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Received: 2 April 2022 | Revised: 18 July 2022 | Accepted: 18 July 2022

DOI: 10.1002/jsde.12614

ORIGINAL ARTICLE

AOCS WILEY

Synthesis of tri-cationic surfactants containing two amide functional groups and inhibition effectiveness against metal corrosion in acidic medium

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Abstract

In this study, two tri-cationic surfactants containing two amide functional groups and three positively charged quaternary aminium nitrogens were synthesized. The structures of the synthesized surfactants were proved by various spectroscopic methods (FT-IR, ^{1}H NMR, and ^{13}C NMR). Inhibition activities of two synthesized products against mild steel corrosion were determined using the weight loss method in 1.0 M HCl acidic medium. As a result of corrosion tests performed by immersing metal coupons in acidic solutions with different inhibitor concentrations for 24 h at room temperature, close and effective inhibitions were obtained for both surfactants. It has been found that the number of carbons in the long carbon chain in their structures is an effective factor in corrosion protection. In the acid environment, the corrosion inhibition efficiency of the inhibitor with more carbon in the long chain was found slightly higher than the other. While the compound containing 16 carbon atoms in its long chain showed corrosion inhibition over 92%, the compound containing 12 carbons in its long chain exhibited corrosion inhibition of 90% and below. In addition, some physicochemical parameters such as critical micelle concentration, surface tensions at this concentration, and free energy of micelle formation were calculated for the tri-cationic surfactants in order to correlate them with their anti-corrosion activities. Surface images were also taken by scanning electron microscope, which is an important supporting evidence that surfactants adsorb on the metal surface and protect the surface from corrosion, to determine the roughness conditions on the metal surfaces immersed in the acid solution.

KEYWORDS
acidic media, amide, corrosion inhibition efficiency, synthesis, tri-cationic surfactants

INTRODUCTION

Mild steel, which is in the class of metals and alloys, has a wide range of uses in machinery and other industrial applications containing elements of this class. Unfortunately, as with many other metals, one of the most unfortunate events that can happen to mild steel is corrosion. Corrosion phenomenon, which affects human health and the environment negatively as well as reducing the life of metals, causing material losses (O'm Bockrıs et al., 2000), is due to the tendency of metals to turn into stable compounds as a result of the reaction of the metals with the elements in the corrosive environment. Metals, which are found in the form of compounds in nature, turn from the high-energy metallic state to their lower and more stable compound states due to corrosion (Erbil, 2012).

Corrosion, which can also be defined as the chemical or electrochemical dissolution of the metal, occurs faster if the metal is exposed to an acidic environment. It is often treated with acid solutions (HCl, H_2SO_4, etc.) in order to clean the mild steel used in the production of storage tanks, reaction vessels and boilers in the industry (Quaraishi et al., 2002). Since there is no oxide or hydroxide layer on the metal surface due to the cleaning of the surface in an acidic environment, the metal surface is usually bare. This ensures that the metal surface interacts more easily with the corrosive

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components in the acidic environment and corrosion is inevitable (Ergun et al., 2008).

In recent years, the use of inhibitors to prevent corrosion has become very common. It has been determined that the use of inhibitor, which has become the most suitable method especially in closed systems, is also economically viable. The strengthening factor for this interpretation is that instead of the corrosion resistant but expensive metal, it is possible to use a cheaper metal by using an inhibitor. Heterocyclic organic inhibitors are often preferred against mild steel corrosion in acidic environments (Doğru Mert et al., 2011; Ergun et al., 2008; Kosari et al., 2014; Sherif, 2014; Solmaz et al., 2011). The corrosion inhibitory effect of such organic inhibitors, which strongly adheres to the metal surface and forms a protective film layer on the surface (Erbil, 1984), is generally achieved by physical or chemical adsorption (Karthikaiselvi & Subhashini, 2014).

In the literature, long carbon chain cationic surfactants have an important place among the compounds synthesized to be used as corrosion inhibitors in corrosive acidic environments. These compounds dissolve well in the corrosive environment and adsorb well to the metal surface by physical and chemical means. For these reasons, cationic surfactants have been effective in combating metal corrosion by reducing the corrosion rate (Öztürk, 2017; Yıldırım et al., 2013). Among the cationic surfactants, there are studies in which dicationic gemini type (symmetric twin structure) containing two positively charged groups (Abd El-Lateef et al., 2016; Asefi et al., 2010; Feng et al., 2018) and non-gemini type (Hegazy et al., 2016; Öztürk, 2018a, 2019) surfactants are used as corrosion inhibitors. The common aspect of these studies is that the anticorrosion effect of the surfactants synthesized in all of them was investigated by weight loss tests based on gravimetric measurements. There are few studies on tri-cationic surfactants, which are used as corrosion inhibitors among cationic surfactants and contain three positive groups in their structure. For example, from these studies Bensajjay et al. (2011) have investigated the inhibitory performance of straight chain ammonium salt against corrosion in 1.0 M HCl environment. In another study, Eldougdoug et al. (2018) have reacted cetyl-2 chloroacetate with di-amide compound for the synthesis of tri-cationic surfactant. They have investigated it as a corrosion inhibitor for carbon steel in hydrochloric acid solutions by using weight loss and potentiodynamic polarization methods. More recently, Öztürk et al. (2021) have synthesized tricationic surfactants derived from vanillin compound and containing three positively charged nitrogen atoms in its structure.

