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### RESEARCH ARTICLE

#### STUDY THE EFFECT OF COATING USING NANOPARTICLES ON THE CORROSION OF ALUMINUM ALLOY IN DIFFERENT SOLUTIONS

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#### Abstract

Corrosion resistance of metals is one of the most important topics for researchers. In this work, epoxy-TiO<sub>2</sub> nanocomposite has been developed for protection of 6061AL alloy. The dip-coating technique used to deposit the coating on 6061 Al alloy substrate. Different concentrations of TiO<sub>2</sub> NPs (1,3,5)Wt% have been used to study the corrosion behavior in 3.5% NaCl medium. Also, 200,150 and 100 mm/min dipping speeds have been employed for 1%wt TiO<sub>2</sub> to show their effects on coating layer and corrosion rate. Characterization of coated surfaces were investigated by AFM, FESEM and EDS, while the electrochemical measurements was using to evaluate protective nature of the coating. The results have shown that the corrosion rate has decreased with 1wt% of TiO<sub>2</sub> NPs concentration. The corrosion rate (mm/y) of (7.131×10<sup>-4</sup>, 1.803×10<sup>-3</sup> and 8.183×10<sup>-3</sup>) have been obtained with (1, 3 and 5 wt% TiO<sub>2</sub>) respectively. The results have been confirmed by using a constant dip-coating speed of 200 mm/min, and the results of the corrosion rate for 200,150 and 100 mm/min have been 7.131×10<sup>-4</sup>, 7.984×10<sup>-4</sup> and 8.293×10<sup>-4</sup> respectively. The morphology of coating film which characterized by SEM, have shown that the TiO<sub>2</sub> NPs spread and was covered by epoxy very well. The AFM test used to study the surface average roughness of coating film where the Ra for sample with epoxy coating is 24.4 nm and for sample with (epoxy\_1%wt TiO<sub>2</sub>) is 1.508 nm.

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#### Introduction:-

A wide range of properties of aluminum and aluminum alloys that can be engineered just to the difficulties of specific applications, such as in advanced nuclear reactors, aerospace, metal/air batteries and surface coating, through the choice of alloy, fabrication process and temper condition. By using numerous combinations of its beneficial properties such as lightness, strength, corrosion resistance, formability and recyclability, Al is being active in any increasing number of applications [1].

Commonly aluminum alloys (6xxx series) are used in railway structures, aircraft, automotive and marine applications where better corrosion resistance and good mechanical properties are desired [2, 3]. Alloys of aluminum are extensively used in seawater industries for their great strength, low density and low price. On the other hand, the application of Al alloys is limited for their poor corrosion resistance and high chemical activity. Even though the

oxide layer formation rises the corrosion resistance of the alloy, this layer is easily eroded. This erosion can be credited to defects in the oxide layers. Chloride ions and atmosphere are exposed suffer attacks, leading to more deterioration [4]. There are numerous corrosion resistant systems obtainable on the market today, coatings are one of these systems have been completely positive in inhibiting corrosion, there has continuously been a question mark on the dependability of some critical properties of the coatings, such as resistance to water permeation, precise control over surface morphology and uniformity and adhesion to the substrate, For over a decade researchers and scientists in the coatings and corrosion fields have turned to nanotechnology in a constant effort to be more precise in explaining the corrosion natural phenomena at the nano level ( $10^{-9}$  m). The nanotechnology application has produced many invaluable coatings and materials to minimize corrosion degradation [5].

Corrosion prevention of metal by nanocoating, its manufacturing process begins with the selection of an appropriate substrate. The substrate can come from the typical metallic elements (e.g., Al, Ti, Cu or Au) and alloys (e.g., Ni-Fe, Ti-Al or Pb-Sn) for industrial applications or from ceramics (e.g., AlN, GaN, TiN, SiO<sub>2</sub>, TiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, ZrO<sub>2</sub>, etc.) and semiconductors (e.g., Si and quartz) for micro electro mechanical (MEMS) devices such as electronic actuators, filter elements, catalytic membranes, micro pumps and drug delivery systems [6].

A. S. Hamdy [7] discussed vanadate compounds friendly eco-environmentally surface treatments as replacements to the toxic chromates for Al alloys corrosion protection. Cleaning, etching by (KOH) process includes, and then treatment by vanadate sol-gel was prepared. The corrosion resistance of AA6061 T6 in 3.5% NaCl solution has been measured before vanadate treatment using DC polarization techniques and AC impedance spectroscopy. Scanning Electron microscopic (SEM), (EDS), (XPS) and (AFM) was performed for surface examination. The results show that the process of etching before vanadate treatment improved creation of a uniformly dispersed compact surface Al-oxide V-rich film of smooth arrival and reserved the active surface sites and thus stopping localized corrosion. Vanadate conversion coatings seem promising as alternatives to toxic chromating for the corrosion defense of Al alloys in sodium chloride solution.

