Preparation and estimation of some tetrahalometallate cationic surfactants modified by adding polyurethane as corrosion inhibitors

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ABSTRACT

A novel tetrahalometallate was created in this study by quaternizing nicotinic acid with 1-bromododecane as the fatty halide to make cationic surfactant. Grinding the obtained cationic surfactant with metal halide stannous chloride ($SnCl_2$) or ferric chloride ($FeCl_3$) and hydrazine sulphate yielded the tetrahalometallate. Proton nuclear magnetic resonance, UV, and Fourier-transform infrared spectroscopy were used to characterise the produced cationic surfactants and the tetrahalometallate cationic surfactant. Effectiveness (π_{CMC}), efficiency (Pc_{20}), maximum surface excess (Γ_{max}), minimum surface area (A_{min}), and critical micelle concentration (CMC) were estimated as surface parameters. The thermodynamic parameters of adsorption and micellization were evaluated, and the results revealed that both processes are spontaneous. It is clear that the produced tetrahalometallate surfactants have a high proclivity for adsorption at interfaces as well as micellization in the bulk of their solutions. The effect of adding polyurethane non-ionic surfactants on produced compounds and the evaluation of these cationic surfactants as corrosion inhibitors using the weight loss method were investigated for improved corrosion inhibitors and surface properties.

Keywords: Cationic surfactant; Tetrahalometallate; Nicotinic acid; Critical micelle concentration; Polyurethane

1. Introduction

Surfactant use is gradually increasing worldwide, and it is expected to reach US$ 28.8 billion by 2023 as a result of its intervention in a variety of industries [1]. Because of their corrosion resistance, low toxicity, stability, and antibacterial qualities, they are used in health care, cosmetics, and other sectors [2]. Surfactants are cationic, anionic, non-ionic, or amphoteric compounds that have both hydrophobic and hydrophilic parts [3]. It possesses the unusual ability of self-aggregation, which results in the formation of micelles and is thought to be an alternative to interfacial adsorption. Micellization results in a decrease in the system's free energy and an increase in entropy, both of which have a continuous mechanism [4].

Cationic surfactants are antibacterial, fabric softeners, and corrosion inhibitors because they have a positive charge on their hydrophilic side [5]. Quaternary pyridinium salts are cationic surfactants that are produced by quaternizing (pyridine) with an alkyl halide to produce quaternary ammonium salts [6,7].

Because cationic surfactants are positively charged and may be adsorbed on negatively charged surfaces, they can

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be utilised as corrosion inhibitors [8]. Tetrahalometallate compounds are made by combining cationic surfactants with metal chlorides like copper, nickel, or stannous chlorides, among others. There are several techniques to create these compounds, including grinding the cationic surfactant with any sort of metal chloride in a mortar, which results in a change in the colour of the mixture, indicating the development of tetrahalometallate complex. [9].

Polyurethanes, for example, are a type of surfactant made when diisocyanate reacts with a long-chain polyol. The endpoints of the prepolymer chain have reactive isocyanate groups, which are subsequently reacted with a diol or diamine chain extender to form the final product [10].

Nonionic polyurethanes are a form of polyurethane distinguishable from other types of polyurethane dispersions by their high resistance to acid and alkali hydrolysis. They are used to manufacture leather surfactants, dye dispersants, and fabric finishing agents [11].

When any of the physicochemical attributes is plotted against the concentration of surfactant molecules, the critical micelle concentration (CMC) is the lowest concentration at which surfactant molecules begin to combine [12,13]. The CMC value can be impacted by a variety of parameters, including structure, chemical nature of hydrophobic and hydrophilic portions, temperature, presence of electrolyte, solvent, nature of counter ions, pH, and others [14–16].

Corrosion occurs when materials deteriorate due to chemical reactions with their environment, such as oxygen and water. Corrosion can also apply to the breakdown of polymers, concrete, and wood, but it's most usually used to describe the deterioration of metals, with iron being the most common (usually as steel) [14]. There are two ways to keep iron from corroding: corrosion inhibitors or coatings. Corrosion inhibitors are a chemical component (made or naturally occurring compounds) that, when given in small quantities, inhibits or stops corrosion [15,16]. Corrosion inhibitors come in a variety of forms, with the surface active agent being one of the most important and effective organic chemicals [17,18].

In the present study we aimed to synthesize some tetrahalometallate cationic surfactant based on stannous and ferric chlorides by quaternizing nicotinic acid as a tertiary amine with 1-bromododecane as fatty halide. The product was grinded with the chosen metal chloride and hydrazine sulphate to produce the tetrahalometallate cationic surfactant. This different cationic surfactants were characterized using different characterization techniques and evaluation of this surfactant as corrosion inhibitors. This cationic surfactants were studied using several characterisation techniques, and its potential as a corrosion inhibitor was assessed. The effect of adding nonionic polyurethane

on corrosion inhibition and surfactants efficiency was investigated.

2. Experimental

2.1. Materials

Nicotinic acid (99%) and 1-bromododecane (98%) were supplied by Alfa Aesar, Germany. The metal chlorides (stannous chloride and ferric chloride) were supplied with high purity by BDH, Dubai, UAE. Hydrazine sulfate was supplied by Sigma-Aldrich, Germany. The used corrosion media is sulfuric acid (Conc. 98%), supplied by ADWIC, Egypt.

