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## Anodic Inhibitor Doped Polypyrrole Coating to Reduce Corrosion in Petroleum Medium

**Abstract:** It is known that corrosion inhibitors and coatings are the best choices to use in oil refinery; therefore, we suggested using both inhibitors and coating to get more efficiency for corrosion control. Polypyrrole coating (PPy) without and with doping by anodic inhibitors were investigated to protect the tank from corrosion in light naphtha unit at Al-Dura refinery. Polypyrrole coating was deposited as nanofibers by electropolymerization using cyclic voltammetry method. Doped polypyrrole coating was applied by adding anodic inhibitor represented by chromate, phosphate and molybdate in acidic electrolyte. The coated layers were examined by SEM/EDS, FTIR and AFM. These examinations showed that coating layers were as nanofibers on carbon steel surface with average roughness in nanoscale as follow: 24.2 nm for  $PO_4^{=}/PPy$ , 108 nm for PPy, 122 nm for  $B_4O_7^{=}/PPy$  and 139 nm for  $MoO_4^{=}/PPy$  coating. Corrosion measurements were investigated by Potentiostat in oil medium at 60°C which represent the average temperature of light naphtha unit; the results showed that the highest protection efficiency was 78.34% in the presence of  $PO_4^{=}/PPy$  coating and the others were 77.85% for PPy, 72.64% for  $B_4O_7^{=}/PPy$  and 68.83% for  $MoO_4^{=}/PPy$  coating. The presence of inhibitor within the structure of polypyrrole coating leads to repairing the defects that may be produced by corrosion process and then the protection will take a long time to be destroyed. The protection by inhibitor doped PPy suggesting reduce the ionic conductivity of coating film especially in the presence of phosphate anions, where the porosity percents was 0.00025 for  $PO_4^{=}/PPy$  compared with other coatings (0.04794 for PPy, 0.00043 for  $B_4O_7^{=}/PPy$ , and 0.13083 for  $MoO_4^{=}/PPy$ ).

**Keywords-** Polypyrrole; Doping; Carbon steel; Oil medium; Anodic inhibitors.

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### 1. Introduction

Corrosion problems in refineries may be caused by various inorganic compounds, such as water, hydrogen sulfide, HF, HCl,  $H_2SO_4$ , and caustic in addition to hydrocarbons. These compounds are produced by feed stock contaminants and process chemicals, including neutralizers, solvents, and catalysts. In addition, corrosion problems by the atmosphere, cooling water, steam condensate, and soil may be caused. The most corrosion control methods are coating and inhibition. Now days, conductive polymers are used to protect many structure against corrosion, these polymers contain carbon and hydrogen atoms in addition to heteroatoms (N or S), also they contain  $\pi$ -conjugation across the polymer backbone such as polyaniline (PANi), polypyrrole (PPy), polythiophene and polyacetylene [1]. Many properties are presented in Polypyrrole include high conductivity, ionic exchange, biocompatibility, ease of preparation, electroactivity, or high yield redox process [2]. These properties have attention to use in many applications such as bioactive platforms [6], supercapacitors [3, 4], biomedicine [8, 9], batteries [5], and corrosion inhibitors [7].

Many authors highlighted on using polypyrrole as coating to reduce corrosion as alone and in the presence of dopants to deposit composite coating on metallic surfaces. Ferreira et al. in 2001 deposited polypyrrole/ $TiO_2$  on steel substrate in oxalic acid medium at different conditions include surface treatment [10].

Tallman et al. in 2002 deposited conducting polymers on iron and aluminum [11]. While Nakayama et al. in 2002 studied the formation of polypyrrole (PPy) as composite by electrochemical process [12]. Yoshihiro et al. in 2006 deposited a uniform composite film of Ni/polypyrrole on a copper substrate [13].

In other studies, polypyrrole films doped with derivative of sulfonic acid with functionalized nanotubes (single-walled) have been deposited [14], also polypyrrole coatings doped with small size sulfate ( $SO_4^{2-}$ ) was applied on NiTi alloy surface [15]. Demétrius et al. in 2015 synthesized nanocomposites of ZnO (nanoparticles) with polypyrrole to protect steel by chemical polymerization [16]. Maido et al. in 2015 prepared a new corrosion-resistant hybrid coatings based on graphene and polypyrrole, the hybrid coatings

showed much better anticorrosion performance [17]. Ruhi et al. in 2015 synthesized polypyrrole-flyash (PPy-flyash) composite by chemical oxidative emulsion polymerization of pyrrole monomer using  $\text{FeCl}_3$  as an oxidant [18]. Mahmoud et al. in 2016 deposited PPy-CNTs and PANI-NiLa nanocomposites on aluminum [19]. The present work aims to investigate anions/polypyrrole coating for reducing corrosion of carbon steel in petroleum medium at  $60^\circ\text{C}$ . These anions include borate, molybdate and phosphate. The characterization of coating film was done by SEM/EDS, FTIR and AFM. The protection efficiency and porosity percentage were calculated for doped PPy films.

