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Evaluation of hardness and wear of surface treated zirconia on enamel wear. An in-vitro study

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A B S T R A C T

Objective: The present study aimed to examine the effect of aged and polished surface treated full anatomical zirconia and veneered zirconia on wear of opposing natural human enamel surface, and investigate the surface hardness of different zirconia.

Methods: Two types of zirconia blocks were used in this study (Incoris ZI and Incoris TZI), Stainless Steel dies with dimensions of maxillary molars were used, duplicated using addition silicon impression material to form twenty four epoxy dies, then dies were scanned by the OmniCam. Software designing of crowns and copings was performed, then milling of the presintered blocks was done followed by separation of crowns and copings from the blocks and finishing. Sintering of zirconia blocks was done afterwards, regarding the copings feldspathic porcelain was used with the layering technique to veneer the copings to produce full anatomic crowns. All samples were then polished using special polishing kit for zirconia and glass ceramics and thermal ageing was done for two group using an autoclave at 135 °C and 2 atmospheric pressure for 5 h. The twenty-four specimens were weighed before wear testing using a sensitive electronic balance then inserted in a custom made wear machine that simulated the wear mechanism that occurs in the oral cavity with natural mandibular molars as antagonists under a weight of 5 kg (49 N) and the rotation was of 240 cycles/min, the total time of wear was 8 h, so the total cycles performed were 120,000 cycles. The twenty-four specimens were weighed again after wear testing for the weight loss measurements determination. One specimen from each tested group was randomly selected and viewed under Scanning Electron Microscope in-order to determine the wear pattern. Vicker's hardness testing was the performed for all the specimens in-order to detect Vicker's hardness number.

Findings: Full anatomical zirconia specimens revealed the lowest wear values when they were polished followed by higher values after thermal aging. The veneered copings showed significantly higher wear values compared to full anatomical zirconia crowns; with the aged veneered group showing the highest wear readings. These results were the same revealed with the natural enamel antagonists. As regarding hardness, full anatomical zirconia revealed better surface mechanical properties in comparison to veneered zirconia whether zirconia was polished or aged.

Conclusions: Yttria Stabilized Zirconia (Y-TZP) is the ceramic material of choice for all ceramic FPDs in the posterior region, since it underscored remarkable mechanical properties results, and this allowed it to be used for highly loaded ceramic restorations with high performance.

1. Introduction

In prosthodontics, esthetics and mechanical properties are paramount, and zirconia has the highest mechanical properties [1]. Zirconia restorations are aesthetically appealing, when compared to metal ceramic restorations, because they have good optical properties, and absence of the black line at the gingival margin,

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despite its translucency and opacity differ between dissimilar systems, its previous properties favor the use of zirconia over PFM restorations [2].

There are numerous efforts for developing translucent zirconia materials, these attempts are to make zirconia nearly translucent as other glass ceramics, as this would avoid the necessity of zirconia coping and veneering porcelain. This can be achieved by modifying the zirconia surface (grinding, abrasion, laser induced, roughening and heat treatment) [3].

Recently, translucent anatomical zirconia has been introduced into dentistry, so-called “Full Contour”, without covering the veneering porcelain, indicating its zirconia surface is exposed to the oral cavity. The concept of this high translucent zirconia made it not only possible to mill full anatomical restoration without veneering but also to resolve the phenomenon of chipping, different coefficient of thermal expansion that lead to debonding of veneering porcelain, and reduce the wear of the material and antagonist [3].

Zirconia full anatomical crown deprived of veneering porcelain has advantages; no fractured veneering porcelain, additional strength, improved hardness constituent, however it also has the disadvantage of abrading the opposing natural tooth upon the formation of the occlusal surface with it [3].

The possibility to use non-veneered, full anatomical zirconia materials is now presented by several manufacturers who have improved the esthetics of zirconia materials, mainly by reducing the opacity of the material and adding coloring pigments. Additionally, to chipping resistance compared with conventional zirconia-based restorations, there are other possible advantages of anatomical zirconia materials; by oversight of the ceramic veneering, a more solid restoration could be fabricated, and a conservative preparation like full-cast gold could be performed, since there would be no need to maintain space for the veneer porcelain.

Surface treatments may disturb the long term stability and the aging sensitivity of zirconia and the success of the restoration, that's why the effect of fabrication procedures of zirconia and changes in its properties under low thermal degradation is of great interest [4].

The introduction of zirconia-based ceramics as restorative dental materials has generated considerable interest in the dental community. The mechanical properties of zirconia are the uppermost testified for any dental ceramic. This may allow the awareness of posterior fixed partial dentures and permit a substantial reduction in core thickness. These capabilities are highly attractive in prosthetic dentistry, where strength and esthetics are paramount.

