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Corrosion inhibition and biocidal effect of some laboratory synthesized cationic surfactants in oil fields

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ABSTRACT

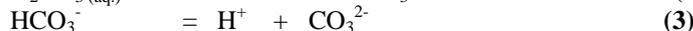
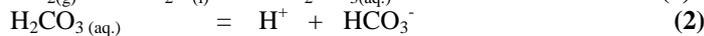
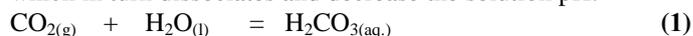
Two cationic surfactants namely; N-(2-hydroxyethyl)-N,N-dimethyldodecane-1-aminium bromide (I) and , N-(2-(2-mercaptoacetoxy)ethyl)-N,N-dimethyl dodecan-1-aminium bromide (II) were synthesized and characterized using FTIR and NMR spectroscopic methods. The two synthesized surfactants were evaluated as corrosion inhibitors for X- 65 type carbon steel in oil wells formation water under CO₂ environment at 50 °C and antimicrobial agents against sulfate reducing bacteria (SRB). It was found that the percentage inhibition efficiency (η%) was increased by increasing the inhibitor concentration until the critical micelle concentration (CMC) is reached. Potentiodynamic polarization curves indicated that the inhibitors under investigation act as mixed type. The data obtained from electrochemical impedance spectroscopy (EIS) were analyzed to model the corrosion inhibition process through equivalent circuit. The nature of the protective film formed on carbon steel surface was analyzed by SEM and EDX techniques. The serial dilution method was used to evaluate the inhibiting effect of these compounds on the sulfate reducing bacteria growth. The results showed that the prepared compounds have good antimicrobial activities against the SRB as well as they have acceptable efficiency as corrosion inhibitors for X- 65 type carbon steel in oil wells formation water.

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Introduction

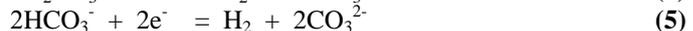
Carbon steel has been widely employed as construction materials for pipe work in the oil and gas production such as down hole tubular, piping systems and transmission pipelines [1,2]. Corrosion in the oil field appears as leak in tanks, casing, tubing, pipeline and other equipment. This process changes 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) [3]. This type of corrosion forms a scale which varies from dense and adherent to less porous and thick [4,5]. Corrosion inhibitors (mainly, surfactants) are widely employed in the petroleum industry to protect iron and steel equipment used in drilling, production, transportation and refining of hydrocarbons[6,7]. The efficiency of the inhibition film depends on the inhibitor concentration and immersion time. The antimicrobial action of cationic surfactants is based on their ability to disrupt and disorganize the integral bacterial membrane by combined hydrophobic and electrostatic adsorption phenomena at the membrane-water interface [8,9]. Recently, a new generation of surfactants, gemini surfactants, which contain two hydrophilic groups and two hydrophobic groups in the molecules rather than one hydrophilic group and one hydrophobic group of conventional surfactants, have attracted great interest. These surfactants are more efficient at reducing surface tension and forming micelles than conventional surfactants [10-14]. Extensive investigation studies had been carried out to understand and control CO₂ corrosion [15]. CO₂

dissolves in oil wells produced water forming carbonic acid which in turn dissociates and decrease the solution pH:



In view of the above mentioned conditions, CO₂ corrosion had been proposed as follow:

Firstly, the cathodic reaction can proceed by one of the following reactions:



Secondly, the anodic reaction can takes place through:



Under such conditions, a ferrous carbonate corrosion scale can be formed according to :



The nature of oxide/passive film resulted from a corrosion process contributes to the overall behavior of it as well as temperature and chemical composition of the aqueous media [16]. This work is aimed to examine the efficiency of new synthesized, environmental friendly cationic surfactants on the corrosion rate of carbon steel in deep oil wells formation water. Also, to study the biocidal effect of these surfactants.

Experimental

Chemical composition of the investigated carbon steel alloy:

The chemical composition of X- 65 type carbon steel alloy used in this study is listed in **Table 1**.

