

## Biocidal Activity and Corrosion Inhibition of Some Cationic Surfactants Derived from Thiol Polyurethane

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SIX series of cationic polyurethane surfactants [PQ10-20] were synthesized by the reaction between different six esters (which prepared by condensation of fatty alcohols namely: octanol, decanol, dodecanol, tetradecanol, hexadecanol and octadecanol with bromoacetic acid) and polyurethane as quaternizing agent. The chemical structures of these surfactants were confirmed using elemental analysis, FTIR spectra, <sup>1</sup>HNMR and UV analysis. The surface activities of the synthesized cationic thiol polyurethane surfactants showed their tendency towards adsorption at the air/water interface. The adsorption tendency was estimated from the values of surface tension and the depression of surface tension at the critical micelle concentration. The studied surfactants were evaluated as antimicrobial agents against pathogenic and sulfur reducing bacteria using inhibition zone diameters and minimum inhibition concentration values. The synthesized cationic thiol polyurethane surfactants showed good antimicrobial activities against the tested microorganisms including Gram positive, Gram negative as well as fungi. The synthesized compounds were tested for the activity as corrosion inhibitors against carbon steel corrosion in 2N H<sub>2</sub>SO<sub>4</sub> at 50, 100, 200, 400 and 600 ppm. The inhibition efficiencies of the tested compounds showed good inhibition and protection of the carbon steel. The corrosion inhibition tendency correlated to the surface activity and chemical structure of the compounds.

**Keywords:** Cationic surfactants, Surface activity, Adsorption, Antimicrobial activity, Corrosion inhibition, Polyurethane.

### Introduction

Corrosion inhibitors are added to prevent metal dissolution during its surface cleaning from oxides and other adherent materials [1]. Also, corrosion inhibitors may be liquids or powder form that effectively reduces the corrosion rate by adsorbing on the metal surface [2, 3]. Corrosion inhibition efficiency of organic compounds is related to their adsorption properties [4]. Corrosion protection of steel in acidic media is of great importance for both industrial facilities and theoretical aspects [5]. The adsorption of these molecules depends mainly on certain physicochemical properties of the inhibitor molecule such as the presence of heteroatoms including: oxygen, sulfur, nitrogen atoms and multiple bonds in the molecule through

which they are adsorbed on the metal surface [6–10].

Heteroatoms such as nitrogen, oxygen, and sulfur are capable of forming coordinate covalent bond with metal owing to their free electron pairs and thus acting as inhibitor. Compounds with  $\pi$  bonds generally exhibit good inhibitive properties due to the interaction of  $\pi$  orbital with metal surface [11-13]. The selection of a suitable inhibitor for a particular system is a difficult task because of the selectivity of the inhibitors and wide variety of corrosive environment. As a result, several types of corrosion inhibitors were developed to fit the different types of corrosion processes and also the medium where the corrosion takes place. Corrosion inhibition of carbon steel in sulfuric

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acid was studied using some cationic surfactants derived from Thiol polyurethane with different molecular structures [14–16].

On the other hand, to overcome the alarming problem of microbial resistance to antibiotics, the discovery of novel active compounds against new targets is a matter of urgency. Many of the crude drugs, which are sources of medicinal preparations, still originate from wild growing material. However, plant-based drugs have shortened the life span of the source of material. There is a continuous search for more potent and cheaper raw materials to feed the industry. Cationic surfactants are organic compounds with great utility in important fields such as medicine, agriculture and cosmetic products [17]. Some cationic surfactants present anticancer, antitumor and antibacterial activity [18].

In this study, six new cationic Thiol polyurethane surfactants were synthesized, characterized and evaluated as corrosion inhibitors against the corrosion of carbon steel in acidic media and also as antibacterial agents to prevent bacterial growth. The relation between the surface activity and the efficiency of these compounds in the different applications was discussed.

## **Materials and Methods**

### *Chemicals*

Fatty alcohols (octanol, decanol, dodecanol, tetradecanol, hexadecanol and octadecanol) were purchased from Sigma (99 %), toluene diisocyanate TDI were purchased from (DOW, 97 %), Triethanol amine (ADWIC, 99 %), Mercapto acetic acid were purchased from (Aldrich, 98 %), Bromoacetic acid were purchased from (Aldrich, 98%), ethyl alcohol (ADWIC, 99 %) and sulfuric acid (ADWIC, 99 %).

### *Instrumentation*

Elemental analysis was done using Vario Elementar Analyzer; IR spectroscopy was done using Perkin Elmer FTIR System (Genesis Fourier Transformer FTIRTM) in the 4,000–400 cm<sup>-1</sup> region using KBr disks; <sup>1</sup>H-NMR spectroscopy was done using Varian NMR-300, Mercury 300 MHz spectrometers in CDCl<sub>3</sub> solvent and TMS as internal standard and the chemical shifts reported in δ (ppm); Ultra violet was done using Perkin-Elmer S52 spectrophotometer; Surface and interfacial tension was done using K6-Kru'ss Tensiometer with platinum ring and weights.

### *Synthesis*

#### *Preparation of Triethanol amine mono Mercapto acetate*

Triethanol amine (0.1 mole) reacts with mercaptoacetic acid (0.1 mole) to form the targeted compound. The solvent used was xylene due to its high boiling point to ensure the completion of the reaction. The reaction procedure involves the addition of the reaction mixtures and suitable dehydrating agent (p-toluene sulfonic acid, 0.1 g) in three necked flasks connected with efficient condenser, dean stark joint and stirrer. The completion of the reaction is indicated as the equivalent amount of water of reaction is trapped in the dean stark connection. The interaction takes about 12 hours. After removal of the solvent under vacuum using rotary evaporator, the catalyst was then removed from the reaction product by extracting them using petroleum ether. Subsequent purification was done by means of vacuum distillation to remove the excess, unreacted and residual materials to afford Triethanol amine mono Mercapto acetate, [19] yield 96%.

#### *Preparation of the targeted Thiol polyurethane compound*

The polymerization was carried out in a four-necked round bottom flask equipped with a mechanical stirrer, and dropping funnel. An excess amount of TDI (0.1 mole) dissolved in acetone; (100 ml) was added to the four-neck flask and stirred. TEA<sub>MER</sub> (0.2 mole) and five drops of triethylenediamine (TEDA) were dissolved in 40 ml of acetone solution and was added slowly during 20 min to the TDI solution [20]. The reaction mixture was then stirred at 30 °C for 30 min to form the pre-polymer. The NCO/OH ratio was kept 1:2 in polyurethane reaction.

