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Coating Evaluation of Nanocomposite Mixture of TiO_2 and ZrO_2 by Electrophoretic Deposition and Dip Techniques on Commercially Pure Titanium

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ABSTRACT

This study aim to Development of implant surface modification by electrophoretic deposition and dipping methods of nanocomposite mixture of TiO_2 and ZrO_2 with different percentage of mixing to increase surface roughness and microhardness. Grade (II) commercial pure titanium rod was machined into discs shaped. Discs were divided into 3 groups according to the types of coating used for each percentage: 1st group coated by nanocomposite mixture of titanium and zirconium using electrophoretic deposition, 2nd group coated by the same mixture former using dip technique, 3rd group uncoated commercially pure titanium. Each group were evaluated by using optical microscope, X-ray diffraction analysis, Atomic Force Microscope, Scanning Electron Microscope, Energy-Dispersive X-ray investigations and Vickers microhardness measurements. By comparing between the three groups (EPD, dip and uncoated samples), the EPD coated sample technique was higher in surface roughness and microhardness test. However, Coating of commercially pure titanium with Nano composite mixture 70% TiO_2 and 30% ZrO_2 having more surface roughness than coated with nanocomposite mixture of 50% TiO_2 and 50% ZrO_2.

Key words: Nanocomposite, Electrophoretic Deposition, Dip Technique

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INTRODUCTION

Titanium dental implants considered as the most advantageous treatment option in replacing the missing teeth. Surface modification of implant materials performed in order to improve biological and biomechanical properties, furthermore accelerate the bone cells respond (osseointegration process); which is considered as the main goal for success of the dental implant treatment. The surface properties of the implants materials can be influenced significantly by the different surface modifications [1-3].

Titanium dioxide (TiO_2) was widely described to show superior biocompatibility and osseointegrated properties by enhancing the osteoblast cells attachment and also increasing the proliferation rate, but their low mechanical properties and poor chemical stability, which result in weakening of the coatings and may lead to implant failure, while adding of zirconia to coating layer due to their high chemical–thermal stability, strong mechanical strength, and stress-induced phase transformation toughening, is significantly improve the toughness and the bonding strength of coating [4]. Electrophoretic deposition process (EPD) is a colloidal deposition process which can be considered as a colloidal forming method in which charged particles of the colloidal suspension are deposited on the opposite charged substrate by the utility of electrical field [5]. Dip coating the other method of coating can produce thin homogenous coating layer, in addition to their good control of the coating chemical composition and macrostructure of the coating layer as well as improving the bioactivity of the titanium implants which enhance bone formation (osseointegration) [6].

This study was performed to evaluate the best percentage of mixing that improve the surface roughness and microhardness for EPD and dip coating technique for nanocomposite mixture of TiO_2 and ZrO_2.

MATERIALS AND METHODS

Commercially pure Titanium (grade 2) was cut into small circular discs (20 mm diameter and 1 mm thickness) with a lathe machine then used as the substrate for coating. These discs were grinded by silicon carbide paper starting from 500 to 1000 grit till uniform smooth surface was obtained. The discs were cleaned by Ultrasonic bath of ethanol used to get rid of contamination and debris in 15 minutes, then for 10 minutes in distilled water bath, after that the specimens left to dry at room temperature [7] .

- Commercially pure titanium disc examined by optical microscope, AFM and SEM before coating procedure.

- Two nanoparticle suspension (A & B) were prepared to coat the samples by EPD and dip coating technique and compare to select the best one.

A-First suspension: equal amount of nanoparticle materials (50% TiO_2 and 50%ZrO_2) which about (2 g) of each nanomaterial. This suspension used in EPD and dip coating technique.

B-Second suspension: 70% nano TiO_2 (2.8 g) and 30% ZrO_2 (1.2 g) also used in EPD and dip coating technique.

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For EPD: Mixture of nano TiO_2 and nano ZrO_2 by prepared mixing in a glass container after weighing each quantity by the electronic balance. Then the suspension prepare by adding 4 g of nanopowder to solvent which is 50 ml ethanol inside glass container over magnetic stirrer, after 10 min dispersing agent 0.05 g iodine and 0.05 g of povidone K-30 as a binder for better deposition and adhesion of coating film were add and the stirring continued (15-20 minute) until a colloidal suspension was obtained [8]. The coating of nanocomposite mixture was obtained after 1minute, for understanding the effect of time on the coating thickness the same procedure was repeated for periods of times 3, 5 and 7 min. The power supply was used with applied voltage 60 V for 1, 3, 5 and 7 min [9].

For dipping : The coating suspension consists from dissolution of 0.01 g of P_2O_5 in 50 ml of absolute alcohol (ethanol) with continuous mixing and heating (45C°) on a hot plate stirrer for half an hour. Then 4 g of nano powder was add to solution the temperature was to be maintained at approximately 45°C then the mixture was left over a stirrer for half hour to gain homogenous solution as [10]. The samples coating was done by dipping in suspension in the first disc for 15 sec, the second disc twice dipping 15 sec for each dipping(total 30 sec) while three time dipping (total 45 sec) was done for the third disc.