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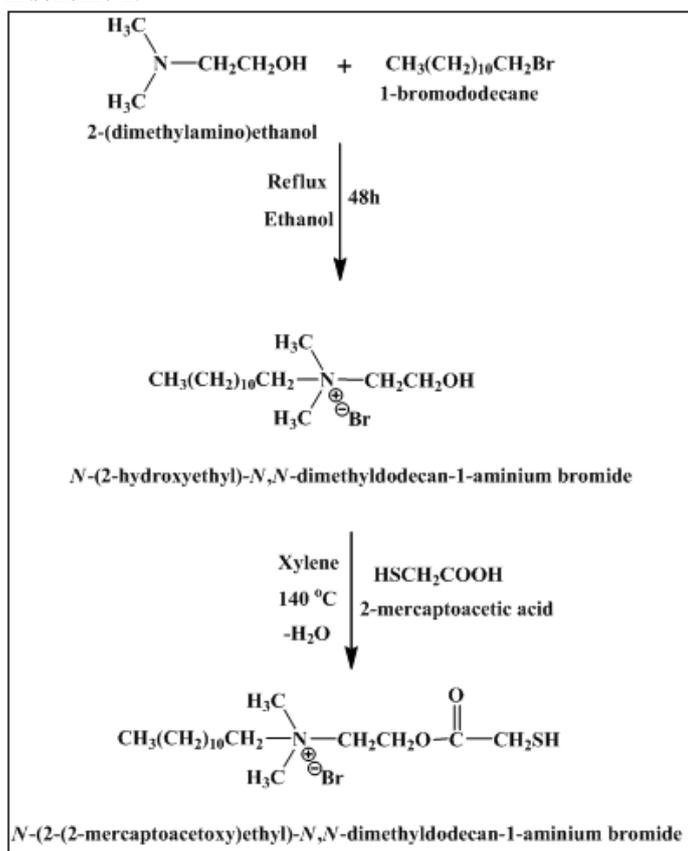
Deep oil well formation water

Deep oil wells formation water naturally exists in the reservoir 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 shown in **Table 2**.

Synthesis of the inhibitors:

The two cationic surfactants used in this study were synthesized as shown in **scheme 1**, by reaction of one mole of 1-bromododecane with one mole of 2-(dimethylamino)ethanol to produce N-(2-hydroxyethyl)-N,N-dimethyldodecane-1-aminium bromide (inhibitor I). The reactants were allowed to reflux in ethanol at 70 °C for 12 h. The mixture was allowed to cool-down. Then, the obtained precipitate was further purified by diethyl ether then recrystallized by ethanol.

One mole of N-(2-hydroxyethyl)-N,N-dimethyldodecane-1-aminium bromide and one mole of 2-mercaptoacetic acid were esterified in presence of toluene as solvent. The reaction was completed when the water removed from reaction system. The reaction mixture was distilled under vacuum to remove the unreacted material and solvent completely to obtain N-2-(2-mercaptoacetoacetoxy)ethyle) N,N-dimethyldodecan -1-aminium bromide (inhibitor II) [17]. The chemical structure was confirmed by FTIR and NMR spectroscopy analysis. The overall steps of the reactions and their conditions are presented in **scheme 1**.



Structure confirmation of the synthesized inhibitors

FTIR spectroscopy

FTIR spectrum of the synthesized cationic surfactant showed the following absorption bands at 722.09 cm^{-1} ($(\text{CH}_2)_n$ rocking), 1086.14 cm^{-1} (C-N^+), 1284.67 cm^{-1} (CH_3 symmetric bending), 1466.02 cm^{-1} (CH_2 asymmetric bending), 1752.52 cm^{-1} (C=O stretching), 1170 cm^{-1} (C-O-C asymmetric stretching),

2589.14 cm^{-1} (SH stretching), 2854.61 cm^{-1} (CH symmetric stretching) and 2920.35 cm^{-1} (CH asymmetric stretching). FTIR spectrum of the synthesized inhibitor is shown in **Fig. 1**.

The FTIR spectra confirmed the expected functional groups in the synthesized cationic surfactant.

