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Investigation Eucalyptus Extraction as Green Corrosion Inhibitor in Crude Oil Pipeline

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Abstract

This study investigate the internal corrosion of low carbon steel (API 5L X52) pipelines in the crude oil, as well as prepare and use natural and locally available plant (the bark of eucalyptus tree) as a natural corrosion inhibitor, The eucalyptus bark extraction achieved by two types of solvents in order to show the solvent effect on inhibition process, the first being distilled water and the second is diethyl ether, the two types of the new inhibitor compared with a chemical inhibitor which used by Missan Oil Company (MOC) in terms of the cost, toxicity, availability and performance. FT-IR spectra and using a chemical reagents achieved to detection the presence of many active groups and the presence of tannins, phenols and alkaloids in the Eucalyptus Bark (EB). Some experiments were achieved to estimate the performance of a new inhibitor, one of these tests include corrosion measurement by simple immersion in crude oil within and without of inhibitors which added in different amounts 30, 40, 50, and 60 ppm at two temperature 300 and 323k, where the best inhibition efficiencies which get when added the inhibitors in a critical amounts or closest to it, since for the aqueous extract (EB-A) the inhibition efficiency reached (94.4) and (86.71)% at 300 and 323K respectively, and for diethyl ether extract (EB-D) reached (82.87) and (84.6)% at 300 and 323K respectively, while the chemical inhibitor (CRW) which used by MOC reach to (84.21) and (88.73)% at 300 and 323K respectively .Optical microscopy examination have been conducted to evaluate the corrosion nature where it show a clear difference in the topography of the immersed samples surface after add the inhibitors at two temperatures. The results show , that the new corrosion inhibitor is not only equivalent to a chemical inhibitor, but have greatly improvement properties such as : high efficiency , low cost , non-toxic , easily to product, and nonpolluting as compared with chemical inhibitor which is considered toxic and cancer inhibitor.

Keywords: corrosion inhibitor, crude oil pipeline, API 5L X52 , simple immersion, weight loss.

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1. INTRODUCTION

In the oil and gas industry, the crude oil pipelines which made of low carbon steel suffer from internal corrosion. The corrosion operation is originally related with the presence of the wastewater mixed with the crude oil, especially when it is accompanied by hydrogen sulfide, carbon dioxide, elemental sulfur, inorganic salts, organic acids, and bacteria. Corrosion inhibitors injection in the oilfield is a most common and beneficial method for prevention the pipelines internal corrosion . Because of a high cost and toxic nature of the chemical corrosion inhibitors which used presently in the oil industry, it is necessary to promote a less expensive and environmentally acceptable inhibitors, natural plants can be treated as a perfect sources for this purpose .

Carbon steels are commonly used as structural materials of piping systems because of their good mechanical properties , lower cost and wider availability despite their relatively lower corrosion resistance [1,2].

The corrosion phenomenon is a permanently existence , this problem originates very important materials and economic losses due to a total or partial replacements of structures and equipment, and plants shutdowns for repaired. Corrosion is not only has an economic impacts, but also social and these engaging the health and safety of the people who either living in the neighboring cities or working in industries . [3].

Corrosion is the essential cause of internal deterioration in pipelines of crude oil transportation , followed by the erosion in a lesser extent [4]. It must be mentioned that the crude oil by itself is not corrosive at pipelines conditions, but the water can infiltration to the crude oil causing corrosion occurrence wherever it piling up [5]. This is because the total removal of wastewater from oil is very difficult therefore, it can work as an electrolyte [6,7].

Crude oil oftentimes includes salt crystals, traces of metals, fine droplets of saline water, and suspended solids [8], these corrosive contaminants are more likely to settle and accumulate on the bottom surface of pipelines. The different means by which sediment, dissolved gases, water, and other materials can cause internal corrosion of crude oil pipeline [4]. In addition to a some corrosive species in crude oil include carbon dioxide (CO_2), elemental sulfur, hydrogen sulfide (H_2S), organic acids, bacteria, and inorganic salts which includes a different metal chloride such as sodium chloride, magnesium chloride, and calcium chloride [7,4].

