.[2]

To eliminate the risk of debonding, many investigations have demonstrated the applicability of monolithic restorations made from zirconia, which are widely used nowadays in dentistry.[2] Different zirconia generations were introduced by modifying their microstructure to have an enhanced material that combines both good esthetics and adequate strength. The elemental changes in microstructure strongly affected zirconia properties when a higher percentage of yttria improved the esthetics by increasing the cubic phase and reducing the tetragonal one.[3]

Monolithic restorations increased their mechanical stability and widened the range of their indications. They eliminated the risk of debonding of the veneer layer and reduced material thickness, consequently conserving the tooth structure. These restorations can be colored or pre-shaded before sintering, followed by characterization by staining, resulting in good esthetic minimally invasive restorations in the posterior region. However, this generation did not provide the desirable esthetics for the anterior region.[4,5]

The recent zirconia types showed a modified crystalline structure to improve translucency, which was achieved by increasing the amount of yttria content to 4 mol % (4Y-TZP) or 5 mol % (5Y-TZP) to increase the percent of cubic phase. Elevating the cubic phase within the microstructure resulted in enhanced translucency.[5] Recently, gradient zirconia was introduced in the dental field, which provides gradient shades to mimic natural teeth and combines both 3Y-TZP with the highest strength and 5Y-TZP with the highest translucency. It was claimed that because of its 650–1200 MPa flexural strength and fracture toughness of greater than 5 MPa $m^{1/2}$, their "gradient technique"[6] made it suitable for monolithic restorations in the esthetic zone.

Wear of dental restorations is a continuous phenomenon always present in the oral cavity in the form of progressive loss of the original structure form. Physiologically, all hard dental tissues have a natural tendency to experience a degree of wear throughout life. Nevertheless, pathological wear presents itself as surface partial loss of the restorative materials as well as the opposing enamel and dentin in most severe cases. The mechanism of wear relies on various factors such as friction, chemical influences, or the mechanical load due to compression, flexion, and tension.[7, 8]

The wear behavior of zirconia restorations was investigated for many years**.** Reduced wear resistance of any restorative material has an impact on the surface properties. It was mentioned that the wear behavior of different zirconia generations is relatively unaffected by their difference in microstructure with different yttria content, as all are considered wear-resistance materials. Despite the low fracture toughness and flexural strength of 4Y-TZP and 5Y-TZP when compared to 3Y-TZP, the cubic zirconia can maintain its surface integrity. Nevertheless, the pattern of grain dislodgment seen under magnification is particularly different in 5Y-TZP due to the larger grains than those of 3Y-TZP, leaving larger pits.[9]

Surface roughness is one of the main consequences of wear and is directly related to the wear behavior of different restorations. Therefore, maintaining a smooth surface of the restoration ensures its longevity and durability by permitting a uniform stress distribution for the restoration's surface.[10]

Thus, this study aimed to evaluate the wear and surface roughness of cubic and gradient zirconia under simulated oral conditions.

Two null hypotheses were suggested for this study: the first one was that there would be no statistical difference in wear between cubic and gradient zirconia under simulated oral conditions, and the second one was that there would be no difference in surface roughness between cubic and gradient zirconia under simulated oral conditions.

MATERIALS AND METHODS

Power analysis was designed to have adequate power to apply a two-sided statistical test of the null hypothesis that there was no statistical difference between the intervention (gradient zirconia) and control groups (cubic zirconia) regarding wear and surface roughness effect on the natural teeth. By adopting an alpha (α) level of (0.05), a beta (β) of (0.2) (i.e., power=80%), and effect size (d) of (1.20) calculated based on the results of a previous study, the minimum required sample size (n) was found to be (24) samples (i.e., 12 samples per group). Sample size calculation was performed using G*Power version 3.1.9.7 2. Control (n=12), intervention (n=12).[11,12]

Sample preparation

For sample standardization, all samples were designed and manufactured using a 5-axis dental milling machine (Roland DWX-510) following the manufacturer's instructions to produce samples with dimensions 10 mm diameter and 2 mm thickness.[13] A digital caliper (INSIZE digital caliber, Insize measuring tool, India) was used to ensure the dimensions of the samples with a 20% increase in size according to the manufacturer's instructions to compensate for sintering shrinkage.