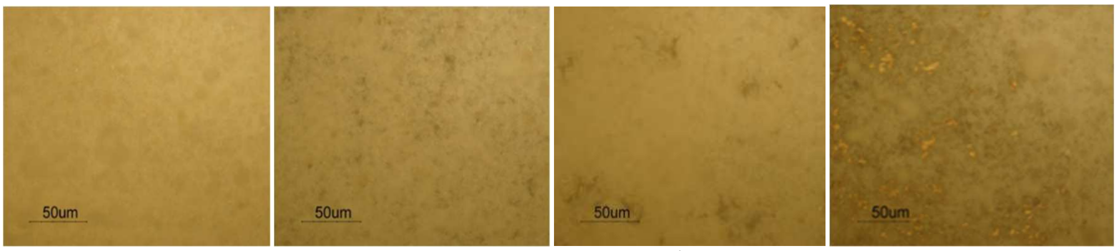
Heat treatment:
Thermally treating of coated discs (Sintering) was carried out by using carbolated furnace (tube furnace) for densification and remove additive. For EPD the samples were dried at room temperature to prevent cracking and then sintered at 300°C for 30 min [8] while for dip coating the samples were heat- treated in air at a temperature of (550) °C for (2) hours [11] .

RESULTS

Optical microscope findings
A series of optical micrographs reveal the microstructure of nanocomposite mixture of 50% TiO_2 with 50% ZrO_2 and also for mixture of 70% TiO_2 with 30% ZrO_2 of coated discs by EPD (for 1, 3, 5, 7 min) and dip (for 15, 30, 45 sec) techniques under magnification powers 50x which illustrated homogeneous thickness layer over the surface of titanium disc and there are no coating cracks at 7 min in both mixture in EPD and 30 sec (twice dipping each dip 15 sec) in dip coating Figure (1, 2, 3 and 4).

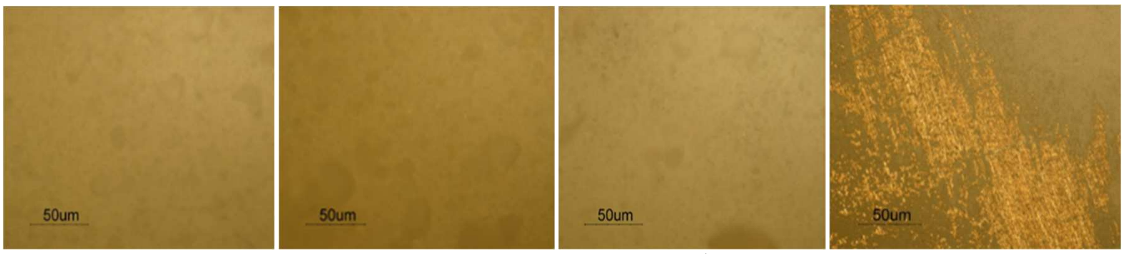
Atomic force microscope
The surface topography analysis including surface roughness analysis were provided by the atomic force microscope for machine cut surface Cp Ti sample and the two suspension of EPD and dip coating techniques (A and B) for selection the best one. Scanning probe microscope analysis shows peaks and projections with the average roughness, average grain size and the granulation distribution charts of the sample

Sample surface	Average roughness	Average grain size
Machine cut (control)	0.029 nm	75.65 nm
EPD coated surface with 50% nano$TiO_2$and 50% nanoZrO_2.	0.34 nm	92.39 nm
EPD coated surface with 70% nano$TiO_2$and 30% nanoZrO_2	1.37 nm	68.86nm
Dip coated surface with 50% nanoTiO2and 50% nanoZrO2	0.638 nm	70.03 nm
Dip coated surface with 70% nanoTiO2and 30% nanoZrO2	1.08 nm	79.07 nm



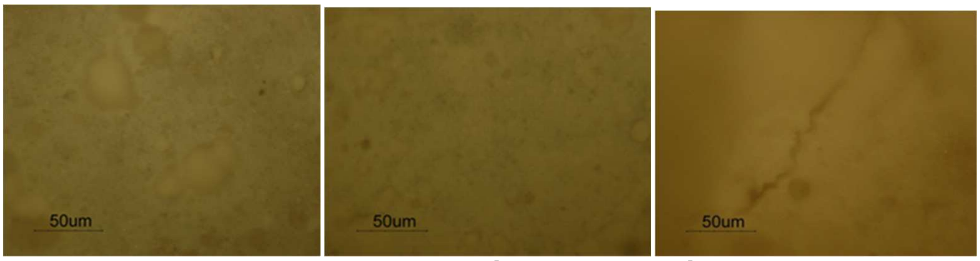
1min **3 min** **5min** **7min**

Figure 1: EPD optical micrographs of CPTi coated by nanocomposite mixture of 50% TiO_2 and 50% ZrO_2 for different times