#### *Preparation of the fatty alcohol ester of Bromoacetic acid*

Bromoacetic Acid (0.1 mol.) and octanol, decanol, dodecanol, tetradecanol, hexadecanol and octadecanol (0.1 mol.) were esterified respectively in xylene in presence of 0.01% p-toluene sulphonic acid as catalyst, the mixture was refluxed until the azeotropic amount of water (1.8 mL) was removed. The interaction takes about 8-16 hours according to the hydrophobic chain length (fatty alcohol). After removal of the solvent under vacuum using rotary evaporator, the catalyst was then removed from the reaction product by extracting them using petroleum ether. Subsequent purification was done by means of vacuum distillation to remove the excess and

unreacted and residual materials [21] to afford the different fatty alcohol esters of Bromoacetic acid. The yield of the products, octyl bromoacetate (C10), decyl bromoacetate (C12), dodecyl bromoacetate (C14), tetradecyl bromoacetate (C16), hexadecyl bromoacetate (C18) and octadecyl bromoacetate (C20) esters were 95-98%.

#### *Preparation of cationic Thiol polyurethane surfactants (PQ)*

A solution of Thiol polyurethane compound contained tertiary amine and octyl, decyl, dodecyl, tetradecyl, hexadecyl, octadecyl esters were mixed. The mixtures were refluxed in presence of dimethyl formamide (DMF) for a period ranging from 1 day to 5 days according to hydrophobic chain length. Then left to stand overnight and then filtered. The product was recrystallized three times from ethanol and dried in vacuum oven at 40°C to afford a brown viscous liquid (PQ10, PQ12, PQ14, PQ16, PQ18 and PQ20) yield 82-89%. [22, 23] (Scheme 1)

#### *Chemical structure*

##### *Elemental analysis*

The elemental analysis of the synthesized cationic Thiol polyurethane surfactants showed that the expected and found values of the different elements are very close to each other, indicating the purity of the synthesized compound. (Table 1)

##### *FTIR Spectroscopy*

The chemical structure of Triethanol amine mono Mercapto acetate was confirmed using FTIR as follows:-

Absorption band at 3435  $\text{cm}^{-1}$  corresponding to OH stretching group, very weak band at 2550  $\text{cm}^{-1}$  corresponding to Thiol S-H group, absorption band at 2925  $\text{cm}^{-1}$  corresponding to symmetric stretching of C-H group, band at 1018  $\text{cm}^{-1}$  corresponding to C-N stretching group of aliphatic amine and absorption band at 1732  $\text{cm}^{-1}$  corresponding to C=O group.

The chemical structure of the targeted Thiol polyurethane compound confirmed using FTIR as follow:

Absorption band at (1660 – 1663)  $\text{cm}^{-1}$  corresponding to C=O in urethane group, absorption band at (1458 – 1462)  $\text{cm}^{-1}$  corresponding to N-H binding in urethane group, (1508 – 1510)  $\text{cm}^{-1}$  corresponding to C=C of aromatic ring of toluene diisocyanate moiety.

The chemical structure of fatty alcohol esters confirmed using FTIR as follow:

The IR spectra of these esters showed complete disappearance of the absorption band at 3400  $\text{cm}^{-1}$  which characterizes the aliphatic hydroxyl groups of fatty alcohols and acids and the appearance of a new absorption band at (1736-

1738)  $\text{cm}^{-1}$  indicates the formation of the carbonyl of ester group. Absorption band at (1275-1277)  $\text{cm}^{-1}$  corresponding to ether linkages -C-O-, absorption band at (2920-2922)  $\text{cm}^{-1}$  and (2849-2850)  $\text{cm}^{-1}$  corresponding to methyl (CH<sub>3</sub>) and methylene (CH<sub>2</sub>) groups respectively. On the other hand, the characteristic band of C-Br bond was appeared in the range of 663-667  $\text{cm}^{-1}$ .

The chemical structure of cationic Thiol polyurethane surfactants confirmed by using FTIR as follow

All bands which appear in FTIR spectra of fatty alcohol bromo esters and Thiol polyurethane appeared in FTIR spectra of cationic polyurethane surfactants. Two specific bands appeared at 1460  $\text{cm}^{-1}$  and 2960  $\text{cm}^{-1}$  corresponding to vibration and elongation of [N<sup>+</sup>] group.

##### *<sup>1</sup>H NMR as follows:*

The <sup>1</sup>H NMR spectra of the synthesized cationic surfactants (representatively for PQ10 & PQ14) showed the appearance of signals at:-

$\delta$  = 0.85 ppm assigned to terminal methyl group of fatty alcohol (CH<sub>3</sub>).

<sup>1</sup>H NMR also showed an increase in the integration of the signal at 1.25 ppm, which is attributed to the presence of the methylene groups of the fatty alcohols moiety (CH<sub>2</sub>)<sub>n</sub>.

$\delta$  = 2.5 ppm assigned to methyl group on aromatic rings (toluene diisocyanate).

Three intense signals centered at  $\delta$  = 7.8 ppm, 8.0 ppm and 8.2 ppm attributed to the aromatic proton.

##### *UV spectra as follow:-*

UV spectra were recorded with a Perkin-Elmer S52 spectrophotometer. The cationic thiol polyurethane surfactants were confirmed by the appearance of new bands in UV spectra (Table 2).