2.2. Analyses and description

Percentage of O, C, H, Br, and N were determined by micro elemental analysis for QC12. Vario Elementar Analyzer was used for micro elemental analysis, to detect the functional groups in QSnT and QFeT, Fourier-transform infrared spectroscopy (FTIR, Genesis Fourier transformer FTIR™) was used for infrared spectroscopy, Perkin-Elmer S52 spectrophotometer for ultra violet, and The chemical structure was determined by using ^{1}H NMR Varian NMR – 300 model, Mercury 300 MHz for proton nuclear magnetic resonance (^{1}H NMR) spectroscopy and the metals in this prepared surfactants were detected by using UV.

2.3. Preparation procedure of tetrahalometallate cationic surfactants

2.3.1. Quaternizing nicotinic acid

The quaternization of nicotinic acid by reacted 0.08 mol (10 g) with 0.08 mol (19.9 g) of 1-bromododecane were dissolved in acetone (50 mL) and reacted in 250 mL flask under reflux for 12 h. The cationic surfactant (QC12) was produced and washed numerous times with ethanol before being dried completely in a heated oven (Fig. 1) [19].

2.3.2. Addition of metal chloride

The prepared cationic surfactant 0.04 mol (14.9 g) QC12 was mixed and grinded in morter with 0.02 mol of stannous chloride $SnCl_2$ (3.8 g), to obtain quaternary stannous chloride QSn and by adding 0.04 mol (10.8 g) of ferric chloride ($FeCl_3 \cdot 6H_2O$) to obtain quaternary ferric chloride QFe (Fig. 2).

2.3.3. Preparation of tetrahalometallate

By grinding 0.015 mol (5.6 g) of QC12, 0.015 mol (2.8 g), 0.03 mol (8.1 g) of stannous chloride ($SnCl_2$) and
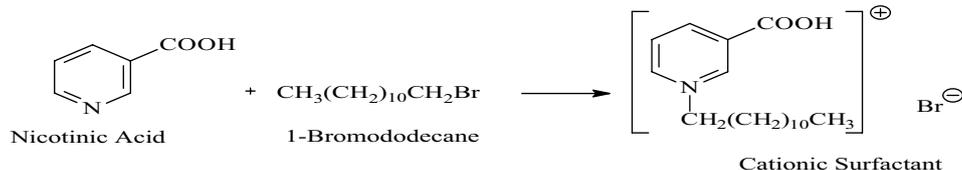
Fig. 1. Synthesis of the cationic surfactant by quaternization process.

FeCl$_3$.6H2O respectively and 0.015 mol (2 g) of hydrazine sulphate NH$_2$NHSO$_4$H in morter to obtain tetrahalometallate cationic surfactant named QSnT, QFeT (Fig. 3).

2.3.4. Non-ionic polyurethane preparation

The nonionic polyurethane surfactants were synthesized as early reported [20–22]. The synthesized nonionic polyurethane referred (PU) shown in Fig. 4.

2.3.5. Development of the synthesized tetrahalometallate

By grinding of the prepared tetrahalometallate cationic surfactant QSnT, QFeT (4 g) and 20% wt/wt of the

previously synthetic polyurethane (1 g) in mortar until colour changes of to get QSnT/PU and QFeT/PU.

2.4. Surface tension measurements

Surface tension measurements were obtained using De-Nöuy Tensiometer Krüss K-6 with a platinum ring. 10^{-1} mol/L Newly aqueous solutions of the synthesized surfactant QC12, QSn, QFe, QSnT and QFeT (3.72, 8.79, 9.82, 6.37, 7.4 g in 100 of distilled water respectively) and 10^{-3} mol/L of QSnT/PU and QFeT/PU (4.1g, 4.06 g in 100 mL of distilled water correspondingly) were synthesized and varied concentration variety of (10^{-1} to 10^{-7} mol/L) at 25°C. Seeming surface tensions were estimated about
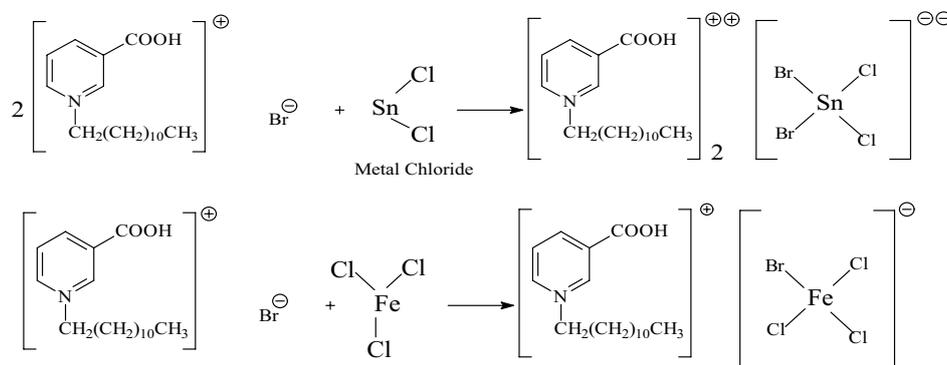
Fig. 2. Synthesized quaternary metal chloride (QSn and QFe).
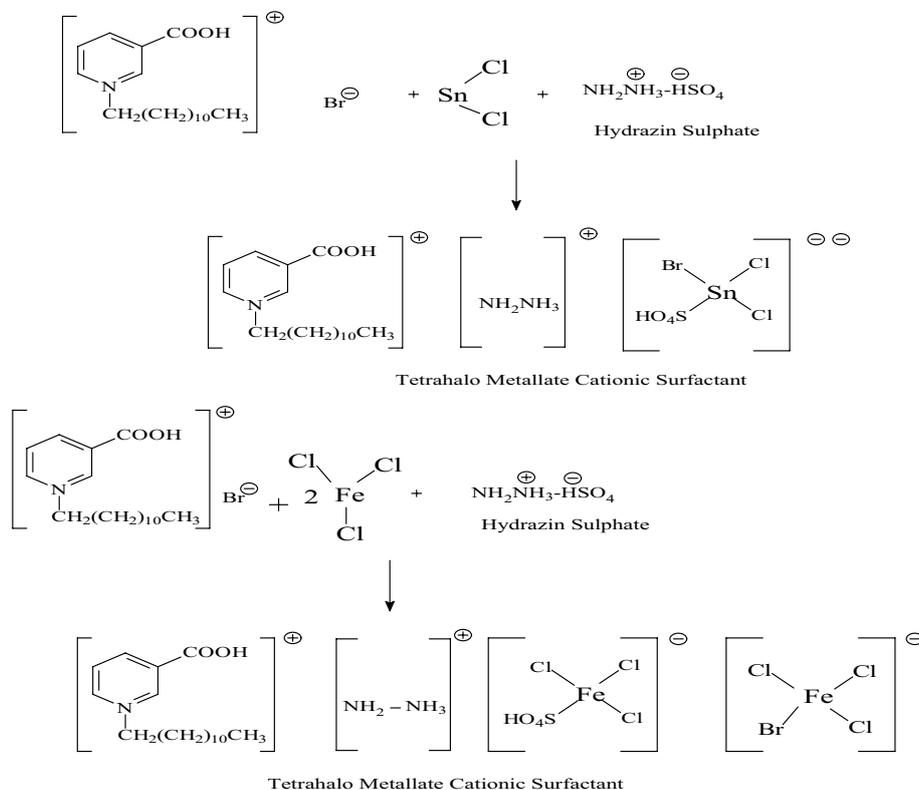
Fig. 3. Preparation of tetrahalometallate.