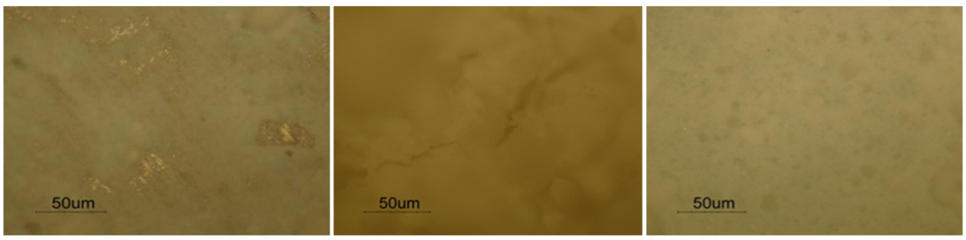
1min **3min** **5 min** **7min**

Figure 2: EPD optical micrographs of CPTi coated by nanocomposite mixture of 70% TiO_2 and 30% ZrO_2 for different times



One time **twice dipping** **three time**

Figure 3: Dip coating optical micrographs of CPTi coated by nanocomposite mixture of 50% TiO_2 and 50% ZrO_2 for different times of dipping



One time **twice dipping** **three time**

Figure 4: Dip coating optical micrographs of CPTi coated by nanocomposite mixture of 70% TiO_2 and 30% ZrO_2 for different times of dipping

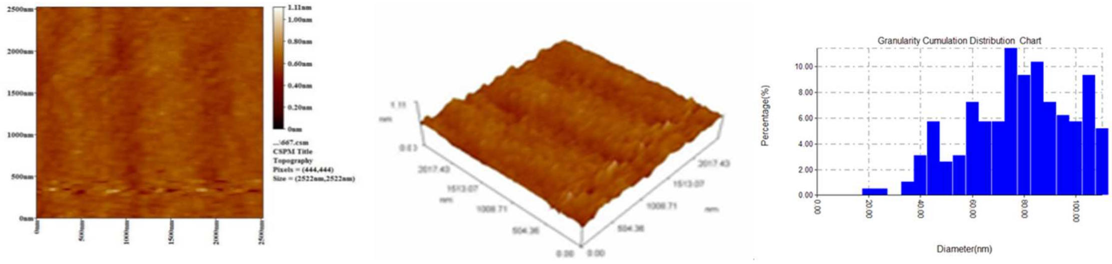


Figure 5: AFM topographies and granulation distribution charts of machine cut CPTi

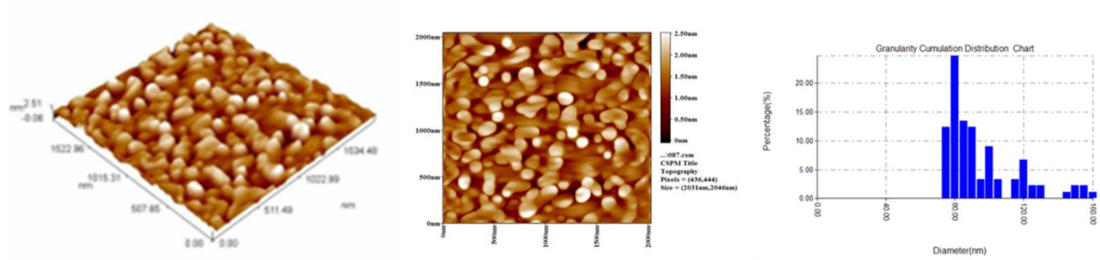


Figure 6: AFM topographies and granulation distribution charts of the EPD coated surface with nanocomposite mixture 50% $TiO_2$and 50% ZrO_2

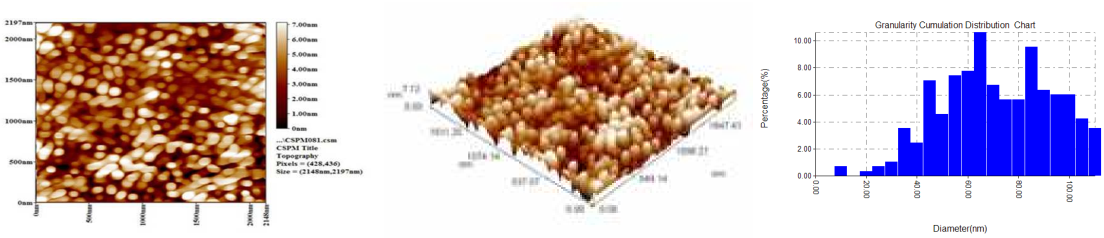


Figure 7: AFM topographies and granulation distribution charts of the EPD coated with nanocomposite mixture of 70%$TiO_2$and 30% ZrO_2