In this study, it was aimed to synthesize two tricationic surfactants containing amide functional groups with 12 and 16 carbon atoms in their long chain and three positively charged quaternary amine structured nitrogen atoms. At the same time, it was aimed to examine whether the synthesized surfactants showed an inhibitory effect against the corrosion of mild steel in 1.0 M HCl environment. Some physicochemical parameters of these compounds such as critical micelle concentration (CMC), surface tension at this concentration value and micelle formation free energy (ΔG_{mic}) value were tried to be determined. Finally, it is planned to take scanning electron microscope (SEM) images of the metal surface after the corrosion test to confirm and support the inhibition efficiency results.

MATERIALS AND METHODS

Chemicals

Fatty acids containing 12 and 16 carbon atoms, thionyl chloride ($SOCl_2$), toluene, N,N-dimethyl-ethylenediamine, ethanol, and 37% HCl chemicals and solvents were purchased from Merck (Merck, Darmstadt, Germany). Reagents named triethylamine (TEA), dichloromethane (CH_2Cl_2), 3-(dimethylamino)-1-propylamine and (2-bromoethyl)trimethylammonium bromide were obtained from Sigma-Aldrich company (St. Louis, MO, USA).

Instrumentation

The FT-IR spectra of the tri-cationic surfactants synthesized as the final product were obtained with the Perkin Elmer Spectrum 100 (Shelton, CT, USA) FT-IR spectrophotometer. NMR spectra (^{1}H NMR and ^{13}C NMR) of the same compounds were obtained with Agilent 600 MHz Premium Compact NMR spectrometer (Santa Clara, CA, USA) in dimethylsulfoxide (DMSO) solvent. The elemental analyses were performed using an LECO CHNS-932 elemental analyzer (Saint Joseph, MI, USA). NUVE EV 018 vacuum oven (Ankara, Turkey) was used to dry the metal coupons until they reach constant weight.

Synthesis of compounds

First, 2-chloro-N-(3-(dimethylamino)propyl)acetamide (**1**), which is one of the starting materials, was synthesized. For this, 3-dimethylamino-1-propylamine, trimethylamine and dichloromethane liquids were placed in the double-necked balloon. The mixture in the flask was cooled in an ice bath, and a solution of chloracetyl chloride in dichloromethane was added through the dropping funnel. The reaction mixture was heated at 70°C for 6 h. At the end of the period, the dichloromethane solvent was evaporated and the remaining residue was washed with ethanol, while the precipitated trimethylamine salt was filtered off. Then, ethanol was evaporated from the filtrate and the remaining material was set aside for reuse.

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In the synthesis of compounds **2a**–**b**, which is the second starting material used in the preparation of the product, acid chlorides were firstly obtained by heating fatty acids containing 12 and 16 carbons in their long chain with $SOCl_2$ in toluene. In the continuation of the reaction series, amide compounds (**2a**–**b**), prepared by treatment with acid chlorides, N,N-dimethyl-ethylenediamine, were synthesized using the same experimental methods previously reported for similar compounds (Öztürk, 2019).

2-((3-(dimethylamino)propyl)amino)-N-(2-dodecanamidoethyl)-N,N-dimethyl-2-oxoethanaminium chloride (**3a**)

Compound **1** (0.8286 g, 4.63 mmol), compound **2a** (1.2546 g, 4.63 mmol) and 30 ml of ethanol were placed in a round bottom flask. The mixture was refluxed for 48 h and at the end of time ethanol was evaporated. The residue was washed by adding ether and the precipitated product was filtered in vacuo. A beige solid wax was obtained. Yield: 80%.

2-((3-(dimethylamino)propyl)amino)-N,N-dimethyl-2-oxo-N-(2-palmitamidoethyl) ethanaminium chloride (**3b**)

Compound **1** (0.75 g, 4.20 mmol), compound **2b** (1.3712 g, 4.20 mmol) and 30 ml of ethanol were placed in a round bottom flask. The synthesis of compound **3b** includes the same process steps used in the synthesis of compound **3a**. A beige solid wax was obtained. Yield: 37%.

N^1-(3-(2-((2-dodecanamidoethyl) dimethylammonio)acetamido)propyl)-N^1,N^1, N^2,N^2,N^2-pentamethylethane-1,2-diaminium dibromide chloride (**4a**)

Compound **3a** (1.64 g, 3.65 mmol), (2-bromoethyl)trimethyl ammonium bromide (0.9019 g, 3.65 mmol) and 30 ml of ethanol were placed in a round bottom flask. The mixture was refluxed for 48 h and at the end of time ethanol was evaporated. The residue was washed two times with hot acetone and the unreacted (2-bromoethyl)trimethyl ammonium bromide compound was filtered off. A yellow solid product was obtained. The yield and melting point of the product are shown in Table 1.

N^1-(3-(2-(dimethyl(2-palmitamidoethyl) ammonio)acetamido)propyl)-N^1,N^1,N^2,N^2,N^2-pentamethylethane-1,2-diaminium dibromide chloride (**4b**)

Compound **3b** (0.772 g, 1.53 mmol), (2-bromoethyl)trimethyl ammonium bromide (0.3774 g, 1.53 mmol) and 30 ml of ethanol were placed in a round bottom flask. The synthesis of compound **4b** includes the same process steps used in the synthesis of compound **4a**. A yellow solid product was obtained. The yield and melting point of the product are shown in Table 1.

Corrosion tests performed in 1.0 M HCl solution

Metal plates prepared from cold rolled steel with low carbon content were used for corrosion tests carried out in an acidic environment. This steel material complies with the DIN EN 10130 (Anonymous, 2007) norm and it contains 0.07% C, 0.35% Mn, 0.015% P, and 0.015% S. Metal plates were cut into rectangles to be 0.1 cm thick, 2.2 cm wide, and 5.0 cm long.