Both groups were sintered in a TABEO M1 zirconia furnace according to the manufacturer's instructions; the caliper was used again after sintering to confirm the accurate dimensions (2mm x10mm) after sintering shrinkage. To standardize the finishing and polishing process of the zirconia samples, a dental surveyor was used to apply constant pressure, speed and time for each sample. Two polishers from the EVE DIAERA polishing kit were sequentially[14] used, first H2DCmf (4x13mm dimension) for coarse polishing then H8DC (4x13mm dimension) for high gloss polishing. To maintain grinding integrity, the following parameters for each polisher were obtained: speed 8000:10000 rpm for 60 seconds to achieve a luster finish.[14,15]

Wear and surface roughness measurement.

Weighing of the discs before wear simulation:

The wear quantification process was done for all samples by measuring the amount of substance loss. An electronic balance with a glass housing to prevent air entrapment, with an accuracy of .0001 gm was used to weigh the values of the zirconia discs before starting the two-body wear simulation.[11] It had a micro-weighing scale with fully automated technology to ensure accuracy. Each sample was cleaned in an ultrasonic cleaner and dried with a clean napkin before weighing, then the plastic housing was closed to weigh the samples avoiding the effect of air accurately.

Surface roughness assessment before wear simulation:

Baseline surface roughness measurement was done before wear simulation testing to ensure the qualitative analysis of two-body wear for all samples. An optical profilometry was used, and a digital camera and image processing software were combined. It provided a surface topography for each sample without touching it, thus ensuring accurate measurements.[16] A specific software program (WSxM) was used to identify all sizes, measured characteristics and frames, revealing them in pixels.[17] The system converted the pixels into absolute real-world units. All the captured magnified cropped three 3D photos were taken at a (10 × 10) µm size in three different sites (middle and on each side). The software was used to measure the average heights (Ra) and then express them in µm, which was considered a reliable quantity indication of surface roughness.[18] Then a surface topography assessment was made, which is considered a reliable method to qualify the surface by evaluating the worn areas and surface changes in terms of peaks and valleys.

Wear simulation

After baseline measurement of weight and surface roughness, all samples were stored in distilled water one day before starting the wear test.

A chewing simulator device (ROBOTA) that was integrated with a thermo-cyclic protocol and operated through a servo-motor (**Figure 1**) was used to perform the 2-body wear test.[19] It has four chambers to accommodate four samples at the same time. Each chamber has an upper part (Jacob's chuck) that holds natural premolar antagonists and a lower plastic holder in which the samples were embedded in a Teflon housing.