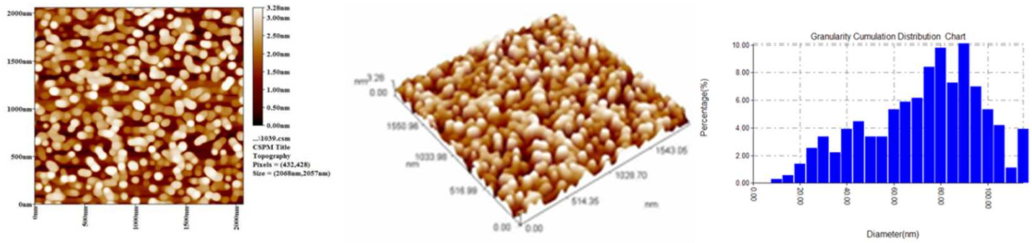


Figure 8: AFM topographies and granulation distribution charts of the dip coated surface with nanocomposite mixture of 50% $TiO_2$and 50% ZrO_2

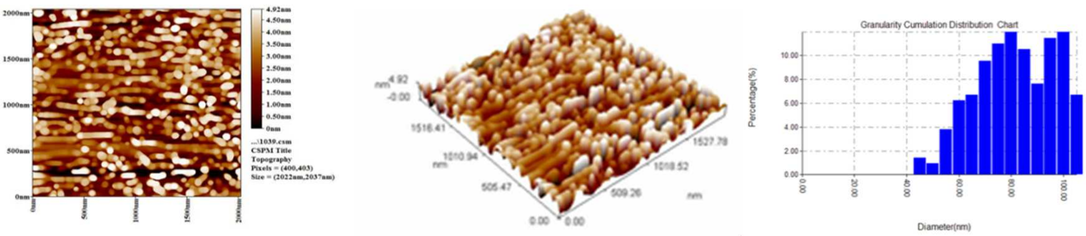


Figure (9) AFM topographies and granulation distribution charts of the dip coated surface with nanocomposite mixture of 70% $TiO_2$and 30% ZrO_2

-According to the results of optical microscope and AFM we select the following parameters to complete the study.

1- **For EPD** the best coating in four samples (1, 3, 5, 7 min) was after 7 min and the applied voltage was 60V in nanocomposite mixture of 70% TiO_2 & 30% ZrO_2 suspension.

2-For dip technique the best coating in three samples (15, 30, 45 sec) after 30 sec in twice dipping (15 sec for each dip) in in nanocomposite mixture of 70% TiO_2 & 30% ZrO_2 suspension 70% TiO_2 & 30% ZrO_2 suspension

Film thickness

The thickness of the coated layers was measured by the Erichsen mini test micro process thickness gauge. The coating thickness of the coated film were increased with the increasing of the coating time.

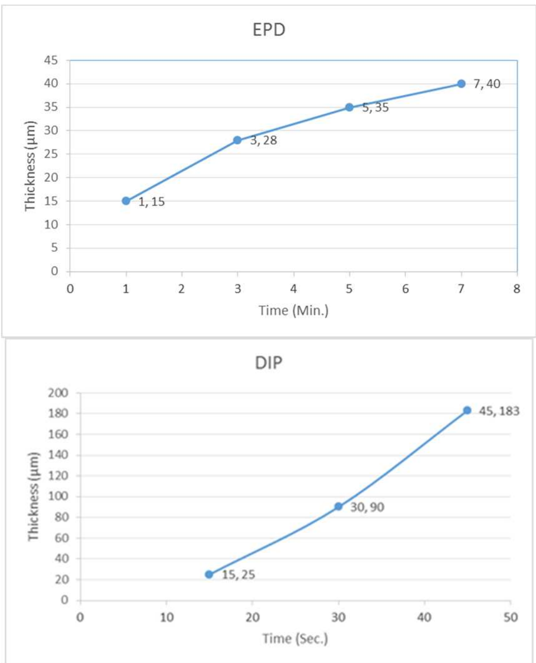


Figure 19: Thickness of EPD and dip coating film at different times

X-ray Diffraction analysis

The results of X-ray diffraction patterns of the three group for samples coated with nanocomposite mixture of 70% TiO_2 and 30% ZrO_2 shown in figure (3.21). The peak was indexed according to the JCPDS (joint committee on powder diffraction standards) International Centre for Diffraction Data, ICDD file # 44-1294 for titanium, #21.1276 for TiO_2 and #37-1484 for ZrO_2.

A- The strongest peaks for uncoated CpTi disc at 2Ө: (35.4), (38.6), (40.5), (53.3), (63.3).

B- The strongest peaks for EPD coated titanium

1- The strongest peaks for TiO_2 at 2Ө: (27.5), (36.1), (41.4), (54.5), (56.7),(68.9).

