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Preparation and Characterization of PEVA/ TiO_2 Nanocomposites Coating for Oil Pipes Protection

Balqees M. Aldabbagh, Hanaa J. Kadhim*

[1]University of Technology/ Applied Science Dep, [2]University of Babylon/ Factuality of Materials Engineerin

*Corresponding Author E-mail: hanajawad21@yahoo.com

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Abstract

The spin coating process was used in this study to create nanocomposites of ethynevinyl acetate copolymer reinforced with nano titania nanoparticles at various ratios (50, 100, & 150 part/million). Using Fourier-transform infrared spectroscopy (FTIR), different types of reactions between TiO_2 and PEVA were investigated. Deferential Scanning Calorimetry was used to study melting point of thin films. As well as, anti-corrosion resistance of thin films using electrochemical method were studied.

Results show that the adding of TiO_2 nanoparticles enhanced the thermal characteristics of nano composite thin films, Tm increasing from 66°C to 71°C, the results of the FTIR analysis demonstrate that the nanoscale TiO_2 and PEVA are physical change ocuure. Anti-corrosion behavior shows that the corrosion current reduces from 188 mA to 1 nA and corrosion resistance was increased. Additionally, the efficacy of inhibition increased from 0.0 to 99%. with adding Nano TiO2 with 150 ppm to thin films.

Keywords: nano composite thin film, corrosion, EVA, coating. Nano titania nanoparticles.

تحضير وتوصيف طلاء نانوي من مركبات $PEVA/TiO_2$ لحماية أنابيب النفط

الخلاصة:

تم استخدام عملية الطلاء الدوراني في هذه الدراسة لإنشاء مركبات نانوية لبوليمر إيثيل فينيل أسيتات المقوى بجسيمات التيتانيا النانوية بنسب مختلفة (50، 100، و 150 جزء/ مليون). تم استخدام التحليل الطيفي للأشعة تحت الحمراء (FTIR) لمعرفة الاواصر ونوع التفاعل بين مكونات الطلاء، تم فحص أنواع مختلفة من التفاعلات بين TiO_2 و PEVA. تم استخدام مسعر المسعر التفاضلي الحراري لدراسة درجة انصهار الأغشية الرقيقة. كما تمت دراسة مقاومة الأغشية الرقيقة للتآكل باستخدام الطريقة الكهروكيميائية.

أظهرت النتائج أن إضافة جزيئات TiO_2 النانوية عزز الخصائص الحرارية للأغشية الرقيقة المركبة النانوية، وزاد Tm من 66 درجة مئوية إلى 71 درجة مئوية، وأظهرت نتائج تحليل FTIR أن TiO2 و PEVA يتفاعلان فيزيائيا. يظهر السلوك المضاد للتآكل أن تيار التآكل ينخفض من 188 مللي أمبير إلى 1 نانو امبير ويزيد مقاومة التآكل. بالإضافة إلى ذلك، زادت فعالية التثبيط من 0.0 إلى 99٪. مع إضافة التيتانيا النانوية مع 150 جزء في المليون إلى الأغشية الرقيقة.

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1. Introduction:

One useful technique for improving a variety of domains, including the industrial, medicinal, environmental, and biological ones, is nanotechnology [1-2]. Oil pipe protection is one area that nanotechnology may be improved to produce high performance materials with suitable features [3]. Recently, attention to hydrophobic materials and coatings has increased due to advances in nanotechnology, leading to the acquisition of materials with the desired qualities [4]. The nanocomposite material consists of two phases. One phase is in the nanoscale (10-9 m) range, and the second phase is in the macro meter range. A significant majority of interaction at interfaces and an increase in surface area are two things that happen when using nanoscale materials [5]. In addition, the inclusion of nanoparticles increased a number of attributes, including thermal characteristics and anti-corrosion resistance [6]. One of the most advanced and innovative techniques for creating coatings with superior performance characteristics is the nano fluid mode, which consists of two phases: a polymeric solution media phase and a nanoparticle phase [7]. Ethylene-vinyl acetate (EVA) copolymers are thermoplastic, amorphous and linear copolymers produced during ethylene and vinyl acetate (VA) are copolymerized by solution addition polymerization. It thus improves flexibility, stress-crack resistance, and toughness, especially at low temperatures; increases clarity and gloss; widens the temperature range at which heat seals can be made; boosts adhesion and hot-tack properties; and offers improved barrier properties. EVA copolymers are often used for extrusion coating and as co-extruded heat seal layers in multilayer constructions due to their simplicity of production and good adherence [8]. Nano particles like TiO_2 NPs are used to improve the adhesion forces of coating, hydrophobicity, surface roughness parameters, anti-corrosion resistance and anti-bacterial properties of coating [9]. Materials, particularly metals (such as carbon steel, galvanized steel, and cast iron), are worn down by electrochemical and chemical processes throughout the corrosion process. Metals have anodic and cathodic regions, as well as oxygen, water, and the conducting medium all contribute to corrosion. Anti-corrosion coatings work to isolate the metal's surface from ambient oxygen and moisture, which inhibits surface corrosion [10]. An inhibitor coating system is made up of several coating layers with various characteristics and uses. Depending on the desired coating system properties. A top coat, one or more intermediate coats, and a primer often make up an inhibitor coating. [11]. There are many previous studies which published about using the PEVA and TiO_2 as a coating materials for anti-corrosion applications. In 2010 Tambe et al. published a paper about preparing of EVA and VAc blend for