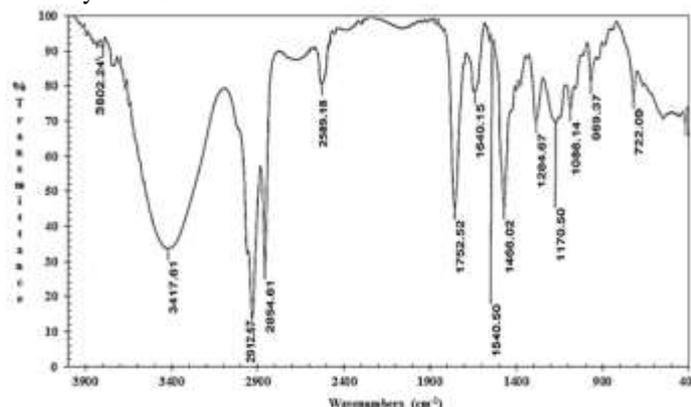


Fig 1. FTIR spectrum of N-(2-hydroxyethyl)-N,N-dimethyldodecan-1-aminium bromide

¹HNMR spectroscopy

The ¹HNMR (DMSO) spectrum of N-(2-hydroxyethyl)-N,N-dimethyldodecane-1-aminium bromide showed different bands at $\delta=0.81$ ppm (t, 3H, $\text{NCH}_2(\text{CH}_2)_9\text{CH}_2$ **CH**₃); $\delta=1.21$ ppm (m, 18H, $\text{NCH}_2\text{CH}_2(\text{CH}_2)_9\text{CH}_3$); $\delta=1.63$ ppm (m, 2H, NCH_2CH_2 (CH_2)₉**CH**₂); $\delta=3.27$ ppm (t, 2H, $\text{NCH}_2\text{CH}_2(\text{CH}_2)_9\text{CH}_3$); $\delta=3.02$ ppm (s, 6H, **CH**₃**N** **CH**₃); $\delta=3.32$ ppm (t, 2H, $\text{NCH}_2\text{CH}_2\text{OH}$); $\delta=5.22$ ppm (t, 2H, $\text{NCH}_2\text{CH}_2\text{OH}$); $\delta=3.78$ ppm (s, 1H, $\text{NCH}_2\text{CH}_2\text{OH}$). ¹HNMR spectrum of the synthesized inhibitor is shown in **Fig. 2**. The data of ¹HNMR spectra confirmed the expected hydrogen proton distribution in the synthesized cationic surfactant.

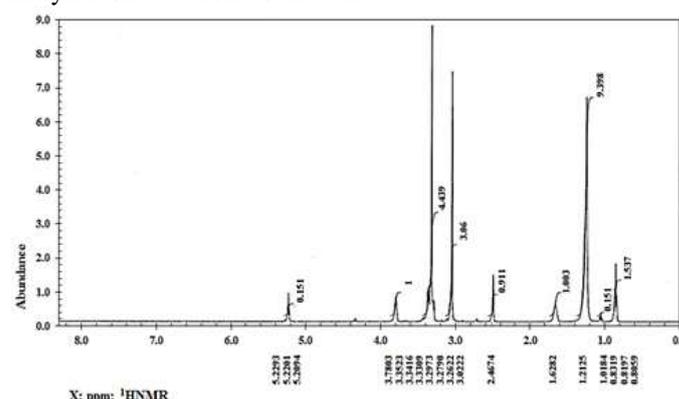


Fig 2. ¹HNMR Spectrum of N-(2-hydroxyethyl)-N,N-dimethyl dodecan-1-aminium bromide

Potentiodynamic polarization measurements

All electrochemical measurements were carried out using Volta lab 80 (Tacussel-radiometer PGZ402) controlled by Tacussel corrosion analysis software 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 (50 °C).

Electrochemical impedance spectroscopy (EIS)

Impedance spectra were obtained in the frequency range between 100 KHz and 50 m Hz using 10 steps per frequency decade at open circuit potential after 3 h of immersion time. AC signal with 10 mV amplitude peak to peak was used to perturb the system. EIS diagrams are given in both Nyquist and Bode representations.