## 2. Experimental Procedure

### I. Materials and Chemicals

Carbon steel (St 44-2-DIN 17100) which has a chemical composition of (wt%: 0.115 C, 0.228 Si, 0.54 Mn, 0.008 S, 0.012 P, 0.018 Ni, 0.017 Cr, 0.067 Al, 0.022 Cu, 0.013 Mo, 0.008 V, 0.004 Co and remained Fe) obtained by AMETEK, SPECTRO MAXX in State Company for Inspection and Engineering Rehabilitation (SIER)–Ministry of industry and minerals. The specimens ( $20 \times 20 \times 3$ ) mm were grinded with SiC emery paper up to 600 grit.

### II. Preparation of PPy and Doped PPy Films

The PPy film was synthesized in 0.1 M  $\text{H}_2\text{C}_2\text{O}_4$  which containing 0.1 M pyrrole monomer (Fluka AG, Buchs SG) at room temperature using the three-electrode cell by cyclic voltammetry method at potentials (-100 to +1500) mV at scan rate 40 mV/sec using Potentiostat. Doping by anions was achieved by adding 0.01 M of  $\text{Na}_2\text{B}_4\text{O}_7$ ,  $\text{Na}_2\text{MoO}_4$  and  $\text{NaHPO}_4$  to solution of pyrrole.

### III. Characterization of the Polypyrrole Films

The coated surfaces were characterized using some techniques. The morphology of the obtained thin films was characterized by SEM microscope with EDAX type (VEGA3 TESCAN). The FTIR spectra were recorded by FTIR-8400S Shimadzu Fourier Transform Infrared Spectrophotometer.

Atomic force microscopy (AFM) technique was used to exam the coated surfaces (Veeco dinnova model) using cantilever with linear tips at scanning area and scan rate of  $5 \times 5 \mu\text{m}$  and 0.6 HZ/sec. respectively.

### IV. Corrosion Protection Test

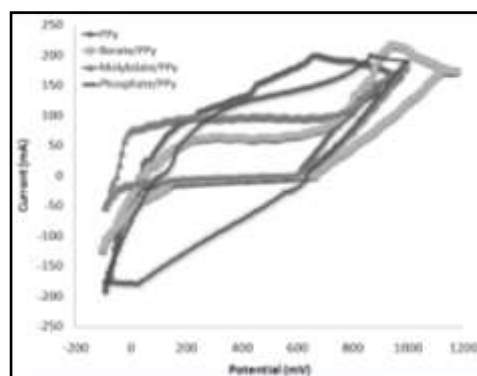
Corrosion measurements for uncoated and coated carbon steel specimens were tested in petroleum medium using potentiostat (WENKING MLab 200/

Germany) which is connected to the computer device with software program. Working electrode holder was used to fix uncoated and coated specimens. Reference and counter electrode were Pt and saturated calomel electrode (SCE) respectively. The results of corrosion were calculated using Tafel extrapolation method. The base electrolyte of corrosion test was petroleum medium obtained from Iraqi oil refinery with contents of metals: 48.67% C, 8.73% H, 0.13% N, and 0.71% S. The pH of this medium is 5.25, electrical conductivity is 300  $\mu\text{S}/\text{cm}$ , and TDS 296 mg/L.