5 times for every sample inside 2 min break amid every evaluation.

2.5. Corrosion inhibition

The tests were conducted on a carbon steel with the following composition: 0.07% carbon, 0.3% manganese, 0.022% phosphorus, 0.010% sulphur, 0.01% silicon, 0.030% aluminium, and the remainder Fe. 1 M H_2SO4 is the aggressive media were prepared by dilution of analytical grade 97% H_2SO_4 with distilled water. The concentration range of recently prepared cationic surfactants and their derivatives that acts as inhibitors used was 600, 800, 1,000 and 1,200 ppm. The carbon steel sheets of dimensions 3.5 cm × 1.4 cm × 0.3 cm were abraded with a series of emery paper and then washed with distilled water and acetone [15]. The specimens were immersed in a 250 mL beaker containing 250 mL of the produced aggressive acid solutions with and without addition of varying quantities of the tested inhibitors after being precisely weighed. All of the corrosive acid solutions were exposed to air. The specimens were taken out, washed, dried, and weighed properly after different immersion time intervals of 2, 4, 6, 8, 10, 12, 14, and 16 d at 25°C. It was possible to obtain the weight loss of sheets. Eq. (1) was used to compute the corrosion rate (R) [14]:

$$R = \frac{W}{AT}\left(mg.cm^{-2}/d\right) \quad (1)$$

where W is the average weight loss of three parallel carbon steel sheets, A is the total area of the specimen, and T is the immersion time. The calculated corrosion rate, the inhibition efficiency (IE%) of the used corrosion inhibitors for carbon steel was calculated as follows in Eq. (2) [23]:

$$IE\% = \left[\frac{\left(R_o - R\right)}{R_o}\right] \times 100 \quad (2)$$

where R_o and R are the corrosion rates before and after the inhibitor addition, respectively.

3. Results and discussion

3.1. Characterization

The micro-elemental analysis, FTIR, ^{1}H NMR, and UV spectra were used to determine the chemical structure of the prepared parent cationic surfactants (QC12) and their derivatives.

3.1.1. Micro-elemental analysis

Micro-elemental analysis of the synthesised QC12 shown in Table 1. It indicates theoretical and experimental values of micro analysis are quite near to each other, implying that these yields are relatively pure and that their preparation was completed correctly.

3.1.2. Fourier-transform infrared spectroscopy

Fig. 5 represents the FTIR spectra for the prepared cationic surfactants. The spectra of the prepared cationic surfactants QC12 and their derivatives reveal a band at (1,580 and 1,600 cm^{-1}) linked to C=C of the pyridine, a band nearby 1,720 cm^{-1} conforming to acid's carbonyl (C=O), and a band at near 1,420 and 2,800 cm^{-1} conforming to aliphatic chain. Preparation of the quaternary ammonium salt of nicotinic acid is demonstrated by a band at almost 3,000 cm^{-1} related to OH acidity of nicotinic acid and two bands at 2,900 and 1,400 cm^{-1} relating to an ammonium ion N$^+$. In the case of QSnT and QFeT, a band at 3,290 and 3,650 cm^{-1} respectively corresponding to NH group of hydrazine sulphate. A band at 1,108 cm^{-1} related to S=O and for S–O there is a band at 640 cm^{-1}.

3.1.3. ^{1}H NMR spectra

Chemical structure of the produced QC12 was determined by ^{1}H NMR spectroscopy and showed peaks

Fig. 4. Prepare of nonionic polyurethane surfactants as in [19–21].