S.S. Pathak [8] has established a Sol-gel, Thermal-cured, waterborne organosilane-polyester coatings using polyester resin and methyltrimethoxysilane, 3-glycidoxytrimethoxysilane for increasing corrosion resistance of AA6011 AL alloy. The coatings morphological features and structural were examined by (AFM) and (FT-IR). Results display that the coatings on Al were smooth, defect-free and continuous. Performance of the SiE coatings were examined and matched with pure polyester coating and organosilane coating using contact angle measurement, pencil hardness test and potentiodynamic polarization studies. SiE coatings provided better hydrophobicity and hardness than the polyester coating. Furthermore, polarization studies have shown that the SiE coated substrate provided a better corrosion resistance than the polyester coated substrate due to creation of aluminum-oxygen-silicon covalent bond at coating-Al interface.

N. F. Atta, et al. [9] have prepared TiO<sub>2</sub> film by Sol-Gel and working as a corrosion protective coating for aluminum alloy (AA2024) in 3.5% sodium chloride. The coatings morphology was examined using (FESEM). The impedance spectroscopy measurements and electrochemical polarization used to evaluate the protective nature of the coating. Modification of the sol-gel coating with titanium shows that the corrosion resistance of the alloy improved. The electrolyte penetrated with slower rate to the underlining of the coating related to the non-permeable and insulating nature of the coating. Instead, the modification of the sol-gel structure enhanced with presence of TiO<sub>2</sub> that allowed ended with a high cross-linked structure. However, high chance for galvanic corrosion founded with increasing the content of titanium that resulted in increasing the corrosion rate. Increasing resistance of Corrosion of the sol-gel coating include titanium dioxide explained by electrochemical impedance spectroscopy measurements provided. Changes imparted to the surface with and without coating revealed by the surface morphology before and after corroding solution.

F. Yu et al. [10] have modified sol-gel coating containing hybrid titania, corrosion protection of AA2024-T3 alloy investigation by pigments of a co-polymer polyvinyl butyral insert to a conductive polymer polyaniline and a corrosion resistant glass flake (GF). Coatings effect tested by (EIS) during immersion in 3.5% sodium chloride solution. A volume ratio of sol-gel/PVB = 4:1 revealed that modification by PVB can increase the resistance to electrolyte ingress of the sol-gel layer, intermediate layer of the PANI between coating and substrate produced a chemically stable as agglomerate-type particles. Additionally, the modification of the sol-gel with GF provided a physical barrier properties to water uptake. GF increases the thermal stability and decreases the affected the

condensation reactions taking place during the sol-gel synthesis which indicated by water contact angle measurements and (DSC/TG) analysis.

M. H. Hussin [11] has evaluated a sol-gel coatings consist of hybrid (APTES-TEOS) and single (TEOS) to investigate anti-corrosion performances of Al alloys exposed to 3.5wt% sodium chloride. Electrochemical impedance spectroscopy and potentiodynamic polarization employing to study the effect of coating. The single precursor silanol coating offered the lowest current density and corrosion rate in comparison with Hybrid sol-gel coating showed by three corrosion analysis techniques. According to Tafel curves the hybrid sol gel improved performance of Al alloys against corrosion considerably.

### Experimental Part:-

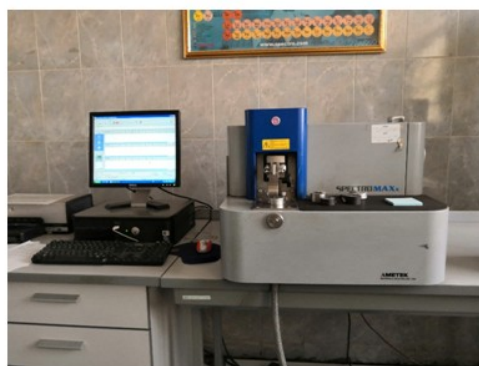
#### Materials:-

##### Substrate

In this work, a main substrate of 6061-T6 aluminum alloy has been used for coating process, the alloy has been tested by chemical composition analysis device. Table 1 shows the chemical composition of the alloy, Fig. 1 shows chemical composition device.

**Table 1 shows the chemical composition of the alloy.**

Si%	Fe%	Cu%	Mn%	Mg%	Cr%	Ni%	Zn%	Ti%	Pb%	Al%
0.668	0.409	0.292	0.0668	0.864	0.0867	0.0148	0.0174	0.0194	0.0148	Bal.



**Fig.1. chemical composition device.**

#### Materials used for coatings:-

Table 2 contains the materials that have been used in this work.

**Table 2 The materials used for the sol-gel coating process.**

Material	Formulation	Purity	Origin of manufacturing
Ethanol	C <sub>2</sub> H <sub>5</sub> OH	99.9%	Scharlau, Spain
Titanium dioxide	TiO <sub>2</sub>	99.5%	Sky spring Nanomaterials, Inc., USA 10-30 nm (CAS# 1317-70-0)
Epoxy A Hardener B		100%	Sika Australia Pty Limited

#### Substrate Preparation:

The first step is cutting a rod from 6061-T6 aluminum alloy of size (15 mm) radius and (4 mm) length for each sample used as the main substrates in this investigation. The second step is grinding of samples by silicon carbide papers (220-800) using grinding and polishing device (MetaServ 250, China) as shown in Fig. 2, and then

ultrasonically cleaned by ultrasonic cleaner(MTI corporation, USA) as shown in fig. 3 for 10 min with ethanol and distilled water.