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anticorrosion application as a binder for the development of an anticorrosive coating, blends of various grades of ethylene vinyl acetate (EVA) with low density polyethylene (PE) and vinyl acetate (VAc) contents of 18%, 28%, and 40% as well as hydrolyzed EVA (EVAl) have been employed in their research. The melting point, degradation temperature, and flow behavior of the blend compositions were determined using the (DSC), (TGA), and melt flow index (MFI) procedures, respectively. The blend compositions were sprayed using a flame spray technique to mild steel specimens that had been subjected to grit blasting. The coated specimens were then tested for corrosion resistance in seawater, humidity, and salt spray. After eight weeks of exposure, compositions based on EVA/PE outperformed compositions based on EVAl/PE in terms of corrosion resistance under all conditions [12].

In 2021, Laurentiu and Lidia published a paper about using the TiO2 for enhancing the corrosion resistance of polymeric coating. In this study, the researchers compare the corrosion rates of welded joints on flat, uncoated steel, welded joints on flat steel protected by polymeric film, and welded joints on flat steel covered by nanocomposite polymeric film (primer reinforced with TiO_2 nanoparticles). For the examination of corrosion, the electrochemical impedance spectroscopy, linear polarization resistance, and open circuit potential approaches were used. The findings of the electrochemical experiments show that the corrosion protection of polymeric film rises when titanium oxide is used to reinforce it and create nanocomposite layers over naval welded steel. This is in contrast to primer that has not been modified [13].

In 2022, Chenhao et al. published a paper about creating a self-healing coating for anti-corrosion application using epoxy resin with PEVA, they developed a unique self-healing technique that can restore coating adhesion strength and corrosion resistance concurrently. The coating was created using a shape memory epoxy resin and EVA microspheres that had been loaded with Ce $(NO_3)_3$ inhibitors. It was then dried at a high temperature to encourage the fusing of nearby microspheres, strengthening the self-healing effect. The electrochemical impedance spectroscopy and scanning electrochemical microscopy results showed that the epoxy matrix's shape memory effect, the filling of molten EVA microspheres, and the release of $Ce(NO_3)_3$ inhibitors all worked together synergistically to suppress the corrosion reaction at the coating damage. After healing, the low frequency impedance modulus of the coatings containing $Ce(NO_3)_3$-EVA microspheres exceeded that of the blank epoxy coating by three orders of magnitude [14].

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This study aims to contribution between thermoplastic PEVA and TiO2 nanoparticles for high performance anticorrosion coating preparation.

2. Methodology

2.1 Materials

Ethylene vinyl acetate as a white granules with chemical formula $(C_2H_4)n(C_4H_6O_2)m$, molecular weight about 342.43 , density about 0.95 g/cm3 and thickness of granular about 2-3 mm were obtained from Chania , Ali Baba Company and used for preparing the coating in this study . TiO_2 nanoparticles with a diameter about 25-40 nm were purchased from Ali Baba company, Chania and used as a reinforcement material. Tetrahydrofuran (THF) with chemical formula $(CH_2)_4O$ and boiling point about 68 o C from Indian origin was used as a solvent in this study. Table (1). Shows the chemical and physical properties of the materials that used through this study:

Table (1) Physical and chemical properties of the used materials

Material	Properties	Value	Material	property	value	Material	property	value
Thermoplastic PEVA	density	0.95 g/cm^3	THF	T_b	66 °C	TiO_2 Nanoparticles	Particle size	**25-40 nm**
	Mw	342.43		density	0.9 g/cm^3		shape	**White powder**
	Tm	50 ° C		Molar mass	72.107 $g{\cdot}mol^{-1}$		Chemical resistance	**excellent**
	Chemical resistance for Oil and Greases, Dilute Acid, Dilute Alkalis Aliphatic Hydrocarbons,	good		Viscosity	0.48 cP		Transparency	**dim**
	Water absorption	0.1% low						

2.2 Coatings Formation

Ten grams of polyethylene vinyl acetate were dissolved in fifty milliliters of tetrahydrofuran using a magnetic mixer. The mixture was then heated to fifty degrees Celsius to speed up the dissolution process and create a homogeneous solution. Following another 15 minutes of mixing, the mixture was allowed to cool to room temperature.

