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Efficiency of some non ionic surfactants based on tolyl triazole as corrosion inhibitors for oil tubing steel during production process

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ABSTRACT

This work was aimed to prepare four non ionic surfactants based on tolyl triazole (I, II, III and IV), and evaluate their efficiency as corrosion inhibitors for X - 65 type carbon steel in deep oil well formation using potentiodynamic polarization, electrochemical impedance spectroscopy (EIS) and weight loss measurements. The experimental results showed that these inhibitors revealed a very good corrosion inhibition even at low concentrations. It was found that, the adsorption ability of the surfactant molecules on carbon steel surface increased with the increase of both ethylene oxide units and molecular size. Critical micelle concentration (cmc) represents the key factor for the selected surfactants. Potentiodynamic polarization curves indicate that the inhibitors under investigation act as mixed type i.e., promoting retardation of both anodic and cathodic discharge reactions. Also, EIS data showed that both (I %) and charge transfer resistance (R_t) are increased where as electrochemical double layer resistance (Cdl) is decreased. Finally, scanning electron microscope (SEM) and energy dispersive X- rays analysis (EDX) were used to study the nature of the protected film formed on carbon surface.

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Introduction

Carbon steel has been widely employed as construction materials for pipe work in the oil and gas production such as down hole tubular, flow lines and transmission pipelines [1]. Corrosion phenomenon in e oil fields appears as leak in tanks, casing, tubing, pipe line, and other equipment. This process changes the base metal to another type of materials. The most corrosive environment in oil field operations is caused by trace amounts of oxygen entering into a sour brine system, as well as the large amounts of carbon dioxide and hydrogen sulfide present in a deep oil well water (formation water). The corrosion rate depends on the fluid composition; e.g. Oil type, oil water ratio, water salinity and the acidity caused by CO₂ and H₂S [2]. Corrosion inhibitors (mainly, surfactants) are widely employed in the petroleum industry to protect iron and steel equipment used in drilling, production, transport, and refining of hydrocarbons [3- 6]. The efficiency of the inhibition film depends on the inhibitor concentration and contact time with the metal surface. In fact, introducing of ethylene oxides into surfactant molecule (ethoxylation) increases the inhibitive effect of surfactant [7]. The presence of these groups increases the solubility of surfactant and hence the extent of its adsorption on the metal surface increases and consequently its inhibitive action improves. Many studies on the inhibition of the corrosion of carbon steel by some ethoxylated surfactants have been carried out in different corrosive environments [8 - 12]. This work is aimed to examine the effectiveness of the new synthesized, environmental friendly non ionic surfactants on the corrosion rate of carbon steel in deep oil wells formation water. It is an onset of series of works currently under investigation in our labs.

Experimental

Tele:

2.1. Chemical composition of the investigated carbon steel alloy

X - 65 type carbon steel specimens used in this investigation were cut from unused petroleum pipeline as regular edged cuboids with dimensions 2.5 cm \times 2.5 cm \times 0.7 cm. The chemical composition of X- 65 carbon steel alloy is listed in Table 1.

Deep oil well formation water

Deep oil wells formation water is naturally in the rocks before drilling. Most oil field water contains a variety of dissolved organic and inorganic compounds. The major elements usually present are sodium, calcium, magnesium, chloride, bicarbonate, and sulfate. The Chemical composition of the oil wells formation water used in this investigation and its physical properties are show in Table 2.

Synthesis of the inhibitors

Tolytriazole (1 mol) was added to (1mol) of citric acid in three necked round bottomed flask equipped with a mechanical stirrer, condenser and a thermometer. The reaction mixture was heated to 155°C until the theoretical amount of water has been collected using Dean Stark apparatus. The obtained intermediate reacts with poly ethylene glycol (PEG 400, 3000, 4000 and 6000) to give the inhibitors (I, II. III. IV) according to scheme1:

The chemical structure of the obtained product was confirmed by various spectroscopic techniques.