## 3. Results and Discussion

### I. Characterization of Coating Film

Figure 1 illustrates the cyclic voltammograms of pyrrole polymerization in a prepering solution of 0.1 M oxalic acid on carbon steel surface. The polymeric film was electrodeposited using potentiodynamic method with potentials in the range of -100 mV to +1500 mV at scan rate of 40  $\text{mV}\cdot\text{s}^{-1}$  for a period of 6 cycles. During electropolymerization, the oxidation of monomer occurs and appears as a single peak in CV curve; on the other hand, the reduction takes place to form of oligomers and polymer on steel surface. The polymerization process is influenced by the presence of any anion for the following reasons: the redox potential of the anion, adsorption of the anion on the steel surface, ionic charge and ionic size of anion. The presence of borate anion gave two peaks in the anodic region. While the presence of molybdate did not effect on CV curve, i.e., the oxidation of monomers was not affected by the presence of  $\text{MoO}_4^-$  anion, but the growth of PPy depended on the nature of the anions. The presence of phosphate anion gave a high and wide anodic peak. Generally, the presence of inorganic anions confirms the incorporation of additives in the monomer matrix.



**Figure 1:** shows the SEM images of PPy growth on steel surface without and with doping; this image indicates that the aggregation of bulk polymer may take place as large particles.

This means that the inter-chain interactions increase in the form of stabilized particles to act as a limiting factor for such an interaction. Generally, the film has a uniform granular morphology and the average grain size is in the range of 0.35 – 1.54  $\mu\text{m}$ . The presence of dopants did not affect the surface morphology of the PPy films but giving more refining in film. Where the borate/PPy film gave particle size ranged between 93.02 to 131.74 nm, molybdate/PPy film gave 56.33–171.05nm and phosphate/PPy film was between 70.85 and 104.43nm. This means that the doping with phosphate ions gave the lowest particle sizes.

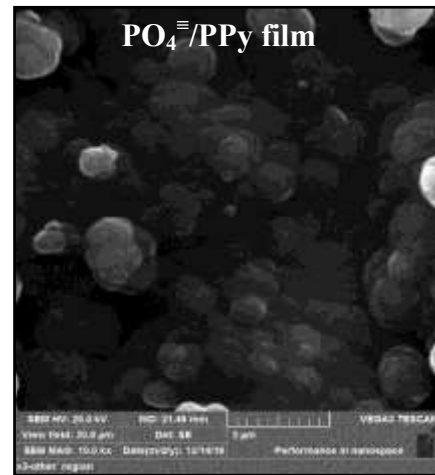
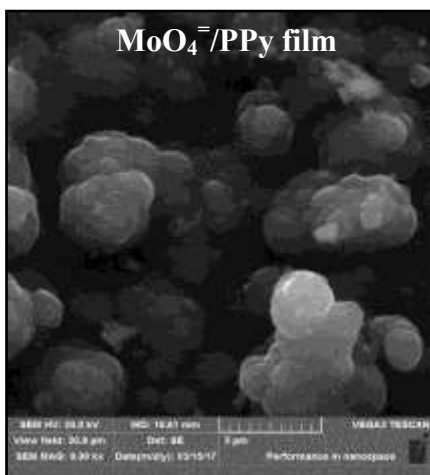
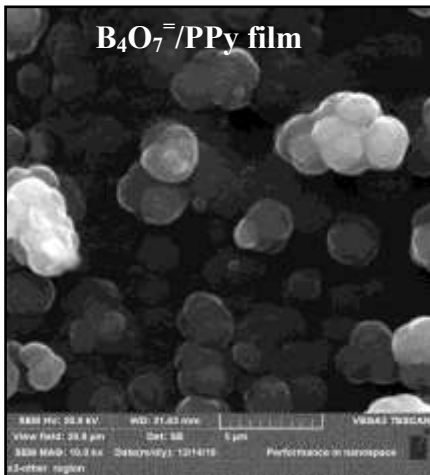
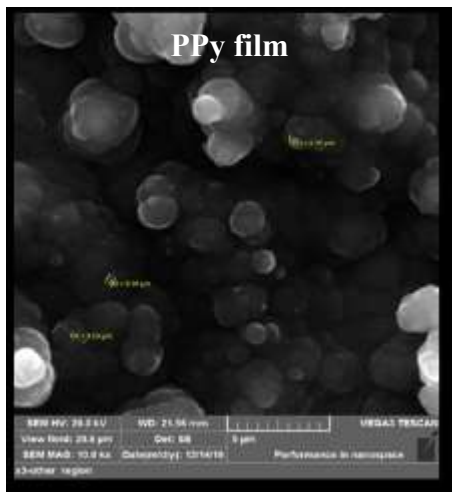
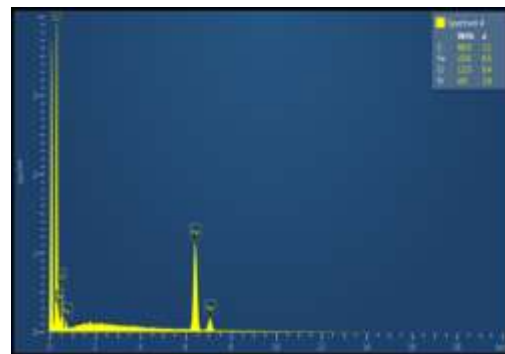
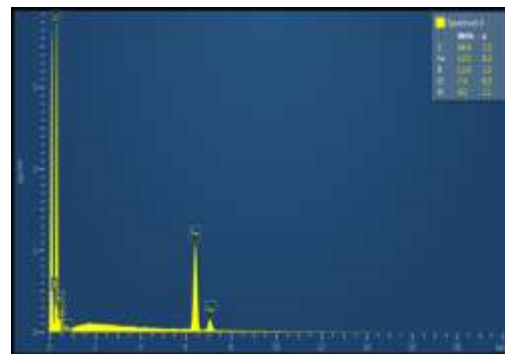