#### *Measurements*

##### *Surface Tension measurements ( $\gamma$ )*

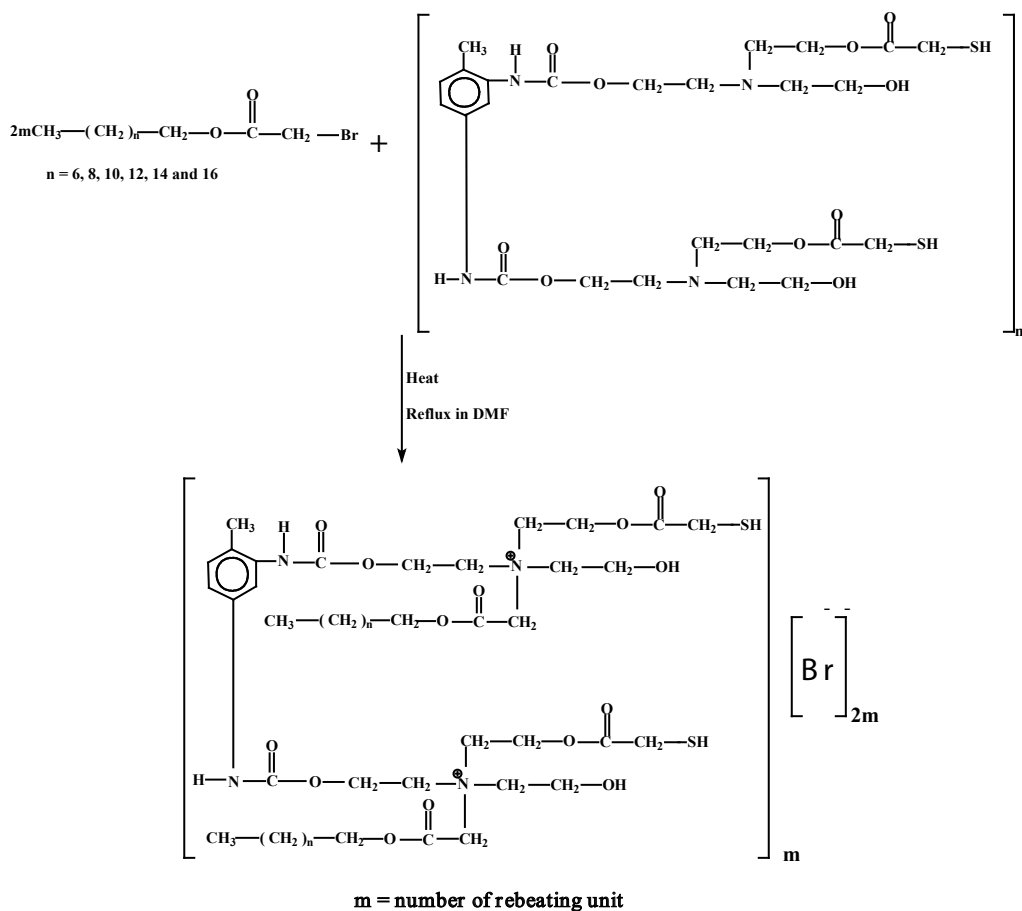
The surface tension of the prepared surfactants was measured as a function of concentration at 25 °C. The CMC and surface tension at CMC were determined. [24].

#### *Antimicrobial activity measurements*

##### *Microorganisms*

The biocidal activity of the synthesized surfactants was tested against different bacterial strains (ATCC: American Type Culture Collection) as follows: Staphylococcus aureus ATCC 29213, Escherichia coli ATCC 25922, Pseudomonas aeruginosa ATCC 27853, Bacillus subtilis ATCC 55422, Desulfomonas pigra ATCC 29098 and Staphylococcus typhimurium ATCC 27948.

The Sulfur Reducing Bacteria (SRB) are a  
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Scheme 1. Cationic Thiol polyurethane Surfactants.

TABLE 1. Elemental analysis of the synthesized cationic Thiol polyurethane surfactants.

	M.wt	C.F.	C%		H%		N%		S%		Br%	
			Calc.	Found	Calc.	Found	Calc.	Found	Calc.	Found	Calc.	Found
PQ <sub>10</sub>	20500 g/ mole	(C <sub>45</sub> H <sub>78</sub> O <sub>14</sub> N <sub>4</sub> S <sub>2</sub> Br <sub>2</sub> ) <sub>n</sub>	46.47	46.42	6.65	6.66	5.56	5.53	6.35	6.30	15.89	15.85
PQ <sub>12</sub>	21400 g/ mole	(C <sub>49</sub> H <sub>86</sub> O <sub>14</sub> N <sub>4</sub> S <sub>2</sub> Br <sub>2</sub> ) <sub>n</sub>	48.54	48.50	7.05	7.06	5.27	5.25	6.02	5.98	15.05	15.01
PQ <sub>14</sub>	22300 g/ mole	(C <sub>53</sub> H <sub>94</sub> O <sub>14</sub> N <sub>4</sub> S <sub>2</sub> Br <sub>2</sub> ) <sub>n</sub>	50.36	50.31	7.41	7.43	5.00	4.97	5.71	5.68	14.28	14.25
PQ <sub>16</sub>	23200 g/ mole	(C <sub>57</sub> H <sub>102</sub> O <sub>14</sub> N <sub>4</sub> S <sub>2</sub> Br <sub>2</sub> ) <sub>n</sub>	52.04	52.00	7.74	7.76	4.76	4.73	5.44	5.41	13.60	13.56
PQ <sub>18</sub>	24100 g/ mole	(C <sub>61</sub> H <sub>110</sub> O <sub>14</sub> N <sub>4</sub> S <sub>2</sub> Br <sub>2</sub> ) <sub>n</sub>	53.57	53.54	8.03	8.05	4.54	4.51	5.19	5.17	12.99	12.96
PQ <sub>20</sub>	25000 g/ mole	(C <sub>65</sub> H <sub>118</sub> O <sub>14</sub> N <sub>4</sub> S <sub>2</sub> Br <sub>2</sub> ) <sub>n</sub>	54.97	54.93	8.31	8.29	4.35	4.32	4.97	4.95	12.44	12.39

TABLE 2. UV adsorption maxima of Cationic Thiol polyurethane surfactants.

Compound	$\lambda_{\max}$ (nm)
PQ <sub>10</sub>	208
PQ <sub>12</sub>	212
PQ <sub>14</sub>	210
PQ <sub>16</sub>	234
PQ <sub>18</sub>	216
PQ <sub>20</sub>	214

common problem in the oilfield systems that should be protected. On the other hand, the selected species cause dangerous diseases for the humans and animals.