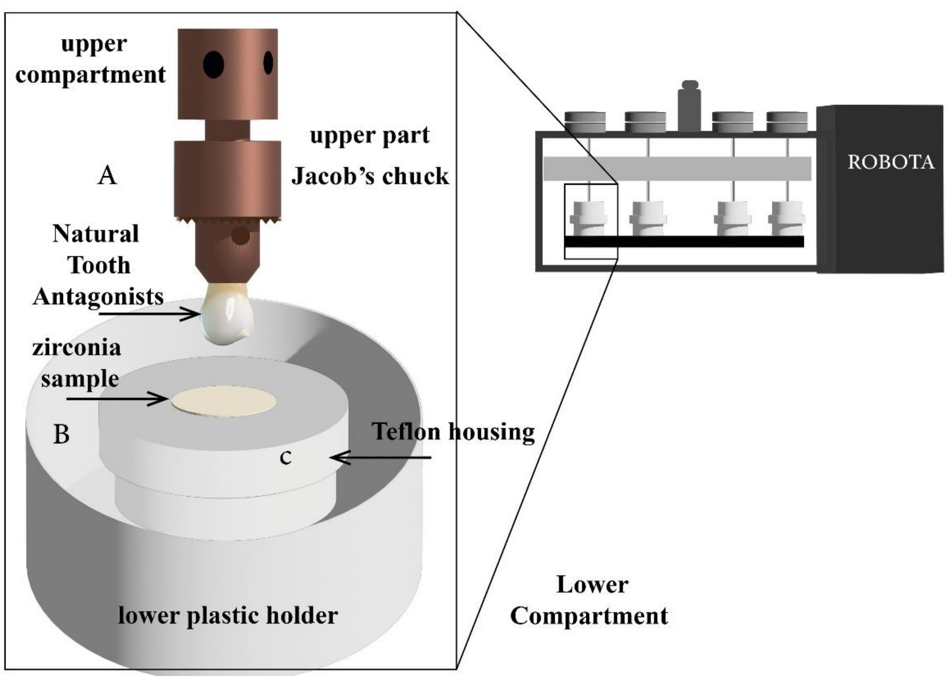

Figure (1): Illustrating diagram of ROBOTA wear testing device.

The two-body wear test was repeated for 75000 cycles,equivalent to six months[20,21] of clinical use for both cubic and gradient zirconia samples. ROBOTA chewing simulator device applied 5 kg per sample simulating horizontal and vertical movements in the thermo-dynamic conditions with the following parameters.[11,22] (**Table 1)**

Table (1): Parameters of Chewing simulator cycle.

Temperature fluctuation	(5ºC/55ºC)	Forward speed	90mm/s
Vertical movement	1mm	Backward speed	40mm/s
Horizontal movement	3mm	Cyclic frequency	1.6Hz
Rising speed	o 90mm/s	Torque	2.4 Nm
Descending speed	40mm/s		

Weighing the samples after wear simulation:

All samples were then re-weighed after the wear simulation to measure the amount of substance loss in the same way as mentioned before.

Surface roughness measurement after wear simulation:

Surface roughness measurement was done after wear simulation to ensure the qualitative analysis of two-body wear for all samples together with the surface topography in the same way as mentioned before.

Statistical analysis

All data were explored for normality by using Shapiro Wilk and Kolmogorov Normality test and presented as means and standard deviation (SD) values. A Paired t-test was used for intragroup comparison and independent t-test was used for intergroup comparison. The mean and standard deviation values of weight loss before and after wear in Group C (BruxZir Esthetic) and Group G (IPS e.max ZirCAD Prime) were done using the Paired t-test. The mean and standard deviation values (± SD) of weight loss before and after wear comparing the two groups were done using an independent t-test. The mean and standard deviation values (± SD) of surface roughness before and after wear in Group C (BruxZir Esthetic) and Group G (IPS e.max ZirCAD Prime) using a Paired t-test. The mean and standard deviation values (± SD) of surface roughness before and after wear comparing the two groups were done using independent t-test t. The significance level was set at $P \leq 0.05$. Statistical analysis was carried out with IBM SPSS Statistics for Windows, Version 20.0. Armonk, NY: IBM Corp.

RESULTS

Weight loss measurements

Intragroup comparison **(Figure 2)**:

In group C, there was a statistically significant decrease in weight (mean 0.8933 ± SD 0.0117) before wear compared to (mean 0.8931 ± SD0.0017) after wear. The decrease in weight was (0.0002 ± 0.00007) as P value =0.0001. In group G**:** There was a statistically significant decrease in weight (mean 0.9023 ± SD 0.0117) before wear compared to (mean 0.9021 ± SD 0.0018)

after wear with a decrease in weight (0.0002 ± 0.00007) as P value =0.0001.

Intergroup comparison: There is no statistical significant difference in weight before and after wear was found between the two groups as P=0.57. **(Table 2)**

Surface roughness

Intragroup comparison: In group C, there was a statistical significant increase in