Figure 2: SEM images of polymeric film deposited on carbon steel.

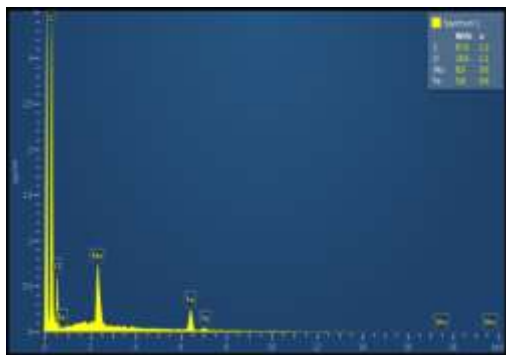
The deposition of polypyrrole on steel surface was confirmed by EDS analysis as shown in Figure 3 which containing the carbon and nitrogen that presented in pyrrole structure in addition to appear iron through electrochemical process on steel electrode. The presence of dopant elements within polypyrrole film indicates the presence of B, Mo, P and O elements that appear in EDS analysis corresponding to type of added anion.



PPy film



Borate/PPy film



Molybdate/PPy film

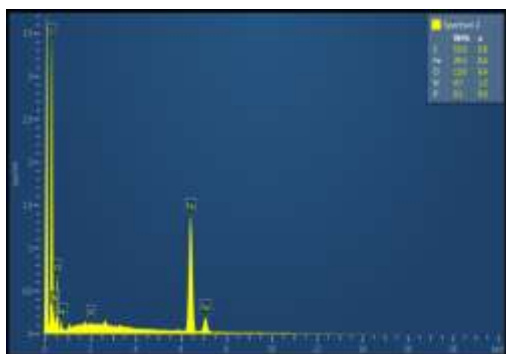


Figure 3: EDS analysis of polymeric film deposited on carbon steel.

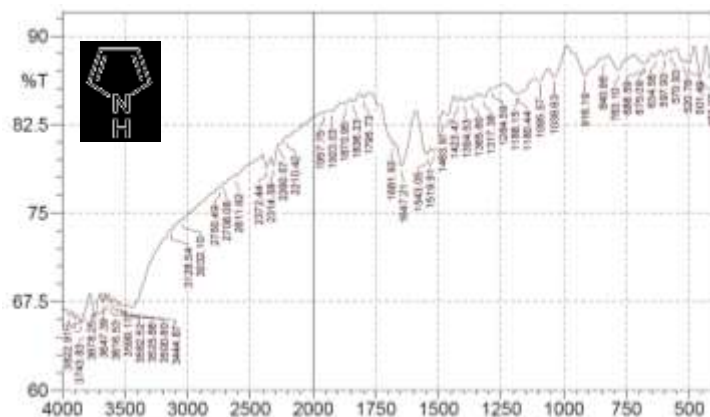
Figure 4 shows the FTIR spectrum of PPy film deposited on carbon steel surface without and with doping, the spectrum of PPy film indicates the peaks of C–H wagging that appear at 783.1 and 916.19  $\text{cm}^{-1}$ [20, 21]. While the C=C stretching peaks occur at 1543.05  $\text{cm}^{-1}$  and 1463.97  $\text{cm}^{-1}$ , whereas peaks at 1647.21  $\text{cm}^{-1}$  and 1317.38  $\text{cm}^{-1}$  are attributed to the bonds of C=N and C–N respectively [22]. The occurrence of small peaks at 3616.53  $\text{cm}^{-1}$  is assigned to presence of N–H stretching vibrations. The observed peaks in Figure 4 are agreement with