Even though carbon steels can develop a passive layer of iron oxides, ingress of aggressive environment such as chloride which causes passive layer breakdown accelerating the corrosion of unprotected surfaces finally leading to formation the rust ($Fe_2O_3.H_2O$), a hydrolyzing agent for chloride phases leading to formation of acidic environments, which accelerate the corrosion rate [7].

Corrosion phenomena are very common in the oil industry and lead to a great harms to the oilfield equipment. a considerable efforts have been conducted by petrochemical industries to prevent corrosion damage. The practice of corrosion protection by injecting a substances which can dramatically delay the corrosion when added in a very little amounts is called inhibition [9].

The investment of the corrosion inhibitors considered as one of the most economical ways to protect metals surfaces against corrosion , reduced the corrosion rate, and maintain the industrial facilities [3].

It is certainly that the plants products and natural compounds will be emerge out as an effective corrosion inhibitors in the next years due to their non-toxic nature, inexpensive, biodegradability, and easy availability. Natural compounds as a green corrosion inhibitors having a restriction on their production volume on a large number of industrial metrology, therefore, their economic sides must be evaluated for the industrial utilization [10].

This study aims to investigate the internal corrosion of API 5L X52 low carbon steel pipelines in crude oil, as well as prepare and use natural and locally available plant as green corrosion inhibitors, and study the effect of the temperature and the adding amount of inhibitor on the efficiency of inhibition process. Also aims to study the effect of solvent type of plant on the inhibition process and compared the new inhibitor with a chemical inhibitor used in Missan Oil Company (MOC) in terms of the cost, toxicity, availability and performance.

2. EXPERIMENTAL WORK

A pipeline made of API 5L X52 low carbon steel (0.077%C) with 18 inch outer diameter and 0.5 inch wall thickness was obtained from Missan(province in Iraq) Oil Company (MOC) it has been cutting for a small samples in size (3×1×0.7cm) in order to use in simple immersion test as shown in Fig.1. A 2mm hole was make in all specimens, then its tied by a plastic thread in order to hanging it in the conical beakers which contain the crude oil.

Fig. 1 carbon steel specimens.

2.1. SPECIMEN PREPARATION

All specimens were grind with SiC emery papers in sequence of 180, 320, 400, 600, 800, 1000,1200 and 2000 grit to get flat and scratch-free surface, after that it polished by using polish cloth , washed with distilled water and alcohol respectively , dried and preserved it in a cans with a silica gel in order to kept it dry until used for corrosion investigation.

2.2. ELECTROLYTE SOLUTION

Crude oil used as a corrosion medium in simple immersion test, it was obtained from Al-Halfaya oil field in MOC. And it have a density equals 0.916, with salt content about 25ppm, 0.2% Water content , Sediment by extraction 0.016 %w/w and Sulpher 4.1 % wt/wt .

2.3. CORROSION INHIBITORS

2.3.1 CRW85282

A chemical inhibitor have a traditional name known as CRW85282 was obtained from the "PetroChina International Iraq FZE-Iraq Branch" which used by MOC, and we use it in corrosion test to comparing it with the new inhibitor in terms of cost, toxicity, performance and availability.

2.3.2 EUCALYPTUS BARK EXTRACTS

Eucalyptus bark (EB) used as a green corrosion inhibitor, the bark of eucalyptus tree was collected from several trees and then it was washed with distilled water, dried and grinding to a powder. The process of extraction achieved by two types of solvents in order to show the role of the solvent type on the inhibition process, the first being distilled water and the second is diethyl ether.

The amount of 10g dissolved in 200ml of solvent and put in the stirrer for 4 hours and leave soaked for 24 hours and then filtered with a piece of gauze to separate the solids, after filtration the liquid nominate by filter papers two times and then dried both of distilled water and the diethyl ether extracts to get a black powder and brown paste extracts, respectively, as shown in Fig.2 which keep cool until used, This process is repeated several times until we get on the enough quantity. When we need to use the inhibitor we must be dissolved an amount from the extract in the dissolvent to get on an extract with 10% concentration. We will be symbolized the aqueous extracts by a symbol (EB-A) and the diethyl ether extract by a symbol (EB-D).