Weight loss measurements

The specimens were polished with different grade emery papers, degreased with ethanol [13]. Then washed with bidistilled water and finally dried. The weight losses (mg cm⁻²) of rectangular carbon steel specimens in deep oil wells formation water in the absence and presence of various concentrations of the inhibitors were determined. Triplicate specimens were exposed to each condition and the mean weight lose was reported.



Scheme 1Potentiodynamic polarization measurements

The electrochemical measurements were carried out using Volta lab80 (Tacussel-radiometer PGZ402) controlled by Tacussel corrosion analysis soft ware model (Volta master 4). A platinum electrode was used as auxiliary electrode. All potentials were measured against a saturated calomel electrode (SCE) as a reference electrode. All the measurements were carried out in air-saturated solutions and at ambient temperature $(25\pm^{\circ}C)$.

Electrochemical impedance spectroscopy (EIS)

Electrochemical impedance (EIS) measurements were carried out using Volta lab 80 potentiostat (Tacussel-radiometer PGZ402) controlled by_Tacussel corrosion analysis soft ware model (Volta master 4). Impedance spectra were obtained in the frequency range between 100 KHz and 10 mHz using 10 steps per frequency decade at open circuit potential after 4 hours of immersion time. AC signal with 10 mV amplitude peak to peak was used to perturb the system. EIS diagrams are given in the both Nyquist and Bode representation.

Surface tension measurements

The surface tension (γ) was measured using (DuNouy Tensiometer, Kruss Type 8451) for various concentrations of the investigated surfactants.

Scanning electron microscopy

The surface examination was carried out using scanning electron microscope (Jeol 5400, Japan). The energy of the acceleration beam employed was 30KV. All micrographs were taken at a magnification power (X 750).

Energy Dispersive Analysis of X-Rays (EDX)

EDX survey spectra were used to determine which elements present on the electrode surface before and after exposure to the inhibitor solution

Results and discussion

Weight loss measurements

Fig. 1 shows weight loss-time curves for carbon steel immersed in deep oil wells formation water in the absence and presence of various concentrations of the inhibitor (IV) as a representative sample. It is apparent that, the increase in additive concentration was accompanied by a decrease in weight loss until critical micelle concentration is reached. This behavior can be attributed to the adsorption of the surfactant molecules on carbon steel surface. The maximum inhibition efficiency (IE %) was exhibited at 450 ppm of inhibitors. This concentration is considered as critical micelle concentration of the surfactant (CMC).



Fig. 1: Weight loss-time curves for carbon steel dissolution in Formation water in the absence and presence of various concentration of inhibitor (IV) at 303 K.

The degree of surface coverage (θ) , the percentage inhibition efficiency (IE %) were calculated as follow [14]:

$$\theta = \frac{\mathbf{W}_{\text{free}} - \mathbf{W}_{\text{inh.}}}{\mathbf{W}_{\text{free}}}$$
(1)
$$= \frac{\mathbf{W}_{\text{free}} - \mathbf{W}_{\text{inh.}}}{\mathbf{W}_{\text{free}}} \mathbf{X} \mathbf{100}$$
(2)

Where, w_{free} and w_{inh} represent the values of weight loss of carbon steel in formation water in the absence and presence of the inhibitor, respectively.

(3)

Corrosion rate was calculated from the following relation [15]:

Corrosion rate(mpy) =
$$\frac{\mathbf{K} \mathbf{W}}{\mathbf{D} \mathbf{A} \mathbf{T}}$$

IE %

Where, $K = 3.45 \times 10^6$, T is the exposure time in hour, A is the surface area of the test specimen, W is the weight loss in gram and D is density of the test specimen in g/cm3, respectively. Complete data are summarized and listed in Table 3. **Potentiodynamic polarization measurements**





Figs. 2 show the cathodic and anodic polarization curves of carbon steel immersed in deep oil wells formation water in the absence and presence of different concentrations of the inhibitor (IV) as a representative sample.