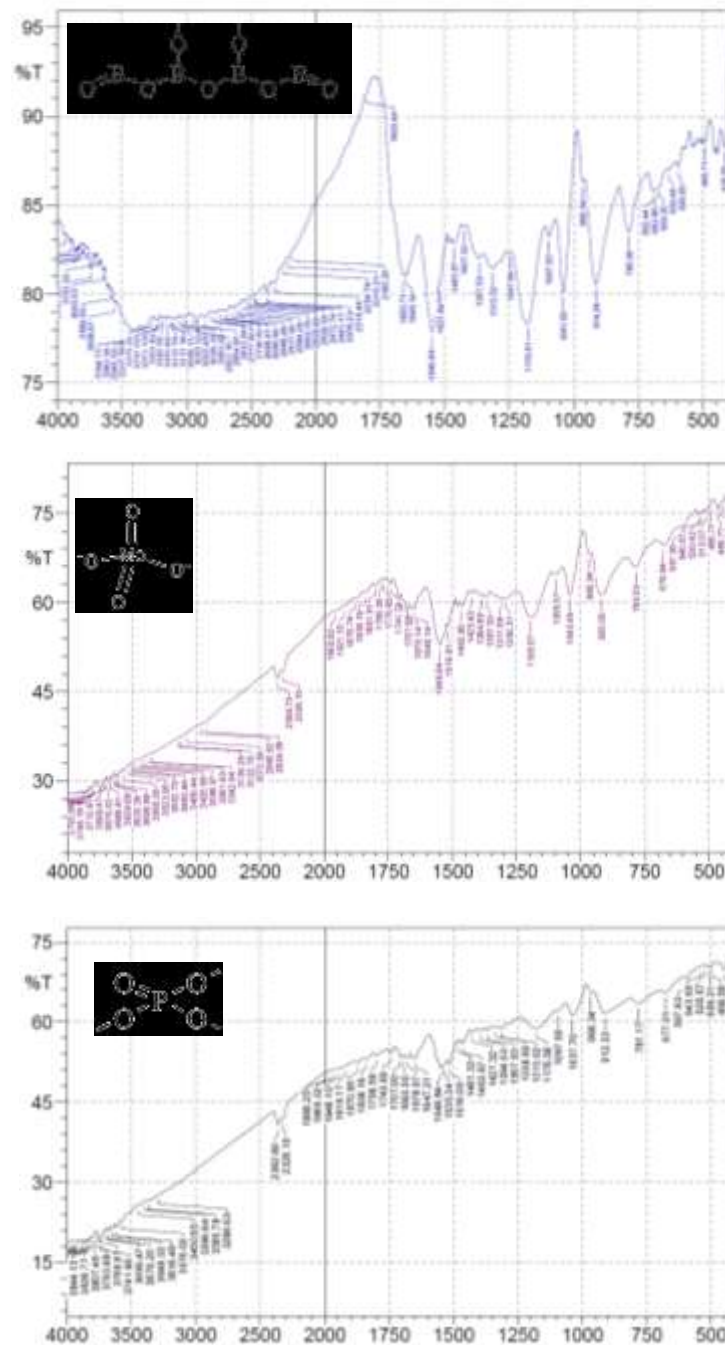
the ones observed in the literature [21, 22] confirming the formation of Polypyrrole (PPy). In addition to appear the peaks at 1188.15  $\text{cm}^{-1}$  for C-H in-plane deformation and 1039.63  $\text{cm}^{-1}$  for N-H in-plane deformation.

FTIR spectra for anions doped PPy coatings are related to type of solution and additive. In FTIR spectrum of  $\text{B}_4\text{O}_7^{=}$ /PPy film can be seen that the spectrum of borate is very clear within PPy with broad band at 2500-3500  $\text{cm}^{-1}$  due hydrogen bonding in presence of borate ions with more polarity. The peaks at 632.65 and 682.80  $\text{cm}^{-1}$  are attributed to bending of  $\text{BO}_3$  triangles and stretching of  $\text{BO}_3$  units with non bridging oxygens respectively. The band around 786.96  $\text{cm}^{-1}$  can be due to vibration of the linkage between tetrahedral and triangle borate groups. The peaks at 1463.97 and 1548.84  $\text{cm}^{-1}$  are attributed to B-O stretching mainly due to the linkage between oxygen and different groups as well as B-O bridging between boroxol rings and trigonal  $\text{BO}_3$ . The peaks of O-H bending are appearing around 1650  $\text{cm}^{-1}$  and 2378.23  $\text{cm}^{-1}$  to give rise to absorption in this region and the possibility of some adsorbed water.