Table 1
Micro-elemental analysis of the synthesized parent cationic surfactant QC12

	M. wt	C.F.	C%		H%		N%		Br%	
			Calc.	Found	Calc.	Found	Calc.	Found	Calc.	Found
QC12	372.34	$C_{18}H_{30}O_2NBr$	58	57.8	8	7.9	3.7	3.4	21.5	21.3
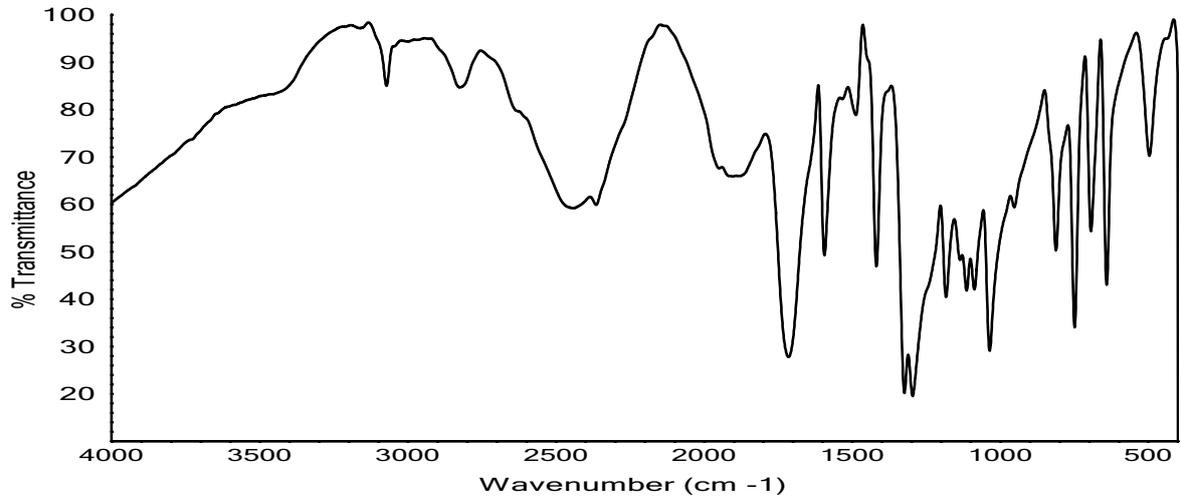
QC12

QSn

QFe
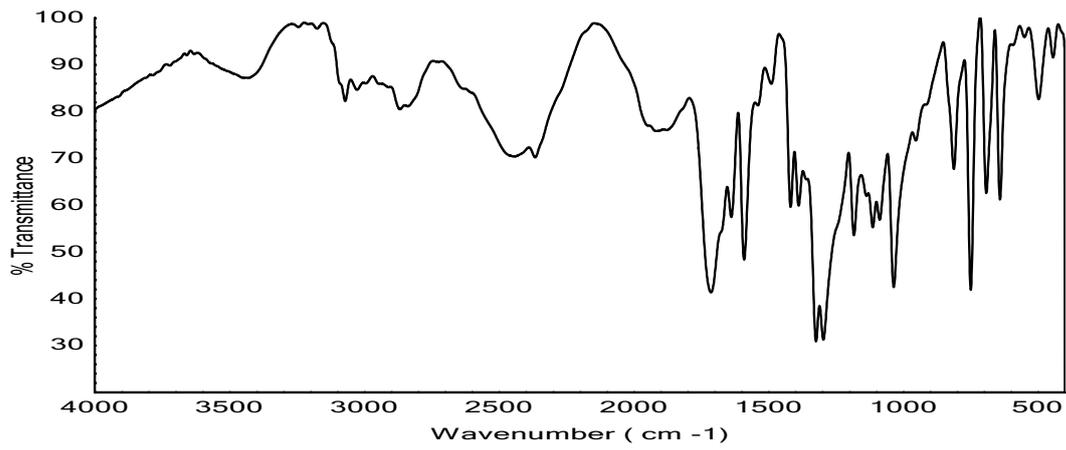
(Continued)
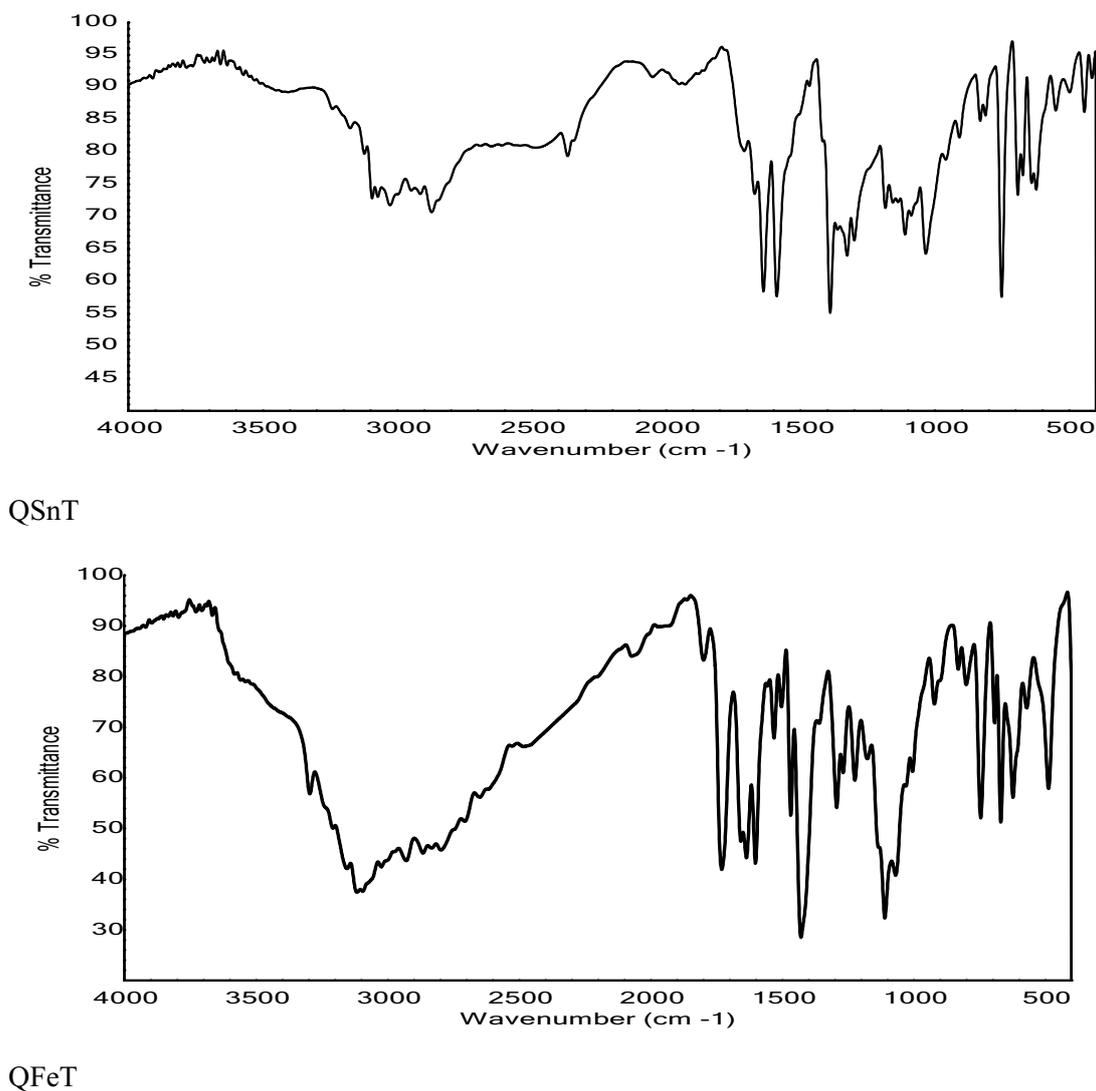
QSnT