Table1: Chemical composition of carbon steel alloy									
Element	С	Si	Р	S	Ni	Mo	V	Al	Fe
Content (wt/wt)	0.07	0.18	0.03	0.06	0.02	0.004	0.002	0.04	Rest

Table 2: Chemical composition and Physical properties of deep oil well formation water used in this investigation

Chemi	cal composition		
Ionic concentration	Concentration, ppm (wt/wt)		
$Na^+ \& K^+$	61978		
Ca ²⁺	5800		
Mg^{2+}	1180		
Ba ²⁺	8.50		
Sr^{2+}	443		
Cl	91350		
SO_4^{-2}	400		
HCO ₃ ⁻	247		
T.D.S	153000		
Physi	ical properties		
Property	Value		
Specific gravity	1.109		
pH	6.23		
Salinity as NaCl	150581 ppm (wt.)		
Total alkalinity	350 ppm (wt.)		
Total hardness	19321		

Table 3: Data obtained from weight loss measurements for carbon steel dissolution in Formation water in absence and presence of various concentrations of the inhibitors (I, II, III and IV) at 303K

Inhibitor	Concentration, (ppm)	Corrosion rate, (mpy)	θ	%IE
Formation water (blank)	-	0.921	-	-
	150	0.344	0.521	52.1
	200	0.301	0.621	62.1
Inhibitor I	250	0.280	0.722	72.2
	350	0.222	0.815	81.5
Inhibitor I Inhibitor II Inhibitor II	450	0.181	0.856	85.6
Formation water (blank) Inhibitor I Inhibitor II Inhibitor II	150	0.310	0.662	66.2
	200	0.270	0.714	71.4
Inhibitor II	250	0.230	0.743	74.3
Inhibitor I Inhibitor II Inhibitor III	350	0.150	0.832	83.2
	450	0.120	0.886	88.6
	150	0.300	0.684	68.4
	200	0.260	0.723	72.3
Inhibitor III	250	0.220	0.778	77.8
	350	0.140	0.847	84.7
	450	0.110	0.895	89.5
Inhibitor II Inhibitor III	150	0.280	0.704	70.4
	200	0.250	0.775	77.5
Inhibitor IV	250	0.210	0.824	82.4
	350	0.132	0.863	86.3
Inhibitor I Inhibitor II Inhibitor III Inhibitor IV	450	0.100	0.902	90.2

Table 6:	Surface active	properties of	f the four synt	thesized inhibitors	(I, II, III	and IV
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Surfactants	CMC, (mmol/l)	Гстс, (mN/m)	Пстс, (mN/m)	$\frac{\Gamma max \times 10}{(mol/cm^2)}$	Amin (nm ²)
Inhibitor I	2.8×10 ⁻⁴ .	35	37	7.49	0.22
Inhibitor II	4.5×10 ⁻⁵	41	31	7.4	0.22
Inhibitor III	3.5×10	41	31	7.1	0.23
Inhibitor IV	3.2×10	43	29	6.7	0.24

 Table 4: Data obtained from potentiodynamic polarization measurements of carbon steel in formation water solution in absence and presence of various concentrations of the inhibitors (I, II, III and IV) at 303K

Inhibitor	Concentration (ppm)	R _p , (k ohm cm ⁻²)	I _{corr.} (mA cm ⁻²)	E _{corr} , mv, (vs.SCE)	b _c . (mV dec ⁻¹)	b _a , (mV dec ⁻¹)	IE %
Blank	0	0.97	26	-793	-113.0	102	-
	150	1.18	13.1	-781	-112.2	101	49.5
	200	1.19	10.2	-780	-116.5	103	60.7
1	250	2.18	8.4	-771	-124.0	101	67.7
	350	2.68	6.5	-764	-136.6	104	75.0
	450	2.76	4.3	-758	-140.0	103	83.5
	150	0.24	9.4	-707	-168.2	103	63.8
	200	1.68	8.7	-714	-130.8	101	66.5
II	250	1.91	6.9	-713	-124.7	100	73.0
	350	2.91	5.3	-696	-151.8	102	79.6
	450	2.95	3.8	-673	-120.0	109	85.4
	150	2.60	8.3	-806	-125	112	68.0
	200	2.62	7.1	-774	-136	113	72.7
III	250	2.63	5.8	-821	-124	102	77.7
	350	2.66	2.3	-816	-132	116	91.1
	450	2.95	1.1	-773	-120	101	95.7
	150	6.8	7.5	-774.7	-121	113	71.1
	200	2.62	6.8	-774	-132	105	73.8
IV	250	6.94	5.2	-745.2	- 123	103	80.0
	350	9.31	2.1	-755	- 128	102	92.0
	450	9.10	1.05	-821.9	-124	101	96.2