Fig.2. Grinding and Polishing devise.



Fig. 3. Ultrasonically cleaner device.

#### Preparation of Nanocomposite:-

Nano Composite polymer consists of epoxy/TiO<sub>2</sub>nanoparticles has been prepared. Liquid epoxy was blended with the hardener at weight ratio 2:1 Epoxy/Hardener . Nanoparticles of TiO<sub>2</sub> were added with different weight % to the blended polymer as revealed in Table 3. Themixing of Nanocomposite coating done by stirrer for 30 minutes.

**Table 3 Preparation of nanocomposite of epoxy/TiO<sub>2</sub>.**

No.	Epoxy(wt%)	TiO <sub>2</sub> (wt%)	Dipping speed(mm/min)
Sample A	100	0	200
sampleB	99.00	1	200
sampleC	97.00	3	200
sampleD	95.00	5	200

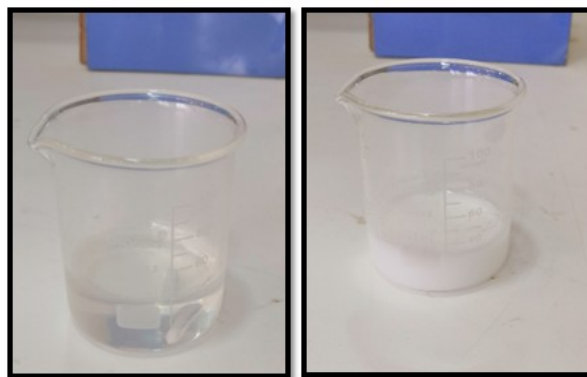


Fig.4 epoxy solution and nanocomposite solution of TiO<sub>2</sub> NPs.

The specimens have been used for applying (Epoxy:TiO<sub>2</sub> ) nanocomposite coating by dipping process on6061 aluminum alloy the specimens as shown in Fig.5.The dipping with automatic process has been processed at speed 200 mm/min for 10 seconds. The samples are left for curing at atmosphere condition. To study the effect of dipping speed on the behavior of corrosion, the coating condition that gave best corrosion resistance had been repeated on other specimens but with different dipping speeds as indicated in Table4.



Fig.5 Dip coater device and specimen during coating.

Table 4 Specimens at different dipping speeds.

No.	Epoxy (wt%)	TiO <sub>2</sub> (wt%)	Dipping speed(mm/min)
Sample B1	99	1	200
Sample B2	99	1	150
Sample B3	99	1	100

**Characterization Tests:**  
(FESEM/EDS)



Fig.6 Field Emission Scanning Electron Microscopy.

In this study, Field Emission scanning electron microscopy (TESCANMIRA 3FRANCE) Fig.6,has been used to identify the surface structure and the morphology of the coated substrates. The local chemical composition of coatings have been analyzed using EDS, which is found as an accessory with SEM.

**(AFM) :**

The surface morphology including 2D and 3D images and partial size of uncoated and coated substrates have been observed by AFM (JPK nanowiziped) Fig.7.

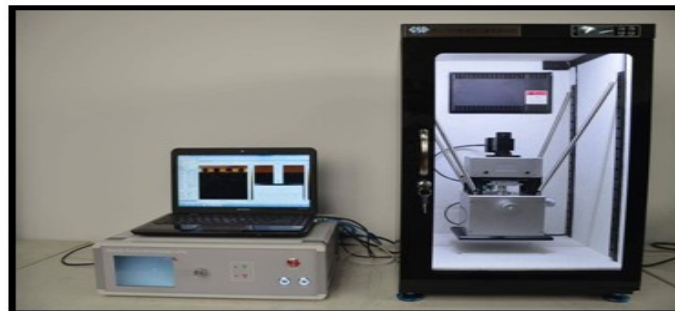


Fig.7 Atomic Force Microscope device.

**Electrochemical test:**

The test has been performed at the Ministry of Science and Technology through the employment of electrochemical system PARSTAT 2273 as shown in fig.8. The testing completed by Conventional three-electrode cells. Al alloy sample the first electrode as (working electrode), a reference electrode of Ag/ AgCl (second electrode) using in cell, and platinum is used as a counter electrode as third electrode, the three electrodes cells have been immersed in 3.5% sodium chloride solution. In this testing, fresh solutions are used for all the electrochemical tests. Also 1cm<sup>2</sup> areas have been left for exposure to the electrolyte. The variation of open circuit potential is measured Since the first minute of the immersion process,. The curves of the potentiodynamic polarization have been obtained at 1 mV/s .