QFeT

Fig. 5. FTIR spectra for (a) QC12, (b) QSn, (c) QSnT, (d) QFe and (e) QFeT.

at = 0.84 ppm (t, 3H, CH$_3$), = 1.26 ppm (t, 18H, CH$_2$), = 2.01 ppm (m, 2H, CH$_2$), = 5.01 ppm (t, 2H, CH$_2$), = 8.24 ppm (t, 1H, CH), = 8.93 ppm (d, 1H, CH), δ = 9.09 ppm (d, 1H, CH), δ = 9.31 ppm (s, 1H, CH), δ = 12.75 ppm (s, 1H, OH) as given in Fig. 6.

^{1}H NMR spectra of QCuT and QCoT shown a peak at a peak at δ = 0.84 ppm (t, 3H, CH$_3$), δ = 1.26 ppm (t, 18H, CH$_2$), δ = 1.5 ppm (s, 2H, NH$_2$), δ = 2.01 ppm (m, 2H, CH$_2$), δ = 4.2 ppm (s, 1H, HSO$_4$), δ = 5.01 ppm (t, 2H, CH$_2$), δ = 7.2 ppm (s, H, NH$^+$), δ = 8.24 ppm (t, 1H, CH), δ = 8.93 ppm (d, 1H, CH), δ = 9.09 ppm (d, 1H, CH), δ = 9.31 ppm (s, 1H, CH), δ = 12.75 ppm (s, 1H, OH) as shown in Fig. 7.