Table 5: Data obtained from electroe	chemical impedance spectroso	copy (EIS) measurements	s of carbon steel	in Formation
water solution in absence and	presence of various concentra	ations of the inhibitors (I	II, III and IV)	at 303K

Inhibitor	Concentration (ppm)	R _t , (ohm cm ⁻²)	C _{dl} (mF cm ⁻²)	IE%
Blank	0	0.30	1.31	-
	150	1.43	7.51	79.2
	200	1.84	6.90	83.6
Ι	250	1.83	6.32	83.6
	350	1.85	6.10	83.7
	450	2.38	5.90	87.3
	150	1.48	1.50	78.3
	200	2.46	1.29	87.8
II	250	2.61	1.21	88.4
	350	2.62	0.85	88.5
	450	2.79	0.11	89.2
	150	2.17	1.25	85.7
	200	2.51	1.20	88.0
III	250	2.74	1.16	89.0
	350	2.96	1.07	89.8
	450	3.78	0.84	91.8
	150	1.56	1.70	82.3
IV	200	1.61	1.63	88.4
	250	2.77	1.57	89.1
	350	2.89	1.48	89.6
	450	3.10	1.32	93.3

Electrochemical parameters such as corrosion potential $(E_{corr.})$, corrosion current density $(i_{corr.})$, cathodic and anodic Tafel slopes (b_c and b_a) and polarization resistance (R_p) were calculated. From the obtained polarization curves, it is clear that the corrosion current densities $(i_{corr.})$ were decreased with increasing the concentration of the inhibitor (IV) with respect of the blank (inhibitor free solution). This behavior confirms a greater increase in the energy barrier of carbon steel dissolution process.

The degree of surface coverage (θ) and the percentage inhibition efficiency (IE %) were calculated using the following equations [16]:

$$\theta = \frac{\mathbf{I}_{o} - \mathbf{I}}{\mathbf{I}_{o}}$$
(5)

$$IE\% = \frac{I_o - I}{I_o} \times 100 \tag{6}$$

where i_0 and i are the corrosion current densities in the absence and presence of the inhibitor, respectively.

The values of polarization resistance (Rp) were calculated from the well known Stern – Geary equation [17]:

$$R_{p} = \frac{b_{a}b_{c}}{2.303 I_{cor}(b_{a} + b_{c})}$$
(7)

From the obtained corrosion data, it is clear that the Tafel lines are shifted to more negative and more positive potentials for the anodic and cathodic processes, respectively relative to the blank curve. This means that the selected compound acts as mixed type inhibitor, i.e., promoting retardation of both anodic and cathodic discharge reactions. Also, the slopes of the cathodic and anodic Tafel lines are approximately constant and independent on the inhibitor concentration. This means that, the selected inhibitor has no effect on the metal dissolution Complete data obtained from polarization mechanism. measurements are summarized and listed in Table 4. The results indicate, that the percentage inhibition efficiency (IE %) of the inhibitor (IV) is greater than that of the inhibitor (I, II and III). This could be attributed to the increase of the number of actives sites, the electron densities and the molecular size.