To achieve a uniform distribution of the nanoparticles, they were dispersed in 40 ml of tetrahydrofuran for 15 minutes at 35°C using a dispersing equipment. To create a homogeneous

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nano solution, the first and second solutions were then continuously stirred in a magnetic mixer at a temperature of 40 degrees Celsius. Table (2) shows the contents of nano fluid solutions.

Table (2) Coating contents

Sample no.	Contents EVA+Nano TiO2 ppm
NC0	PEVA
NC50	PEVA + 50
NC100	PEVA +100
NC150	PEVA +150

2.3 Tafel's method

The Tafel's test is performed using an aqueous (NaCl) salt solution that is 5 weight percent. The Al base surface was covered with thin films of PEVA and PEVA/TiO_2 nanocomposites with varied ratios of nanoparticles (50, 100, and 150 ppm) for a corrosion test with a 1 cm^2 area. Then, insulated copper wire was used to connect it. Current of corrosion was determined, the rate of corrosion was computed using equation (1) [15] and inhibition effectiveness was computed using equation (2) [16]. 20 minutes are needed to finish each test.

$$C.R = \frac{I_c * K.\ E_W}{D\ A} \dots\dots\dots\dots\dots (1)$$

K = a constant [= 3272 mm/(amp-cm-year), I_c = corrosion current (Amp), D = density (2.7 g / cm^3, E_W = equivalent weight (= 9), A = specimen area (cm^2) = for each sample is equal to (1 cm^2)

Inhibitor efficiency (η) is equal to:

$$\eta = [I_{1\,C} - I_{2\,c} / I_{1\,C}] * 100\% \dots (2)$$

3. Result and Discussion

3.1 FTIR Spectrum

Figure (1) and Table (3) depict the results of an FTIR analysis of thin films made of EVA and EVA/TiO_2 coatings, respectively.

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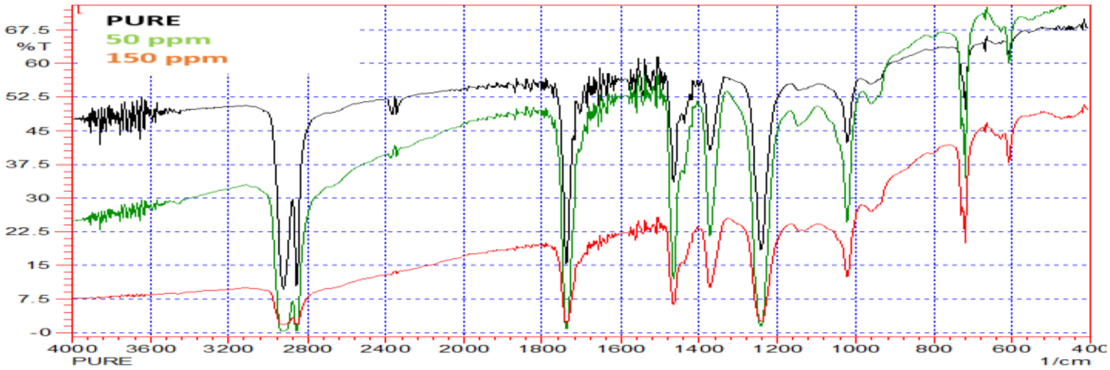

Fig. (1): FTIR analysis of thin films

Table (3) Bonds of coatings

Wave number cm^{-1}	Bonds	Groups
PEVA		
2980	OH	**Hydroxyl group**
1679	C== O stretch	**Carbonyl group**
1517	C-C stretch	**Carbon group**
1478	H-C-H	**Ethylene**
1117	C-O	
710	O-C-O	
PEVA + TiO_2 NPs		
620	Ti – O - O	**Metal oxide**

The results demonstrate a physical interaction between ethylene vinyl acetate and nano TiO_2 by demonstrating a decrease in intensity values when infrared absorption values are present. Due to the fact that nano-TiO_2 is opaque to light and does not transmit it, adding it to an ethylene vinyl acetate polymer reduced the amount of light that may pass through again. More light is reflected and diffracted by TiO_2 particles than is transmitted. As a result, the presence of nanoparticles causes a decrease in the transmitted infrared radiation's intensity and causes divergence of the transmittance peaks, as seen in Figure (1). this is matched [17-18].

3.2 2D Surface images of coatings

Figures (2 a, b, c, & d) show the 2D images of thin film surfaces:

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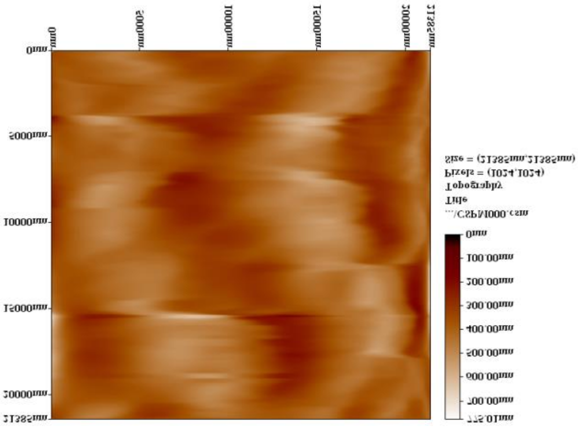
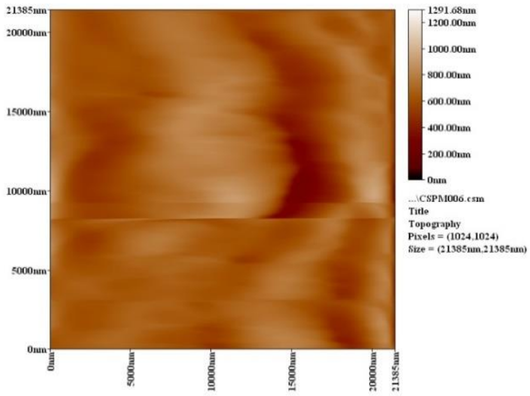
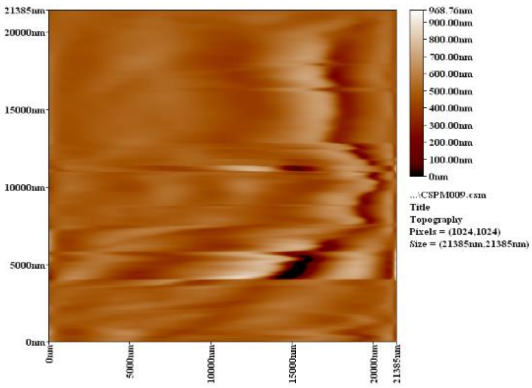


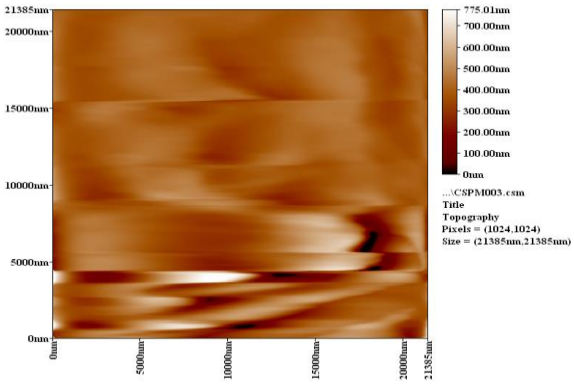
(a): Pure PEVA thin film



(b): PEVA/ 50 ppm TiO_2 NPs nanocomposite thin film



(c): PEVA/ 100 ppm of TiO_2 NPs nanocomposite thin film



(d): PEVA / 150 ppm TiO_2 NPs nanocomposite thin film

Fig. (2): AFM images of nanocomposites thin films

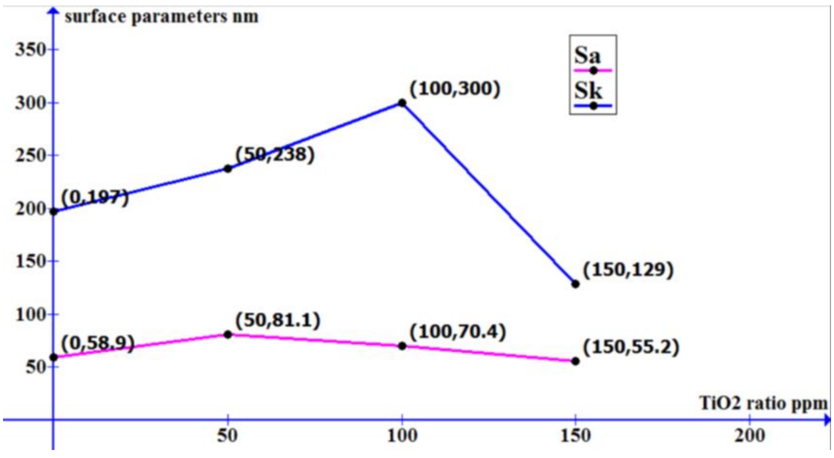


Fig. (3): Roughness parameters of coating

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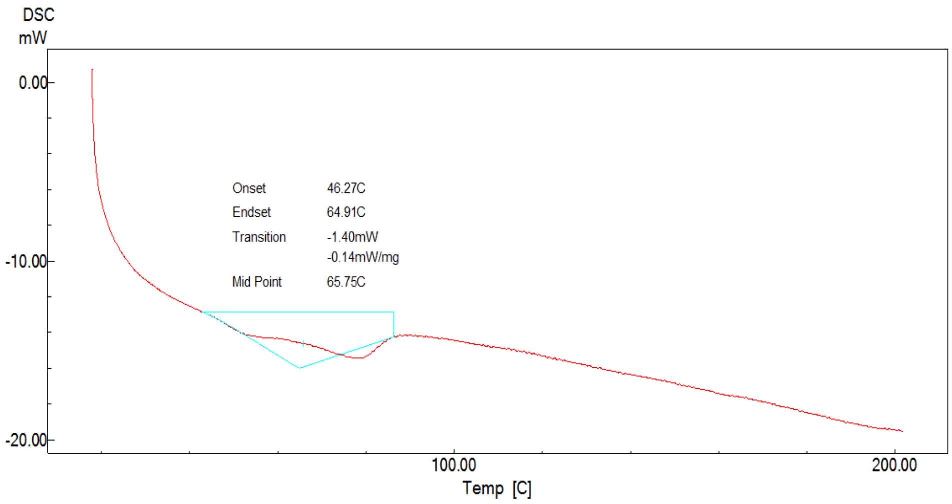
Table (4) The AFM result of nanocomposites thin film.