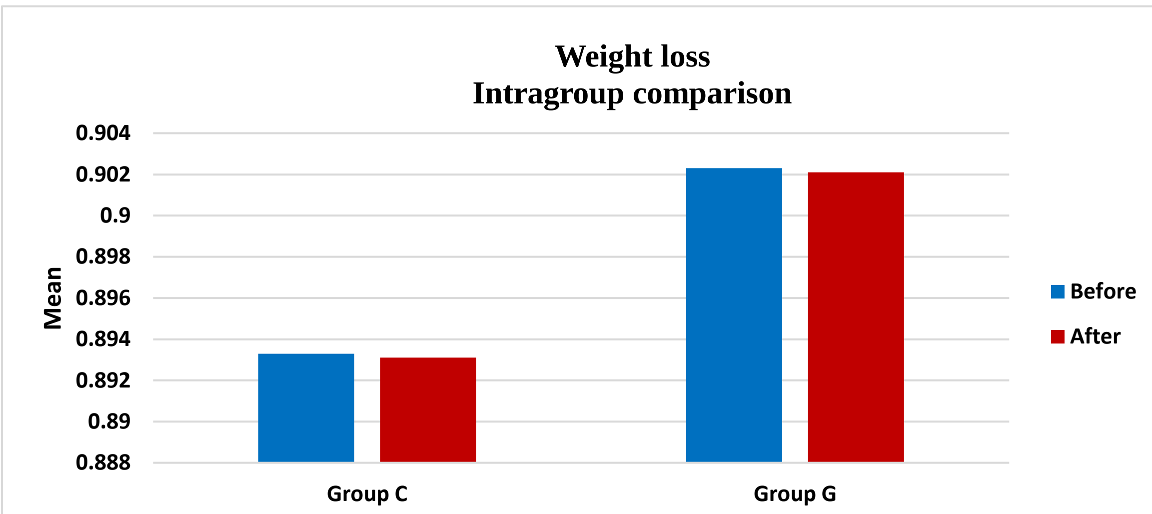

Figure (2): Bar chart showing weight loss in group C and group G before and after wear (Intragroup comparison).

Table (2): Intergroup comparison of weight loss before and after wear.

	Group C		Group G				95% Confidence Interval of the Difference		
	Mean	± SD	Mean	± SD	Mean Difference	Std. Error Difference	Lower	Upper	P value
Before	0.8933	0.0117	0.90234	0.00174	0.00904	0.00342	-0.01614	-0.00195	0.01*
After	0.8931	0.0117	0.90213	0.00177	0.00903	0.00342	-0.01613	-0.00194	0.01*
Difference	-0.0002	0.0001	-0.00023	0.00008	0.00002	0.00003	-0.00004	0.00008	0.57

**Significant difference as P<0.05.*

surface roughness (mean 0.249 ± SD 0.0014) before wear compared to (mean 0.2575 ± SD 0.0016) after wear. In group G**,** there was a statistical significant increase in surface roughness from (mean 0.2497 ± SD 0.014) before wear compared to (0.2572 ± 0.0013) after wear.

Intergroup comparison**:** There is no statistically significant difference was found in surface roughness before and after wear for both groups P>0.05

DISCUSSION

Nowadays, the incorporation of CAD/CAM technology has been increased in daily dental practice, which facilitated the use of different esthetic materials, including zirconia. It has become a prevalent material to be used as a restoration in the oral cavity due to its high strength. Despite the excellent mechanical properties of 3 mol yttria-stabilized (3Y-TZP), it has an opaque white color and lacks translucency. Therefore, a glass-ceramic layer was needed to mask the opacity and enhance aesthetics.[23] Recently, monolithic restorations made from zirconia are widely used in dentistry. The new types were concerned with enhancing the aesthetic appeal without comprising its strength.[2] The newest types showed a modified crystalline structure to improve translucency, which was achieved by increasing the amount of yttria content to 4 mol % (4Y-TZP) or 5 mol % (5Y-TZP) to increase the percent of cubic phase. Elevating the cubic phase within the microstructure resulted in a significant increase in translucency.[5]

Wear behavior is an important property and vitally related to the long-term success of zirconia monolithic restorations under occlusal forces. The clinical implication of the present study is represented by understanding the wear behavior of two types of zirconia and its relation to surface roughness.[24,25]

It was proven in many previous studies that there is a relation between wear and surface roughness[11,25] and the long-term success and durability of zirconia restoration are directly related to maintaining its smooth surface.[24]

Therefore, this study aimed to evaluate the wear and surface roughness of cubic zirconia and gradient zirconia under simulated oral conditions.