#### *Growing of microorganisms*

The bacterial strains were cultured according to the standards of the National Committee for Clinical Laboratory (NCCLS) [25]. The bacterial species grow on nutrient agar. The nutrient agar medium consisted of Beef extract (3.0 g/L), Peptone (5.0 g/L), Sodium chloride (5.0 g/L), Agar (20.0 g/L); then the volume was completed to one liter distilled water, the mixture heated until boiling and the media sterilized by autoclave. The bacterial strains were kept on nutrient agar medium and showed no inhibition zones.

#### *Measurements of resistance and susceptibility*

For preparation of discs and inoculation, 1.0 mL of inocula were added to 50mL of agar media (40 °C) and mixed. The agar was poured into 120 mm petri dishes and allowed to cool to room temperature. Wells (6mm in diameter) were cut in the agar plates using proper sterile tubes and filled up to the surface of agar with 0.1mL of the synthesized cationic Thiol polyurethane surfactants (PQ10-20) dissolved in DMF (5mg/mL DMF). The plates were left on a leveled surface, incubated for 24 h at 30 °C and then the diameters of the inhibition zones were read. The inhibition zone formed by these compounds against the particular test bacterial strain determined qualitatively the antibacterial activities of the synthetic compounds. The mean value obtained for three individual replicates was used to calculate the zone of growth inhibition of each sample. The antimicrobial activity was calculated as a mean of three replicates. The tested

compounds were completely compatible with the medium of agar and no turbidity was observed during the mixing process [25, 26].

#### *Weight loss measurements*

The weight loss experiments were performed with carbon steel specimens having a composition of (wt %): 0.21 C, 0.035 Si, 0.025 Mn, 0.082 P and the remainder is Fe. The carbon steel sheets of 2.5 cm \* 2.0 cm \* 0.6 cm were abraded with a series of emery papers (grade 320, 500, 800 and 1200) and then washed with acetone and distilled water. After weighing accurately, the specimens were immersed in 250 mL solution of 2N H<sub>2</sub>SO<sub>4</sub> without and with the tested inhibitors (PQ10-20) at different concentrations (50, 100, 200, 400 and 600 ppm) for 14 days. Then, the specimens were taken out, washed, dried and weighed accurately [27]. The test was performed for three specimens and the weight was the average of the three specimens. The coupons were polished by hard plastic brush, hot water then by ethanol to remove the corrosion products. The corrosion rate (R) and the inhibition efficiency (IE %) were calculated using Eqs. (1 and 2) [28]:

$$R = W / At \quad 1$$

$$IE \% = [(R_0 - R) / R_0]100 \quad 2$$

where W is the average weight loss of three parallel carbon steel sheets, A is the total area of the specimen, t is immersion time, R<sub>0</sub> and R are the values of the corrosion rate without and with addition of the inhibitor, respectively [29, 30].

## **Results and Discussion**

### *Surface activity*

Surface tension is a characteristic property of liquids. This phenomenon appeared originally from the attraction between the molecules at the surface.

The surface tension value of the bidistilled water at 25°C was found to be 71.8 mN/m which is attributed to the attraction forces between water molecules at the water surface due to the hydrogen bonds. If there are any foreign molecules present at the water surface, this results in disturbing the tension leading to a decrease to lower values. Meanwhile surfactant molecules tend to be adsorbed at the air–water interface at lower concentrations. Hence, by increasing the surfactant concentration, the surface tension of the resulted solution generally decreased. Figures 1&2 show the relation between the surface tension values in (mN/m) and the concentration (Log C) at 25°C. These curves are characterized by two regions, one at lower concentration which is very sensitive towards the concentration reaction; the other surface tension curves are almost constant with variation of the surfactant concentration. Extrapolation of these two regions determines the so-called critical micelle concentration CMC. [31].

The critical micelle concentration is defined as the concentration of the surfactant at which no further decrease in the surface tension could be obtained upon addition of any further amounts of surfactant in the solution. There is an equilibrium between the singly adsorbed surfactant molecules at the interface and the micellized surfactant molecules. That equilibrium occurred at the concentration of complete surface tension (CMC) [32, 33]. The micelle formation is the most vital point of view in the surfactant fundamental, because it is the most effective geometrical arrangement of the molecules at the desired concentrations of the synthesized surfactants given from their surface tension isotherms as listed in (Table 3).

Obviously, the CMC values are dependent on the chemical structure of the surfactant molecules [34]. The different CMC values of this surfactant depend on the hydrophobic moieties in its structure. Increasing the hydrophobic chain length, the number of repeated methylene groups (CH<sub>2</sub>) decreases the CMC values. That effect could be explained by the view of the hydrophobicity of the surfactant molecules. Increasing the hydrophobic chain length of the surfactant molecules increases their nonpolar characters, which result in increasing the repulsion forces between them and the polar water molecules (thermodynamically unstable). Hence, the molecules tend to reduce this unfavorable situation by forming more stable aggregative structures which are micelles (thermodynamically stable). Therefore the CMC was at a lower concentration [35].

#### *Antimicrobial activity*

In this work we aim to evaluate the prepared cationic surfactants as biocide against some pathogenic Gram-positive bacteria (*Bacillus subtilis*, *Staphylococcus aureus*), Gram-negative bacteria (*Salmonella typhimurium* and *Escherichia coli*) and some pathogenic fungi (*Candida albicans* and *Aspergillus niger*).

The results of antimicrobial activity are recorded in Table 4, indicating that the synthesized compounds have antimicrobial activity range from a moderate to high effect on Gram negative bacteria, Gram positive bacteria, yeast and weak effect on fungi compared to the drug reference used. Where the inhibition zone for *Bacillus subtilis* ranges from 15–27 mm/mg compared to 26 mm/mg for Chloramphenicol drug, for *Staphylococcus aureus*, it ranges from 0–22 mm/mg compared to 25 mm/mg for Chloramphenicol drug, for *Salmonella typhimurium* ranges from 0–15 mm/mg compared to 28 mm/mg for Cephalothin drug, for *Escherichia coli* ranges from 10–26 mm/mg compared to 27 mm/mg for Cephalothin drug, for *Candida albicans* ranges from 17–27 mm/mg compared to 28 mm/mg for cycloheximide drug, for *Penicillium chrysogenum* ranges from 0–13 mm/mg compared to 26 mm/mg for cycloheximide drug.

The biological activities of surfactants often show a non-linear dependence on their chain length, where bactericide and fungicide activities increase by increasing hydrophobic chain length.