Sample No.	Sa : nm	Surface area ratio	Bearing index	Sk nm
1	58.9	1.17	0.369	197
2	81.1	1.7	0.597	238
3	70.4	4.91	0.35	300
4	55.2	4.74	0.6	129

The optimal sample is (NC150), which comprises (PEVA+150 ppm TiO_2 NPs), as shown in Figure (2d) and Table (4). It has the highest surface area ratio, the best bearing index, and the least average and deep core roughness. In addition, TiO_2 nanoparticles are used, which boosts the surface area ratio, improves surface film uniformity by lowering surface roughness, and strongly combines with other materials due to their high surface area to tiny size. This is matches with Aldabbagh et al. 2022. [20]

3.3 Melting point of thin films by DSC

Figure (4) and Table (5) show the DSC curve of free coating and nano composite thin film samples:



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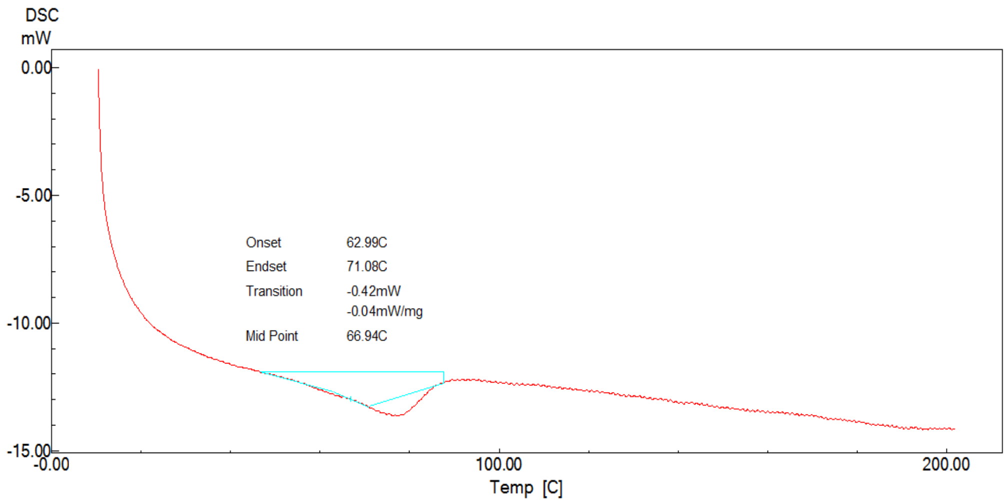

Fig. (4): DSC curve of thin films samples

Table (5) Melting points of samples (with & without Nano TiO2)

Coating	Tm Onset °C	Mid point °C	Tm Endest °C
EVA	46.27	64	66
EVA/ N.TiO_2	62.99	66.94	71.08

It is clear from Figure 4(a, b) and Table (5) that the TiO_2 nanoparticles improved the thermal characteristics of PEVA. PEVA's melting point range rises from (46-66) °C to (66-71) °C, which is due to the thin film's increased thermal stability following the addition of TiO2 nanoparticles. TiO_2 NPs increase the thermal stability of the thin films produced by nanocomposites by linking more cross-linkers to the polymer chains in PEVA. Increased interfacial area in the nanocomposite may have increased the strength of the material, which in turn has increased thermal stability [19-20].

3.4 Hydrophobicity of thin films

Figures 5(a–d) depict the contact angle of thin films made of nanocomposites; take note that the presence of TiO_2 NPs causes this angle to rise. PEVA had a contact angle of about 64 degrees, which rose to 71, 77, and 84 degrees, respectively, with additions of 50, 100, and 150 ppm of TiO_2 NPs. This is because the presence of TiO_2 NPs through the PEVA structure reduces the intensity of the hydroxyl group and prevents hydrogen bonds from forming between the coated surface and the water [21].