The preparatory phase of the corrosion tests based on weight loss measurements in the metal was carried out with the method previously reported in the literature (Öztürk, 2018b). For this purpose, the metal plates cut in the specified dimensions were kept in 15% HCl solution for 5 min, and rust stains and oxide layers were removed. The plates were then cleaned with distilled water. After the surface of the dried metal plates were sanded with sandpaper (150), they were kept in acetone for a while. The plates removed from the acetone were dried first in the vacuum oven and then in the open air for a short time until they reached constant weight, and the mass of the metal plate was weighed before the test. Before the corrosion tests, 1.0 M HCl solution was freshly prepared to create an acidic aqueous corrosive medium. Tri-cationic surfactants, which were subjected to the test, were weighed in certain amounts and then dissolved in 100 ml 1.0 M HCl and placed in 150 ml glass containers with lids. In this way, 25, 50, 100, and 200 ppm solutions of surfactants in

TABLE 1 Characterization data of the cationic surfactants

Compounds	Chemical formula	MW (g mol^{-1})	*wi*(calc.)/%−*wi*(found)/%			Yield (%)	Melting point (°C)
			C	H	N		
4a	$C_{28}H_{62}Br_2ClN_5O_2$	696.09	48.31−48.39	8.98−8.91	10.06−9.98	52	113–114
4b	$C_{32}H_{70}Br_2ClN_5O_2$	752.19	51.10−51.21	9.38−9.29	9.31−9.25	61	125–126

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acidic medium were prepared. On the other hand, for the control test and comparison, 100 ml of 1.0 M HCl was placed in another glass container and no inhibitor was added into it. The metal plates, weighed before the test, were immersed in these prepared, inhibitor-free and acidic solutions with inhibitor, tied with a rope, and left at room temperature for 24 h without mixing the solutions. After the test period, the metal plates removed from the corrosive environment were rinsed with distilled water, were cleaned with acetone, dried in a vacuum oven and the post-test mass of the metal was weighed. By taking the difference between the pre-test and post-test metal masses weight loss was calculated for each metal coupon.

DETERMINATION OF PHYSIOCHEMICAL PARAMETERS FOR TRI-CATIONIC SURFACTANTS

Critical micelle concentration

The CMC of the synthesized tri-cationic surfactants was determined using the conductometric method. Conductivity measurements were carried out using OHAUS Starter 3100M brand digital conductivity meter. To measure the specific conductivity in each surfactant, a step-by-step dilution-extraction method was followed. For this, the specific conductivity of 30 ml of pure water was determined first. Then, 0.5 ml of 10^{-2} M inhibitor solution was added to this water and specific conductivity measurements were made after each added 0.5 ml inhibitor solution. A graph was created from the conductivity measurements determined for each inhibitor concentration. CMC values were determined from the breakpoint on each conductivity curve versus surfactant concentration.

Surface tension

Surface tensions of aqueous solutions of tri-cationic surfactants at CMC were measured by capillary rise method (Martino et al., 2006; Sugden, 1921). The capillary used for measurement is Na-heparinized micro hematocrit tube (BRIS, Soda Lime glass, diameter = 1.0 mm, length = 75 mm, Vitrex Medical, Herlev, Denmark). The corresponding values were measured at 25°C.

Imaging metal surface with SEM

The SEM image of the metal surface was taken on metal plates cut into squares with a thickness of 0.1 cm, a width of 2.0 cm, and a length of 2.0 cm. After the corrosion tests carried out in an acidic environment, the metal plates were washed with acetone and dried in a vacuum desiccator, and then SEM images of the 1000 times magnified environment of the metal surfaces were obtained with a SEM device (Carl Zeiss EVO 40, Oberkochen, Germany) under high vacuum.

RESULTS AND DISCUSSION

Synthesis of tri-cationic surfactants

Compound **1**, which will be used as a starting material in synthesis, was obtained by the amide formation reaction of chloracetyl chloride, an active reagent, with 3-dimethylamino-1-propylamine. On the other hand, amide formation reaction was also used for the synthesis of **2a**–**b** compounds, which is another starting material used in the synthesis. For this, acid chlorides prepared from fatty acids containing 12 and 16 carbons in their structure were reacted with N,N-dimethyl-ethylenediamine, ant the targeted amide starting compounds (**2a**–**b**) were obtained. Preparation reactions of the two starting compounds (**1** and **2a**–**b**) are given in Figure 1. As seen in Figure 1, these starting materials interacted with each other to synthesize **3a**–**b** compounds containing the first positively charged nitrogen atom in their structure. In the last step of the reaction series, the targeted corrosion inhibitor products (**4a**–**b**) were obtained as a result of the quaternization reaction between the related amide compounds (**3a**–**b**) containing one positive nitrogen atom and (2-bromoethyl)trimethylammoniumbromide. With this last quaternization reaction, the number of positively charged nitrogen atoms in the structure of surfactants increased to three (Figure 1).

The FT-IR, ^{1}H NMR, and ^{13}C NMR spectra of the synthesized tri-cationic surfactants are given in the "Supplementary Material" (Appendices 1, 2, and 3, respectively). The spectral data of the characteristic peaks seen in the FT-IR and NMR spectra of the synthesized corrosion inhibitors are given in Table 2. For compound **4a**; the amide N—H peak at 3273, the aliphatic C—H peak at 2921, the carbonyl (C═O) group peak at 1642 and the N—H bending peak (2nd amide band) at 1553 $\tilde{\nu}$/cm^{-1} are given in Table 2. The same peak values for compound 4b were observed at 3240, 2917, 1638, and 1566 $\tilde{\nu}$/cm^{-1}, respectively (Table 2). It was observed that the peak values of N—H, aliphatic C—H and carbonyl (C═O) for compound **4a** were slightly higher than those of **4b**. In Table 2, the characteristic ^{1}H NMR and ^{13}C NMR spectral data of two tri-cationic surfactants are also shown. In the ^{1}H NMR spectrum, the most important peaks for the synthesized compounds are the triplet peak of the hydrogen in the amide N—H bond, the triplet peak of the hydrogens in the N^{+}—$\underline{CH_2}$—$\underline{CH_2}$—N^{+} bond, and the singlet peak of the hydrogens in the N^{+}—$\underline{CH_2}$—CO—NH bond. The values of these peaks were observed at 8.15, 4.07, and 3.14 ppm for compound **4a**, and 8.04, 4.07, and