The following equation has been used to estimate corrosion rate based on corrosion current:

$$\text{Corrosion rate (mm/y)} = 3.271 * 10^{-3} * I_{\text{corr}} * \frac{E}{\rho}$$

Where:

**I<sub>corr</sub>** : represented current density (A/cm<sup>2</sup>)

**ρ** : represented Density (g/ cm<sup>3</sup>)

**E** : Equivalent weight



**Fig.8 An electrochemical system.**

**The Results and Discussion:-**

(AFM)

the surface roughness of the coated layers revealed by (AFM) analysis. Table 5 presents the average values of the surface roughness of uncoated and coated substrates measured by AFM analysis.

**Table 5:-** Average surface roughness obtained by AFM analysis.

Samples	Average Roughness (nm)
Uncoated	255.1 nm
Epoxy	24.4 nm
1% TiO <sub>2</sub>	1.508 nm
3% TiO <sub>2</sub>	2.609 nm
5% TiO <sub>2</sub>	5.212 nm

Fig.9 (a–e) shows the AFM images of samples. Fig. 9a revealed that higher value of R (255.1 nm) of uncoated substrate as compared to that of coated substrates. This indicates the significant effect of different coatings produced by dipping process on the surface of 6061-T6 alloy. On the other hand, the AFM analysis showed that the coated substrates are fully covered by nano-scale layers.

In case of epoxy, high value of Ra (24.4 nm) characterizes in topography of coating as a rough surface, having. This might be owing to decreased homogeneity and bigger agglomerations along with in the surface structure of epoxy coated substrate, as shown in Fig.9 b.

The topography of sample 1% TiO<sub>2</sub> NPs coated substrate discloses a greater homogeneity, as shown in Fig. 9c. The surface of the 1% TiO<sub>2</sub> NPs coating is smoother (Ra= 1.508 nm) than other films as it possesses less amount of agglomeration comparing with these coatings.

The topography of sample 3% TiO<sub>2</sub> NPs coated revealed that highly incorporated of TiO<sub>2</sub> with epoxy. Fig. 9d show that coatings obtained with a nanoscale structure and higher homogeneity, along with surface roughness (Ra= 2.609 nm). In case of 5% TiO<sub>2</sub> NPs coating, the film is composed of nano-particles shown in fig. 9e. The topography of this coating characterizes porous structure with a value of Ra (5.212 nm) lower to that of epoxy and uncoated substrate. The presence of larger agglomerations along with reduced homogeneity of TiO<sub>2</sub> particles in coated surface structure (see Fig. 9) is the main factor affected on the surface roughness of this coating.

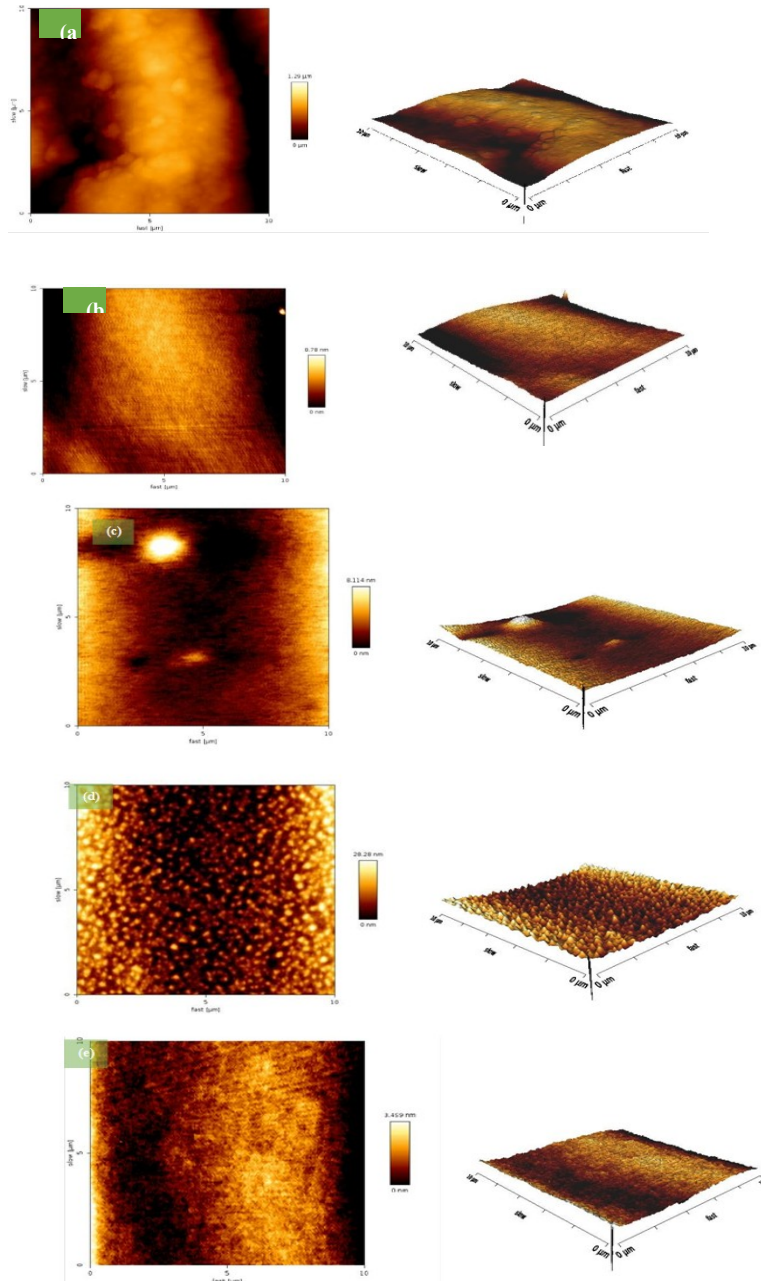
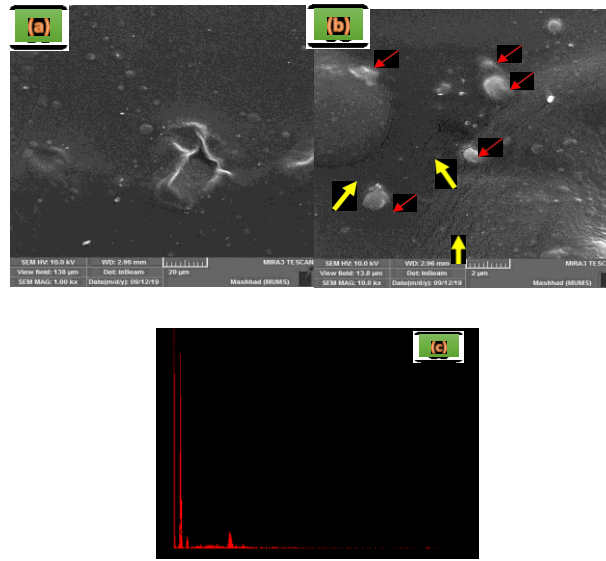