3.1.4. UV

UV-VIS absorption spectra were used to detect the formation of metal complexes and the presence of metal ions stabilised by cationic tetrahalometallate. The UV-Vis absorption spectra of synthesized Stannous and Ferric cationic surfactants in aqueous solution in distilled water as the solvent were recorded to monitor their production of metal complexes and stability. Because surface Plasmon excitation confirmed the appearance of a new band in the UV spectra of tetrahalometallate cationic surfactants with stannous and Ferric, UV-Vis spectroscopy is particularly sensitive to the creation of metal complex stannousated and ferricated cationic surfactants. The UV-Vis spectrum of stannous cationic surfactants, with a large peak matching to the stannous range of 260–365 (291 nm) nm, is used to determine their metallic nature [14,24]. Fig. 8 shows the UV-Vis spectrum of ferric cationic surfactants for determining their metallic character, with a broad peak corresponding to the Ferric range 240–305 nm (289 nm) [25] For QSnT and QFeT, this appearance from UV spectra shows the existence of metal Sn and Fe.
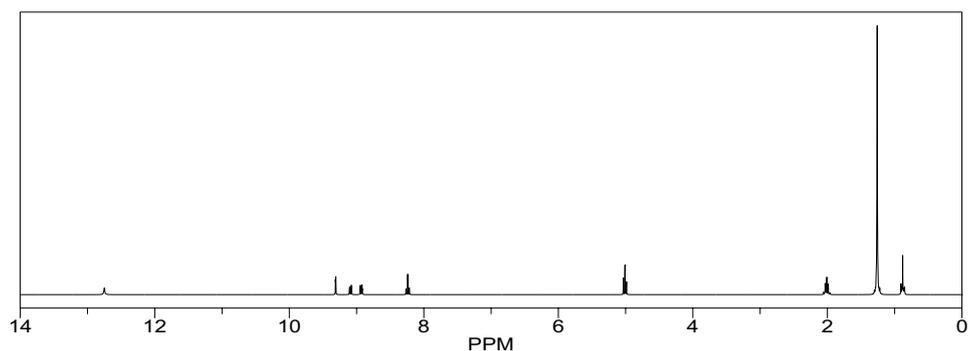
Fig. 6. ¹H NMR of the parent cationic surfactant (QC12).
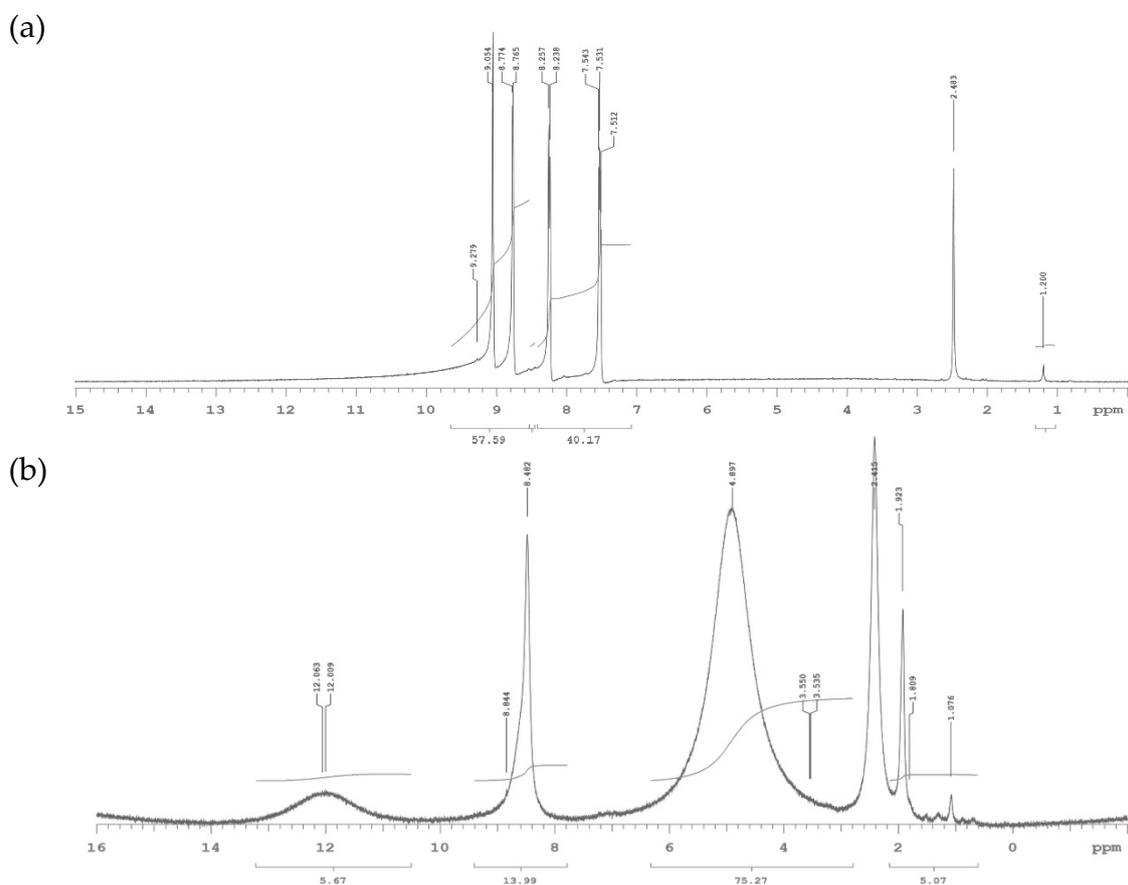
Fig. 7. ¹H NMR of (a) QSnT and (b) QFeT.

3.2. Surface measurements

Fig. 9 displays the relationship between measured surface tensions (γ) and the logarithm of measured concentrations for each of the generated cationic surfactants QC12 and their tetrahalometallate derivatives (log$C$). For QC12, QSn, QFe, QSnT, QFeT, QSnT/PU and QFeT/PU, it reflects the relationship between measured surface tension (γ) and log$C$. The observed concentration at the breakpoint is known as the critical micelle concentration (CMC). Table 2 and Fig. 9 show the final results. The CMC values were raised when the cationic surfactants were complexed with stannous or ferric ions. Between the hydrophobic chain and the water molecules, repulsion occurs. At the same time, that repulsion aids two processes. 1. Adsorption of molecules (metal complexes) at extremely low quantities at the air/water interface (below CMC). 2. Micellization of molecules (metal complex) at low concentrations by adding 20% polyurethane non-ionic surfactants to tetrahalometallate compounds Due to an increase in the carbon chain of polyethylene glycol employed in the manufacturing of polyurethane, CMC values fall compared to tetrahalometallate and the attraction between the hydrophilic chain and the water molecules increases [26–28]. From the parent
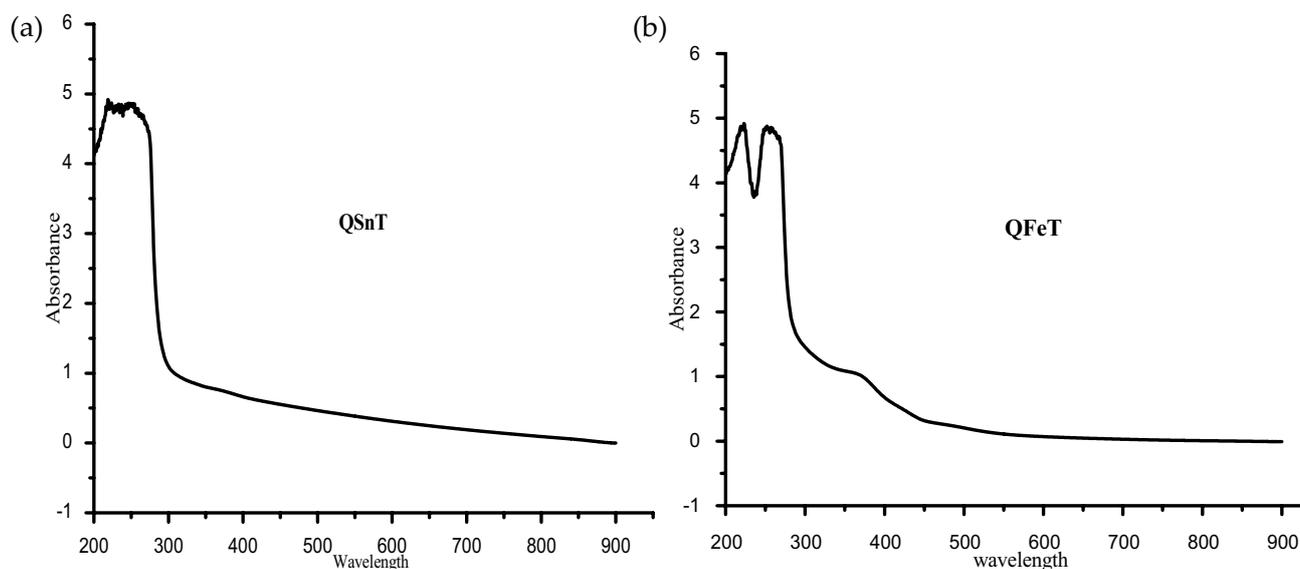
Fig. 8. UV for the prepared tetrahalometallate QSnT and QFeT.