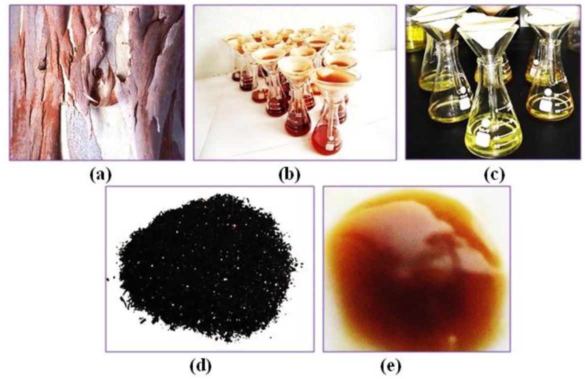


Fig. 2 (a) eucalyptus tree bark, (b) EB-A during filtration, (c) EB-D during filtration, (d) powder of EB-A, (e) paste of EB-D.

2.4. INHIBITOR EVALUATION

2.4.1 FT-IR SPECTRA

Fourier transform infrared (FT-IR) spectra were recorded to detection the active groups. A 0.002 gm from EB powder was mixed with 0.02 gm KBr and triturate the mixture well and then put it in a spatial mold with pressing strongly for the purpose of obtaining a pellets to put in the devise to take the FT-IR spectra, Fig.3 .

Fig. 3 Fourier transform infrared spectroscopy (FT-IR).

2.4.2 DETECTION OF TANNINS, PHENOLS AND ALKALOIDS

1. Mayer's Reagent : Mayer's reagent is an alkaloid precipitating reagent used to detect the alkaloids groups in the natural product. It is prepared freshly by dissolving a mixture of the mercuric chloride (13.5 g) and the potassium iodide (5 g) in distilled water (1 liter) .

2. Tannic Acid Reagent : Tannic acid is also used to detect the alkaloids groups in the natural product, added 1.5 ml of tannic acid in concentration of 1% to the EB extract to indicated the presence of alkaloids .

3. The Ferric Chloride Test : The ferric chloride is used to determine the presence or absence of phenols and tannins in a given sample (for instance natural phenols in a plant extract) .

2.5. SIMPLE IMMERSION TEST

Simple immersion test or weight loss measurement measures the advance of corrosion damage which gained when immerse the specimens into corrosive solution , i.e. studying the corrosion rate to see the behavior of material in corrosive environment.

A weighed samples of the low carbon steel are immersed in the crude oil with and without different inhibitors (EB-A, EB-D, and CRW85282) with a different amounts 30,40,50 and 60 ppm at two temperatures (300 and 323k) as shown in Fig.4, and then removed after a sensible time interval, later the samples was cleaned of all corrosion product, washed by alcohol, dried with hot air and take a weight after cleaning.

Fig. 4 Simple Immersion of specimens in crude oil.

2.6. OPTICAL MICROSCOPY EXAMINATION

Low carbon Steel API 5L X52 specimen was examined by optical microscope to show the steel microstructure , also the specimens immersed in crude oil examined after corrosion and inhibition experiments at a magnification of 800x by optical microscopy .

3. RESULTS AND DISCUSSION

3.1. FTIR SPECTRA OF INHIBITOR POWDER

The FTIR spectrum of Eucalyptus bark (EB) is given in the Fig.5 , which demonstrates the presence of many chemical functionality groups. Table 1 show the wavenumbers and the interview active group. Since the wavenumber at 3400 cm^{-1} may be a trait to **−OH** phenol, with regard to **C−H** aromatic and aliphatic, they appear at 3100 and 2950 cm^{-1} respectively, it seems that aromatic cycle appear at 800 cm^{-1}. These peaks show that the EB may be including aliphatic, aromatics and phenols which have an inhibition properties, several literature research prove that the compounds with aromatic structures and long aliphatic chain are considered as among the more prevalent corrosion inhibitors [10,3].

The **C=O** functionality group appear at 1748 cm^{-1}, while **C=N** and **C=C** shows at 1650 and 1600 cm^{-1}, the presence of double bonds in the EB structure means that this organic compound may be an effective corrosion inhibitor, where the existence of the double bonds makes the inhibitor tends to adsorption on the surface of the metal significantly and thus be able to prevent the metal corrosion , this is agreement with other research [10].

