Assessment of Coating Zirconium Implant Material with Nanoparticles of Faujasite

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https://doi.org/10.26477/jbcd.v33i4.3016

ABSTRACT

Aim: To evaluate the wettability and microhardness of Zirconium (ZrO_2) dental material when coated with different concentrations of Faujasite.

Materials and methods: 30 circular disks produced from ZrO_2, then each group is classified into 10 control groups, 10 coated groups with 3% Faujasite, and 10 coated groups with 7% faujasite by electro-spun tool to study variable properties in hardness and water contact angle of implant materials.

Results: This study stated the high hardness in 7% of faujasite concentration for ZrO_2, in addition, the contact angle decreased gradually until reach 0 ° in 7% concentration of faujasite with ZrO_2

Conclusion: Water contact angle (WCA) declined till disappeared in (7% wt.) of faujasite coated with the ZrO_2 group, also in the same group the microhardness became high compared with other groups due to alteration in surface morphology of substrate, and properties of coated material.

Keywords: electrospinning, microhardness, wettability, circular disk, Polyvinylpyrrolidone (PVP), water contact angle. **(Received: 28/9/2021, Accepted: 30/10/2021)**

INTRODUCTION

Titanium was used as a dental material for several decades and considered as standardization of dental implants but because of disadvantages such as galvanic corrosion and cellular sensitivity associated with the saliva of humans [(1)], it was an alternative with ceramic dental material such as zirconia(zircon strong transition metal, grey-white, and lustrous named Zirconium), it was the oxide form of Zircon [(2)].

[2]In 1824 Jones Jakob Berzelius was the first to produce zirconium in the form of impure. In dentistry, it was utilized to fabricate esthetic orthodontic brackets, crown/bridge, endodontic posts, implant abutments for rehabilitation of partial and complete arches, and restorations. Faujasite was classified into Y had a Si/Al ratio of more than 1.5, and X had a Si/Al from (1_1.5). Zeolitic materials were characterized mainly by their ion exchange and adsorption capacities. In addition, they can be produced in the laboratory using low-, cost raw materials. These materials have been widely used as adsorbents, molecular sieves, and ion exchangers in the treatment of wastewater, air purifiers, catalysts, and catalyst support [(3)]. The hardest of ceramic was a Zirconia. It was widely produced in a monolithic phase for many clinical usages, was known as yttria-stabilized tetragonal zirconia polycrystal (Y-TZP). Different types of Y-TZP found can be based on heat treatment, sintering, dopants, and additives [(4)]. Absence of toxicity and good mechanical properties of Y-TZP was forced to choose it in dental usage, though it had one problem about matching it with natural teeth to improve esthetic [(5)]. Electrospinning was one way to produce filaments (ultrafine fiber) from many types of materials like composite, polymer, and ceramic. The machine was composed of three parts, conductive collector, syringe with metallic needle, and voltage power supply. This process was divided into various techniques such as bubble electrospinning, Siro-electrospinning, vibration electrospinning, and magneto-electrospinning [(6)]. Various devices were utilized to analyze surface texture and surface properties such as water contact angle (WCA) and microhardness by the X-Ray diffraction and Energy diffraction spectroscopy [(7)].

MATERIALS AND METHODS

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Partially sintered zirconia from VITA was supplied in block disc with a dimension of 98.4 mm in diameter and 12 mm in height, the white shade (A1), was cut into small circular discs (10 mm diameter and 1 mm thickness) as shown in figure (1) the substrates prepared with exo-cad dental CAD Software. To prepare zirconia samples, fractional sintered zirconia block disc was cut according to the selected measurements utilizing a dental computerized three-axis milling system, computer-aided design /computer-aided manufacturing (CAD/CAM) imes-icore, Germany), substrates sintered in sintering heater of imes-icore up to 1650℃, according to manufacture instruction. Then, substrates were cleaned ultrasonically in ethanol for half an hour and then put aside in the air before coating [8]. polyvinylpyrrolidone (PVP) (MW: 40,000) was produced by alpha chemical (made in India), PVP, and distilled water was considered as a solvent for the faujasite manufactured by China with a particle size of about 286.7 as shown in the figure (2) and measurements of parameters device mentioned in table (1). The sample of Zirconia coated with Faujasite by using the electrospun technique is shown below

Figure 1 Zirconium disk

By using an electrospinning machine (made in the USA), filaments was formed after mixing 40% wt. of PVP with concertation of Faujasite (3% & 7%), and addition D.W. to become the mixture solution 5 gm for each percentage, the parameters of the machine were 20 K.V. of the voltage supply, flow rate (1.5 ml/h.), and the distance (13 Cm) between detector with samples and head of needle syringe found in the electrospun machine after the filaments were formed, all samples were kept in room temperature to evaporate the solvent [9].