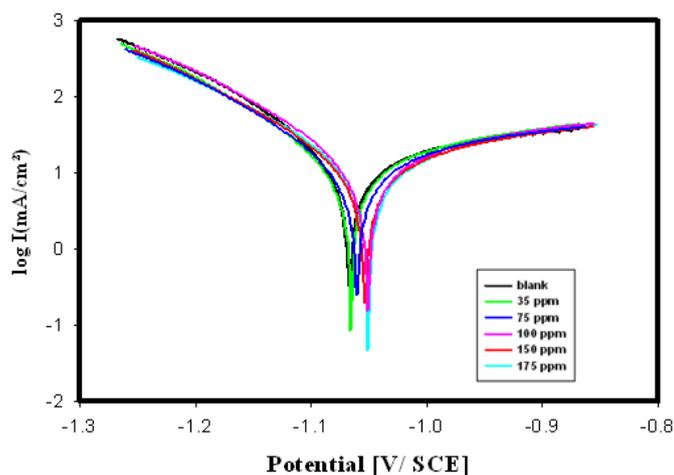


Fig. 3: Potentiodynamic polarization curves obtained for carbon steel immersed in oil wells formation water in the absence of various concentrations of the inhibitor (I) at 50°C under CO₂ environment

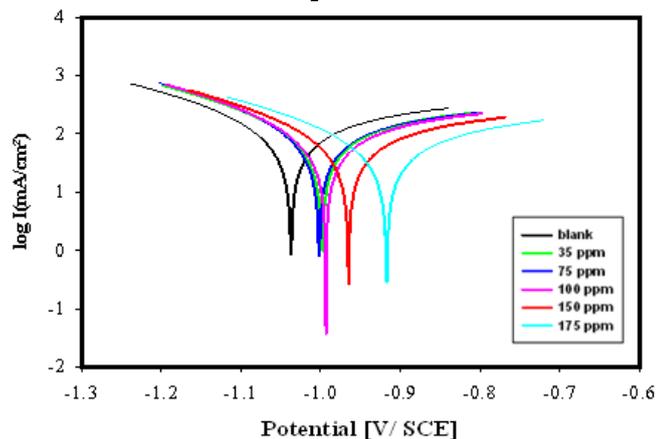


Fig. 4: Potentiodynamic polarization curves obtained for carbon steel immersed in oil wells formation water in the absence of various concentrations of the inhibitor (I) at 50°C under CO₂ environment

Surface tension measurements

The surface tension (σ) was measured using (Krüss K 6 Tensiometer type, a direct surface tension measurement using ring method) for various concentrations of the investigated surfactants.

Energy dispersive analysis of X-rays (EDX)

EDX system attached with a JEOL JSM-5410 scanning electron microscope was used for elemental analysis or chemical Evaluation of the synthesized compounds (I & II) against Sulfate Reducing Bacteria, SRB.

This test has been conducted according to ASTM D4412-84(re-approved 1990) [18]. The tested water has been subjected to growth of about 10^7 bacterial cell/ml. Two chemical samples (I & II) were tested as biocides by doses of 35, 75, 100, 150, 175 ppm and the system was incubated to contact time 3.0 hours, each system was cultured in SRB specific media.

Results and discussion

Potentiodynamic polarization measurements

Figs. 3 & 4 show the cathodic and anodic polarization curves of carbon steel immersed in deep oil wells formation water under CO₂ environment at 50 °C in the absence and presence of different concentrations of the inhibitor (I & II). 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 concentration of inhibitor (II) with respect to the blank (inhibitor free solution).. The degree of surface coverage (θ) and the percentage inhibition efficiency ($\eta\%$) were calculated using the following equations:

$$\Theta = 1 - i/i_0 \quad (9)$$

$$\eta\% = (1 - i/i_0) \times 100 \quad (10)$$

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

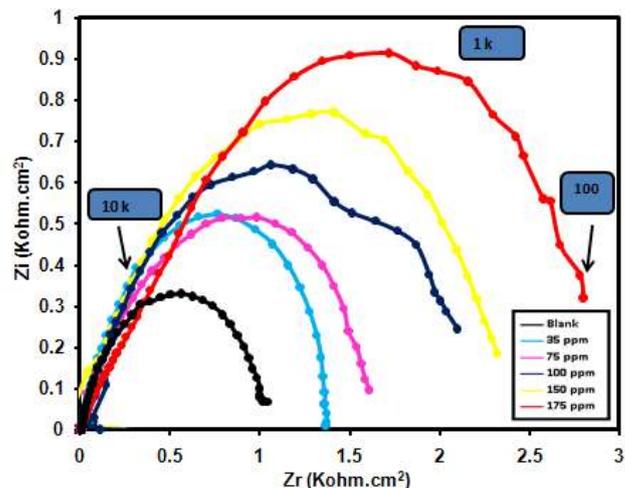


Fig. 5: Nyquist plot for carbon steel in oil wells formation water under CO₂ environment at 50 °C in the absence and presence of different concentrations of inhibitor (I)

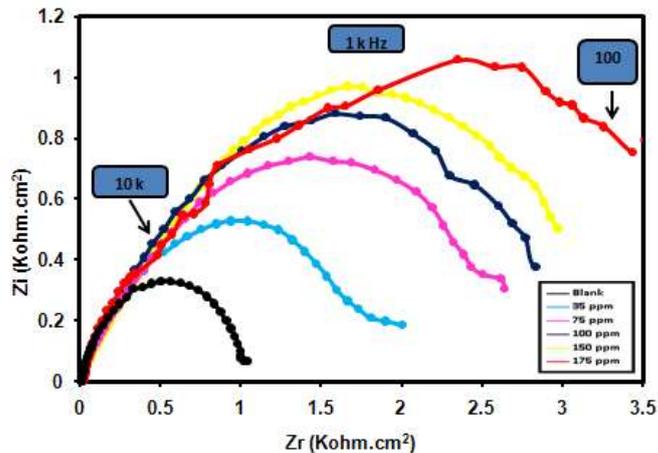


Fig. 6: Nyquist plots for carbon steel in oil wells formation water under CO₂ environment at 50 °C in the absence and presence of different concentrations of inhibitor (II)

From the obtained data, it is clear that 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 mechanism. Complete data obtained from polarization measurements are summarized and listed in **Table (3)**. The results indicate that the percentage inhibition efficiency ($\eta\%$) of the inhibitor (II) is greater than that of inhibitor (I). This could be attributed to the introducing of S - atom which can form additional $d\pi - d\pi$ bond with the metal surface [19].

Electrochemical impedance spectroscopy (EIS)

The corrosion behavior of carbon steel in deep oil wells formation water under CO₂ environment at 50 °C in the absence and presence of various concentrations of inhibitor (I) and inhibitor (II) were investigated by EIS technique. Nyquist is shown in **Figs 5&6**. 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. Various parameters such as the charge transfer resistance (R_t), double layer capacitance (C_{dl}) and percentage inhibition efficiency(n%)were calculated according to the following equations and listed in **Table 4**. The values of R_t were given by subtracting the high frequency impedance from the low frequency one as follows [20]:

$$R_t = Z_{re}^1 \text{ (at low frequency)} - Z_{re}^1 \text{ (at high frequency)} \quad (11)$$

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:

$$C_{dl} = \frac{1}{2\pi f_{max} R_t} \quad (12)$$

The percentage inhibition efficiency (n%) was calculated from the values of R_t using the following equation:

$$n\% = [1 - (R_t / R_{t(inh)})] \times 100 \quad (13)$$

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 [21]. For analysis of the obtained impedance spectra, the equivalent circuit (EC) was obtained using Boukamp program as shown in **Fig. 7**, where R_s is the solution resistance, R_t is the charge transfer resistance, C_{dl} is the electrochemical double layer capacitance, R_f is the film resistance and C_f is the film capacitance. From EIS data it was found that the percentage inhibition efficiency of inhibitor (II) is greater than that of inhibitor (I) thereby, agreeing with aforementioned results of potentiodynamic polarization measurements.