From the FT-IR results noted that the EB contains different atoms such as oxygen and nitrogen and as is common in other research [3,10,11], this atoms allows adsorption on the surface of the metal , and they are able to form bonds with the metal surface and thus reduce the corrosion rate.

TABLE-1-Wavenumbers of different active groups.

Positive number cm^{-1}	Active groups
3402.43 to 3537.45	−OH phenol
3105.39	C−H aromatic
2916.37	C−H aliphatic
1734.01	C=O
1635.64	C=N
1616.35	C=C
831.32	Aromatic cycle

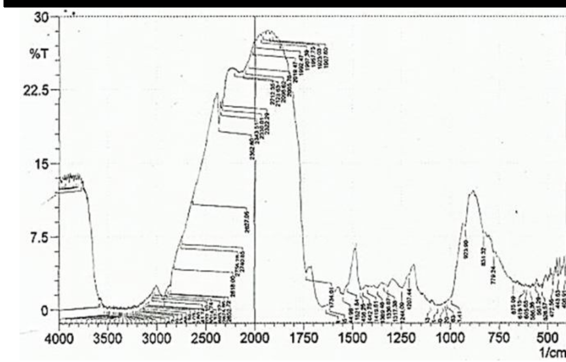


Fig.5 FT-IR spectrum for eucalyptus bark.

3.2. MICROSTRUCTURE OF CARBON STEEL

The sample was prepared by grinding, polishing, and etching using 3% Nital solution. The X52 steel's microstructure was shown in the Fig.6, the image indicated the pearlite laminates (the dark and bright laminate areas) with the ferrite (the bright areas) at the grains boundary, and with comparatively the higher content of the ferrite. This is consistence with an analogous microstructures gained by others [12,13,14].

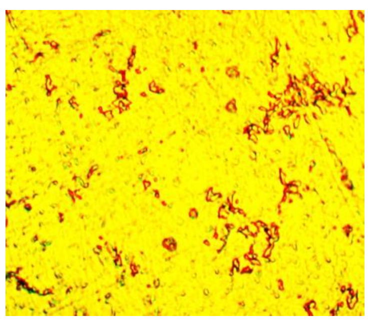


Fig. 6 The microstructure of X52 steel .

3.3. WEIGHT LOSS MEASUREMENTS IN CRUDE OIL

The weight loss measurement simulates the corrosion environment and the degree of acceleration. This test was made by the simple immersion of the specimens in crude oil in the absence and presence inhibitors (EB-A ,EB-D, and CRW85282) with a various amounts (30, 40, 50, and 60 ppm) at two temperatures 300 and 323 K. The findings of this test are shown in Fig.7 , which reveals the corrosion rates of specimens with time . From figure, it is quite clear that the weight loss increases with the time, where it well know that the materials have one phase is more corrosion resistance than the multiphase as result of forming galvanic corrosion.

The loss rate of Fe^{2+} ions gradually increases with the time, since, there is a clear increase in the loss rate with increase the temperature. The crude oil contain CO_2 and H_2S, the presence of CO_2 in the crude oil can lead to the reaction with the water to form carbonic acid (H_2CO_3) which accelerates the corrosion, i.e. this acid is dissociates into CO_3^{2-} and $2H^+$ to accelerate the cathodic reaction at the specimens surface, [15,16,17,18]. CO_2 corrosion was vary widely when more complex effects are included (e.g. protective scales, water entrainment/wetting, H_2S, etc), since, the presence of H_2S can lead to formation of various forms of iron sulfide scales that can be either very protective or cause localized attack [19].

3.3.1 EFFECT OF TEMPERATURE AND INHIBITOR AMOUNT

When immersing the sample in crude oil in the absence of inhibitors at 300 and 323 °k , a continuous decrease in the weight loss was happen as a result of corrosion products are thick, porous and weak adhesion so it does not provide protection for the surface of the steel, and this led to a search for ways to protect a steel. But after add a various amounts of different inhibitors there is a clear decreasing in the weight loss as shown in Fig.7 That the corrosion inhibitors (EB-A and EB-D) will adsorb, or stick to the surface of the metal significantly to the formation of membrane insulator, this separation membrane composed of the inhibitor will be able to reduce the cathodic and / or anodic corrosion reactions for steel surface.