FTIR spectrum of  $\text{MoO}_4^{=}$ /PPy film, the hydrogen bonding is not significantly increases. In FTIR spectrum can be seen the peak at 1043.49  $\text{cm}^{-1}$  which is characteristic for Mo=O stretching and the  $\text{MoO}_x$  particles appear upon deposition that are Mo=O terminated. In addition, the vibrational transition at 785.03  $\text{cm}^{-1}$  has been reported as O–Mo–O in deposited film.

The lowest change in IR spectrum was showed for  $\text{PO}_4^{=}$ /PPy film. In this figure, can be seen the  $\text{PO}_4^{=}$  group forms intensive IR absorption peaks at 597.93 and 677.01  $\text{cm}^{-1}$  and at 1000 – 1100  $\text{cm}^{-1}$ . This result suggests the good incorporation between phosphate anions and PPy film.



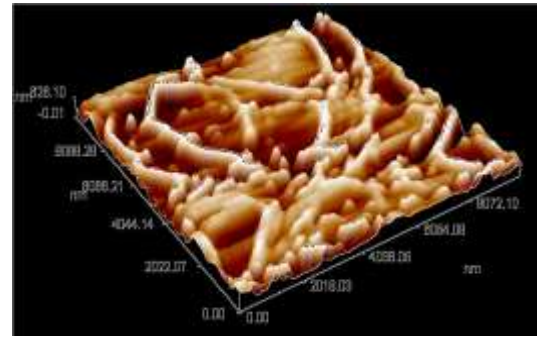
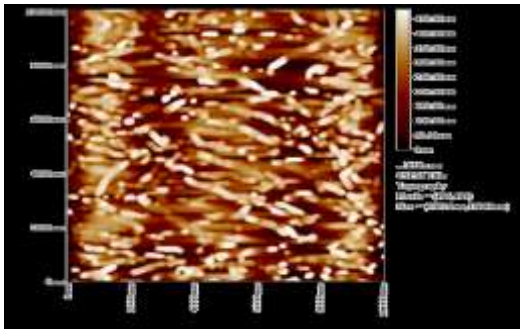


**Figure 4: FTIR of PPy film without and with doping by borate, molybdate and phosphate.**

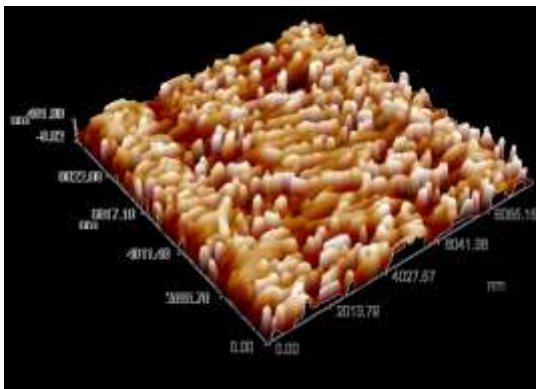
AFM images of PPy film and its nanocomposites are shown in Figure 5. These images indicate the better growth of PPy as nanofibers, also the surface morphology shows that there are a high number of very small polymer mountains as illustrated in 3D images, significantly contributing to the roughness as observed by adding dopants. Roughness average ( $R_a$ ) of these films were estimated from AFM and the results indicated that the  $R_a$  of PPy was 108 nm, while was 122, 139 and 24.2 nm in the presence of

borate, molybdate and phosphate respectively. The roughness of the film was effected by type of added anion and the increasing in roughness in the presence of borate and molybdate is attributed to tetrameric boron structure ( $B_4O_7^{2-}$ ), and a very large range of oxoanions of molybdate ( $MoO_4^{2-}$ ) which can be discrete structures or polymeric extended structures, in addition to Mo element as transition metal. While the decreasing in roughness for  $PO_4^{3-}/PPy$  film is due to the simple structure of phosphate anions and the higher