2- The strongest peaks for ZrO_2 at 2Ө: (24.5), (31.6), (44.1), (50.2), (62.8), (64.1), (65.8).

C- The strongest peaks for dip coated titanium

1- The strongest peaks for TiO_2 at 2Ө: (27.7), (36.2), (41.3), (54.5), (56.7), (69.09).

2- The strongest peaks for ZrO_2 at 2Ө :(24.2), (31.4), (44.1), (50.5), (62.8), (64.2), (65.7).

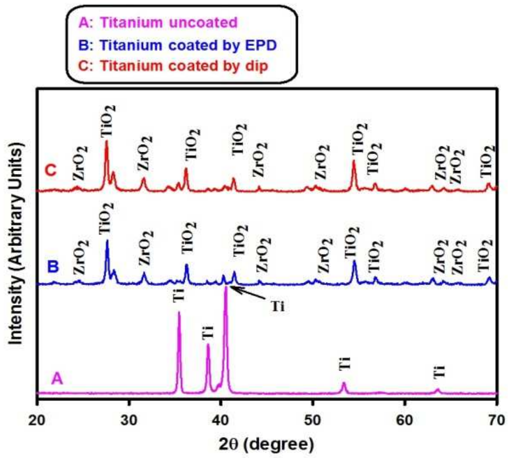


Figure 10: x ray diffraction of three groups

Scanning electron microscope (SEM) analysis:

Field emission scanning electron microscope (FESEM) used for morphological analysis of CP Ti discs (uncoated, coated by EPD and dip coating with nanocomposite mixture of 70% TiO_2 and 30% ZrO_2). In these figures (10, 11, 12) the changes in the surface were observed at different magnification. In the FESEM micrograph of coated discs, there are many irregular projections, and the picture of the surface had a feature or a structure nanoTiO_2 and nanoZrO_2 partials in coated disc.