Electrochemical impedance spectroscopy (EIS)

The corrosion behavior of carbon steel in deep oil wells formation water in the absence and presence of various concentrations of inhibitor (IV) as a representative sample was investigated by EIS technique. Nyquist and Bode plots are shown in Figs. 3&4. It is clear from the plots that the impedance response of carbon steel in formation water was significantly changed after the addition of the inhibitor molecules. For analysis of the impedance spectra containing one capacitive loop, the equivalent circuit (EC) giving in Fig. 5 was used, where, R_s represents the solution resistance, Rt represents the charge transfer resistance and C_{dl} represents the electrochemical double layer capacitance. Various parameters such as the charge transfer resistance (R_t) and double layer capacitance (C_{dl}) and percentage inhibition subtracting efficiency IE % were calculated accordion to the following equations. and listed in Table 5.

The values of R_t were given by the high frequency impedance from the low frequency one as follow [18]:

 $R_t = Z'_{re.}$ (at low frequency) $- Z'_{re.}$ (at high frequency) (8)

The values of C_{dl} were obtained at the frequency f_{max} , at which the imaginary component of the impedance is maximal – Z_{max} using the following equation [19]:



Fig.3. Nyquist plots for carbon steel in oil wells formation water in absence and presence 0f different concentration of inhibitors (IV)



Fig. 4. Electrochemical impedance curves (Zr–logZ relationship) of carbon steel in formation water in absence and presence of different concentrations of the inhibitor (IV).



Figure 5. The equivalent circuit model for the electrochemical impedance measurements.

$$C_{dl} = \frac{1}{2\pi f_{\max}} \frac{1}{R_t}$$
(9)

The percentage inhibition efficiency IE % was calculated from the values of R_t using the following equation [20]:

$$IE \% = \frac{Rt (inh) - Rt}{Rt (inh)} X 100$$
(10)

Where R_t and R_t (inh) are the charge transfer resistance values in the absence and presence of inhibitor, respectively. Increasing the value of charge transfer resistance (R_t) and decreasing the value of double layer capacitance (C_{dl}) by increasing the

inhibitor concentration indicate that the surfactant molecules inhibit corrosion rate of carbon steel in deep oil wells formation water by adsorption mechanism [18]. From EIS data it was found that the percentage inhibition efficiency of inhibitor (IV) is greater than that of inhibitors (I, II and III). Thereby, agree with aforementioned results of weight loss and the potentiodynamic polarization measurements.

Surface tension measurements

The values of surface tension (γ) were measured at various concentrations of the inhibitors (I, II, III and IV). The measured values of (γ) were plotted against logarithm of surfactant concentration; log *C*, as shown in Fig. 6. These plots indicate that each surfactant is molecularly dispersed at low concentration, leading to a reduction in surface tension until certain concentration is reached the surfactant molecules form micelles, which are in equilibrium with the free surfactant molecules. The intercept of the two straight lines designates the critical micelle concentration (cmc), where saturation in the surface adsorbed layer takes place. The surface active properties of the surfactants (I, II, III and IV); effectiveness (π_{cmc}), maximum surface excess (Γ_{max}) and minimum area per molecule (A_{min}) was calculated using the following eq. [21]:



Fig.6. Surface tension (γ) vs. logC at different concentrations of the inhibitor (I, II, III and IV) at 303 K.



$$\Gamma_{\max} = \frac{-1}{RT[\partial \gamma / \partial \ln C]_{\rm T}} \tag{12}$$

$$A_{\min} = \frac{1}{\Gamma_{\max} \times N_{A}}$$
(13)

Where γ_0 is the surface tension of pure water, γ_{cmc} the surface tension at critical micelle concentration and *NA* is the Avogadro's number. The data obtained from surface tension measurements were summarized and presented in Table 6.