As well as the existence of atoms of oxygen and nitrogen in the synthesis of (EB-A and EB-D) inhibitors will facilitate the process of adsorption and adhesion to the surface of the metal and therefore the extracted plant will be able to reduce the decomposition of steel and thus increase the efficiency of inhibition.

Some curves start with an increasing in the weight instead of decreasing , this may be due to the adsorption of the inhibitor on the metal surface directly and configure a protective membrane can be considered as a layer of coating, but over time, one or more layers form over this layer, causing increasing the thickness of the layer of coverage and makes them vulnerable to loss, Where the adhesion of coverage layer with metal surface decreases with increasing thickness of this layer, this explains why decrease in the sample weight.

The oscillators in the weight loss, sometimes increase and decrease again as its clear in many curves, may be a result of the alternation of building the inhibitor coverage layer and then fallen due to increased inhibitor layer thickness after construct other layers above it. As is evident from the curves, the samples that start with an increasing instead of the decreasing in the weight would have the lowest weight loss at the end.

The added amount of the inhibitor must be suitable , equal to or close to the critical value , since , if it exceeded the critical value then the efficiency of this inhibition would be reduced due to form a thick layer of the inhibitor on the metal surface which makes the coverage layer prone to falling easily where the adhesion of coverage layer decreases with increasing thickness of this layer , but if the inhibitor was added with less critical value, the quantity will be not enough to cover the entire surface and so will remain parts of the surface without protection.

It is an obvious that the weight loss was increase with increasing the amounts of EB-A and CRW inhibitors, that mean an amount equals 30 ppm was a best amount of both inhibitors, while the best added amount of EB-D inhibitor equals 40ppm and it can be shown that any increasing above the best amount lead to the weight loss increase .

The increase of temperature cause an increasing in the weight loss, this is consistent with the findings of the other researchers [20,21].