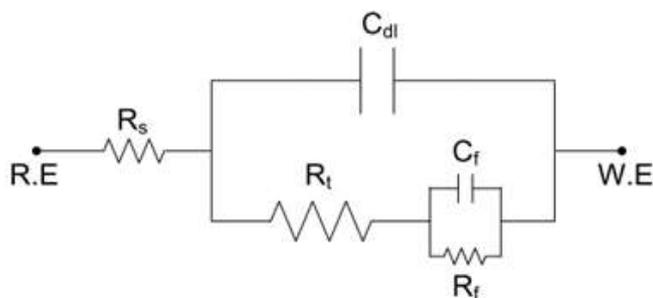


Fig. 7: Equivalent circuit used to model impedance data of carbon steel in oil well formation water at 50°C under CO₂ environment.

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 [22]. **Fig. 8b** shows SEM of the surface of carbon steel specimen after immersion in formation water for 70 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 the presence of 175 ppm of the compound II. The resulting scanning electron micrographs reveal that, the surface was strongly damaged in the absence of the inhibitor, but in the presence of

175 ppm of the compound II, there is less damage in the surface. This confirms the observed high inhibition efficiency of compound at this concentration.

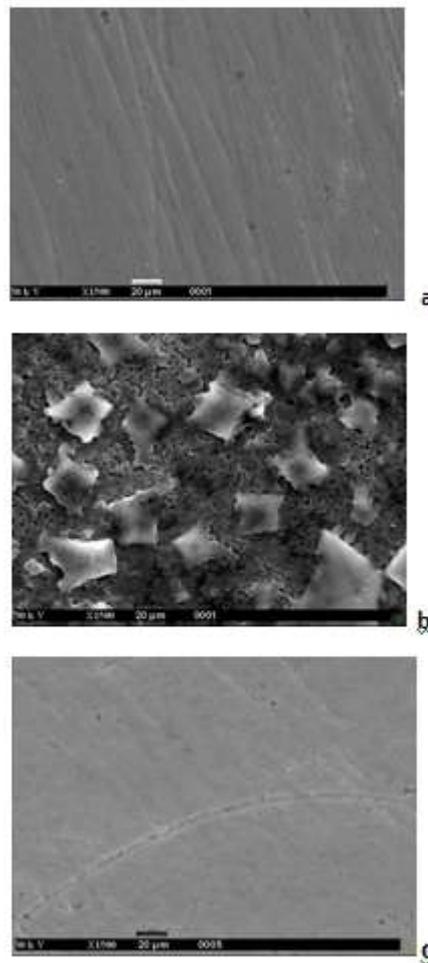


Fig 8: SEM images for the carbon steel surface: (a) polished sample, (b) after immersion in the oil wells formation water under sour conditions and (c) after immersion in the oil wells formation water at 50°C under CO₂ environment in the presence of inhibitor(II)

Energy dispersive analysis of X-rays (EDX)

The EDX spectrum in **Fig. 9a** shows the characteristic peaks of some of the elements constituting the polished carbon steel surface. The spectrum of the polished carbon steel surface after immersion in the formation water in the absence and presence of inhibitor (II) for 70 days, is shown in **Figs. 9b and 8c**, respectively. The spectrum of **Fig. 9c** shows that the Fe peak is considerably decreased relative to the samples in **Figs. 9a and b**. This decreasing of the Fe band is indicated that strongly adherent protective film of inhibitor (II) formed on the polished carbon steel surface, which leads to a high degree of inhibition efficiency[23]. The oxygen signal apparent in **Fig. 9b** is due to the carbon steel surface exposed to the formation water in the absence of inhibitor (II). Therefore, the EDX and SEM examinations of the carbon steel surface support the results obtained from the electrochemical methods that the synthesized surfactant inhibitors are a good inhibitor for the carbon steel in the oil wells formation water.

Efficiency as biocide

The antimicrobial activity of the two studied biocide (I,II) against SRB was determined by a serial dilution method at dosages of (35,70,100,150,175 ppm by weight) and the results are listed in table 5.