The low temperature degradation (LTD) of zirconia is a well-documented phenomenon, impaired notably by the presence of water. The consequences of this aging process are various and include surface degradation with grain pullout and microcracking as well as strength degradation [5].

By means of the existing movement in increasing the life expectancy, wear of dental enamel is becoming an important topic as teeth are required to last longer. An understanding of the mechanisms and controlling factors in tooth wear is therefore, critically noteworthy [6]. Wear rate of ceramics should if possible match that of posterior tooth enamel which is in the range of 20–40 μm per year. Unveneered zirconia ceramic might be an antagonist for ceramic restorations, so its behavior as an antagonist material would be of interest. The use of ceramic has been believed to produce wear under high occlusal force, yet it was less sensitive to wear owing to its crystalline matrix [7]. Zirconia provide high hardness and structural reliability, but their properties differ decisively from those of veneering porcelain resulting in different wear behavior [8].

So, the present study aimed to find whether aged and polished translucent full-anatomical zirconia crowns will wear the opposed enamel surface differently than veneered zirconia frameworks or not, and whether different materials before and after aging will have different hardness characteristics.

2. Material and methods

2.1. Master dies fabrication

Stainless Steel die was machine crafted according to the average dimensions of maxillary molars following the company recommendations for clinical abutment preparation. The die assembly was composed of a Teflon base measuring 15 mm height, 57 mm long and 30 mm width. The base is holding the machined die by means of friction grip.

The die was machined to the average dimension of a molar (11 mm diameter before preparation) and the prepared portion had 7-degree taper and a 1 mm thick rounded shoulder finish line. After reduction, the occluso-gingival height of the molar was 6 mm long having a slight depression on the occlusal aspect simulating the central fossa. The molar die was fixed in place by friction grip through the base not allowing any movement in any direction. The die was snugly fitted in the Teflon base by means of parallelometer, to make sure the die is perpendicular to the base.

2.2. Duplication of the dies

A special rectangular metallic impression tray was used to duplicate the stainless steel die using addition silicon replica impression material to form four impressions.

The impressions were poured with Epoxy resin material to form twenty four epoxy dies in order to use them during the fracture resistance test.

2.2.1. Die scanning

Designing and milling was done by Cerec software. In the administration section the single restoration was chosen, the restoration type was crown and in the design mode was the bigeneric individual for fabrication of crowns, while for fabrication of copings the designing mode was framework. After pressing next the scanning section appeared and the scanning was done by the OmniCam. The software reassembled the images to form the virtual die.

2.2.2. Crown and coping design

After following the software prompts, the next step was to virtually trim the die, outline and locate the finish line of the die. The software guided by the milling parameters i.e. 60 μm spacer thickness and 25 μm proximal, occlusal, dynamic contact strengths. Same steps were done for the copings with same parameters.

2.2.3. Milling of the presintered blocks

After pressing the next button the software chooses the appropriate size of the milling block which was 20/19. The block was secured in place in the milling machine. The same procedure was repeated twenty four times to form the study samples.

2.2.4. Finishing the milled copings

After all restorations were milled, a finishing bur was used to separate the restorations from its corresponding blocks. Finishing burs were used since they produce a smoother finished surface than finishing stones.

2.2.5. Sintering process

Preset program number one was chosen intended for sintering the crowns and copings reaching 1580 °C. The cycle takes around 2 h to be terminated and it's fully automated without any intervention.

2.2.6. Veneer application

Feldspathic porcelain was used by the layering technique to veneer the Incoris ZI copings to produce a full anatomical crowns.

2.2.7. Polishing the samples

All samples were polished following the manufacturer instructions by using polishing kits. The special polishing kit for zirconia was used for the full anatomical zirconia crowns while for the veneered zirconia crowns the glass ceramic polishing kit was used, starting with coarse followed by fine then extra fine finishing stones.

The crowns were divided into four groups each:

- Group 1(GP1) intervention 1: Eight anatomical polished zirconia crowns.
- Group 2 (GP2) intervention 2: Eight anatomical aged zirconia crowns.
- Group 3 (GP3) control: Four Veneered zirconia polished crowns.
- Group 4 (GP4) control: Four Veneered zirconia aged crowns.

2.2.8. Ageing the samples

Thermal ageing was done for both aged groups using an autoclave at 135 °C and 2 atmospheric pressure for 5 h.

2.2.9. Testing procedures

2.2.9.1. Wear testing

2.2.9.1.1. Antagonists collection. Twenty four natural mandibular molars were chosen to function as natural antagonists (Enamel) against the crowns, teeth were examined under digital microscope, magnification (35 X) to ensure the absence of microcracks, stains, or fractures. Any tooth with cavities or restorations was not included in the study.