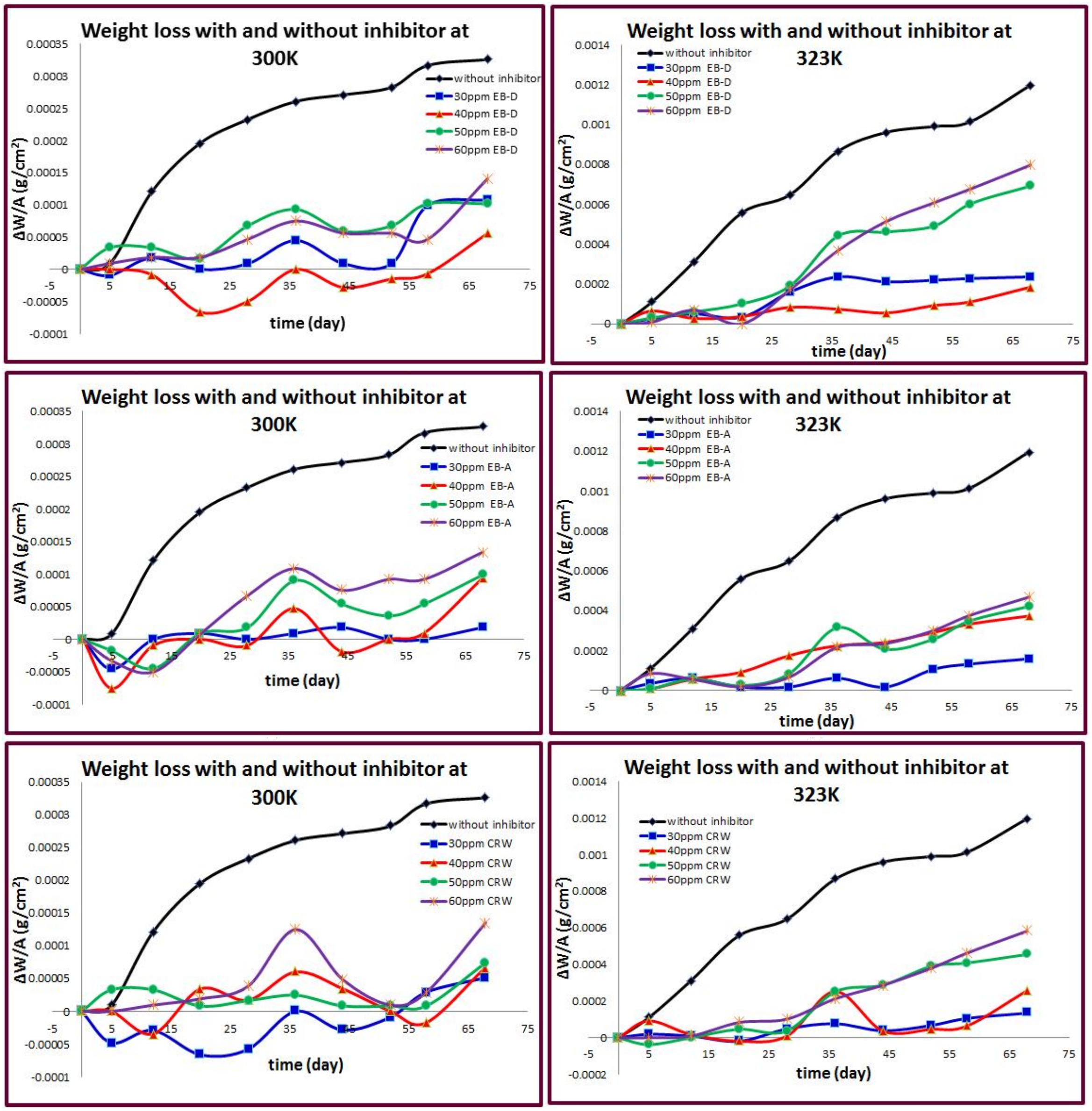


Fig. 7 Weight loss versus time curves for carbon steel in crude oil with and without different inhibitors at different amounts and temperatures

3.3.2 CORROSION RATE AND INHIBITOR EFFICIENCY

The corrosion rate (C.R) may be found according to the equation , [22]:

$$C.R = (K*W) / (A*t* \rho)$$

where K:constant indicate the corrosion rate unit (equals $3.45*10^6$), W:the mass loss in gram, A:area in m^2, t:time of exposure in hour, and ρ:density in g/cm^3. While the inhibition efficiency of inhibitors have been calculate according to the equation [23]:

$$IE = (C.R^o – C.R)/(C.R^o) *100\%$$

Where IE:the inhibitor efficiency, $C.R^o$:corrosion rate without inhibitor, and C.R:corrosion rate with an inhibitor.

The tables 2 and 3 illustrate the inhibition efficiency of inhibitors which added in different amounts and it also illustrate the corrosion rate of steel samples immersed in the crude oil in the presence and absence of inhibitors at two temperatures 300 and 323 k.

From the tables show that the rate of corrosion increases with increasing temperature in all curves, also note that the highest inhibition efficiency of the EB-A inhibitor was when add 30ppm from it since the efficiency reached to (94.4)% this mean that the amount of 30ppm is the critical value or closest to the critical value of the added inhibitor amounts since the increase most of the critical amount caused the opposite effect.

TABLE 2- the corrosion rate and inhibitor efficiency of the samples immersed in crude oil with and without inhibitors at 300k.

Sample	ΔW/A (g/cm^2)	CR ($g/(cm^2.h)$)	IE %
without inhibitor	0.000326	0.0878	-
30ppm CRW	5.15E-05	0.0139	84.21
40ppm CRW	6.74E-05	0.0181	79.35
50ppm CRW	7.43E-05	0.0200	77.23
60ppm CRW	0.000135	0.0364	58.53
30ppm EB-D	0.000108	0.0291	66.79
40ppm EB-D	5.59E-05	0.0150	82.87
50ppm EB-D	0.000102	0.0274	68.76
60ppm EB-D	0.000141	0.0379	56.79
30ppm EB-A	1.83E-05	0.0049	94.4
40ppm EB-A	9.44E-05	0.0254	71.07
50ppm EB-A	9.9E-05	0.0266	69.66
60ppm EB-A	0.000134	0.0360	58.95

TABLE 3- the corrosion rate and inhibitor efficiency of the samples immersed in crude oil with and without inhibitors at 323k.