The critical micelle concentration (CMC) considers a key factor in determining the effectiveness of surfactants as corrosion inhibitors [22]. Below the CMC, as the surfactant concentration increases, the surfactant molecules tend to adsorb on the metal surface, leading to increase the inhibition efficiency of the surfactant. On the other hand, increasing the surfactant concentration above CMC does not affect the surface tension, as shown in Fig. 7, which is, in turn, does not influence the value of inhibition efficiency. This could be attributed to the fact that above CMC the surface of carbon steel is covered with a monolayer of surfactant molecules and the additional molecules combine to form micelles in the bulk of solution. It has to be noted that for a surfactant to be an excellent corrosion inhibitor it's should exhibit a low CMC value, since the inhibition effectiveness decreases as the CMC value increases. On the basis of this view, among all the studied compounds, the surfactant (IV) which shows the lowest CMC value and hence it considers the most effective corrosion inhibitor for carbon steel in oil well formation water.



Fig.7. Schematic representation for adsorption process of surfactant molecules on carbon steel surface:(A) at zero concentration of the inhibitor (B) before CMC (C) at CMC (D) after CMC.

Scanning electron microscopy (SEM)

Fig. 8a shows SEM image of polished carbon steel surface. The micrograph shows a characteristic inclusion, which was probably an oxide inclusion [23]. Fig. 8b shows SEM of the surface of carbon steel specimen after immersion in formation water for 6 days in absence of inhibitor, while Fig. 8c shows SEM of the surface of another carbon steel specimen after immersion in formation water for the same time interval in presence of 450 ppm of the inhibitor (IV). The resulting scanning electron micrographs reveal that, the surface was strongly damaged in absence of the inhibitor, but in presence of 450 ppm of the inhibitor (IV), there is much less damage of the surface. This confirms the observed high inhibition efficiency of the inhibitor (IV) at this concentration.

Fig. 8. Scanning electron micrographs of carbon steel samples (at X = 750).



(a)After polishing.



(b) after immersion in deep oil wells formation water for 6 days.



 (c) after immersion in deep oil wells formation water for 6 days in the presence of 450 ppm of inhibitor (IV).
 Energy Dispersive Analysis of X-Rays (EDX)

The EDX spectrum in Fig. 9a shows the characteristics peaks of some of the elements constituting the polished carbon steel surface. The spectrum of the polished carbon steel surface after immersed in the formation water in absence and presence of inhibitor IV for 60 days, are shown in Figs. (9b & 9c) respectively. The spectra of Fig. (9c) show that the Fe peak is considerably decreased relative to the samples in Figs. (9a & 9b). This decreasing of the Fe band is indicated that the strongly adherent protective film of inhibitor IV formed on the polished carbon steel surface, which leading to a high degree of inhibition efficiency [24]. The oxygen signal apparent in Fig. (9b) is due to the carbon steel surface exposed to the formation water in absence of inhibitor IV. Therefore, the EDX and SEM examinations of the carbon steel surface support the results obtained from the chemical and electrochemical methods that the synthesized surfactants inhibitors are a good inhibitor for the carbon steel in the oil wells formation water.



Fig. 9. EDX of carbon steel surface:

(a) after polishing

5

KeV



(b)) after immersion in deep oil wells formation water for 6 days.



(c) after immersion in deep oil wells formation water for 6 days in the presence of 450 ppm of inhibitor (IV).

Conclusions

From the obtained results one can conclude the following points: 1- The synthesized compounds act as good corrosion inhibitors for carbon steel in oil wells formation water formation water.

2- The adsorption of the inhibitor molecules obeyed the Langmuir adsorption isotherm.

3- The percentage inhibition efficiency (IE %) of surfactants increased by increasing both the number of ethylene oxide units and molecular size of the molecule.

4-By increasing concentration of inhibitor, R_t was increased while C_{dl} was decreased.

5 The critical micelle concentration considers a key factor in determining the effectiveness of surfactants as corrosion inhibitors due to large reduction of surface tension at CMC. 6-The inhibition mechanism is attributed to the strong adsorption ability of the selected surfactants on carbon steel surface, forming a good protective layer, which isolates the surface from the aggressive environment as seen from SEM and EDX techniques.

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