Table 1. Chemical composition of X- 65 type carbon steel alloy

Element	C	Si	Mn	P	S	Ni	Cr	Mo	V	Cu	Al	Fe
Content (Wt %)	0.09	0.22	1.52	0.01	0.05	0.04	0.02	0.004	0.002	0.02	0.04	Rest

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

Test	Unit	Value
Density	g/cm ³	1.0269
Turbidity	NTU	745
PH		6.69
Salinity as NaCl	mg/l	31694
Conductivity@25°C	µs/cm	76.5
Total hardness	mg/l	2814
Sulphate	(mg/l)	910
Phosphate	(mg/l)	330
Carbonate	(mg/l)	Nil
Bi-carbonate	(mg/l)	540
Chloride	(mg/l)	19227
Iron	(mg/l)	2.2
Calcium	(mg/l)	810
Magnesium	(mg/l)	193
Barium	(mg/l)	Nile
Copper	(mg/l)	Nil
Potassium	(mg/l)	1500
Zinc	(mg/l)	0.35
T.D.S.	(mg/l)	37250

Table 3. Potentiodynamic polarization parameters obtained for carbon steel in oil wells formation water in the absence and presence of various concentrations of the inhibitor[I], inhibitor[II] at 50°C

Inhibitor	Conc., Ppm	-E _{corr} , mV vs. SCE	I _{corr} , µAcm ⁻²	Corrosion rate µm/Y	η%
I	0	1055.8	47.9	528.2	---
	35	965.3	26.3	290.1	45.1
	70	952.1	24.7	272.5	48.4
	100	962.2	19.1	210.3	60.2
	150	960.7	16.8	185.2	64.9
	175	965.4	15.4	169.8	67.8
II	0	1055.8	47.9	528.2	---
	35	955.4	18.4	202.9	61.5
	75	962.6	14.9	164.3	68.8
	100	964.2	12.7	140.0	71.2
	150	958.3	11.5	126.8	75.9

Table 4. Nyquist plots for carbon steel in oil wells formation water in the absence and presence of different concentrations inhibitor[I], inhibitor[II]at 50°C.

Inhibitor	Conc., ppm	Coefficient	R _f , Kohm.cm ²	C _f , μF/cm ²	R _t , kΩcm ²	C _{dl} , μFcm ⁻²	η %
I	Blank	0.99	---	---	.49	113.4	---
	35	0.98	0.37	43.7	1.45	87.3	35.1
	70	0.97	0.40	38.5	1.73	81.1	45.6
	100	0.98	0.47	32.1	2.26	66.1	58.4
	150	0.98	0.49	24.2	2.84	55.6	67.1
	175	0.96	0.52	22.9	3.32	34.8	71.6
II	35	0.97	0.39	45.1	1.95	44.1	51.7
	70	0.98	0.46	39.8	2.73	37.7	65.5
	100	0.98	0.53	33.7	3.14	36.1	70.1
	150	0.97	0.58	27.6	3.47	28.0	73.0
	175	0.95	0.63	25.4	3.98	14.2	76.3

Table 5. Efficiency of the two synthesized inhibitors (I &II) as biocides.

Inhibitor	Dose, ppm				
	35	75	100	150	175
SRB count for (I)	10 ⁵	10 ⁴	10 ³	10 ³	Nil
SRB count for (II)	10 ⁴	10 ³	Nil	Nil	Nil

Table 6. Surface active properties of the synthesized compounds I, II

Inhibitor	Temp.	CMC mole.dm ⁻³	γ _{cmc} mN.m ⁻¹	Γ _{max} × 10 ⁻⁷ , mol.m ⁻²	A _{min} nm ²	Π _{CMC}	ΔG _{mic} kJ. Mol ⁻¹	ΔG _{ads} kJ. Mol ⁻¹
I	25°C	2.75x10 ⁻⁴	34	1.3x10 ⁻¹⁰	126	38.3	-20.3	-23.2
II	25°C	3.7x10 ⁻⁵	33	8.25 x10 ⁻¹¹	201	39.3	-26.17	-30.8