2.2.9.1.2. Concealment mechanism. A checklist was designed to identify each tooth used as antagonist. A series of sequentially numbered (1–24), opaque envelopes were used. A participant who had the checklist and the twenty four envelopes transferred the chosen teeth to the envelopes and sealed them to prevent bias.

2.2.9.1.3. Customized wear testing machine, and its components. A custom made machine (Fig. 1) was fabricated to simulate wear mechanism, developed and calibrated at the faculty of engineering. The machine is classified as a reciprocating machine, which transforms rotational motion to linear motion.

2.2.9.1.4. Cleaning samples. All samples were ultrasonically cleaned using ultrasonic cleaner for 20 min to insure removal wear particles created by the wear testing from the contact surface.

2.2.9.1.5. Wear assessment using the weight loss technique. The crowns and natural molars that previously prepared were tested for the weight loss measurements. Samples were weighed before and after wear test using a sensitive electronic balance with 0.001g balance sensitivity.

2.2.9.1.6. Scanning electron microscope. Scanning Electron Microscope was used in this study. Specimen from each tested group was selected. The surface of each sample was recorded photographically at different magnification (10000X and 20000X.) to record the wear pattern.

2.2.9.2. Vicker's hardness testing. Fragments from the groups were randomly selected. The diamond pyramid head of a Duramin-1 Vickers hardness tester was applied to the modified surface under a predetermined load (200 N) over 15 s to induce a diamond-shaped indent. The size of each diagonal distance as a result of the impression of the diamond pyramid head indenter was measured using a micrometer screw-gauge within the microscope attached to the Vickers hardness indenter to obtain an average diagonal distance (D) measured in micrometers (mm). The Vickers hardness (V.H) was obtained by calculating the surface area of the indent according to Equation, where deeper impressions were indicative of decreased hardness, identified by an increased D.

$$VH = \frac{2P \ \sin(68^\circ)}{D^2} = \frac{1:854P}{D^2}$$

Where P is the predetermined load applied in Newton (N), D is the average diagonal distance (mm), 1.854 is the Vicker's constant and 688 is the angle of indentation of the diamond pyramid head indenter of the Vicker's hardness tester.

3. Results

Data presented as means and standard deviation (SD) values. Weight Loss (Crown), Weight Loss (tooth), Fracture resistance (N) and Hardness (VHN) showed parametric distribution so; Two way-ANOVA was used to study the effect of Ceramic materials and Aging on mean Weight Loss (Crown), Weight Loss (tooth), Fracture resistance (N) and Hardness (VHN). Tukey's post-hoc test used for pair-wise comparison between the means when ANOVA test is significant. One-Way ANOVA used to compare between the Interaction between variables for mean Weight Loss (Crown), Weight Loss (tooth), Fracture resistance (N) and Hardness (VHN). Independent *t*-test used to compare between different ceramic materials within each Aging and Aging within each ceramic materials on mean Weight Loss (Crown), Weight Loss (tooth), Fracture resistance (N) and Hardness (VHN).

3.1. Interaction between variables on mean weight loss (crown) (gm)

Mean and standard deviation (SD) for the Weight Loss (Crown) (gm) for the interaction between variables presented in Table 1 and Fig. 2.

It was found that translucent polished zirconia showed lower weight loss in grams than translucent zirconia aged than veneered zirconia polished and veneered zirconia showed the greatest weight loss but this was statistically non-significant ($p > 0.05$).

3.2. Interaction between variables on mean weight loss (tooth) (gm)

Mean and standard deviation (SD) for the Weight Loss (Tooth) (gm) for the interaction between variables presented in Table 2 and Fig. 3.

It was found that tooth weight loss opposing Translucent polished zirconia recorded lower values than tooth opposing translucent aged zirconia than tooth opposing veneered polished zirconia and tooth opposing veneered aged zirconia showed the higher values.

3.3. Interaction between variables on mean hardness (VHN)

Mean and standard deviation (SD) for the Hardness (VHN) for the interaction between variables presented in Table 3 and Fig. 4.

Translucent polished zirconia showed higher hardness values greater than translucent aged zirconia, greater than veneered polished zirconia and veneered aged zirconia showed least hardness values and this was statistically significant ($p < 0.05$).