Data in Table 4 reveal that the synthesized surfactants have antimicrobial activities, and the difference in their activities depends on the length of hydrophobic chains of prepared surfactants. The optimal length of alkyl chain has been noted to be 10 carbon atoms, which exhibit the maximum inhibition zone (PQ10). These results are in agreement with results obtained previously, which indicates that the optimal activity toward a variety of bacterial species for numerous structural variations of water soluble surfactants occurs with an alkyl chain between 10 and 14 carbon atoms. These results are in agreement with those reported elsewhere [36–39].

The biological activities of the synthesized cationic Thiol polyurethane surfactants were generally decreased by increasing the hydrophobic chain length.

This could be referred to the adsorption ability of the surfactants containing long chains

**TABLE 3.** Surface properties of the synthesized cationic Thiol polyurethane surfactants in bidistilled water at 25°C.

Compound	CMC, Mole/L (x10 <sup>-6</sup> )	$\gamma_{cmc}$ , mN/m	$\pi_{cmc}$ , mN/m	$T_{max}^2 \times 10^{-11}$ Mol.K <sup>-1</sup> cm <sup>-1</sup>	$A_{min}^2$ , nm <sup>2</sup>	$Pc_{20}^2$ , Mole/L (x 10 <sup>-6</sup> )
PQ <sub>10</sub>	12.6	40	31.8	10.8	15.2	1.58
PQ <sub>12</sub>	10	35	36.8	10.2	16.2	15.8
PQ <sub>14</sub>	3.1	35	36.8	7.9	20.9	158
PQ <sub>16</sub>	12.5	37	34.8	18.5	8.9	11.2
PQ <sub>18</sub>	7.9	40.5	31.3	16.3	9.9	11.2
PQ <sub>20</sub>	6.3	43	28.8	15.2	10.8	8.9

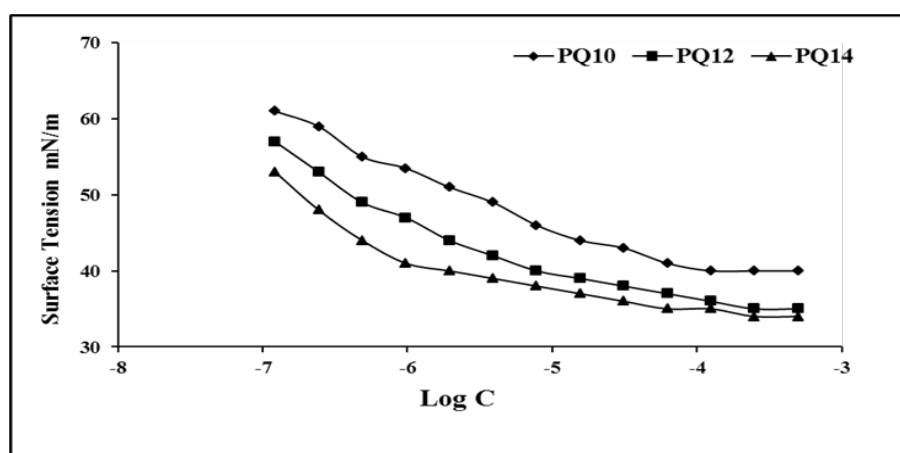
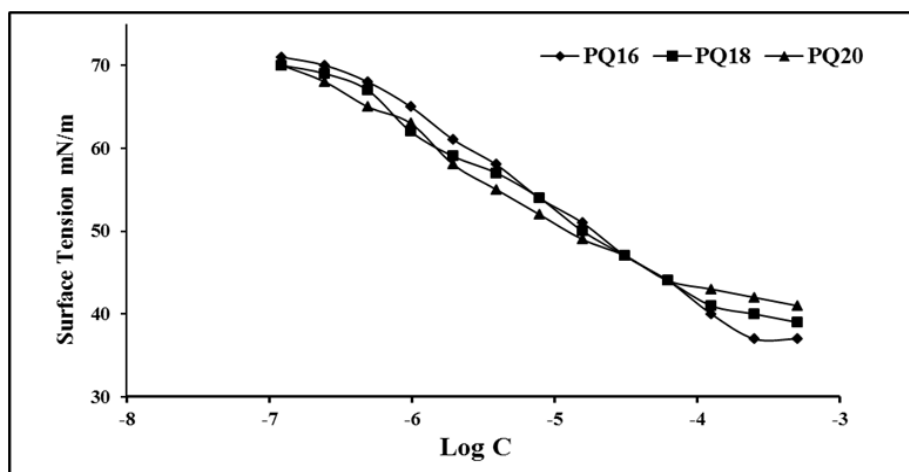

**Fig. 1.** Surface tension vs. log concentration profile of the synthesized cationic Thiol polyurethane surfactants (◆ PQ10, ■ PQ12, ▲ PQ14) at 25 °C.

**Fig. 2.** Surface tension vs. log concentration profile of the synthesized cationic Thiol polyurethane surfactants(◆ PQ16, ■ PQ18, ▲ PQ20) at 25 °C.

TABLE 4. Antimicrobial activity of synthesized surfactants against pathogenic bacteria, yeast and fungi.

Compounds	Bacteria				Yeast	Fungi
	Gram positive		Gram negative			
	Bacillus subtilis	Staphylococcus aureus	Salmonella typhimurium	Escherichia coli	Candida albicans	Aspergillus niger
Control	26	25	28	27	28	26
PQ <sub>10</sub>	27	22	15	26	26	0
PQ <sub>12</sub>	17	20	13	16	25	12
PQ <sub>14</sub>	15	16	10	14	24	13
PQ <sub>16</sub>	23	14	0	10	17	0
PQ <sub>18</sub>	25	9	0	23	19	0
PQ <sub>20</sub>	22	0	0	21	27	0

at the interfaces. Increasing the hydrocarbon content in the surfactant molecules increases their hydrophobicity; hence the molecules will tend to form micelles, which will decrease their biological activities more than the more adsorbed surfactant molecules (with shorter hydrocarbon chain lengths).