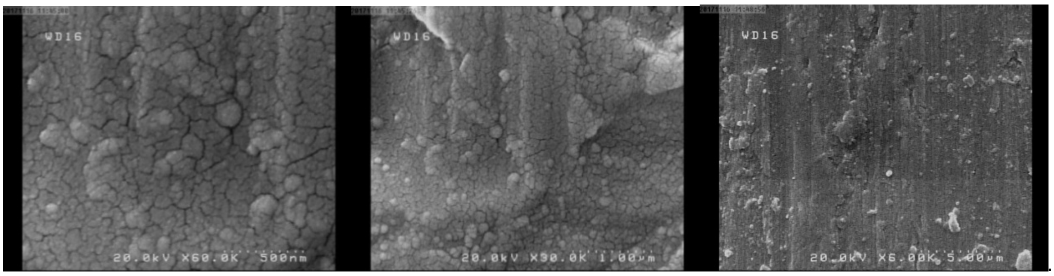


Figure 11: SEM analysis of un coated disc

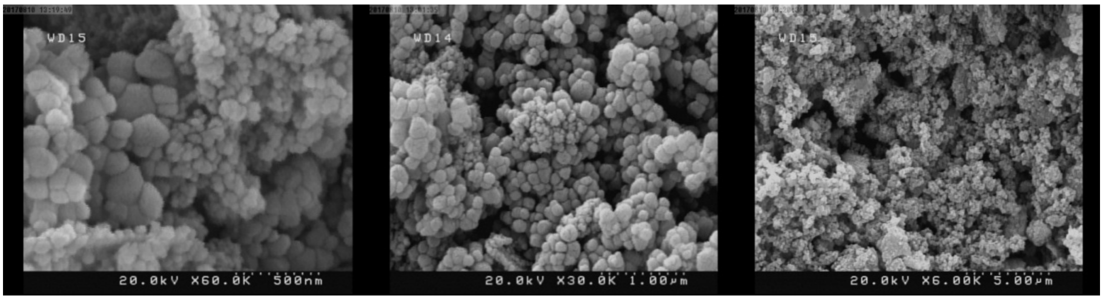


Figure 12: SEM analysis of EPD coated disc

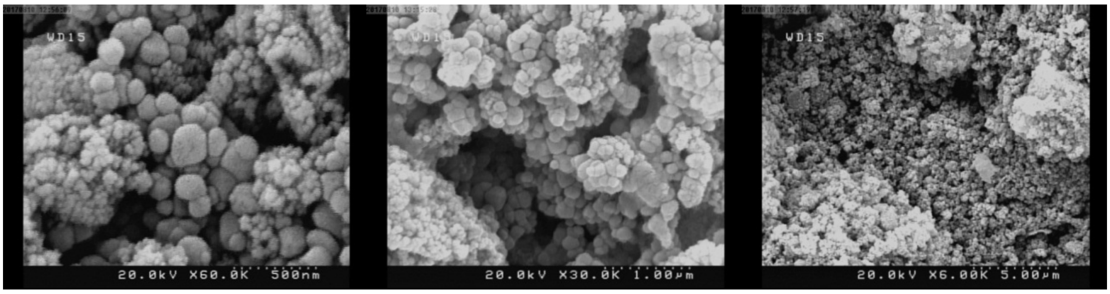


Figure 13: SEM analysis of dip coated disc

Energy Dispersive X-ray spectroscopy (EDX) analysis

EDX analysis for the main Components of the three groups on Cp Ti discs (uncoated, EPD and dip coating techniques with nanocomposite mixture of 70% TiO_2 and 30% ZrO_2) shows the surface chemical composition including weight and atomic percentages of the main elements, are shown in figures (14, 15, 16). An EDX spectrum normally displays peaks corresponding to the energy levels of which the most X-rays received. Each of these peaks is unique to an atom, and therefore corresponds to a single element. The higher the peak in a spectrum, the more concentrated the element is in the specimen [12]. SEM/EDX mapping showed presence of Titanium & Zirconium particles in all coated discs in even distribution. As shown in figure 16 & 17.

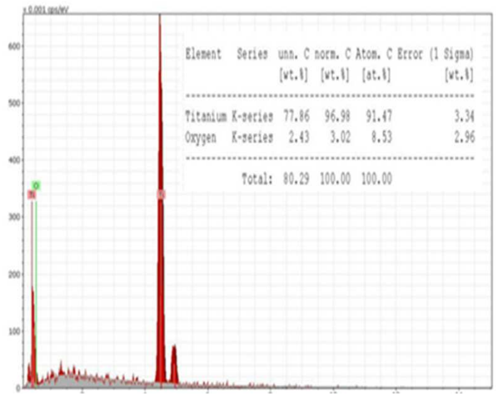
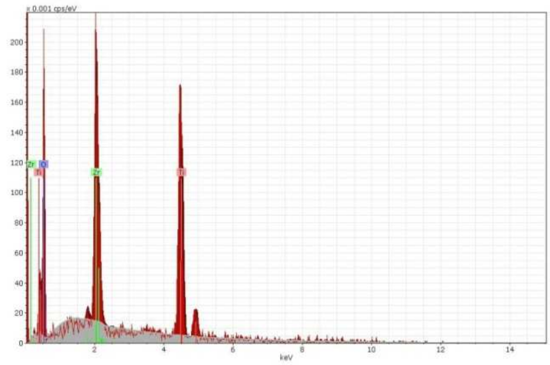
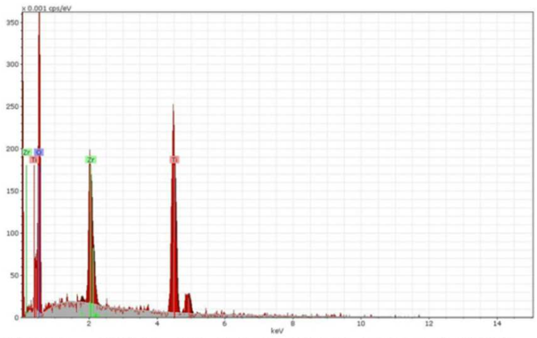


Figure 14: EDX analysis plot for uncoated CP Ti disc



Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
Titanium	K-series	32.33	38.57	24.41	1.75
Zirconium	L-series	21.86	26.08	8.66	1.24
Oxygen	K-series	29.63	35.35	66.93	9.05
	Total:	83.81	100.00	100.00	

Figure 15: EDX analysis plot for EPD coated CP Ti disc



Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
Titanium	K-series	33.71	36.18	19.08	1.68
Zirconium	L-series	14.19	15.23	4.22	0.86
Oxygen	K-series	45.28	48.59	76.70	10.92
	Total:	93.17	100.00	100.00	