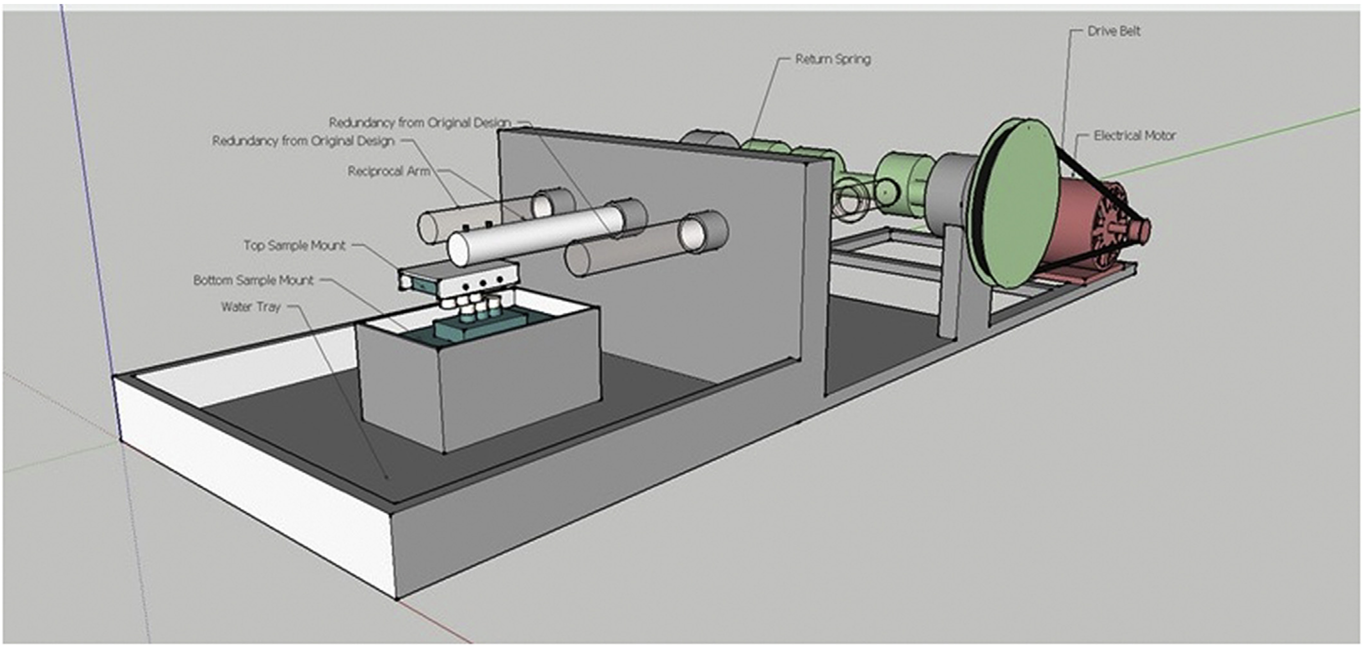

Fig. 1. Customized wear testing machine.

Table 1
Mean and SD for Weight Loss (Crown) (gm) for the interaction between variables.

		Weight Loss (Crown)		Rank	p-value
		Mean	SD		
Interaction	Translucent Zirconia + Polished	0.0043	0.0010	c	≤0.001*
	Translucent Zirconia + Aging	0.0073	0.0015	c	
	Veneered Ziconia + Polished	0.0250	0.0087	b	
	Veneered Zirconia + Aging	0.0403	0.0064	a	

Means with the same letter within each column are not significantly different at p = 0.05.
NS= Non-significant, * = Significant.

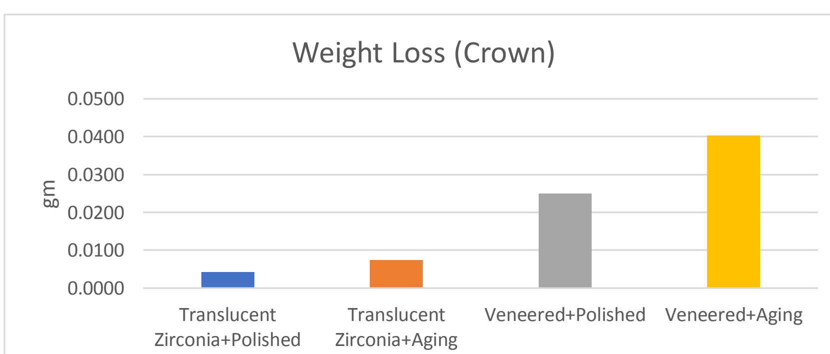

Fig. 2. Histogram showing the mean Weight Loss (Crown) (gm) for the interaction between variables.

3.3.1. Scanning electron microscope

Surface scanning of the different groups by magnification of 3000X revealed that the TZIP group had the least wear patterns and by scanning the corresponding antagonists of the different groups, it was revealed that the antagonist of the TZIP group also showed the lowest wear patterns (Fig. 5). This indicated that the TZIP group has the highest wear resistance and the least wear to opposing enamel.