BruxZir Esthetic Zirconia was selected as a control material in this study due to its popularity in the dental market, which has proven to be an enamel-friendly material with minimal tooth wear. It is composed of a nearly 50% cubic structure with 5 mol % of yttria (5Y-TZP). It is used frequently as a monolithic restoration and characterized by its good flexural strength (500-800 MPa) and good esthetic appearance.[26]

IPS e-max ZirCAD Prime is an upgraded zirconia material that relies on gradients technology to provide both strength and esthetics; it was selected in this study as it consists of two zones. The first one is the enamel zone with the highest amount of cubic zirconia (5Y-TZP) in the occlusal or incisal area to enhance translucency and mimic the natural appearance. 3Y-TZP at the cervical zone, on the other hand, provides strength which is approximately 1200 MPa.[27]

The blanks of both BruxZir Esthetics and IPS e-max ZirCAD Prime were designed using A 3-D builder designing software program to design the discs to simulate the standard fabrication technique and following the manufacturer's recommendations to ensure high intrinsic strength.[28] All the disc samples were dry milled to avoid moisture absorption as the blanks were pre-sintered. DWX-510, which is a 5-axis milling machine, was used for milling the samples due to its high accuracy, trueness and precision.[29] The milling process was made using the same burs recommended by the manufacturer to achieve standardization.[27,30]

Zirconia discs were milled in a larger dimension by approximately 20% to compensate for the sintering shrinkage according to the manufacturer's recommendations, which was confirmed using a digital caliper. Sintering cycles of all samples were done according to the manufacturer's instructions, as most monolithic zirconia ceramics should be sintered at a temperature between 1400–1550 ◦C and no higher than that, as at temperatures of 1600 or 1700 ◦C or after prolonged sintering, as result grain boundary cracks can be generated. This could lead to alteration in the surface topography of the material, especially the surface roughness.[31]

The digital caliper was used to check the dimensions of each sample before and after the sintering process to exclude any exterior factors that might affect the accuracy of the results of wear and surface roughness.[13]

In this study, polishing rather than glazing of zirconia discs was done for many reasons: first to produce a smooth surface and remove any irregularities that increase surface roughness.[14,32] Second, excluding the effect of the glaze material on the wear behavior of zirconia. According to many previous studies, polishing zirconia discs produces a smoother surface than the glazed surface.[12,32] When the glaze material is worn off it leaves a rougher surface than polished surfaces and it also creates a third medium between the tested materials claimed by Chong B et al in 2015.[32]

In-vitro methods have been used to study the wear behavior of restorative materials, taking the advance of the current technology of the chewing simulator devices and allowing full control of confounding variables.[33]

There are different in-vitro methods to quantify wear of dental restorations, such as measuring the amount of weight loss, volume loss, or height loss. All of them are reliable and suitable for quantification of wear of different dental materials with flat surfaces reported by Heintze et al.[19] In the current study, wear of zirconia was assessed quantitatively by accurately measuring the amount of weight loss of the samples before and after wear simulation. This method was used due to its validity to provide reliable results in measuring the weight loss of the materials to quantify wear according to many studies.[11,21,25] An electronic analytical balance was used to weigh the samples, as it is very sensitive and had a micro-weighing scale with fully automated technology to ensure the accuracy of,0001 gr[2]. The measured amount of weight loss was calculated based on the difference between the initial and final weight (before and after the chewing simulation).

Chewing simulator devices were developed to conduct in-vitro studies as they can simulate oral conditions using specific loads and frictional forces.[34,35] Therefore, a dual-axis chewing simulator (ROBOTA) was used to perform the two-body wear test, which provides specific parameters, and a thermocycling protocol operated through a servomotor gives reliable results, as reported by many studies.[11,25,36]

The intermedium demineralized water was used during the cycles to mimic the natural chewing process with a wet oral environment and to ensure that the byproducts were washed away and had no corrosive effects on any tested samples to guarantee the two-body wear test.[5] A five kg load (49 N) was employed in the present study to represent normal physiological masticatory load during function.[33] The repeated horizontal and vertical movements were used in this study to replicate mandibular closure and lateral excursion.[33]