Fig.9 AFM topography images under different presentation mode and resolutions of: (a) uncoated, (b) Epoxy, (c) 1% TiO<sub>2</sub> NPs, (d) 3% TiO<sub>2</sub> NPs and (e) 5% TiO<sub>2</sub> NPs coated substrates.

### SEM-EDS Observations:-

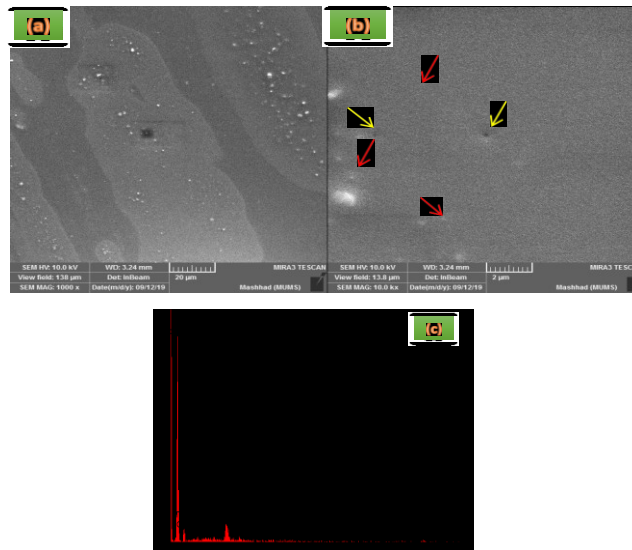
Figures 10, 11, 12 and 13 exhibit different magnifications of SEM micrographs and EDS analysis for epoxy, 1%, 3% and 5% coatings, respectively. It is expected that the properties of the films formed by dipping coating, which might be changeable in a larger extent, depend greatly on the surface morphology.

Fig.10 (a & b), the epoxy coating covered the surface totally and consists of different sized irregular agglomeration (Figure2b, red arrows) This agglomeration occurs because the mixing was carried out by for short time, micro-cracks (Figure2b, yellow arrows) distributed on the epoxy film. the major elemental peaks of alloy and epoxy explained in (Fig.10c) corresponding to EDS analysis.