Sample	ΔW/A (g/cm^2)	CR ($g/(cm^2.h)$)	IE%
Without inhibitor	0.001197	0.3220	-
30ppm CRW	0.000135	0.0363	88.73
40ppm CRW	0.000258	0.0694	78.46
50ppm CRW	0.000456	0.1225	61.95
60ppm CRW	0.000583	0.1569	51.28
30ppm EB-D	0.000236	0.0635	80.28
40ppm EB-D	0.000184	0.0496	84.60
50ppm EB-D	0.000692	0.1861	42.22
60ppm EB-D	0.000797	0.2142	33.47
30ppm EB-A	0.000159	0.0428	86.71
40ppm EB-A	0.000374	0.1007	68.74
50ppm EB-A	0.000423	0.1137	64.71
60ppm EB-A	0.000469	0.1262	60.80

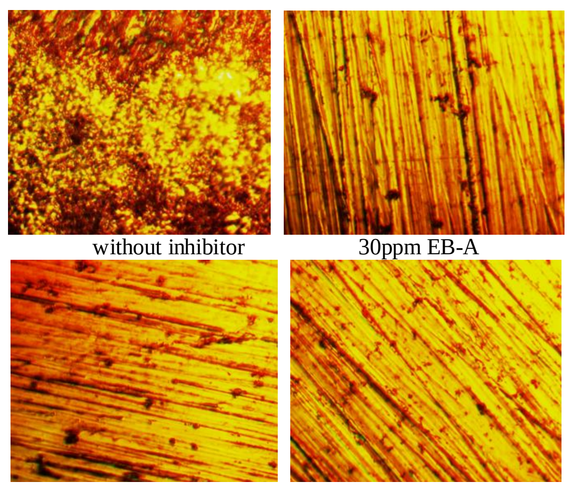
Note that the highest inhibition efficiency of the EB-D inhibitor was when adding in an amount of 40ppm, reaching efficiency to (82)% but when increase the added amount a decrease in the efficiency was happened until reaching (56.79) % efficiency when adding 60ppm, this mean that the amount of 40ppm is the critical value or closest to the critical value of the added inhibitor amounts since the increase most of the critical amount caused the opposite effect.

3.4. Optical Microscopy Examination

Optical microscopy examination was carried out to study the topography of the surface of immersed samples in crude oil with and without inhibitors at 300 and 323K. Optical microscopy examination employed in this work to investigate the actual effects caused by the immersion the low carbon steel in crude oil and the effects of inhibitor type, concentration of inhibitor and temperature.

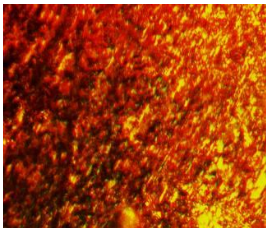
The presence of more pearlite enhances the corrosion rate of ferrite by a galvanic effect. Since the cementite contained in pearlite is electrochemically more stable than ferrite, a galvanic effect between the cementite and ferrite will enhance the corrosion of the ferrite, resulting in a higher corrosion rate (low charge-transfer resistance), as evidenced in other research [24]. The microstructure has an effect on corrosion through the galvanic effect between pearlite and ferrite, which enhances the corrosion of the ferrite because the cementite which is contained in the pearlite is electrochemically more stable than ferrite, this is consistent with the findings of the other researchers [13,24].

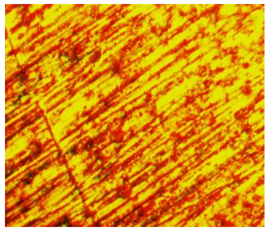
There is a clear difference in the topography of the surface of immersed samples in crude oil after add the inhibitors at 300 and 323K , as shown in Fig 8 (a and b)



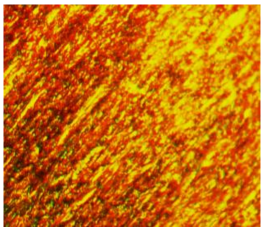
without inhibitor — 30ppm EB-A — 40ppm EB-D — 30ppm CRW

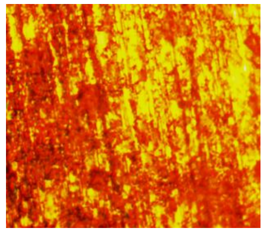
(a_ at 300°K)





without inhibitor 30ppm EB-A





40ppm EB-D 30ppm CRW

(b_ at 323°K)

Fig. 8 Changes in the topography of surface samples due to the addition of different type of inhibitor , the samples immersed in crude oil for 68 days at 323°K.