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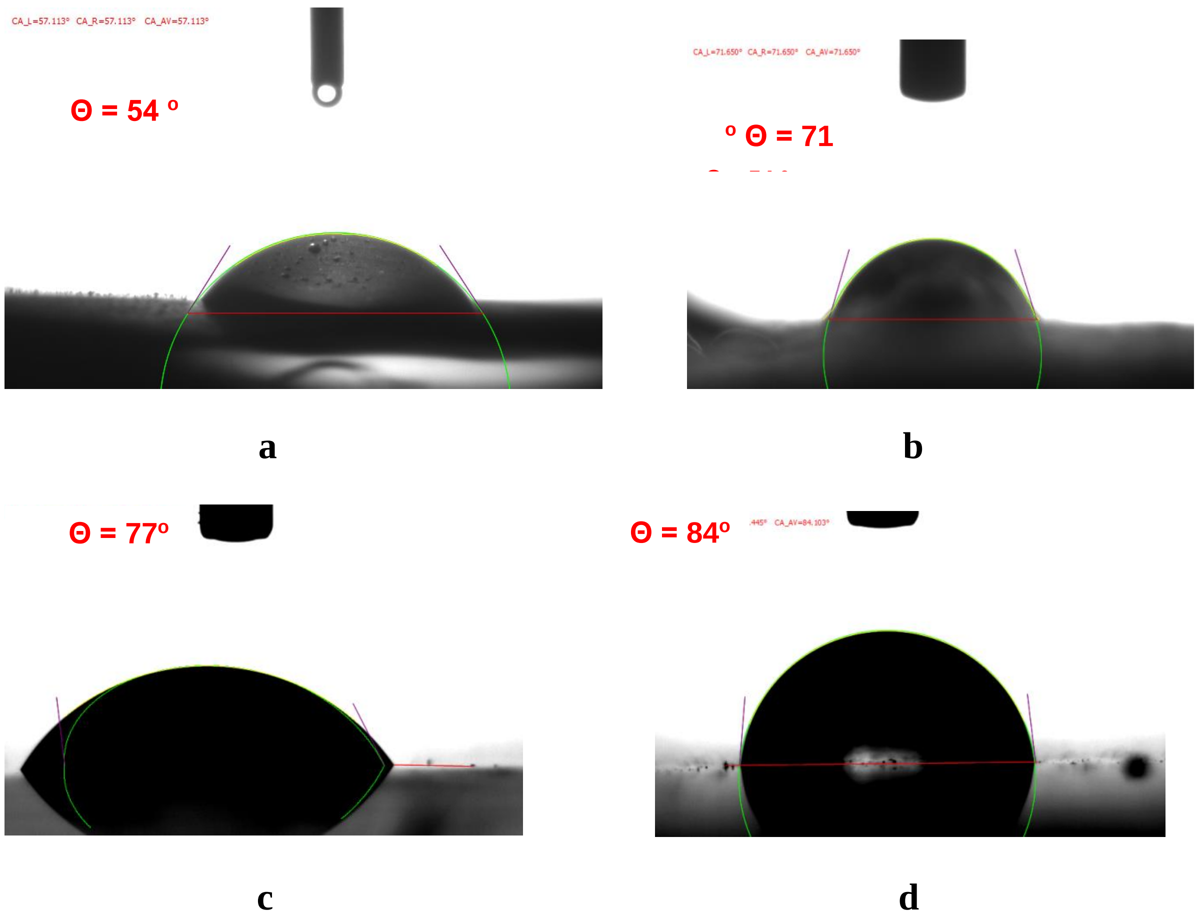

Fig. (5): Contact angles of coatings

3.5 Tafel's Results

The results of the samples' corrosion currents and rates are shown in Table (6) and Figure (6).

Table (6) Tafel results of thin film

No. of S.	E_c	I_c	J_c	η %	Corrosion rate mils/year
NC0	-723	187 µA	188	0.0	2
NC50	-210	3 nA	3	95	0.04
NC100	-211	2 nA	2	96	0.03
NC150	-212	1 nA	1	99	0.02

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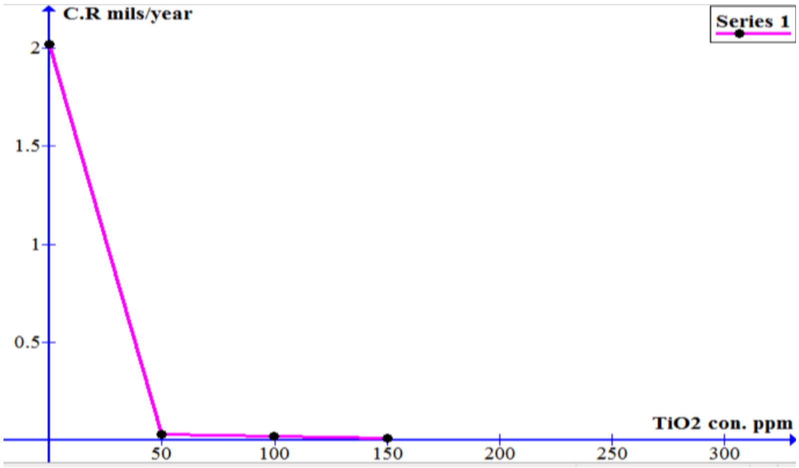

Fig. (6): Relationship between corrosion rate and concentration of TiO_2 NPs

The correlation between TiO_2 NP concentration and inhibitory effectiveness is illustrated in Figure (7).