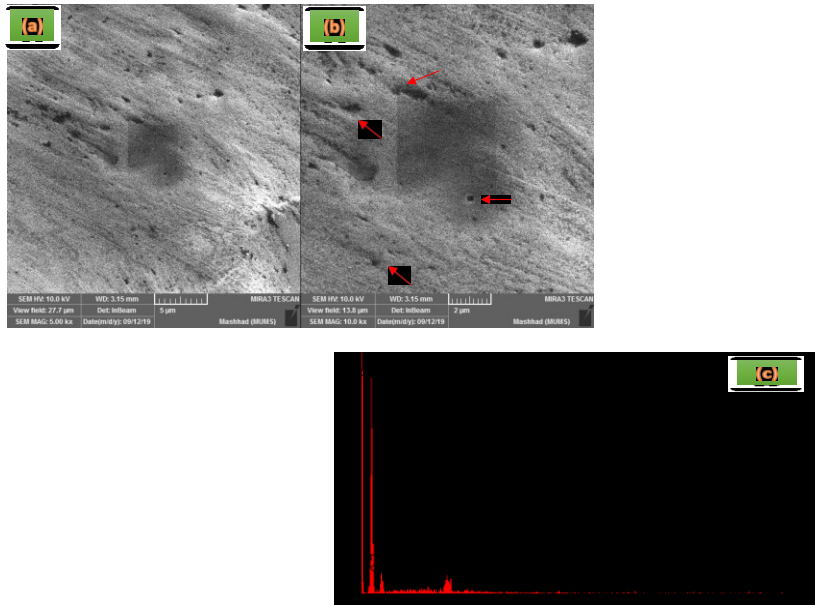


**Fig. 10 SEM images of epoxy coating at (a) low magnification, (b) high magnification and (c) point EDS spectrum ( → clusters, → micro-cracks).**

Fig.11 showing low and high magnification for 1%wt  $\text{TiO}_2$  nano composite containing .homogeneity and higher compact for coated surface can be seen in Fig.11(a & b). Good incorporation of  $\text{TiO}_2$  with epoxy matrix leading to enhanced homogeneity of coated surface. Fig.12 demonstrates the SEM micrographs of epoxy/ $\text{TiO}_2$  Nano composite containing 3% of  $\text{TiO}_2$  coating in low and high magnification along with analysis of EDS of deposited film. non homogeneous structure in Fig.12 (a & b) and numerous micro-pores (Fig.12b, red arrows), and in Fig.12c, the EDX spectrum evidently shows the existence of Ti, O, Mg, Si, C and Al.

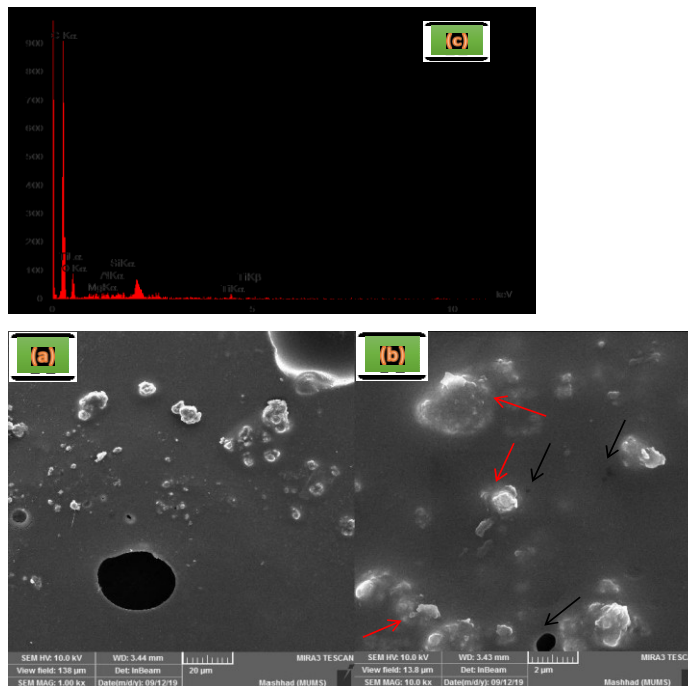


**Fig. 11 SEM images of 1%  $\text{TiO}_2$  at (a) low magnification, (b) high magnification and (c) point EDS analysis ( → granular-like particles, → nano-pores).**



**Fig.12 SEM images of 3% TiO<sub>2</sub> at (a) low magnification, (b) high magnification and (c) point EDS analysis ( → micro-pores).**

Fig.13 (a & b) illustrates the SEM of epoxy/TiO<sub>2</sub> Nano composite containing 5% of TiO<sub>2</sub>. coalescence of many nanoparticles give the structural aggregates which developed on the coated surface to form coarse particles (Fig.13b, red arrows), and micro-pores (Fig.13b, black arrows). The corresponding EDX spectrum for epoxy/TiO<sub>2</sub> coating (Fig.13c) shows that the presence of TiO<sub>2</sub> coating with the presence of major peaks related to TiO<sub>2</sub> elements coating and AA6061 alloy , i.e. Al, Mg, Si, C, O and Ti, were detected.



**Fig.13 5% TiO<sub>2</sub> at (a) low magnification, (b) high magnification and (c) point EDS spectrum ( micro-cracks, micro-pores ).**

**Ocp-Time Measurements:**

Fig.14 show the vales of open circuit potential for all samples .The OCPoscillated with time in the initial stages and then decreased with time until reaching to the stable potentials as observed in fig.14 .However, higher oscillations were in uncoated samples than in coated samples. Moreover, the coated samples show more noble potentials with a shift towards the noble direction. It is of a important note that the coating with 1% TiO<sub>2</sub> NPs exhibited the best results as in fig.14 and fig.15.