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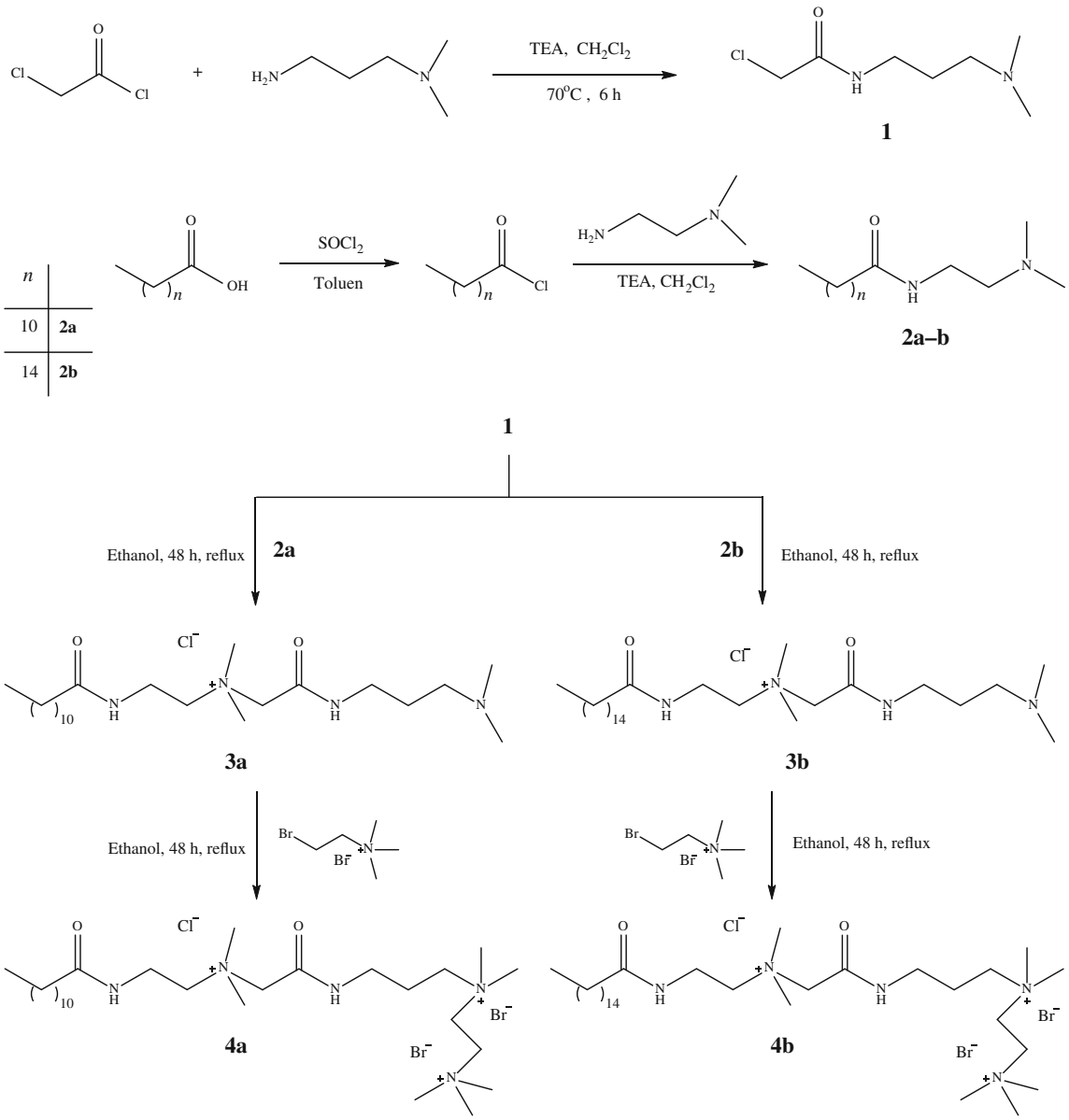


FIGURE 1 Synthesis steps of tri-cationic surfactants

3.12 ppm for compound **4b**, respectively. In the light of all these spectral findings, the structures of two corrosion inhibitors were elucidated and their synthesis were determined.

Corrosion rate and corrosion inhibition efficiencies calculated by gravimetric method

The corrosion rate (CR) and inhibition efficiency (IE) results of tri-cationic surfactants (**4a**–**b**), which are adsorbed on the metal surface at different concentrations, are shown in Table 3. To calculate the CR following inhibition tests by dissolving corrosion inhibitors directly in 1.0 M HCl solution and immersing metal coupons in this corrosive acid solution at room temperature for 24 h, Equation (1) has been used (Abdelrazik et al., 2015; Lashgari et al., 2010).

$$\mathrm{CR} = \frac{\Delta W}{A \times t} \tag{1}$$

ΔW is the weight loss in mg, A is the metal plate surface area in cm^2, and t is the immersion time in hours. Corrosion IE results were determined by the following formula (Equation (2)) (Gerengi et al., 2018).