Figure 16: EDX analysis plot for dip coated CP Ti disc

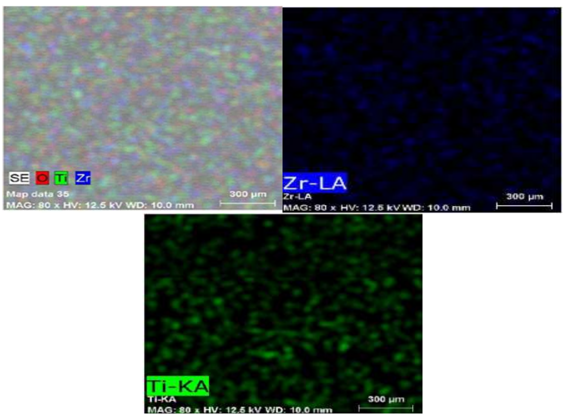


Figure 17: Show SEM/EDX mapping of the two elements Ti and Zr in EPD coating

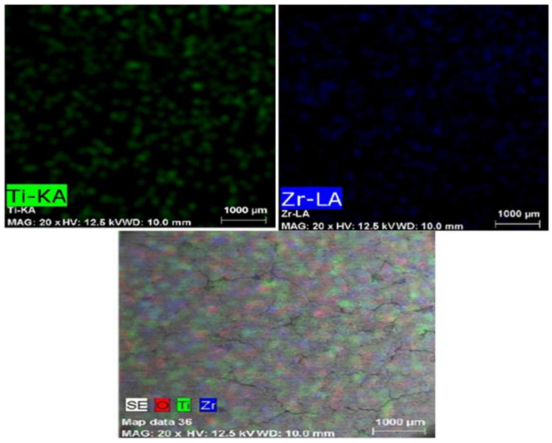


Figure 18: Show SEM/EDX mapping of the two elements Ti and Zr in EPD coating

Micro hardness test

A thirty samples of CpTi discs ten for each group (uncoated, EPD, dip) were tested by using the Vickers micro hardness tester. In order to determine the indentation hardness, the sample subjected to 1.961 N (200 g) load for 15 sec.

Descriptive statistics were obtained for the three experimental groups control, EPD and dip for microhardness shown in table (1). The table shows the lowest mean in control group and the highest mean in EPD.

Table 1: Descriptive statistic for microhardness in Vickers Pyramid Number (HV)

	N	Mean In (HV)	SD	SE	95% Confidence Interval for Mean		Min	Max
					Lower Bound	Upper Bound		
Control	10	214	22.9	7.26	197.28	230.69	179.5	251.3
EPD	10	266.3	20.4	6.45	251.70	280.91	238.9	296.4
DIP	10	222.4	8.7	2.75	216.20	228.69	210.5	235.5

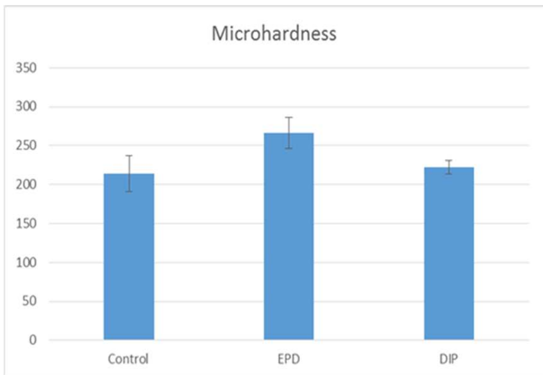


Figure 20: Chart Compare between different groups in microhardness

ANOVA test was used to test the micro hardness of three group, see in table (2). It has been found that there is highly significant difference among the groups ***P≤ .05*** at 2 degrees of freedom.

Table 2: ANOVA test for microhardness.

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	15668.97	2	7834.486	23.02	.000
Within Groups	9186.77	27	340.251		
Total	24855.75	29			

Multiple comparison revealed that there is highly significant difference between the control and EPD and between EPD and dip ***P≤ .05*** while the multiple comparison between the control and dip showed a non- significant difference ***P ≥ .05*** . As shown in table (3)

Table 3: Multiple Comparison

		Mean Difference (I-J)	Sig.
Control	EPD	-43.8700	.000
	DIP	8.1800	.330
EPD	DIP	52.050	.000

DISCUSSION

EPD play important role in biomaterials treating, handling and control the deposition of a range nanomaterials in the biomedical and biotechnology fields [13] .

In this study the suspension of coating with nancomposite mixture was diluted by using of ethanol in order to increase their stability, ability to diminish the initial viscosity and avoid the agglomeration of Nano particles. Using an organic solvent (ethanol) in EPD has benefit of obtaining better quality of coating layer than aqueous suspension due to lack of the H2 and /or O2 gases as a byproduct of electrolysis of water. This agree with [14-16] when they used ethanol organic solvent as diluted material for EPD suspension to obtain a homogenous coating film. Addition of povidone K-30 and iodine for better deposition of Nano partials material on CpTi surface and provide surface charge for composite this agree with findings of [8] who found the using of povidone K-30 and iodine as additive for obtaining a homogenous suspension and successful deposition by EPD method, when using a composite suspension having 70% of nano-ZrO_2 and 30% of ZnO for coating on Ti-6Al-4V.

The samples of titanium coated by EPD showed that the coating layer thickness increases with increase time of deposition under a constant voltage condition. These consequence may be due to raising in the chance of the particles migration to be deposit on the metallic substrate, and this agreed with the findings of [9,14, 17].

The size of partials in coating suspension is of very importance in responsible the strength of the deposited layer. So, deposit uniformity is limited by the particle size of the powders used in the deposition process [18] .

In this study where the samples were coated with nano TiO_2 and nano ZrO_2 displayed a continuous homogenous surface texture layer, which could be due to fine particle size (nano size), and this may give more opportunity to the nano composite mixture for charging during the electrophoretic this result agreed with findings of [14].