In the polished translucent zirconia scanning images the original surface state was not changed from the original situation. Minimal crack growth could be detected. (Fig. 6). Aged translucent zirconia images showed rough ploughed surface and deep wear traces and facets. (Fig. 7). Veneered polished scan images in contrast to translucent zirconia, veneered polished specimens showed rough surfaces with cracks and revealed circular defect which was supposed to be abraded cone cracks; defects that appear on the porcelain surface that is indirect contact to the antagonistic contact points. (Fig. 8).Veneered aged scanning microscope images

Table 2
Mean and SD for Weight Loss (Tooth) (gm) for the interaction between variables.

		Weight Loss (Tooth)		Rank	p-value
		Mean	SD		
Interaction	Translucent Zirconia + Polished	0.3506	0.0686	c	0.003*
	Translucent Zirconia + Aging	0.4833	0.0390	bc	
	Veneered Zirconia + Polished	0.5693	0.1161	ab	
	Veneered Zirconia + Aging	0.6747	0.1184	a	

Means with the same letter within each column are not significantly different at p = 0.05. NS= Non-significant, * = Significant.

showed rough surface, pronounced wear traces and probability of some chipped enamel particles. (Fig. 9).

Enamel opposing polished translucent zirconia scanning images did not show definite wear effects. Scanning images of enamel opposing aged translucent zirconia showed smooth surfaces with clear wear facets and cracks. Enamel opposing Veneered polished scanning images showed rough surfaces, with chipping and provoked wear facets. Scanning images of enamel opposing Veneered aged showed rougher surface with pronounced grooves and stress marks.

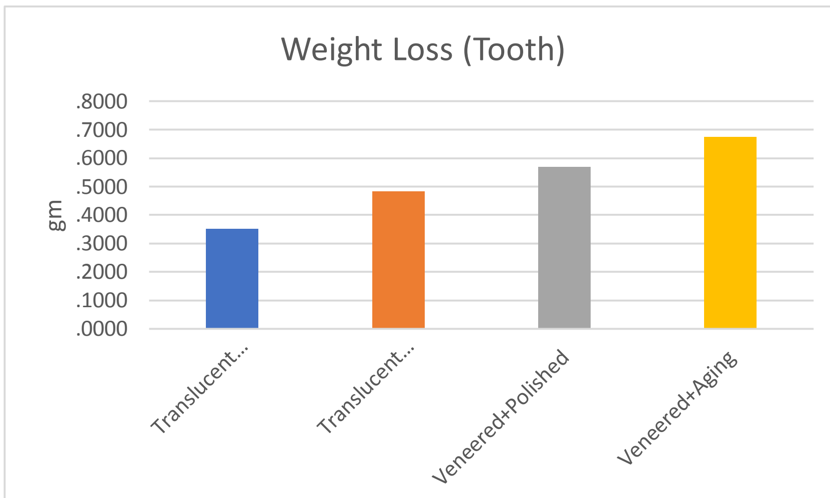

Fig. 3. Histogram showing the mean Weight Loss (Tooth) (gm) for the interaction between variables.

Table 3
Mean and SD for Hardness (VHN) for the interaction between variables.

		Hardness (VHN)		Rank	P-value
		Mean	SD		
Interaction	Veneered Zirconia + Aging	480.60	17.17	d	≤0.001*
	Veneered Zirconia + Polished	640.90	36.50	c	
	Translucent Zirconia + Aging	1103.90	71.29	b	
	Translucent Zirconia + Polished	1424.54	85.21	a	

Means with the same letter within each column are not significantly different at p = 0.05.
NS= Non-significant, * = Significant.

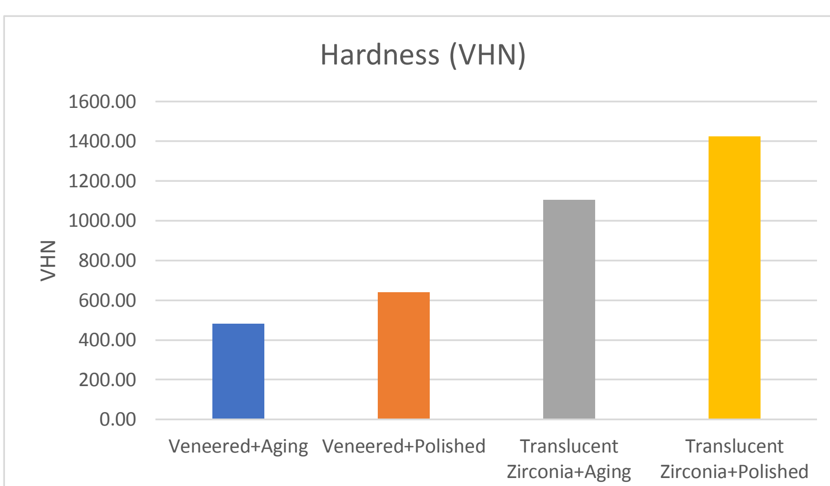

Fig. 4. Histogram showing the mean Hardness (VHN) for the interaction between variables.