$$\mathrm{IE}\% = \frac{W_o - W}{W_o} \times 100 \tag{2}$$

The corrosion IE was calculated as the percent inhibitory efficiency (IE%). W_o is the weight loss of metal in non-inhibitor corrosive solution, and W is the

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TABLE 2 Spectral characterization data of synthesized tri-cationic surfactants

Compounds	FT-IR (/cm^{-1})	^{1}H NMR (δ, ppm) (600 MHz, DMSO-d$_6$)	^{13}C NMR (δ, ppm) (150 MHz, DMSO-d$_6$)
4a	**3273** (amide N—H), **2921** (aliphatic C—H), **1642** (amide C═O), **1553** (amide N—H, second amide band)	**8.15** (t, 1H, Amide N—**H**), **4.07** (t, 4H, N^+—C**H$_2$**—C**H$_2$**—N^+), **3.76** (t, 2H, CO—NH—CH_2—C**H$_2$**—N^+), **3.35** (q, 2H, CO—NH—C**H$_2$**—CH_2—N^+), **3.14** (s, 2H, N^+—C**H$_2$**—CO—NH), **3.08** (t, 2H, CO—NH—CH_2—CH_2—C**H$_2$**—N^+), **3.03** (q, 2H, CO—NH—C**H$_2$**—CH_2—CH_2—N^+), **2.73** [s, 21H, N^+(C**H$_3$**)$_2$—CH_2—CO—NH and N^+(C**H$_3$**)$_2$—CH_2—CH_2—N^+(C**H$_3$**)$_3$], **2.47** (quin., 2H, N^+—CH_2—C**H$_2$**—CH_2—N^+), **2.05** (t, 2H, C**H$_2$**—CO—NH), **1.44** (quin., 2H, CH_2—C**H$_2$**—CH_2—CO—NH), **1.23**—**1.15** [m, 16H, CH_3—(C**H$_2$**)$_8$], **0.81** [t, 3H, C**H$_3$**—$(CH_2)_8$]	**173.22** (CH_2—**C**O—NH and N^+—CH_2—**C**O—NH), **65.35** (N^+—**C**H$_2$—CO—NH), **56.18** (CO—NH—CH_2—**C**H$_2$—N^+), **53.15** (CO—NH—CH_2—CH_2—**C**H$_2$—N^+ and N^+—**C**H$_2$—**C**H$_2$—N^+), **45.86** [(**C**H$_3$)$_3$], **42.68** [N^+(**C**H$_3$)$_2$—CH_2—CH_2—N^+ and CO—NH—CH_2—CH_2—N^+(**C**H$_3$)$_2$], **36.84** (CO—NH—**C**H$_2$—CH_2—CH_2—N^+), **35.78** (**C**H$_2$—CO—NH), **34.19** (CO—NH—**C**H$_2$—CH_2—N^+), **31.72** (CH_3—CH_2—**C**H$_2$), **29.47**—**29.14** [(**C**H$_2$)$_6$], **25.42** (CH_2—C**H$_2$**—CH_2—CO—NH), **22.52** (CH_3—**C**H$_2$— and CO—NH—CH_2—**C**H$_2$—CH_2—N^+), **14.37** (**C**H$_3$—)
4b	**3240** (amide N—H), **2917** (aliphatic C—H), **1638** (amide C═O), **1566** (amide N—H, second amide band)	**8.04** (t, 1H, Amide N—**H**), **4.07** (t, 4H, N^+—C**H$_2$**—C**H$_2$**—N^+), **3.73** (t, 2H, CO—NH—CH_2—C**H$_2$**—N^+), **3.32** (q, 2H, CO—NH—C**H$_2$**—CH_2—N^+), **3.12** (s, 2H, N^+—C**H$_2$**—CO—NH), **3.09** (t, 2H, CO—NH—CH_2—CH_2—C**H$_2$**—N^+), **2.98** (q, 2H, CO—NH—C**H$_2$**—CH_2—CH_2—N^+), **2.67** [s, 21H, N^+(C**H$_3$**)$_2$—CH_2—CO—NH and N^+(C**H$_3$**)$_2$—CH_2—CH_2—N^+(C**H$_3$**)$_3$], **2.47** (quin., 2H, N^+—CH_2—C**H$_2$**—CH_2—N^+), **2.05** (t, 2H, C**H$_2$**—CO—NH), **1.44** (quin., 2H, CH_2—C**H$_2$**—CH_2—CO—NH), **1.24**—**1.16** [m, 24H, CH_3—(C**H$_2$**)$_{12}$], **0.81** [t, 3H, C**H$_3$**—$(CH_2)_{10}$]	**173.21** (CH_2—**C**O—NH and N^+—CH_2—**C**O—NH), **65.40** (N^+—**C**H$_2$—CO—NH), **56.55** (CO—NH—CH_2—**C**H$_2$—N^+), **53.18** (CO—NH—CH_2—CH_2—**C**H$_2$—N^+ and N^+—**C**H$_2$—**C**H$_2$—N^+), **43.06** [N^+(**C**H$_3$)$_3$, N^+(**C**H$_3$)$_2$—CH_2—CH_2—N^+ and CO—NH—CH_2—CH_2—N^+(**C**H$_3$)$_2$], **36.80** (CO—NH—**C**H$_2$—CH_2—CH_2—N^+), **35.77** (**C**H$_2$—CO—NH), **34.53** (CO—NH—**C**H$_2$—CH_2—N^+), **31.72** (CH_3—CH_2—**C**H$_2$), **29.48**—**29.13** [(**C**H$_2$)$_{10}$], **25.45** (CH_2—C**H$_2$**—CH_2—CO—NH), **22.52** (CH_3—**C**H$_2$— and CO—NH—CH_2—**C**H$_2$—CH_2—N^+), **14.38** (**C**H$_3$—)

TABLE 3 Corrosion rate and corrosion inhibition efficiencies (%IE) calculated by weight loss measurement in metal coupons exposed for 24 h at room temperature for different concentrations of surfactants in 1.0 M HCl medium

	Corrosion rate (mg cm^{-2} h^{-1}) × 10^{-2}		Inhibition efficiency (IE%)	
Inhibitor concentration (ppm)	**4a**	**4b**	**4a**	**4b**
0	37.76	37.76	—	—
25	5.34	2.69	85.86	92.88
50	3.90	2.76	89.67	92.68
100	3.56	2.72	90.57	92.78
200	3.98	2.91	89.47	92.28

weight loss of metal in corrosive solution containing inhibitor.