Dip-coating (Sol-gel process) has a number of advantages over other coating processes such as flexibility, control of coating film morphology. This method require simpler than EPD equipment and is of a lower cost, the addition the phosphorous pentoxide (P_2O_5) additive is added to the suspension as a thickening materials this agreed with finding of [19- 21] they demonstrated that using of the dipping method with P_2O_5 as additive to coating suspension can provide many advantage such as control on coating composition and morphology. Poor sticking together of the coating film to substrate regarded as the main problem related with dip coating technique of metallic substrate, this problem usually occur due to large variation among their thermal coefficients, causing the appearance of tensile stresses at interface. This causes failed coatings obtained by higher temperature , so in order to improve the dental implants properties thin biocompatible ceramic films having good mechanical properties enhanced the osteointegration of these implants as found by [22, 20, 23].

The microtopography or fine features consider as an effective factor in encouraging osseointegration by enhancing osteoblasts attachment and proliferation thereby bone formation to the implant surface [9] . The existence of small pits, grooves and machining lines were recognizable on the surface of substrate indicating the presence of rough surface which might enhance osseointegration, this comes in agreement with Albrektsson in1997 [24], who confirmed that a surface with micro irregularities demonstrated a better implant success than a smooth surface. Optical microscope shown that the uniform thickness no cracking and this can suggest that there was no shrinkage in the coated layer as confirm by [20]. In dip coating the best coating layer seen in twice dipping were coating layer appears smooth homogenous with no cracking is due to the controllable way of deposition of the materials and sintering under low temperature also control the amount of P_2O_5 (0.01g in 50 ml of ethanol) this agree with work of [20, 23] and disagree with [25] who verified the presence of crack in the coated layer in optical microscopical examination, with the difference in the coating materials and techniques used other than these in present study.

It is evident from the figure of the XRD in figure: (10) that the surface of the titanium discs is well covered by a mixture of nano TiO_2 and nano ZrO_2 because there was diffraction peaks could be indexed to TiO_2 and ZrO_2 phases according to JCPDS file # 44-1294 for titanium, # 21.1276 for TiO_2 and #37-1484 for ZrO_2 .As XRD shows that the narrower peaks are revealing that the layer consists of highly crystalline form, while broad peaks signify lower levels of crystallinity, this come in agreement with (Ladd and Palmer, 2014) [26].

The coating method using nanocomposite mixture gives nano roughness which appeared from the grain size that was in nano diameter. That was supported by the results of optical microscope. After deposition of coated layer the surface roughness increased which can be estimated from the peaks that appeared on the surface and also the diameter of grains [9, 21].

Results measured by the atomic force microscope revealed that the roughness of the surface increased from 0.029 nm for control sample to 1.37 nm and 1.08 nm for EPD and dip respectively. Surface area increase with increasing the surface roughness on the implant and there by promote the opposition of bone [27] .

Results provided from the scanning electron microscope reveals that the results of the EPD coated samples more homogenous than dip coated sample , it showed more voids and irregularity which may be caused by the evaporation of the liquid phase which largely depends on the type of suspension and also on the thickness of the coating film this agreed with [21] who found the irregular projections, pores and void throughout the sample of dip coating of when coated titanium implant with titanium oxide nanoparticles. Vickers microhardness test for the uncoated CpTi and coated by EPD and dip, samples the results shows that the highest micro hardness value for EPD coated sample while the least value seen in uncoated sample.

The variations in the values of micro hardness showed there is highly significant difference between the control and EPD and between EPD and dip while the multiple comparison between the control and dip showed a non- significant difference this variations belong to the dissimilarities the surface topography between the coated and uncoated samples surfaces. Moreover, the variation in mechanical properties values might occur as a result of the alteration in the nano material distribution and the microstructure of these deposits (such as grain size, porosity and crystallian structure) and that produced by the modification of the substrate surfaces by each technique. These findings are also in agreement with that of [28, 29], who demonstrated that micro hardness values highly influence by the variations in the coating and sintering conditions when used the titanium oxide as coating material on titanium surface.

EDX analysis in figure (14, 15, 16) of the experimental groups (EPD, dip coated) and control uncoated discs revealed that the main elements in the surface of the all groups were Ti and O. However, both EPD and dip coated disc shows a markedly increase in the surface oxide percentages, as compared with uncoated one. This increase in oxygen percentages occurred as a result of the increase in titanium dioxide and zirconium oxide film thickness. This agrees with findings of [30], who showed that an increase in oxygen percentage in the (EDX) plot can be related to the increase in the film thickness of TiO_2 that produced on commercial pure titanium substrates. In energy-dispersive X-ray spectroscopy (EDX) analysis, there is a fairly uniform distribution of particles by mapping the coated plate.

CONCLUSION

A nano composite coating with a mixture of 70 % nano TiO_2 with 30% nano ZrO_2 by EPD and dip coating can be successfully synthesis, with homogenous bioactive layer , uniform thickness of coating and increase surface microhardness**.**

Conflict of Interest

The authors deny any conflicts of interest to this study.

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