In the current study, a two-body wear test was repeated for 75,000 cycles which is equivalent to six months of clinical use as reported by Bayoumi A et al. in 2022.[20] This number of cycles was chosen in this study as the first (50,000 cycles) represents a crucial period that can determine the wear behavior of the restorative materials.[21] Zirconia materials showed a significant increase of wear till 50,000 cycles until progression reached a plateau value or a steady state. It was justified that clinically the wear starts with high values and then decreases after a while due to occlusal adaptation and self-adjustment as reported by Vardhaman et al. in 2020.[37]

In this study, surface roughness of both types of zirconia materials was evaluated as it is directly related to wear of the material, and it shows the surface changes that might occur after wear simulation "qualification of wear process". The selection of the 3D optical non-contact profilometer was advocated to measure surface roughness before and after performing the two-body wear to evaluate surface changes, which is known to be a reliable method, as reported by Monaco C. et al. in 2020.[38] This device is fast and operates without touching the surface to protect the sample and give more accurate measurements. It has a laser beam to illuminate the surface and measure the 3D profile of the restoration material, it defines all the points inside an area of 28.96 mm^2 to include all the defects as cracks, craters, voids, peaks and valleys.[38]

In the present study, WSxM software was used to assess changes in the surface topography. Its goal was to provide a trustworthy estimate of the number of peaks, and valleys.[39]

The results of the present study revealed that there was a statistically significant weight loss and increase in surface roughness of both BruxZir Esthetics and IPS e.max ZirCAD Prime groups after 75000 wear simulation cycles. No statistically significant difference in wear behavior and surface roughness between the studied zirconia groups was found represented numerically in **Table (2).**

These findings could be attributed to the microstructure and the chemical composition of both materials, as both materials are (5Y-TZP) characterized by large grain size (>1.7 μm) and high cubic zirconia content (> 60 wt.%). When they were subjected to frequent friction due to two-body wear, their large-size grains were dislodged leaving surface defects, pits and voids. Consequently, it led to rougher surfaces than the baseline surface roughness, which justifies the decrease in weight and the increase in surface roughness of all zirconia samples, as reported in multiple studies.[9,37,40] The gradient zirconia IPS e.max ZirCAD Prime has the incisal/

occlusal zone composed of 5Y-TZP, which is similar to the microstructure of the cubic zirconia BruxZir Esthetics. The samples were flat surface testing the incisal zone only for that reason the difference in change between the two groups was insignificant.

The results of wear in the present study were in agreement with multiple studies as Kown S et al. in 2018,[9] Zhang F et al. in 2019,[40] Rosentritt M et al. in 2020[41] , and Fouda AM et al. in 2022[42] who reported that different zirconia generations are considered wear-friendly materials regardless of the yttria content and the 5Y-TZP zirconia can maintain surface integrity. They evaluated two body wear of different zirconia materials and investigated whether the microstructure variations of different yttria content would affect the wear performance of zirconia. They stated that the wear behavior of different zirconia generations was similar.

These findings could be attributed to the microstructure of both materials, as they have nearly the same amount of cubic zirconia and a close amount of yttria content of more than 5% mol of yttria.

Hence, the first null hypothesis, which stated that there would be no statistical difference in wear between Bruxzir Esthetics and IPS e.max ZirCAD Prime after 75000 wear simulation cycles, was accepted.

The result of the surface roughness in the present study revealed a statistically significant increase in surface roughness in both studied materials after wear simulation. These results were supported by the qualitative assessment represented by 3D digital topographical micrographs of the non-contact profilometer. The topographical baseline assessment for BruxZir Esthetic group and IPS e.max ZirCAD Prime group represented in **Figure (3)** were significantly increased after wear simulation in the number of peaks becoming with pointed edges and an increase in the number and depth of the valleys as shown in **Figure (4).**

This could be attributed to the repeated friction of the two-body wear test, these grains were pulled out as a result of wear. Accordingly, after the contentious friction due to the wear test; these grains pull out, leaving surface defects, pits, and voids.[11,37,40] Moreover, the detached particles act as abrasives, which leads to an increase in the coefficient of friction between the tested sample and the opposing enamel that might induce a 3-body wear mechanism and an increase in surface roughness.[11]