When the IE values in Table 3 were examined, it was observed that the IE increased significantly when the concentration of tri-cationic surfactant **4a**, which contains a shorter carbon chain in its structure, increased from 25 to 100 ppm. This expected result has been reported in the literature before, and it has been reported that the IE increases with the increase of the inhibitor concentration (Aiad et al., 2016; Öztürk, 2017; Qiu et al., 2005). For the same compound, when the inhibitor concentration increases from 100 to 200 ppm, there appears to be a slight tendency to decrease in inhibitory activity. On the other hand, with increasing concentration of compound **4b**, which contains more carbon atoms in its long chain, the IE did not increase significantly and IE values were calculated very close to each other at each concentration. In addition, when both tri-cationic surfactants were compared with each other, it was determined that the compound with longer carbon chain in its structure exhibited more corrosion inhibition at each concentration value. Obtaining a result of **4b** > **4a** in corrosion inhibition

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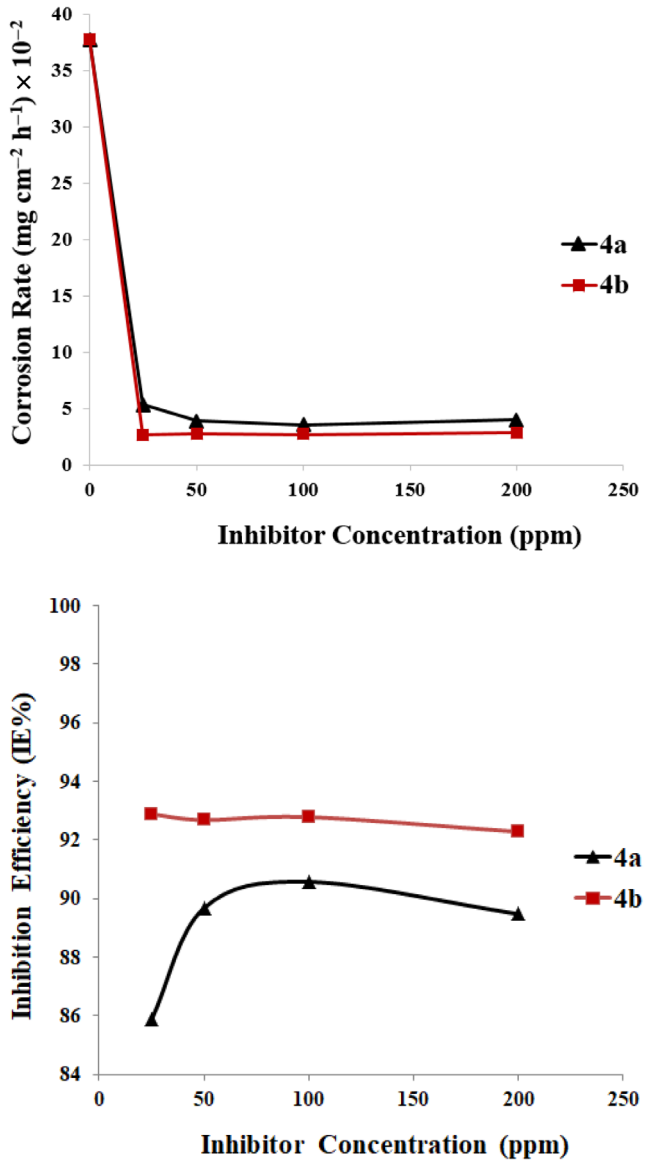


FIGURE 2 Graphical view of changes in corrosion rate and inhibition efficiency depending on the concentration

activities as a result of the comparison between the compounds reveals the fact that the IE increases with the increase in the carbon chain length in the structure, which is valid for surfactants. In general, longer hydrocarbon chains provide better corrosion protection.

When the CR values in Table 3 are examined, it is seen that the rate of corrosion is high in an acidic corrosive medium without inhibitor (0 ppm). It is observed that the CR decreases as the inhibitor concentration increases in those containing inhibitors. As seen in the table, it is clear that the CR and the IE are inversely proportional to each other. In other words, the IE is high when the CR is low. These changes in CR and IE depending on the concentration can be seen graphically in Figure 2.

Physiochemical parameters

The CMC results of the two synthesized tri-cationic surfactants determined by the conductometric method are given in Table 4. The measured conductivities for different concentrations of each surfactant are plotted. Related graphics are given in Figure 3. It is seen in the graphs that the concentrations also increase linearly with the increase in conductivity. In the concentration-conductivity linear trend line, the concentration at which the slope changes was determined as the CMC for the synthesized surfactants (Tyagi & Tyagi, 2011). Conductivity measurements were carried out at 25°C.