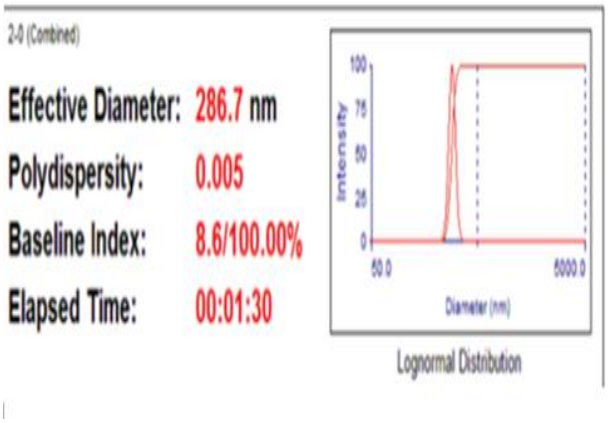

Figure 2 Particle Size Analyzer of Faujasite

Table 1 Measurement Parameter of Particle Size Analyzer Device

temperature	= 25.0 deg. C	Runs completed	= 3
liquid	= water	Burn duration	=00:00:30
viscosity	= 0.890 cp	Total elapsed time	= 00:01:30
Ref. index fluid	= 1.330	Average count rate	=450.1 Keps
angle	90.00	Ref. index real	= 1.590
wavelength	660.00	Ref. index image	=0.000
baseline	Auto (slope analysis)	Dust filter setting	= 30.00

RESULTS

Characterization

Nanofibers were characterized by the energy diffraction spectroscopy (EDS) and x-ray diffraction (XRD) in order to evaluate water contact angle and microhardness for various percentage coating of faujasite and compared with uncoating group.

The energy diffraction spectroscopy (EDS)

It was a method to determine element composition material which formed samples and then analyzed by forming peaks for each ingredient and examination by energy dispersive x-ray spectroscopy (made in Netherland)

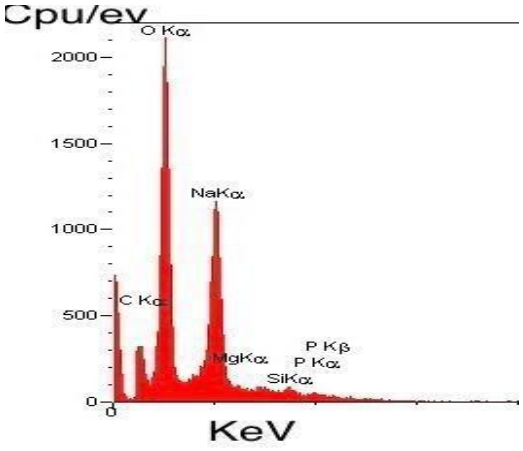

Figure 3 EDX of Uncoated Zirconia

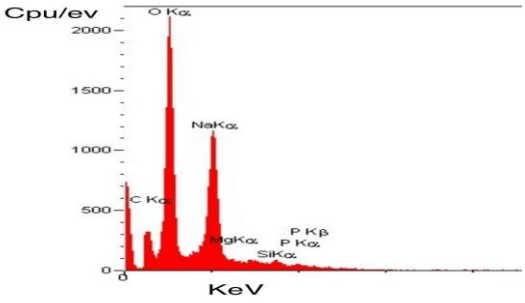

Figure 4 EDX of Zirconia coated 3% Faujasite

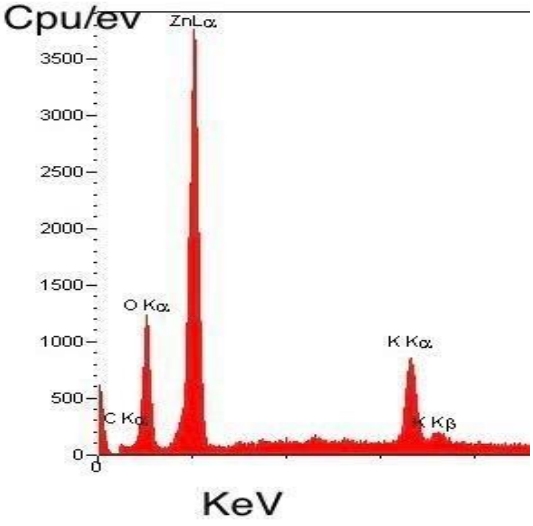

Figure 5 EDX of Zirconia Coated 7% Faujasite

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X-ray diffraction (XRD)