It is believed from recent studies on cationic surfactants that they have an excellent bactericidal activity [40], which depends on the hydrophobic chain length, activity and the dosage of this cationic. Table 4 revealed that the hydrophobic chain length of the cationic surfactants plays an important role in their biological activities against the targeted microorganisms. Several researchers have dealt with the mode of action of these types of compounds on different microorganisms [41]. The most likely explanation is through the surface adsorption mechanism, i.e., the adsorption of quaternary ammonium salt molecules on the outer cellular membrane of the microorganisms due to their amphipathic characteristics. In addition, the similarity between the hydrophobic chains and the lipid layers and the building units of the cell membranes and the monosaccharide in these compounds [42] facilitates that adsorption. The adsorption of these molecules at the cellular membranes reaches its maximum extent at a higher dose, leading to a complete coverage of the membrane by the surfactant molecules, which are able to penetrate it. Furthermore, the positive charges in the cationic molecules neutralize the negative charges on the bacterial cell membranes.

Accordingly, the selective permeability which characterizes the outer cellular membrane is completely lost [43]. Hence, the vital transportation of essential components for cell bioreactions and activities is disturbed. This causes the death of these microorganisms [44]. The high adsorption tendency of these cationics increases their penetration through the cellular membrane, with a boost in activity.

#### *Biocidal activity against sulfate-reducing bacteria (SRB)*

The synthesized cationic Thiol polyurethane surfactants were evaluated for their biocidal action against sulfate-reducing bacterial (*Desulfomonas pigra*) (Table 5). Comparing the inhibition zone diameters of the tested Gram-positive, Gram-negative and *D. pigra* (Gram-negative) regarding the antibacterial activities of the synthesized biocides showed that their efficacies against sulfate reducing bacteria were found at considerably high values. However, these values are still lower than those of the other bacterial strains tested. This may be ascribed to the resistivity of the SRB bacterial strain to the aggressive environmental components due to the rigidity of their cellular membranes. The sulfate-reducing bacteria (as a potent type of Gram-negative bacteria) are characterized by a thick cellular membrane, so that these microorganisms have high resistance against bacterial biocides. Consequently, the creation of efficient antimicrobial agents against the Gram-negative bacteria is considered a promising step in the biocidal field [45].



**TABLE 5. Antimicrobial activity of the cationic Thiol polyurethane surfactants against sulfate reducing bacteria.**

Compounds	Desulfomonas pigra (SRB) inhibition zone diameter (mm/mg sample)
<b>Standard: tetracycline antibacterial agent</b>	<b>25</b>
PQ <sub>10</sub>	9
PQ <sub>12</sub>	12
PQ <sub>14</sub>	14
PQ <sub>16</sub>	11
PQ <sub>18</sub>	13
PQ <sub>20</sub>	15

*Corrosion inhibition efficiency*

The inhibiting efficiencies (IE %) of the synthesized cationic surfactants towards the corrosion of carbon steel in 2N H<sub>2</sub>SO<sub>4</sub> were measured at 25°C using the weight loss technique at different concentrations of the surfactants (50, 100, 200, 400 and 600 ppm).

Inhibition efficiency of the cationic Thiol polyurethane surfactants has higher values than the most other cationic surfactants reported elsewhere at inhibitor dose (600 ppm). Values of inhibition efficiency ranged between 92.12% - 97.73%. This is attributed to the presence of Thiol group and urethane group in the compounds which allow an adsorption on the metal surface. The adsorption of cationic Thiol polyurethane surfactants on the carbon steel surface in 2N H<sub>2</sub>SO<sub>4</sub> solution occurs through the active centers in the inhibitors molecule which contain electronegative of SH, lone pair of N, double bond (urethane group) and π electrons of aromatic ring which they can easily adsorb on the metal surface and then reduce the dissolution of carbon steel.

It has been observed that the adsorption of these inhibitors depends on the physicochemical properties of the functional groups and the electron density at the donor atom. The adsorption occurs due to the interaction of the lone pair and/or p-orbitals of inhibitor with d-orbitals of the metal surface atoms, leading to the formation of a corrosion protection film.

The inhibition efficiencies of the tested inhibitors depend on inhibitor dose and hydrophobic chain length.

*Inhibitor dose*

Values obtained from weight loss

measurements are summarized in Table 6 for the inhibitors (PQ10 – PQ20). It was found that, the gradual increase of the inhibitor dose (from 50 to 600 ppm) gradually increases the corresponding inhibition efficiency. This could be attributed to the adsorption of the inhibitor molecules onto the metal surface [46]. The adsorbed inhibitor molecules form a protective layer at the metal surface, which prevents the metal-aggressive medium contact. Increasing the inhibitor dose increases the amount of the adsorbed molecules at the metal surface [47]. Consequently, thick and compact adsorbed layer is formed, which increases the inhibition efficiency considerably. It is clear, as the dose reached 600 ppm; IE% reached a high value of 97.73% for PQ14 at 25°C, which represented excellent inhibition value.

Surface coverage values were calculated from the corrosion rate data according to Eq. (3):

$$\Theta = [(R_0 - R) / R_0] \quad 3$$

R<sub>0</sub> is the corrosion rate in the absence of inhibitors; R is the corrosion rate in the presence of inhibitors.

The correlation between the inhibitor dose and the percent of metal surface covered by the inhibitor molecules (Θ) is called the adsorption isotherm and provides information about the interaction between the inhibitor molecules and the metal surface [48]. The correlation between Θ and the inhibitor concentration in the corrosive medium was represented by Langmuir adsorption isotherm, which is numerically represented in Eq. (4) [49]:

$$\Theta / (1 - \Theta) = K_{ads} C \quad 4$$

where K<sub>ads</sub> is the equilibrium constant of the

**TABLE 6. Data obtained from weight loss measurements for carbon steel dissolution in 2N H<sub>2</sub>SO<sub>4</sub> solution in absence and presence of various inhibitor concentrations at 25°C.**