As can be seen in Table 4 and Figure 3, the CMC value of surfactant **4a**, which contains a long chain of 12 carbons, is 7.38×10^{-4} M; For compound **4b** containing a 16 carbon long chain, the CMC value was found to be 4.19×10^{-4} M. From these results, it can be concluded that micelle formation is at low concentrations for the synthesized tri-cationic surfactants. Compound **4b**, which has a higher hydrophobic character because it has a longer carbon chain, has a lower CMC value. This is consistent with the result that the CMC value decreases with increasing hydrophobic character (Öztürk et al., 2020; Taleb et al., 2017). This result is also logically compatible with the IE results given in Table 3. The fact that the inhibition activities of compound **4b** are close to each other with the increase in concentration indicates that the CMC value has been reached. However, the increase in the IE values of the surfactant **4a** with the increase of the inhibitor concentration from 25 ppm to 100 ppm causes us to make a prediction that the CMC value has not been reached yet (Yıldırım et al., 2013).

The CMC is dependent on the micelle formation Gibbs free energy (ΔG_{mic}) value, and the micelle-forming tendency of the surfactant increases as the CMC of the surfactant decreases. This parameter, which determines the micelle behavior of the surfactant, was calculated with the following formula (Equation (3)). Considering the ΔG_{mic} values in Table 4, the fact that the free energy value of the compound **4b**, which has a higher hydrophobic character and therefore a lower CMC value, was found to be higher in the negative direction confirms this view (Aiad et al., 2016). That is, the micelle-forming tendency of the **4b** tri-cationic surfactant is greater than that of the other surfactant.

$$\Delta G_{mic} = RT \ln(\mathrm{CMC}) \quad (3)$$

The surface tensions (γ_{CMC}) of the CMCs of the two tri-cationic surfactants shown in Table 4 were measured by the known capillary rise method (Martino et al., 2006; Sugden, 1921). The purpose of determining surface tensions as a physicochemical parameter is to evaluate the surface activity of aqueous tri-cationic

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TABLE 4 Some physical properties of synthesized tri-cationic surfactants

Compounds	CMC ($\times 10^{-4}$ M)[a]	γ_{CMC} (dyn cm^{-1})[a,b]	π_{CMC} (dyn cm^{-1})	ΔG_{mis} (kj mol^{-1})
4a	7.38	16.49	55.48	−17.87
4b	4.19	12.58	59.39	−19.27

[a]Calculated at 25°C.
[b]Calculated at the critical micelle concentration of surfactant.

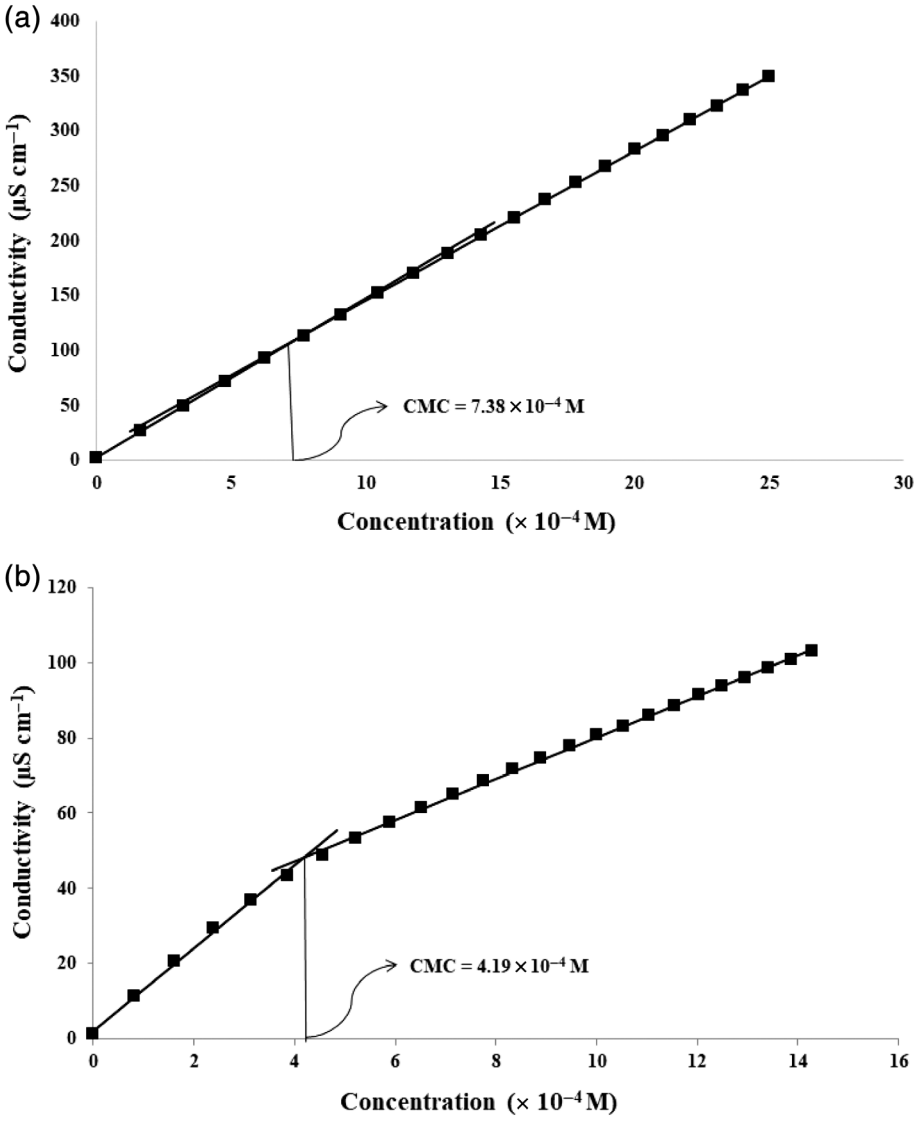


FIGURE 3 Graphs of conductivity values measured against inhibitor concentration

surfactant solutions at CMC. As seen in Table 4, the surface tension of surfactant **4b** in CMC was lower. This result shows that the compound **4b**, containing longer hydrophobic group in its structure, exhibits better surface activity than **4a**. It has been observed that the surface tension increases as the long carbon chain gets shorter. This situation was reflected in the corrosion inhibition results in a consistent manner. Compound **4b**, which showed better surface activity according to the surface tension findings, protected the metal from corrosion more than the other tri-cationic surfactant.