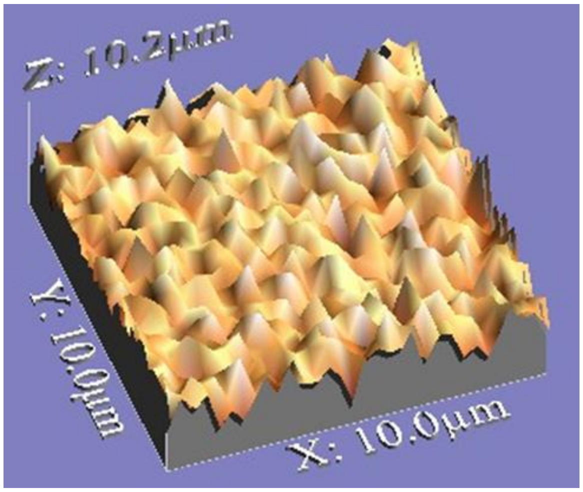

Figure (3): 3D non-contact profilometer interference microscope showing topographic micrograph of IPS e.max ZirCAD Prime disc sample before wear cycles.

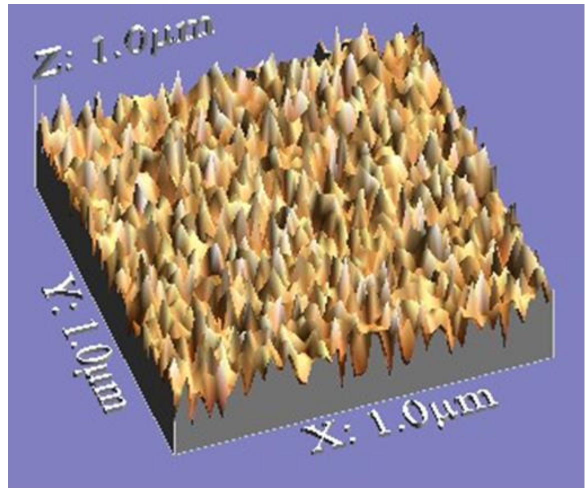

Figure (4): 3D non-contact profilometer interference microscope showing topographic micrograph of IPS e.max ZirCAD Prime disc sample after wear cycles.

These results were supported by the qualitative assessment represented by 3D digital topographical micrographs of the non-contact profilometer, where the topographical baseline assessment of both materials was significantly increased after wear simulation in the number of peaks with pointed edges and an increase in the number and depth of the valleys.

The surface roughness results were in agreement with. Fouda AM et al in 2022[42] who measured the surface roughness of different CAD/CAM materials after 200,000 mastication cycles and reported that no difference in the surface roughness of two types of 5Y_TZP zirconia**.** The results were also in accordance with Zhang F et al. in 2019[40] who reported that the new translucent zirconia with high yttria content showed similar behavior in surface roughness.

The results of this study were in disagreement with the results of Mory N et al. in 2023[16] who reported a significant difference in the surface roughness between two zirconia groups from the fifth generation. These findings could be due to different types of materials used, different sintering parameters or sample size.

Hence, the second null hypothesis, which stated that there would be no statistical difference in surface roughness between **Bruxzir Esthetics** and **IPS e.max ZirCAD Prime** after 75000 wear simulation cycles, was accepted.

The results of the present study showed a positive correlation between wear and surface roughness. 75000 wear simulation cycles caused significant weight loss and surface roughness increase in both types of zirconia materials. However, the difference in changes between both zirconia materials was insignificant.

The limitations of the present study are that it was conducted in vitro, not in vivo where different oral conditions and variations of human beings as different chewing forces, different opposing materials, humidity, PH value, and temperature changes may vary in the amount of wear roughness. The study used 5Y-TZP as a control material, which had a similar composition to the study material. The samples were disc-shaped with flat surfaces rather than anatomical crowns and the test was focusing only on the surface layer.

CONCLUSION

Within the limitations of this study, the following could be concluded:

1- Both materials (cubic zirconia and gradient zirconia) showed a decrease in weight after wear simulation.

2- Both materials (cubic zirconia and gradient zirconia) showed an increase in surface roughness after wear simulation.

3- Both materials showed a similar wear and surface roughness behavior.

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CONFLICTS OF INTEREST: The authors declare no conflict of interest.

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