QC12 through the addition of nonionic polyurethane (QSnT/PU, QFeT/PU), CMC values gradually decrease. Several interfacial characteristics parameters may be estimated using surface tension measurements and CMC calculations, including effectiveness (π_{CMC}, mN/m), efficiency (Pc_{20}, mol L^{-1}), maximum surface excess (Γ_{max}, mol/cm^2) and minimum surface area (A_{min}, nm^2), as shown in Table 2.

The capacity of the surfactant to adsorb at the solution's interface accounts for the decrease in CMC. The maximum surface excess (Γ_{max}), which can be calculated using Eq. (3), refers to the surfactant's ability to adsorb at the solution's interface [2,29,30]. Where, $d\gamma/d\,\log C$: slope of pre-micellar region in surface tension-log$C$ profile, R: universal constant of gases, T: absolute temperature. The maximum surface excess increases as the CMC decreased [2,31].

$$\Gamma_{max} = \frac{\left(\frac{-d\gamma}{d}\log C\right)}{\left(2.303\,nRT\right)} \tag{3}$$

The effectiveness (π_{CMC}) is defined as the difference between the surface tension of the distillate water and the surface tension of the surfactant solution at the critical micelle concentration. Eq. (4) (as illustrated and calculated).

$$\pi_{CMC} = \gamma_o - \gamma_{CMC} \tag{4}$$

$$A_{min} = \frac{10^{16}}{\Gamma_{max}N} \tag{5}$$

Efficiency (Pc_{20}) is the concentration of surfactant at a surface tension of 52 mN/m (surface tension dropped by 20 mN/m). The minimum surface area is the average area filled by adsorbed surfactant molecules at the air/water interface represented in angstrom square (A^2) (A_{min}). Eq. (5) (as illustrated and calculated), where Γ_{max} is the previously determined maximum surface excess and N_{Av} is Avogadro's number (6.023 × 1023 molecule/mole) [32,33].

The standard free energy of micellization (ΔG°_{mic}) and adsorption (ΔG°_{ads}) of synthetic cationic surfactant and its tetrahalometallate derivatives in their solutions at 25°C was estimated using Eqs. (5) and (6) where R is the universal gas constant and n is the mole number. Table 2 shows the