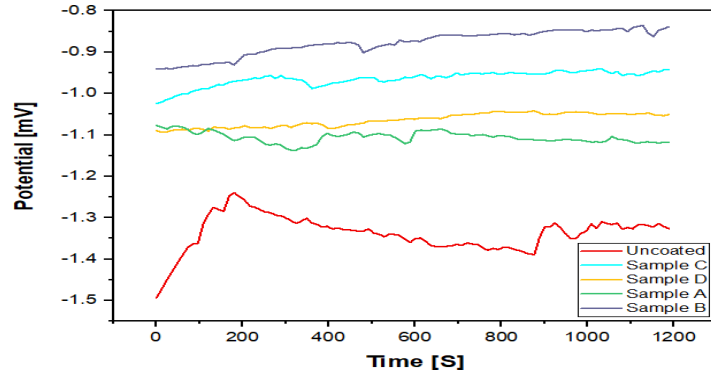


Fig.14:- OCP-time plots of uncoated, sample A, sample B, sample C and sample D.

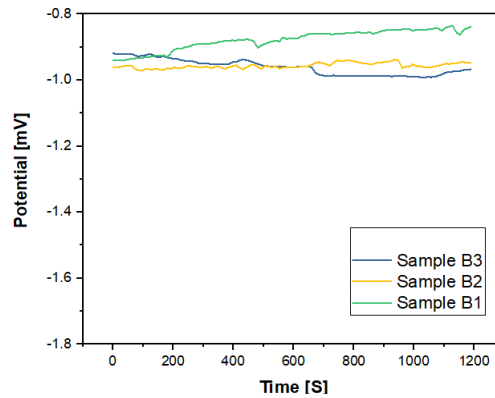


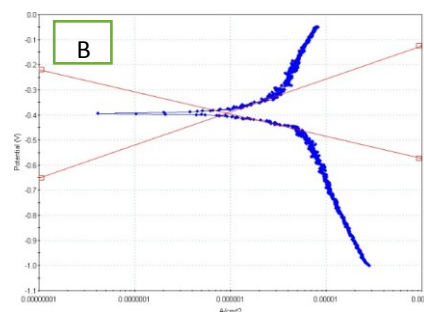
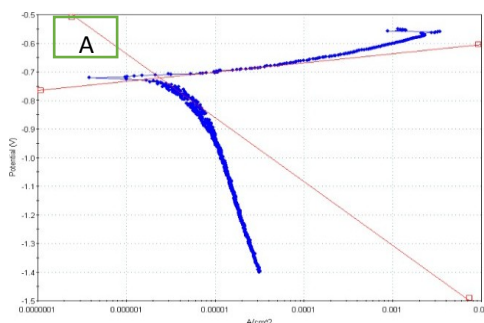
Fig.15:- OCP-time plots of, sample B1, sample B2, sample B3 in different dipping speeds.

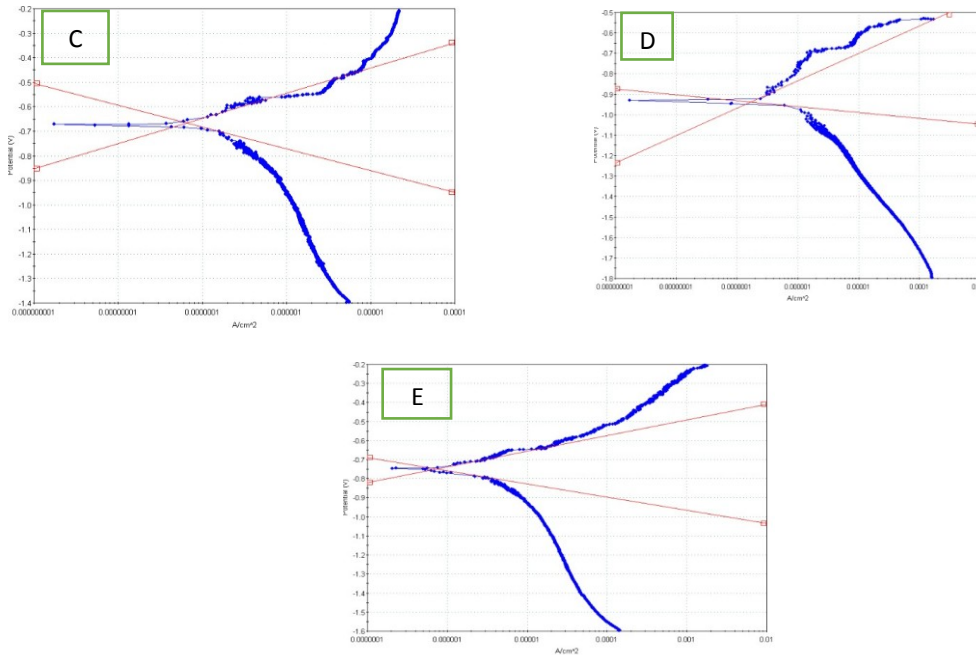
**Potentiodynamic polarization:**

The epoxy /TiO<sub>2</sub> NPs corrosion protection performance shown in Table6 for coating properties on AA6061 electrode was investigated in 3.5% sodium chloride solution using potentiodynamic polarization measurements. Fig.16 which represents the change in polarization curve (Tafel) for different surfaces coating, we can see obviously how all the curves go toward positive direction and becomes positive than uncoated sample, i.e. more passivation tendency.