An automated X-ray Diffractometer was employed for Phase analysis by using Cu - Kα radiation (λ=1.5406 A°), XRD-6000, SHIMADZU (Japan) The operation was done at 30 mA and 40 kV. Ambient laboratory temperature using 10s/angular step (1 angular step = 0.02°) was used for taken diffraction patterns, depending on the joint committee on powder diffraction standards (JCPDS) of the international center for the diffraction data the peak indexing was carried out.

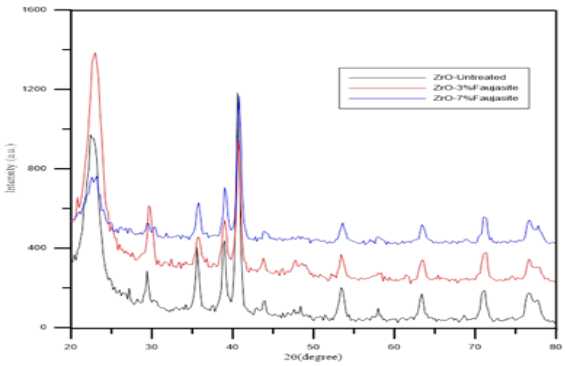

Figure 6 XRD of Uncoated, coated 3% Faujasite(F) and coated 7% Faujasite (F) Zirconia, Groups

Wettability Analysis

In the Department of Chemical Engineering / University of Technology, Iraq. The measurements of wettability were done by goniometer CAM 110, made in Germany, to survey the effect of different percentages of faujasite coating with ZrO_2 and evaluate wettability phenomena on selected disks 30 seconds was needed to capture an image after applying a drop of liquid on an intended surface, the procedure occurred at an ambient temperature.

Microhardness test

Digital Vickers micro-hardness tester Buehler Micrometer 5103, USA, was used to record the micro-hardness of the ZrO_2 disks coated by faujasite with 3% and 7%, and uncoated disks according to (ASTM E92-82, 1997), for 5 seconds 9.8g load was applied to the surface of the disk by using Vickers indenter that joins optical microscopy. An average of 3 different readings was measured from the ten ZrO_2 specimens for each selected concentration to compare between the control and coating groups.

The interpretations EDX for each percentage of faujasite (3% and 7%) in respectively were shown

in fig (4), (5, utilized in the coating of Zirconia and compared it with the control group as seen in fig (3). Also, tables (2), (3), and (4) showed the quantities results for all groups control, coated 3%, and coated 7% of Faujasite in respectively**.**

Table 2 Quantitive Results of Uncoated Group

Elt	Line	Int	K	Kr	W%	A%	ZAF
C	Ka	73.5	0.1726	0.0734	22.25	29.34	0.3298
O	Ka	512.1	0.5986	0.2545	57.49	56.93	0.4426
Na	Ka	434.1	0.1938	0.0824	17.86	12.30	0.4615
Mg	Ka	30.7	0.0128	0.0054	1.11	0.72	0.4908
Si	Ka	28.5	0.0122	0.0052	0.73	0.41	0.7111
P	Ka	20.5	0.0100	0.0042	0.56	0.29	0.7525
			1.0000	0.4251	100.00	100.00	

Table 3 Quantitive Results of Zirconia Coated 3% Faujasite

Elt	Line	Int	K	Kr	W%	A%	ZAF
C	Ka	41.5	0.1049	0.0388	15.87	21.50	0.2442
O	Ka	636.0	0.6807	0.2515	62.48	63.56	0.4025
Na	Ka	483.6	0.1700	0.0628	18.87	13.36	0.3328
Al	Ka	38.2	0.0115	0.0042	0.89	0.54	0.4749
Si	Ka	47.4	0.0145	0.0054	0.88	0.51	0.6067
P	Ka	54.3	0.0184	0.0068	1.00	0.53	0.6776
			1.0000	0.3694	100.00	100.00	