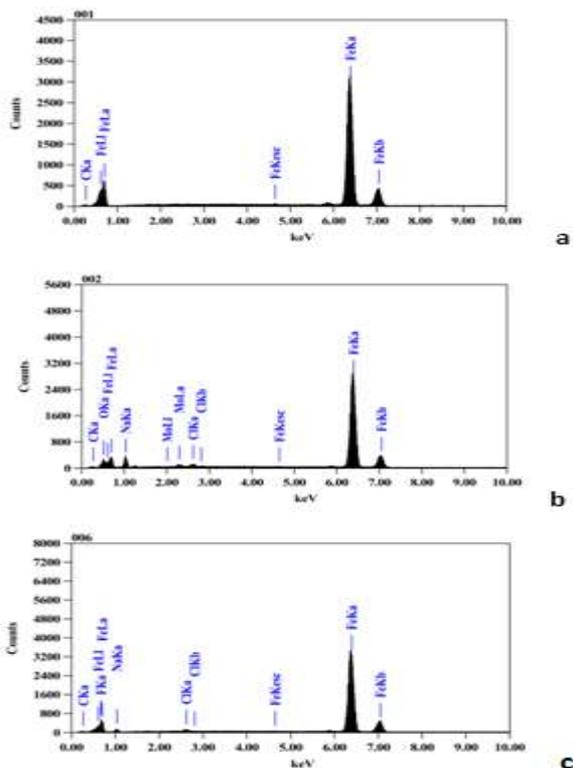


Fig. 9: EDX of the carbon steel surface: (a) polished sample, (b) after immersion in the formation water r and (c) after immersion in the formation water at 50°C under CO₂ environment in the presence of inhibitor (II)

The two studied surfactants were applied as biocides against sulfate –reducing bacteria, showed imposing results due to their relatively high efficiency against this type of bacteria [24]..the

tested compounds I,II have low efficiency at lowest concentrations (35,70 ppm by weight),In the relative high dosages (150,175 ppm by weight), they have high efficiency : the activity of the prepared compounds is dependent on the alkyl chain length which determines the compound solubility in the water , so that when the alkyl chain length increases, the solubility of the compounds decreases , with an increase in their activity .as shown in table 5,the most effective compound is compound II .the two compounds have Biocidal effect at dosage of 150 ppm by weight . This test has been conducted according to ASTM D4412-84(re-approved 1990). The tested water has been subjected to growth of about 10⁷ bacterial cell/ml. Two samples (I & II) were tested as biocides by doses of 35, 75, 100, 150,175 ppm and the system was incubated to contact time 3.0 hours, each system was cultured in SRB specific media. The results are shown in **Table (5)**.

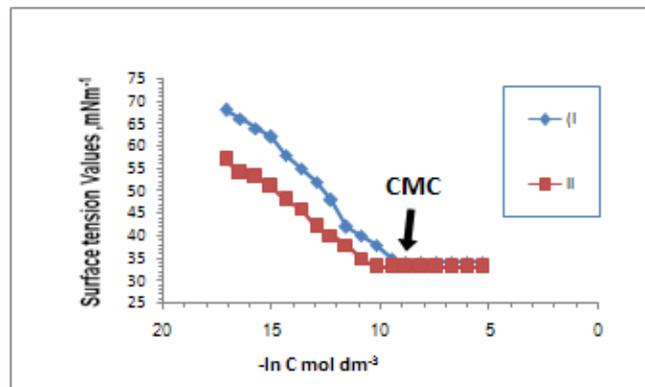


Fig 10. Surface tension vs. log C of compounds(I&II). Surface tension measurements

The CMC values of the synthesized surfactants were determined at various temperatures from the change in the slope

of the plotted data of surface tension (γ) versus the natural logarithm of the solute molar concentration; In C, as shown in **Fig. 10**. The data obtained from surface tension are summarized and listed in **table 6**.

Conclusion:

(1) The two investigated cationic surfactants used in this study can be used as inhibitors for corrosion of carbon steel in oil well formation water under CO₂ at 50 °C, but inhibitor II is more effective.

(2) The values of the percentage inhibition efficiency obtained from potentiodynamic polarization and electrochemical impedance spectroscopy are in good agreement.

(3) The potentiodynamic polarization curves indicated that the inhibitor molecules inhibit both anodic metal dissolution and also cathodic oxygen reduction, so that the undertaken surfactants classified as mixed – type inhibitors.

(4) 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.

(5) The formation of a good protective film on carbon steel surface was confirmed using SEM and EDX techniques.

(6) The two prepared cationic surfactants can be act as biocide in addition to its effect as corrosion inhibitor.

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