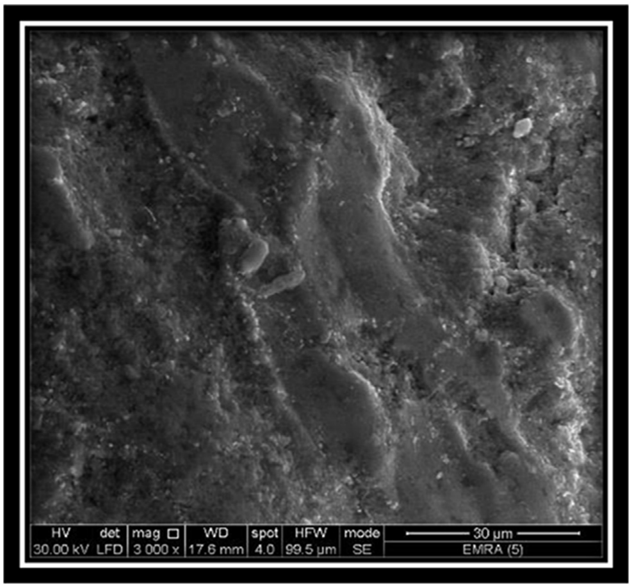

Fig. 5. TZI before wear.

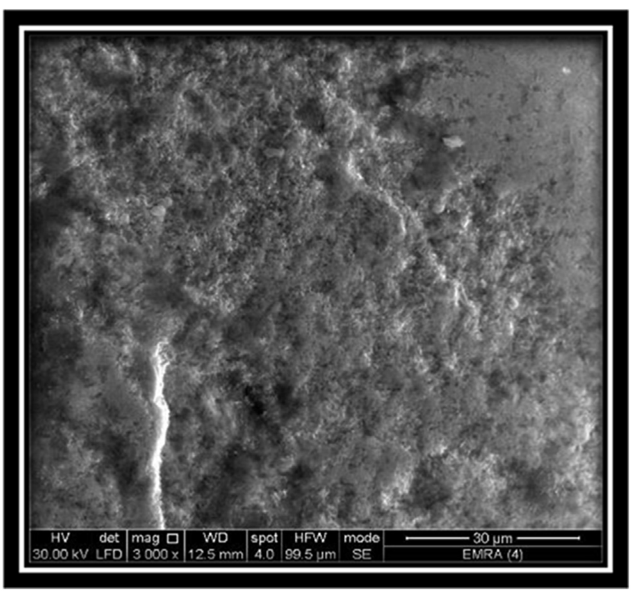

Fig. 6. TZIP crowns after wear.

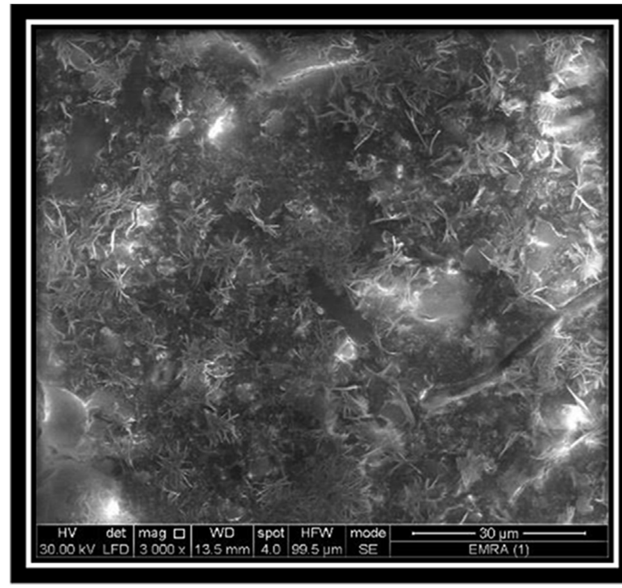

Fig. 7. TZIA crown after wear.

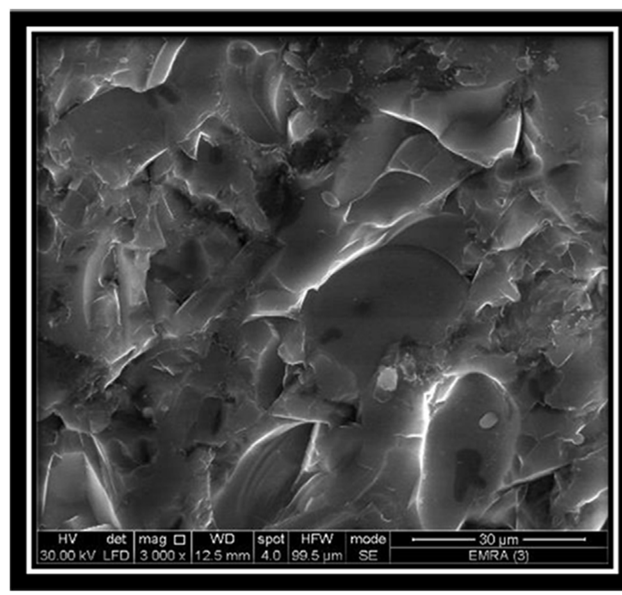

Fig. 8. VP crowns after wear.