Table 6:- the corrosion characteristics for sample at dipping speed 200mm/min.

Substrate	<i>E</i> <sub>corr</sub> (mV)	<i>i</i> <sub>corr</sub> $\mu A\ cm^{-2}$	Corrosion Rate (mpy)
Uncoated	-722	2.413	0.02628
Sample A	-393	0.9107	0.009917
Sample B	-667	0.06549	0. 0007131
Sample C	-983	0.1656	0.001803
Sample D	-750	0.7515	0.008183





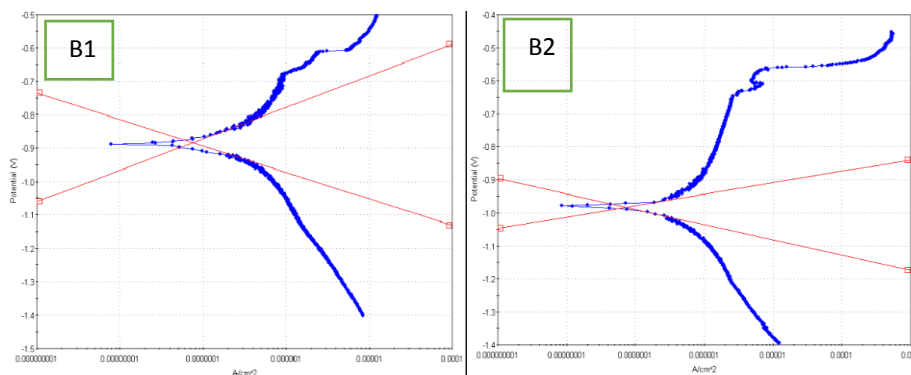
**Fig. 16:-** The polarization curve (Tafel) for different coating on AA6061 surface, A: uncoated, B: coated with epoxy only, C: coated with epoxy and 1% TiO<sub>2</sub>, D: coated with epoxy and 3% TiO<sub>2</sub>, E: coated with epoxy and 5% TiO<sub>2</sub>.

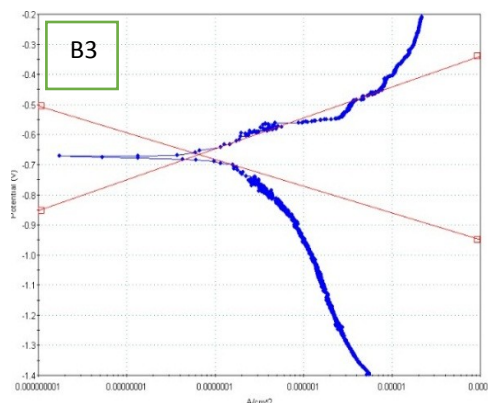
Fig.16 and Table 6, which represent the corrosion characteristics for all samples that the anodic part (especially the passivation region) is more passive and passivation current is more close to cathodic current, i.e. the oxidation reaction on the metal surface is retarded and more difficult to reduce passivation current. The increasing in passivation is related to TiO<sub>2</sub> NPs concentration in epoxy coated layer. the corrosion rate for sample(epoxy without addition TiO<sub>2</sub>NPs ) was  $9.917 \times 10^{-3}$  mm/y while when the concentration was 1% the corrosion rate was  $7.131 \times 10^{-4}$  mm/y and the corrosion rate gradually decreased with increasing the TiO<sub>2</sub> NPs concentration in epoxy coated layers as shown in Fig.16.

Fig.17 and Table 7 represent the change in polarization curve (Tafel) for coated samples with different dipping speeds, It is noticed that increasing the dipping speed led to decrease the corrosion and corrosion rate, that happened because the more decreasing in dipping speed led to sediment the TiO<sub>2</sub> NPs in dipping solution, while the increasing in dipping speed led to more TiO<sub>2</sub> NPs to be in the coated layer and reduce corrosion rate.

**Table 7:-** The corrosion characteristics for sample at different dipping speed.

Substrate	$E_{corr}$ (mV)	$i_{corr}$ $\mu A\ cm^{-2}$	Corrosion Rate (mpy)
Sample B1	-667	0.06549	0.0007131
Sample B2	-983	0.07332	0.0007984
Sample B3	-884	0.07616	0.0008293





**Fig. 17:-** The change in polarization curve (Tafel) for samples coated with different Dipping speeds (mm/min), B1: 100, B2: 150 and B3: 200.

### Conclusions:-

1. EPOXY /TiO<sub>2</sub> nanocomposite coatings with (1 wt % TiO<sub>2</sub>NPs )are found to be the best concentrations capable of improving corrosion resistance of aluminum alloy and decreasing degradation rate.
2. The dipping speed has a reducing effect on corrosion rate and the best dipping speed is at 200 mm/min.
3. The morphology of coating film is characterized by SEM, shows that 1% wt TiO<sub>2</sub> NPs spread and is covered by epoxy very well.

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