Table 4 Quantitive Results of Zirconia Coated 7% Faujasite

ELT	LINE	NIT	K	KR	W%	A%	ZAF
C	KA	5.2	0.0114	0.0066	3.56	7.10	0.1850
0	KA	314.4	0.2937	0.1696	49.33	73.78	0.3438
K	KA	285.1	0. 1147	0.0662	7.61	4.66	0.8707
Zn	KA	351.8	0.5801	0.3350	39.50	14.46	0.8480
			1.0000	0.5774	100.00	100.00	

Phase analysis was applied to the samples before coating surface structuring, and after coating technique for 3 and 7 % concentration of Faujasite besides the control disks as seen in fig. (6) and basic data in the table (5) showed the three strongest peaks in each group which interpreted amount of changing occurred by the effect of treatment with faujasite that included peak number, 2 theta degrees, amount of diffraction, intensity, and full width at a high maximum of XRD profile

Table 5 Basic Data of XRD for Study Groups

Peaks no.	2 Theta (degree)	Diffraction (Angstrom)	Intensity(I/I1)	FWHM
Uncoated ZrO2				
3	30.1423	2.96248	100	0.5726
8	50.1979	1.81596	60	0.6307
5	34.7594	2.57882	29	0.3893
3% Faujasite coated ZrO2				
2	30.2361	2.95351	100	0.6756
5	50.3643	1.81035	54	0.6924
3	34.9005	2.56871	27	0.525
7% faujasite coated ZrO2				
2	29.927	2.9833	100	0.8327
7	50.0502	1.82097	70	0.9082
8	59.2329	1.55871	43	1.0167

The mean water contact angle in ZrO_2 with 7% faujasite was Zero degree, and for the ZrO_2 with 3% faujasite was 21.46° but for control, ZrO_2 was 77.72° and descriptive statistics of water contact angle test of the 3 groups were summarized in table (6). The table shows the lowest water contact in group ZrO_2 7% Faujasite (F) (0)° and the highest value of water contact in the control group of ZrO_2 (78.65°).

Table 6 descriptive statistic WCA for Zirconia groups

Groups	No.	mean	Std	Min	Max
ZrO_2 C	10	77.72	0.579	76.65	78.65
ZrO_2 3% F	10	21.461	1.049	20.121	22.876
ZrO_2 7% F	10	zero	zero	zero	zero

Statistically, the F-test of the one-way ANOVA test showed a non-significance difference in the water contact angle among the 3 groups, because of P> .05 at three degrees of freedom, as shown in table (7).

Table 7 One-way ANOVA Test of WCA

Source of variance	DF	SS	MS	F	P
Between Groups	1	1586	1586	1.969	0.233 N.S
Within groups	4	3222.3	805.575		
total	5	4808.301			

Three readings were obtained from each of the thirty specimens (ten discs for each group) by using the Vickers microhardness tester by applying 9.8 g load for 5-second descriptive statistics for microhardness was seen in the table (8). The table shows the lowest mean value for group Control (C) of ZrO_2 was 1275.38 H.V. and 1683.65 H.V. the highest mean was in group ZrO_2 7 % Faujasite (F).

Table 8 Descriptive statistic of Vickers microhardness test for Zirconia groups

Groups	No.	mean	Std	min	Max
ZrO2 C	10	1275.38	43.446	1190	1349
ZrO2 3% F	10	1646.4	87.398	1521	1763
ZrO2 7% F	10	1683.65	69.824	1587	1790

ANOVA test seen in table (9) revealed that there was a highly significant difference among the groups $P \leq .01$ at 3 degrees of freedom.

Table 9 one-way ANOVA Test for Vickers Microhardness of Zirconia Groups

Source of Variation	DF	SS	MS	F	P
Between Groups	2	6.290	0.645	14.106	0.000 H.S
Residual	27	62.216	4.48		
Total	29	2.507			

DISCUSSION

In XRD analysis between study groups of ZrO_2 as seen in fig. (7) and table (5) especially at the intensity 100 (I/I1) showed a high difference in the full width at a high maximum of XRD profile (FWHM) which was responsible to describe surface and various material properties like plastic deformation, mechanical properties, and changed in microhardness, many surveys stated FWHM was an accurate signal of the surface work hardening compared with another microhardness testing. In this study, the results showed the (FWHM) at some intensity increased from 0.57 degrees in control groups and become in study groups coating 3% F 0.67 degree till reached to 0.83 degrees with a group of 7% F coated of ZrO_2 [10].