3.5. Comparison Between Different Inhibitor

Fig.9 show the corrosion path of the samples immersion in crude oil without and with the best amounts of the three inhibitors adding at 300 and 323k , from the Fig there is a clear difference in the weight loss after adding the inhibitors, when we compared between the three inhibitors we show that the EB-A have the best performance at 300k , while the CRW inhibitor is the best at 323k.

Tables 2 and 3 show the corrosion rate of the samples immersed in crude oil in the absence and presence inhibitors at 300 and 323k, respectively , and show the inhibition efficiency of the three inhibitors which adding at different amounts, as its clear from these tables, the inhibition efficiency of the CRW inhibitor decrease from (84.21) to (58.53)% at 300k and also decrease from (88.73) to (51.28)% at 323k, with increase the inhibitor amount from 30ppm to 60ppm, respectively.

The inhibition efficiency of the EB-A inhibitor also decrease from (94.4) to (58.95)% at 300k and also decrease from (86.71) to (60.8)% at 323k with increase the inhibitor amount from 30ppm to 60ppm, respectively.

While the inhibition efficiency of the EB-D inhibitor increase from (66.79) to (82.87)% at 300k and it also increase from (80.28) to (84.6)% at 323k when the inhibitor amount increase from 30ppm to 40 ppm and then decrease to reached (68.76) and (56.79)% at 300k and (42.22) and (33.47)% at 323k when the inhibitor amount increase to 50ppm and 60ppm, respectively.

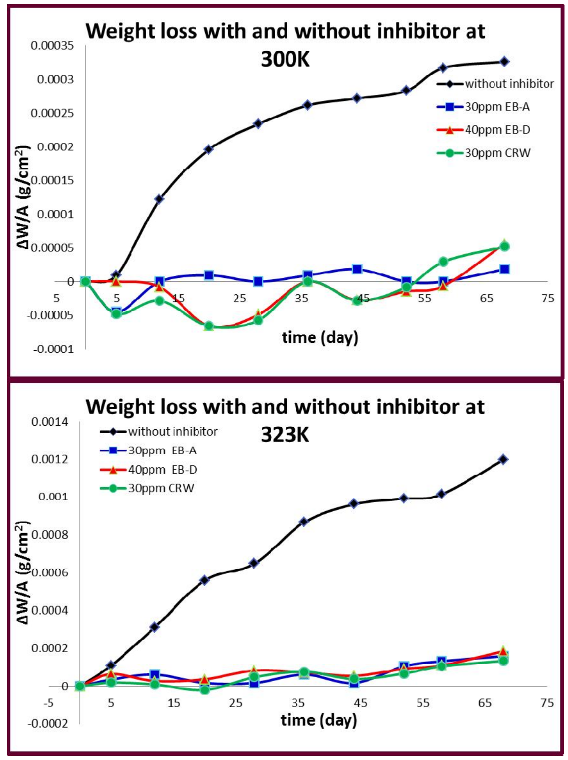


Fig. 9 Corrosion rate versus time curves for carbon steel in crude oil with and without inhibitor at different inhibitors and at different temperatures

4. Conclusions

Many of significant contributions to the corrosion research produce from this study and thus specified the next conclusions:

1- The EB-A and EB-D inhibitors contain an alkaloids, phenols and tannins which make them an effective inhibitors. Also the FT-IR peaks indicate that the EB may be containing aliphatic, aromatics and phenols which have an inhibition properties.
2- The EB extracts made as an effective inhibitors to the carbon steel in the crude oil at very low amounts.
3- The mechanism of inhibition is due to the adsorption on the metal surface, and it is considered to be mixed type inhibitors
4- The EB –A is the best corrosion inhibitor in this research in the terms of cost, toxicity, availability and performance.
5- The results appear a general corrosion type.
6- The highest IE% of the EB-A inhibitor equals (94.4)% when add 30ppm from it to the crude oil at 300°k.

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