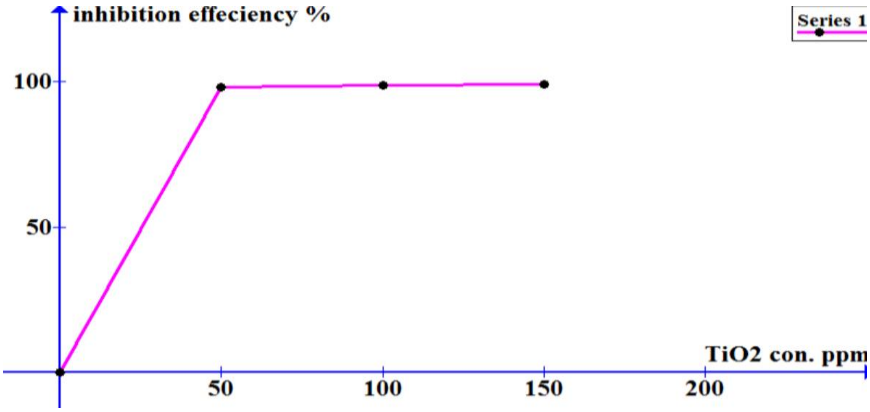

Fig. (7): Inhibition efficiency of nanocomposites thin film

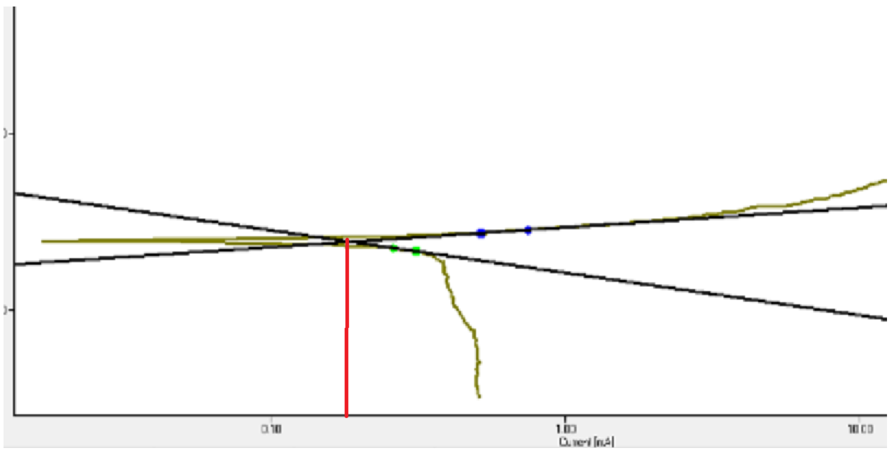

Fig. (8a): Surface without coating

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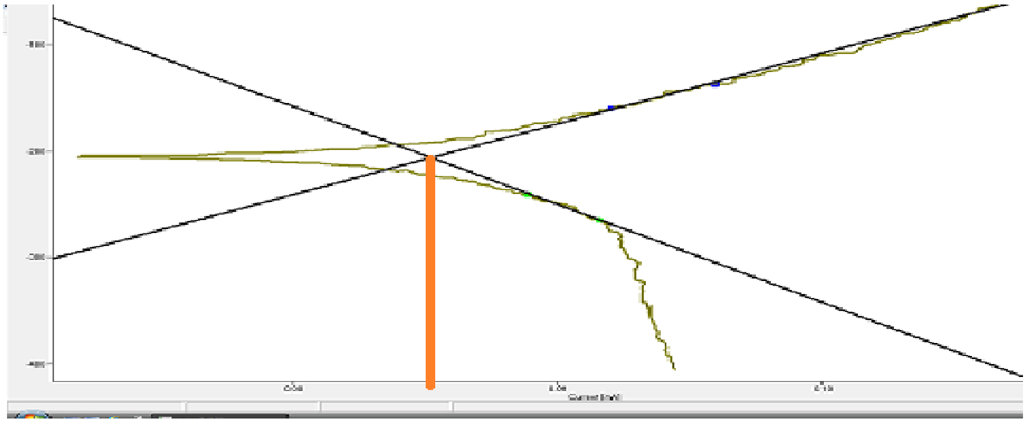