In table (4) compared with table (2), and (3) the EDX interpreted the present zinc clearly with study groups coated with 7% faujasite which was responsible to modify the mechanical and wettability properties, the addition of zinc to the Ti-6Al-4V result in low the modulus of elasticity and enhancement of microhardness, also Zinc (Zn) particles cause improvement in hydrophilicity and biocompatibility through cell proliferation and adhesion [11]. Due to the foundation of oxygen, sodium, silicon, and aluminum in selected samples, there appeared in their low XPS resolution spectra. Faujasite has interpreted the crystallinity via EDS, and FTIR analysis [12].

WCA was decreased gradually with increased concentration of faujasite for dental implant materials until reached WCA = 0^o with coating 7% concentration of faujasite to ZrO_2, interpreted increasing hydrophilicity of materials when coated with Faujasite to improve dental implant properties, was significant with water absorption capability, it was a critical parameter to determine the liquid uptake from the media, Therefore, it can be considered as a significant indicator to evaluate the suitability of biomaterial for tissue engineering usages [13] The θ angle for each scaffold was observed when a drop of water placed on the sample, for PLGA (poly lactic-co-glycolic acid/scaffold), $\theta = 123.8° \pm 5°$, It shows that the structure of pure polymeric scaffold was hydrophobic for PLGA/zeolite 3 (wt.%), θ was declined to $101.83° \pm 6°$, while nano-scaffold was stayed hydrophobic. Water contact angles for PLGA/zeolite 7 (wt.%) and PLGA/zeolite 10 (wt.%) were $94.64° \pm 6°$ and $82.08° \pm 4°$, in sequence. This showed that adding more zeolite to PLGA made it more hydrophilic. These results observed that nanoparticle zeolite powder utilized in the composite had a high tendency to water wettability [14].

In Zirconia groups showed enhancement in microhardness values with coating material due to increase ingrain particle size and surface roughness caused bonding between coating materials and substrate of zirconia [15]. Other investigations may be demonstrated this variation by examination and study increase the microhardness associated with magnifying grain size and diminishing surface roughness due to the load applied diminished upon micro-crack adjacent to the penetration area of diamond tool [16].

CONCLUSION

This study clarified the microhardness and water contact angle (WCA) improved in ZrO_2 groups by enhancement the concentration of nanoparticles of faujasite to develop dental implant material and restriction of implant failure in the future with advanced coated techniques.

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الخلاصة

الهدف:هو تقييم قابلية الابتلال والصلادة لاقراص مادة الزركنيوم دايوكساید المستعمله في زراعة الاسنان عند طلائها بتراكيز مختلفة من مادة الفوجاسيت.

المواد والطرق: ثلاثون قرص مصنع من مادة الزركونيوم دايوكساید تقسم الى عشرة اقراص غير مطليه و عشرة اقراص مطلي ب نسبه aثلاثة بالمية من الفوجاسيت و عشرة اقراص اخرى مطليه بنسبه عشرة بالميه من الفوجاسيت بواسطة جهاز النسج الكهربائي ل بحث اختلاف الصفات في الصلادة وزاوية اتصال الماء لمواد زراعة الاسنان.

النتائج: هذه الدراسة ذكرت زيادة في الصلادة في تركيز السبعه بالمئه من القوجاسيت مع دايوكسيد الزركونيوم بالاضافه الى انخفاض في زاوية اتصال الماء تريجيا حتى يصل الى الصفر في نسبة السبعة بالمئة من الفوجاسيت.

الاستنتاجات: زاوية اتصال الماء تنخفض حتى تختفي في تركيز السبعه بالمئة من الفوجاسيت المطلي لدايوكسيد الزركونيوم وفي نفس التركيز خاصية الصلادة تصبح كبيرة مقارنة بالمجاميع الاخرى نتيجة تغير الشكل الخارجي للمادة المراد طلائها وخواص مادة الطلاء.