Inhibitor	Dose, (ppm)	Corrosion rate, (mg cm <sup>-2</sup> day <sup>-1</sup> )	Surface coverage, (θ)	Corrosion inhibition efficiency, (IE %)
2N H <sub>2</sub> SO <sub>4</sub> (Without inhibitor)	--	29.47	--	--
PQ10	50	13.09	0.49	49.46
	100	9.32	0.63	62.85
	200	5.43	0.78	77.64
	400	3.28	0.88	87.69
	600	2.07	0.92	92.12
PQ12	50	13.73	0.47	47.08
	100	9.77	0.60	60.37
	200	5.62	0.75	75.48
	400	2.57	0.89	89.20
	600	1.77	0.93	92.66
PQ14	50	3.69	0.81	81.10
	100	3.02	0.86	85.96
	200	1.28	0.94	94.06
	400	0.68	0.97	97.30
	600	0.64	0.98	97.73
PQ16	50	11.96	0.53	53.56
	100	9.62	0.62	61.99
	200	4.90	0.77	77.54
	400	2.68	0.87	86.93
	600	0.75	0.96	96.33
PQ18	50	12.98	0.48	47.73
	100	9.89	0.57	57.45
	200	6.34	0.70	70.30
	400	2.11	0.88	87.80
	600	1.21	0.94	94.17
PQ20	50	9.36	0.60	60.04
	100	7.24	0.69	68.79
	200	3.73	0.82	81.96
	400	3.62	0.83	83.04
	600	0.75	0.96	96.33

inhibitor adsorption process and  $C$  is the molar concentration of the inhibitor. This model suggests that the inhibitor molecules occupy only one site on the metal surface and there is no interaction between the other adsorbed molecules [48]. The plot between  $C/\theta$  and  $C$  gave a straight line (Fig.3), which suggests that the adsorption of the inhibitor molecules on the metal surface obeyed the Langmuir adsorption model [50]. The values of the linear regression coefficient ( $r$ ) slope and adsorption coefficients of the different inhibitors are listed in Table 7. The correlation coefficient values ( $r \geq 0.9978$ ) indicate that the inhibition of the carbon steel by cationic Thiol polyurethane derivatives (PQ10-PQ20) was attributed to the adsorption of the inhibitor molecules on the metal surface. The adsorption isotherm slopes are close to unity, which indicates that there is no interaction between the adsorbed inhibitor molecules at the metal interface.

The intercept of Langmuir adsorption isotherm ( $K_{ads}$ ) is related to the adsorption free energy of inhibitor molecules onto the metal surface,  $\Delta G_{ads}$ , according to Eq. (5) [47]:

$$\Delta G_{ads} = -RT \ln (55.5 K_{ads}) \quad 5$$

The value and sign of  $\Delta G_{ads}$  provide more information about the mechanism of the inhibitor molecules adsorption at the metal surface. The negative values of  $\Delta G_{ads}$  indicate that the inhibitor molecules adsorbed spontaneously onto the metal surface [46, 47]. On the other hand, the  $\Delta G_{ads}$  values describe the type of inhibitor molecule adsorption on the metal surface.

Generally, the magnitude of  $\Delta G_{ads}$  is around - 20 kJ/mol or lower are consistent with the electrostatic interaction between charged inhibitor molecules and the charged metal surface (physisorption); those around - 40 kJ/mol or higher involve charge sharing or transfer from the inhibitor molecules to the metal surface to form a coordinate type of bond (chemisorption) [51-55]. The  $\Delta G_{ads}$  values are ranged between -18.12 and -26.49 kJ/mol. The obtained values of  $\Delta G_{ads}$  indicate that the adsorption takes place mainly through the electrostatic interaction between charged inhibitor molecules and the charged metal surface (physisorption and chemisorptions) [56].

#### Hydrophobic chain length

Figure 4 represents the effect of the hydrophobic chain length of the synthesized inhibitors on their inhibition efficiencies towards corrosion of carbon steel in 2N  $H_2SO_4$  solution at 25°C. It is clear that the gradual increase in the hydrophobic chain length from 8-18 methylene group increases the inhibition efficiency of the cationic inhibitors. The longest hydrophobic chain length of PQ20 can inhibit the corrosion of carbon steel in 2N  $H_2SO_4$  at 96.3 in presence of 600 ppm. The surface parameters in Table 3 reveal that inhibitors containing longer hydrophobic chains exhibit more surface activity and display great ability towards adsorption at the interfaces. Increasing the adsorption tendency of the inhibitor molecules increases their accumulation onto the metal interface [47]. This is suggested from the values of the surface coverage at the metal surface, Table 7. Thereby, the inhibitor molecules form condense adsorbed layer on the metal surface,

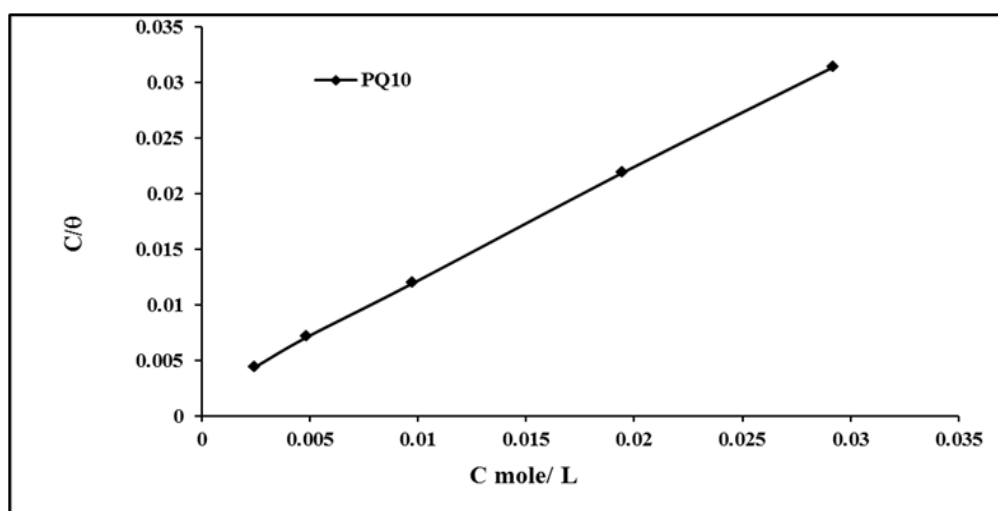


Fig. 3. Langmuir adsorption isotherm of PQ10 inhibitor in 2N  $H_2SO_4$  medium at 25 °C. Data are mean of three replicates with relative error  $\approx 5\%$ .