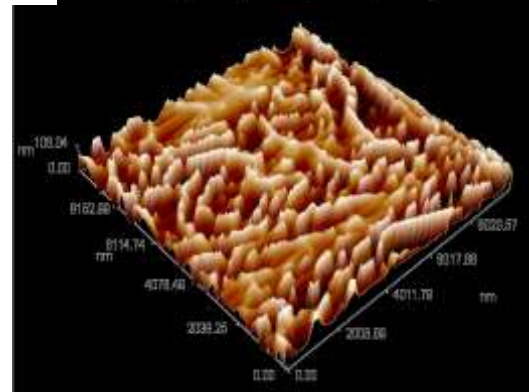
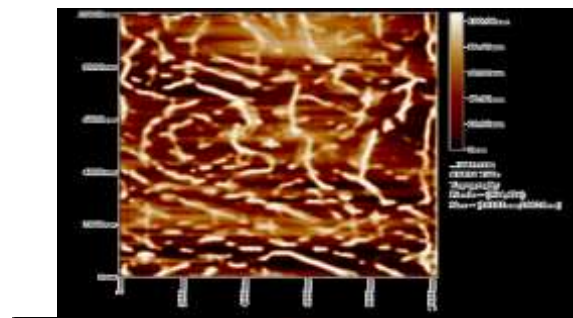
negative charge (3-) in dopant in addition to rupture of PPy nanofibers by phosphor element as illustrated in AFM images that show the more incorporation between the pyrrole and phosphate ions.



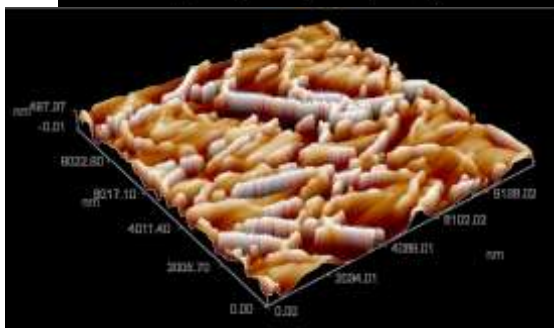
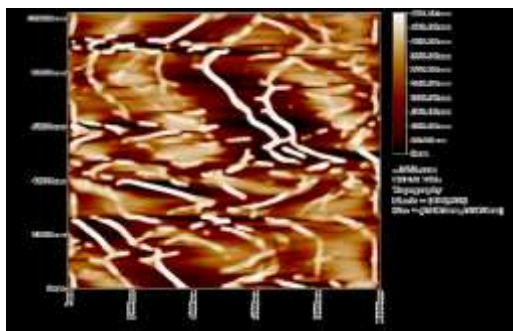
Molybdate/PPy film



PPy film



Phosphate/PPy film



Borate/PPy film

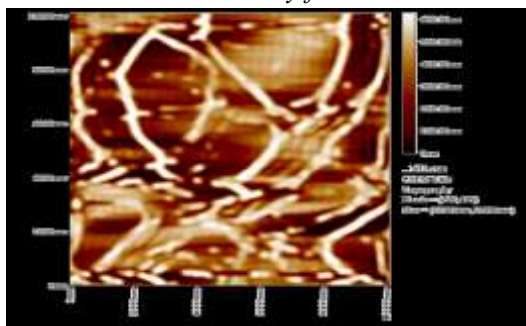


Figure 5: 2D and 3D images of AFM inspection for PPy film with doping.

## II. Corrosion Behavior

Figure 6 shows the polarization curves of uncoated specimen and coated specimens with PPy and doped PPy film in petroleum medium at 60°C. Oxidation of iron occurs at anodic sites, while reduction of hydrogen takes place at cathodic sites. Corrosion data were calculated by Tafel extrapolation method as listed in Table 1. The corrosion potentials ( $E_{corr}$ ) were shifted to positive direction for coated specimens compared with uncoated carbon steel. Corrosion current densities ( $i_{corr}$ ) decreased after coating because of presence the protective film to reduce the diffusion of corrosive species to metallic surface.

The polarization resistance ( $R_p$ ) was determined according to Stern- Geary equation [23, 24]:

$$R_p = \frac{b_a \times b_c}{2.303 (b_a + b_c) i_{corr}} \quad (1)$$

where  $b_a$  and  $b_c$  are anodic and cathodic Tafel slopes respectively. The results indicated than the highest resistance was for  $PO_4^{=}/PPy$  coating as listed in Table 2.