Fig. (8b): PEVA / nano TiO_2 50 ppm composite thin film

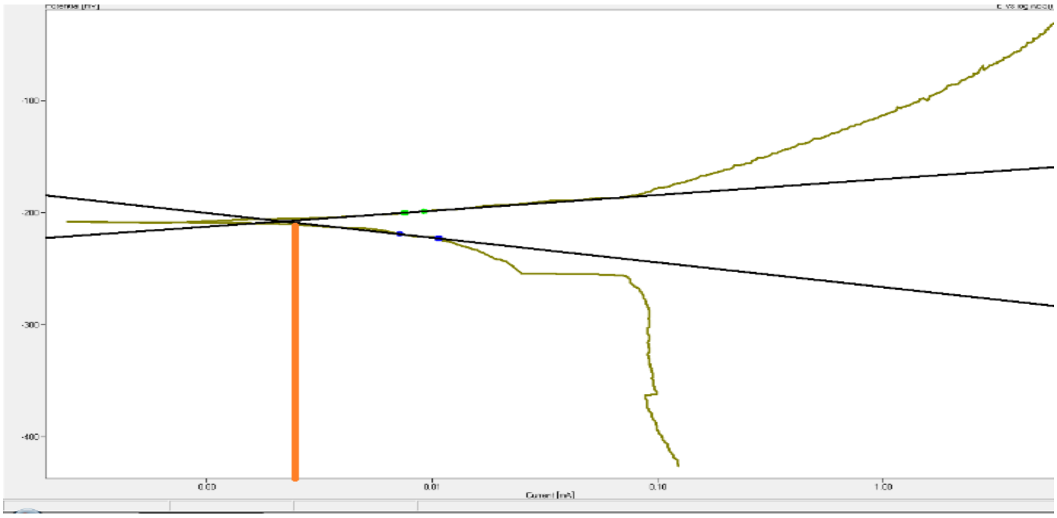

Fig. (8c): PEVA/100 ppm of nano TiO_2 composites thin film

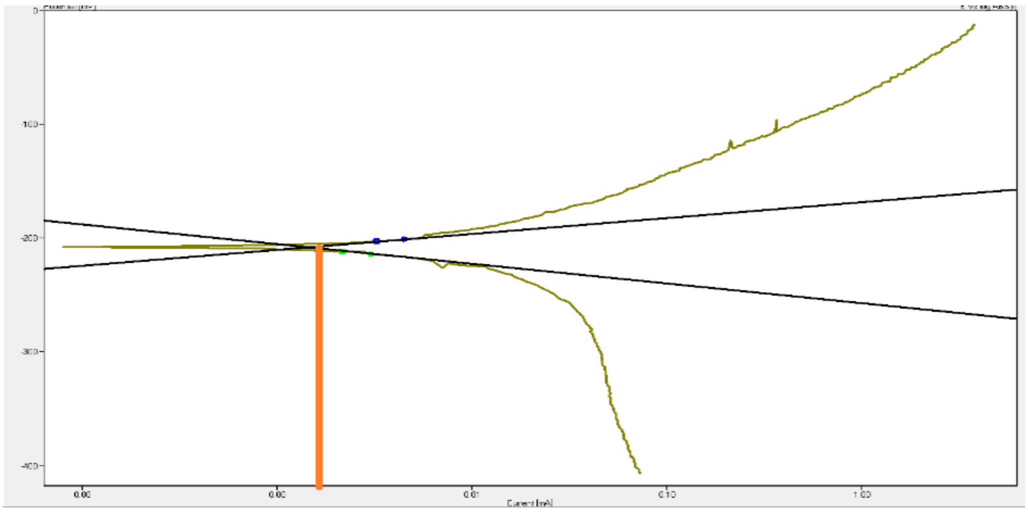

Fig. (8d): PEVA/150 ppm of nano. TiO_2 composites thin film

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Results given that there is no insulation between the aluminum surface and the electrolyte solution media, more corrosion current passes through the aluminum surface, leading to pitting corrosion behavior and oxidation of the aluminum surface, which increases the weight of the aluminum sample and accelerates corrosion. This results in a high corrosion rate for free coating samples. As depicted in Figure (8a) and Table (6).

Figure (8b) illustrates that covering aluminum with an PEVA thin film coating reduces the rate of corrosion on the surface. This is because the EVA coating reduces the rate of corrosion and pitting effects and also preventing the formation of the oxidation cover by shielding the surface from atmospheric oxygen [20].

More evidence of a decrease in corrosion rate and an increase in inhibitor efficiency can be shown in Figure (8c-d). This is accomplished by incorporating TiO_2 nanoparticles into the PEVA thin film coating. I corr decreases when TiO_2 nanoparticles are added. This shows that polymer nanocomposites improved the obtained metal's corrosion resistance in 5% NaCl solution. [22-23]

4. Conclusions

According to our research, PEVA thin coatings are quite effective at preventing corrosion on metal surfaces. Furthermore, by enhancing the thin films' thermal, chemical, and physical properties with a modest amount of TiO_2 nanoparticles, corrosion on metal surfaces is further prevented. The TiO_2 and the PEVA matrix were subjected to a physical modification, according to the results. After the TiO_2 nanoparticles were added, the corrosion rate decreased from 2 miles per year to 0.02 miles per year. By adding TiO_2, the contact angle improved and grew from 54^o to 84^o. Subsequently, by adding TiO_2 NPs, the thermal stability of nanocomposite films was improved, and it can be inferred that by raising the melting point of the nanocomposite thin film by adding nanoparticles to 71^oC.

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