Table 2
Surface tension measurements

Compound	CMC (mol/L)	γ_{CMC} (mN/m)	π_{CMC} (mN/m)	Γ_{max} × 10–11 (mol/cm^2)	A_{min} (nm^2)	Pc_{20} (mol/L)	ΔG°_{mic} (kJ/mol)	ΔG°_{ads} (kJ/mol)
QC12	0.05	44	28	5.34839	3.103735	3.906 × 10^{-6}	–1.77595210	–9.9698
QSn	0.004	51	21	5.34839	3.103735	0.001	–3.273273196	–12.770
QSnT	0.0004	58.4	13.6	4.88137	3.400685	0.001	–4.638308679	–16.554
QSnT/PU	0.00035	47.6	24.4	4.88137	3.400685	0.000031	–4.717469744	–14.429
QFe	0.005	57	15	5.34839	3.103735	0.000062	–3.14098759	–13.755
QFeT	0.001	59	13	5.34839	3.103735	0.000052	–4.09510645	–15.082
QFeT/PU	0.00079	48.4	23.6	5.29082	3.137512	7.812 × 10^{-6}	–4.2348491	–13.346
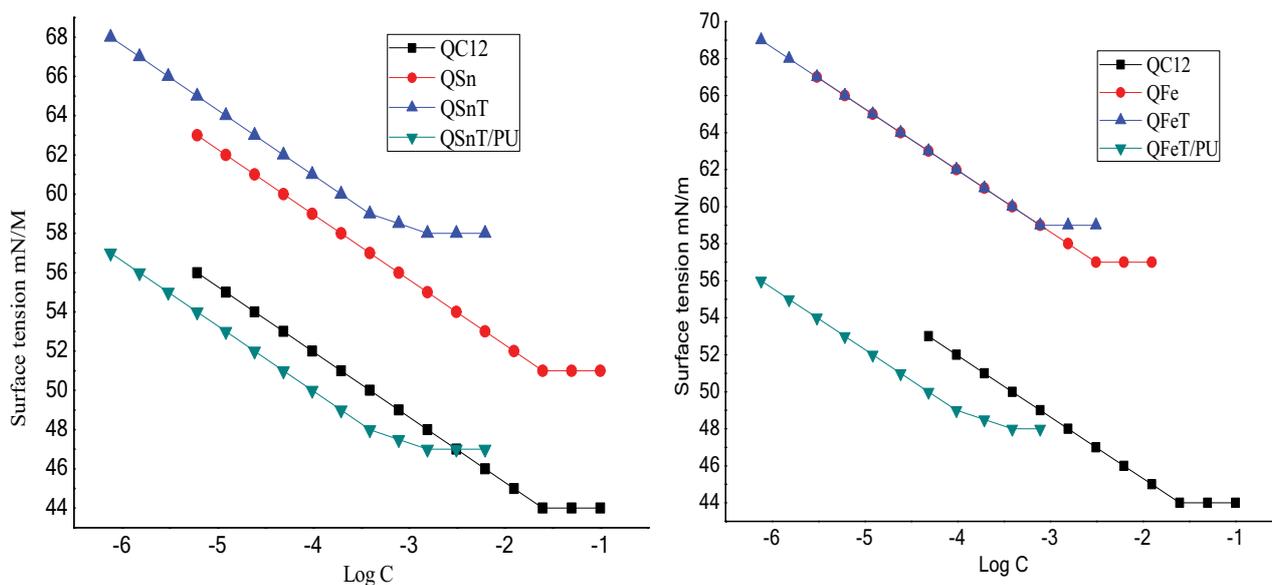
Fig. 9. Surface tension and Log$C$ of prepared cationic surfactants.

results. The mentioned values for the investigated cationic surfactants and polyurethane added derivatives are negative, indicating that adsorption and micelle formation occur spontaneously. As a result, the two stages are in a state of balance. The contact forces between the hydrophobic chain and the polar media lead (ΔG_{ads}°) to be lower than (ΔG_{mic}°), showing that these surfactants prefer adsorption to micellization [34]. When nonionic polyurethane is introduced, the (ΔG_{mic}°) and (ΔG_{ads}°) values decrease.

$$\Delta G_{mic}^{\circ} = 2.303 RT \log CMC \quad (6)$$

$$\Delta G_{ads}^{\circ} = \Delta G_{mic}^{\circ} - \left(0.6\pi_{CMC} A_{min}\right) \quad (7)$$

3.3. Corrosion inhibition

The inhibitory efficiency (I.E) of the generated quaternary pyridinium salts (QC12) and their tetrahalometallate derivatives is shown in Table 3 at various doses. These observed data show a steady improvement in inhibitory efficiency when surfactant concentrations are increased.

Table 3
Inhibition efficiency of cationic surfactant QC12 and the tetrahalometallate

Inhibitor	Inhibition efficiency (IE) %			
	600 ppm	800 ppm	1,000 ppm	1,200 ppm
QC12	55.5	60.7	61.5	62.8
QSn	81.3	83.2	89.3	92.4
QSnT	98.6	99.1	99.4	99.5
QFe	57.1	63.6	65.1	66.4
QFeT	80.6	82.5	83.3	88.4

Because the surfactants studied are pyridinium salt derivatives, quaternized nicotinic acid and their tetrahalometallate cationic surfactants are found to have good carbon steel corrosion inhibition. This is owing to their adsorptive qualities, which cause salts to form insoluble films at the surface and interact with the carbon steel atoms, resulting in the development of insoluble coatings [5].

Table 4 shows the effect of adding 20% (wt/wt) PU to the pyridinium salt tetrahalometallate cationic surfactant at 1,200 ppm. Because of the rise in hydrophobicity in solution, there is a considerable increase in inhibition efficiency [35].

4. Conclusion

Surface active agent is present in cationic surfactants made by quaternizing nicotinic acid with 1-bromododecane QC12 and creating tetrahalometallate cationic surfactants. QC12 has the lowest CMC value, followed by tetrahalometallate with 20% (wt/wt) nonionic polyurethane, and finally tetrahalometallate without 20% (wt/wt) nonionic polyurethane. In this study, these chemicals are employed as corrosion inhibitors for carbon steel. The generated cationic surfactant and its tetrahalometallate component based on stannous and ferric demonstrate substantial corrosion inhibition, especially the tetrahalometallate component, due to their significant adsorptive properties on carbon steel surfaces. The addition of 20% (wt/wt)

Table 4
Effect of adding 20% polyurethane addition on the tetrahalometallate

Inhibitor	Inhibition efficiency (IE) % (1,200 ppm)
QSnT/PU	99.8
QFeT/PU	98

nonionic polyurethane to the tetrahalometallate results in an enhancement in carbon steel corrosion inhibition.

Conflict of interest and authorship conformation form

- All authors have participated in (a) conception and design, or analysis and interpretation of the data, (b) drafting the article or revising it critically for important intellectual content, and (c) approval of the final version.
- This manuscript has not been submitted to, nor is under review at, another journal or other publishing venue.
- The authors have no affiliation with any organization with a direct or indirect financial interest in the subject matter discussed in the manuscript

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