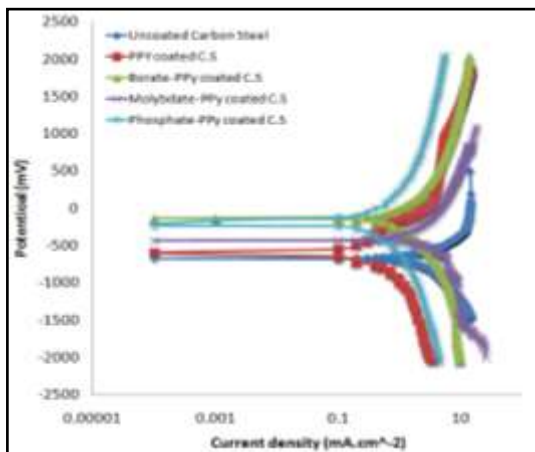


Figure 6: Tafel plots of uncoated carbon steel and coated with PPy and its composites in petroleum medium at 60°C.

Table 1: Corrosion parameters for polarization of uncoated and coated carbon steel in petroleum medium.

Medium	$E_{corr}$ mV	$i_{corr}$ $\mu A.cm^{-2}$	$-b_c$ $mV.dec^{-1}$	$+b_a$ $mV.dec^{-1}$
Uncoated C.S.	-671	2000.0	290.3	223.1
PPy coated C.S.	-600	443.04	843.2	419.5
Borate/PPy coated C.S.	-132	547.29	598.7	607.7
Molybdate/PPy coated C.S.	-433	623.43	55.0	48.5
Phosphate/PPy coated C.S.	-166	433.27	1163.9	1204.8

Table 2: Some calculated parameters for coated specimens.

Medium	$R_p \times 10^2$ $\Omega.cm^2$	PE%	PP%
Uncoated C.S.	0.274	---	---
PPy coated C.S.	2.746	77.85	0.04794
Borate/PPy coated C.S.	2.393	72.64	0.00043
Molybdate/PPy coated C.S.	0.180	68.83	0.13083
Phosphate/PPy coated C.S.	5.933	78.34	0.00025

The Protection efficiencies (PE%) of applied coatings can be estimated by corrosion current densities for uncoated and coated specimens as follow [25, 26]:

$$PE\% = \left[ 1 - \frac{i_{corr, coated specimen}}{i_{corr, uncoated specimen}} \right] \times 100 \quad (2)$$

The protection efficiencies were got good values especially for  $PO_4^{=}/PPy$  coating which was 78.34%. The porosity percentage (PP%) also calculated using the following equation [27, 28]:

$$PP\% = \frac{R_{p, uncoated}}{R_{p, coated}} 10^{\frac{-\Delta E_{corr}}{b_a}} \times 100 \quad (3)$$

where  $R_{p, uncoated}$  and  $R_{p, coated}$  are the polarization resistances of the uncoated and coated carbon steel respectively,  $\Delta E_{corr}$  is the corrosion potential difference between them, and  $b_a$  is the anodic Tafel slop of the uncoated specimen. The lowest porosity percentage was for  $PO_4^{=}/PPy$  coating equal to 0.00025%. This low porosity of coat layer confirms the lowering in electrical conductivity and the good incorporation between PPy and phosphate anions.

These dopants (borate, molybdate and phosphate) can be increased the corrosion resistance of carbon steel by incorporation with PPy coating. Generally, borate, molybdate and phosphate ions act as anodic inhibitor that adsorb on anodic sites and form passive film of iron oxide. In general, molybdate has an ability to be adsorbed by a metal oxide layer, filling gaps and so promoting the formation of an adherent oxide layer [29]. Also phosphate promotes the growth of protective film of iron oxide which assist to healing the defects in protective layer [30].

#### 4. Conclusions

Using anodic inhibitors with polymer coating have an advantage to reduce the corrosion damage for carbon steel in petroleum medium of the light naphtha unit. Three anions were added to polypyrrole film including borate, molybdate and phosphate with 0.01M as concentration. The results showed that the presence of inhibitor with PPy coating had different effect, but the presence of phosphate ( $PO_4^{=}/PPy$ ) gave the best result through the highest corrosion resistance ( $5.933 \times 10^2 \Omega.cm^2$ ), lowest current density ( $433.27 \mu A.cm^{-2}$ ) and the highest protection efficiency (78.34%). This result suggests that the phosphate ions promote the growth of protective films rather than molybdate and borate ions, therefore  $PO_4^{=}/PPy$  film gave the lowest porosity percentage.

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