4. Discussion

Two types of zirconia; full anatomical and veneered, were used in this study to fabricate single crowns, and in order to fabricate these crowns two types of zirconia blocks were used; InCoris TZI partially sintered blocks and InCoris ZI partially sintered blocks. CAD/CAM (computer aided design/computer aided manufacturing) was the technology used in the present study to fabricate the zirconia blocks.

4.1. Aspects of wear testing

Wear is the removal of the material from solid surfaces as a result of mechanical contact between two comparatively moving surfaces [9].

Zirconia is the interest of the current study, so ideally, it should possess both high wear resistance and minimal abrasiveness. However It should also process wear rate that preferably match that of posterior tooth enamel.

In this study, every effort was exerted to mimic the clinical circumstances as closely as possible, and since during manufacturing and clinical use, the surface of zirconia restorations (copings and crowns) could be exposed to plentiful surface treatments, such as grinding, polishing, glazing, and heat, and as a result the surface of Y-TZP ceramics may be transformed, scratched, and defects might be introduced in it [10]. Polishing rather than grinding or glazing, of all zirconia specimens was performed in this study, representing a method of surface treatment.

The oral cavity is a violent environment with saliva, pH changes, and cyclic loading, so investigation of zirconia should occur in

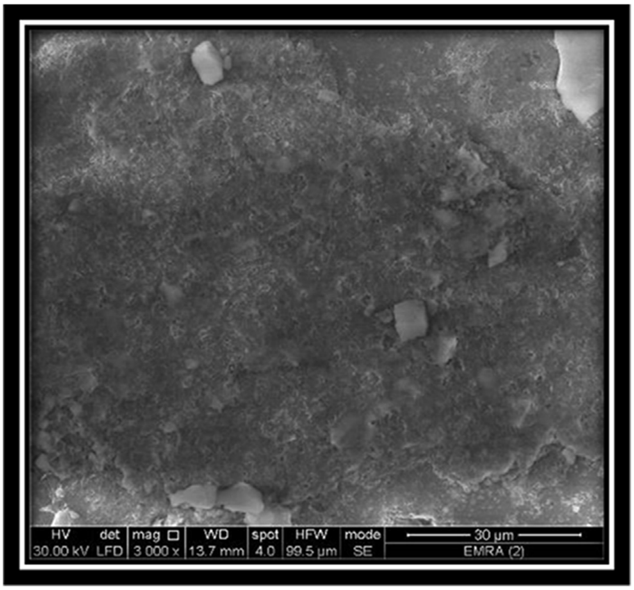

Fig. 9. VA crowns after wear.

conditions that simulate the extended use in the oral environment.

The wear device used in this study provided a sliding action and this sliding motion plays a significant role in simulating intraoral wear; a customized tooth brush simulation device was used, in another word we can say that we performed frictional wear, that is, masticatory attrition, since methodically, attrition is defined as the physiological wearing away of the tooth structure as a result of tooth-to-tooth contact, (two-body wear) as in mastication which this study performed. A loading force of 50 N was applied for 1.2×10^5 cycles with a frequency of about 1–1.6 Hz, and these numbers represented the average mastication loading which is commonly used for in-vitro testing for simulation of the oral situation [11–13].

Wear between the enamel of teeth and zirconia is a very important and zirconia should have a wear degree similar to that of enamel. Therefore, enamel of lower molars was used as antagonists in this study. Water was used as an intermedium fluid to serve as lubricant in order to facilitate mechanical sliding under the lubricated conditions, which was an approach performed also by Kadokawa et al[14].

Assessment of wear was performed in the current study by recording the weight loss of zirconia specimens following wear testing. The findings of this study revealed that polished full anatomical zirconia showed the lowest mean weight loss values of the antagonistic human enamel (0.3506 gm), this was attributed to its extremely smooth surface attained by polishing which maximized its biologic compatibility and diminished its abrasiveness. These findings were in agreement with Mitov et al. [15], Jung et al. [3], Janyavula et al. [16], and Stawarczyk et al. [17].