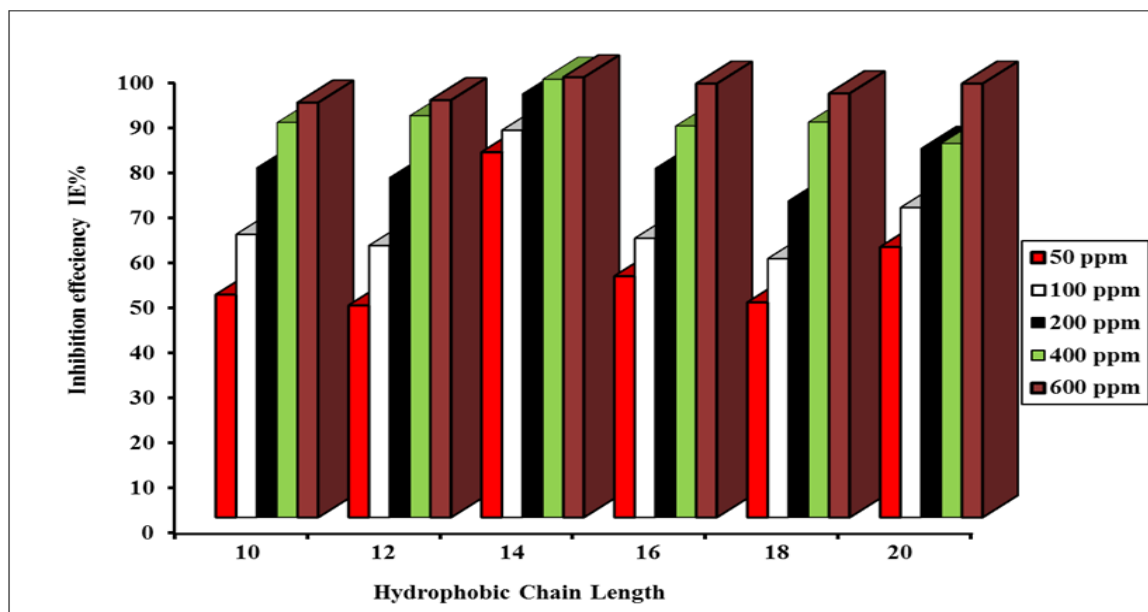


Fig. 4. Effect of the hydrophobic chain length on the inhibition efficiency (IE%) of PQ10-PQ20 inhibitors in 2N  $H_2SO_4$  at 25 °C.

Data are mean of three replicates with relative error  $\approx 5\%$ .

TABLE 7. Thermodynamic parameters for the adsorption of PQ10-PQ20 inhibitors on carbon steel surface in 2N  $H_2SO_4$  at 25 °C.

Inhibitor	Surface coverage $\Theta$	Slope	Linear regression coefficient	$K_{ads}$	$\Delta G_{ads}$ (kJ/mole)
PQ10	0.92	1.008152	0.9999	476.19	-25.22
PQ12	0.94	0.982213	0.9999	446.43	-25.07
PQ14	0.97	1.0071	0.9999	270.03	-18.12
PQ16	0.97	0.960824	0.9992	502.51	-25.36
PQ18	0.96	0.958644	0.9993	469.48	-25.19
PQ20	0.97	0.999035	0.9978	793.65	-26.49

The data with a relative error of  $\approx 5\%$ .  $\Theta$ : Degree of surface coverage by inhibitor molecules at 600 ppm,  $K_{ads}$ : equilibrium constant for the adsorption process of inhibitor molecules,  $\Delta G_{ads}$ : the adsorption free energy of inhibitor molecules onto the metal surface.

which prevents the reaction between the acidic medium and the metal; hence, the inhibition efficiency increases [57-58].

### Conclusions

The main conclusions of the study may be presented in the following points:

1. The synthesized surfactants showed good

surface activity including effectiveness and CMC and also good ability towards adsorption at the interface.

2. The antimicrobial activities of the compounds towards bacteria and fungi were (moderate-high) compared to the blanks used.
3. The compounds showed high inhibition

efficiency for the corrosion of carbon steel in 2N H<sub>2</sub>SO<sub>4</sub> at 25 °C at higher doses (400ppm and 600 ppm).

4. The adsorption of the synthesized cationic surfactant on the carbon steel surface represented a mixture of chemical and physical adsorption, it also obeyed Langmuir isotherm.
5. The antimicrobial activity and corrosion inhibition efficiencies depended on the chemical structure of the synthesized surfactants.

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## النشاط المضاد للحويية وتثبيط التآكل لبعض المواد ذات النشاط السطحي الكاتيونية المشتقة من البولي يوريثان ثيول

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تعتبر المواد ذات النشاط السطحي الكاتيونية من أهم المواد التي لها تطبيقات عديدة في المجالات الصناعية المختلفة. وبالإضافة الى أهميتها كمادة مكونة للمستحلبات والمحفزات فان لها استخدامات مختلفة كمادة مانعة لنمو البكتيريا والفطريات ومواد مانعة أو مثبطة للتآكل.

في هذا العمل:

تم تحضير ستة مواد ذات نشاط سطحي كاتيونية مشتقة من البولي يوريثان ثيول:

وذلك عن طريق تفاعل ستة استرات تم تحضيرها من تفاعل البرومو اسيتيك اسيد مع ستة كحولات دهنية مختلفة. ثم تحضير البولي يوريثان ثيول عن طريق تفاعل ثلاثي ايثانول امين احادي المركبتو اسيتات مع التولوين ثنائي الأيزو سيانات. ثم تحضير المواد ذات النشاط السطحي الكاتيونية المستهدفة عن طريق تفاعل البولي يوريثان ثيول مع استرات الكحولات الدهنية المختلفة.

تم اثبات التركيب الكيميائي للمواد المحضرة باستخدام التحاليل الكمية العنصرية وطيف الأشعة تحت الحمراء والرنين النووي المغناطيسي لذرات الهيدروجين وطيف الأشعة فوق البنفسجية.

تم تقييم المركبات التي تم تحضيرها كمبيدات حيوية ضد أنواع مختلفة من البكتيريا والفطريات وكذلك كمبيدات مانعة لنمو البكتيريا المختزلة للكبريتات في المواد البترولية. وكذلك تم تقييمها كمثبطات لتآكل الصلب الكربوني في الوسط الحامضي باستخدام طريقة الفقد في الوزن.