The wear amount of enamel in this study was higher when enamel was opposed by veneered zirconia (0.6220 gm) mean weight loss in comparison to enamel opposed by full-contour zirconia (0.4004 gm) mean weight loss, this was due to the lower level of enamel hardness when compared to veneering porcelain, and because the fracture toughness of enamel is significantly lower than that of the veneering porcelain. Kim et al. [18] stated the same findings.

The collective wear findings of this study showed higher wear rates of polished veneered zirconia specimens with mean weight loss of (0.0250 gm) and their antagonistic human enamel mean weight loss (0.5693 gm), in comparison to polished full contour zirconia specimens with mean weight loss of (0.0043gm) and only (0.3506 gm) mean enamel antagonistic weight loss. These combined wear findings were in agreement with findings of Stawarczyk et al. [17].

Veneered specimens showed higher wear (0.0327 gm) mean weight loss in contrast to full contour zirconia specimens (0.0056 gm) mean weight loss, which recorded almost no wear values, when natural human enamel opposed both. This may be attributed to the superior physical properties and surface features of zirconia, such as its hardness, bending strength, fracture toughness and density which enabled it to maintain a smooth surface during the wear testing, and this explanation was also in agreement with Kim et al. [18], and Rosentritt et al. [13].

The polished full contour zirconia in this study showed the least wear values of all groups and this was in agreement with the results of Mitov et al. [15].The detected mean weight loss was only (0.0043 gm) after wear simulation in this study.

Wear of the antagonistic enamel was much lower in translucent zirconia than in veneered zirconia, and this was attributable to the fact that zirconia is harder but softer than dental porcelain. This finding was in concurrent with the findings of Jung et al. [3] as well.

When it came to thermal aging, thermal aging led to a decrease in the wear resistance of both zirconia materials tested in this study. Full contour zirconia recorded (0.0043 gm) mean weight loss before aging, and (0.0073 gm) mean weight loss after aging. While veneered zirconia recorded (0.0250 gm) mean weight loss before aging and (0.0403 gm) mean weight loss after aging. Thermal aging as well resulted in higher wear values of the enamel antagonists. So, enamel opposed by full contour zirconia recorded (0.3506 gm) mean weight loss before aging, and (0.4833 gm) mean weight loss after aging. While enamel opposed by veneered zirconia recorded (0.5693 gm) mean weight loss before aging and (0.6747 gm) mean weight loss after aging. But, aged full contour zirconia still revealed lower wear values (0.0073 gm) mean weight loss compared to aged veneered zirconia (0.0403 gm) mean weight loss, and by this translucent full contour zirconia even after aging, was still more wear-friendly in comparison to veneering porcelain, since hydrothermal treatment had only little influence on the smoothness of the ceramic surface, but did not disturb the surface shape, roughness and geometry of ceramic materials to the degree of degradation. These previous findings were in concurrent with findings of Kawai et al. [19] and Papanagiotou et al. [20].

In general enamel in this study achieved wear behavior almost similar to that of zirconia which means that zirconia did not damage natural antagonistic teeth, that's to say, fortunately, zirconia presented wear behaviour similar to that of enamel.

4.2. Aspects of Vicker's hardness testing

Full contour zirconia showed significantly higher mean Vicker's hardness numbers (1264.22) in comparison to veneered zirconia (560.75) in this study, since zirconia ceramic has superior mechanical properties compared to veneering porcelain. This was because of its phase transformation toughening mechanism that prevented crack propagation in the material; the mechanism involved the transformation from tetragonal to monoclinic phase (t-m) at the crack tip and around the crack by localized compressive stresses, this was in agreement with the results of Passos et al. [21].

The hardness testing in this study also revealed higher mean hardness measurements for full-contour (1424.54) and veneered zirconia (940.90) before thermal aging. Thermal aging led to decrease in the hardness values, and so translucent zirconia revealed (1103.90) after aging, while veneered zirconia revealed (480.60). This was due to the t-m transformation which induced micro-cracks [22], but for this transformation to happen, lengthy thermal degradation need to occur, and be able to significantly reduce the values of hardness, and disturb the mechanical properties of the material, this was as well stated by Cattani-Lorente et al. [5], and Miyazaki et al. [23].

5. Conclusions

1. The amount of wear of human enamel is affected by the opposing ceramic materials.
2. Thermal ageing significantly affected the wear loss of full contour zirconia while it insignificantly affects the veneered zirconia.
3. Full contour zirconia was significantly higher than veneered zirconia in wear, and surface hardness.
4. Correlation between the restoration hardness and the degree of wear of antagonistic enamel does not exist, although the surface structure of the restoration affected both.

6. Recommendations

It is recommended to polish the surface of a full-contour zirconia restoration because polished zirconia presents favorable surface properties (wear and hardness) to opposing natural human enamel.

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