The π_{CMC} parameter given in Table 4 is defined as the efficiency in the reduction of surface tension and indicates the amount of reduction in surface pressure obtained in CMC (Öztürk et al., 2019). With this value calculated with the formula given in Equation (4), the effectiveness of the surfactant in reducing the surface tension of water is evaluated (Rosen, 1989).

$$\pi_{CMC} = \gamma_0 - \gamma_{CMC} \quad (4)$$

γ_0 is the surface tension of pure water (72.0 dyn cm^{-1} at 25°C) and γ_{CMC} is the surface tension of the aqueous

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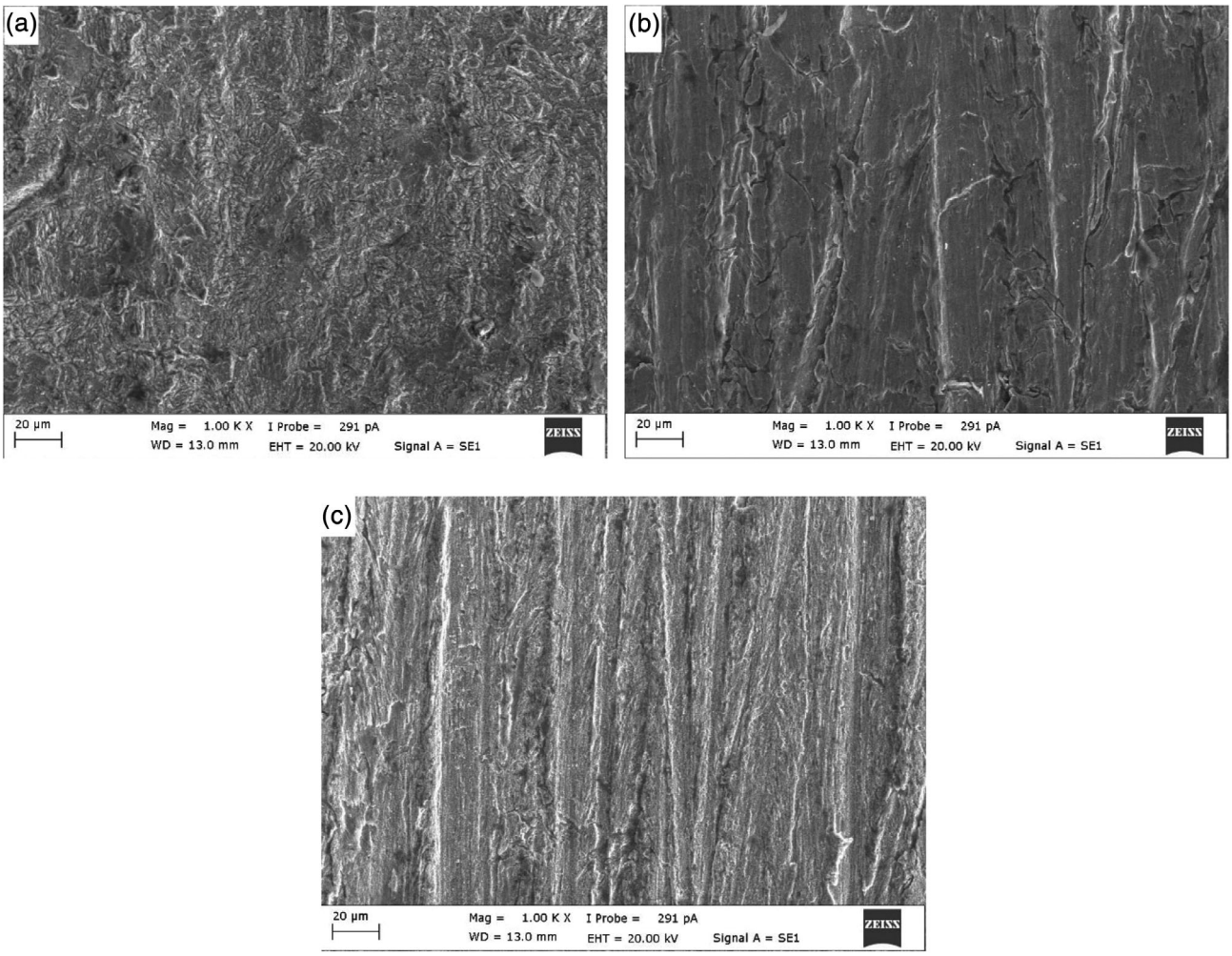


FIGURE 4 Surface images taken with SEM device of the metal coupon surfaces that kept for 24 h in (a) 1.0 M HCl medium without inhibitor, (b) 1.0 M HCl medium containing **4a** inhibitor, and (c) 1.0 M HCl medium containing **4b** inhibitor

solution of the surfactant at the CMC. For compounds synthesized as corrosion inhibitor (**4a–b**), this value was calculated as 55.48 and 59.39 dyn cm^{-1}, respectively. These results indicate that the synthesized tri-cationic surfactants have the potential to reduce the surface tension in the aqueous system. However, since the π_{CMC} value increases as the γ_{CMC} value decreases, it means that the activity of the surfactant increases with the increase of this value.

Scanning electron microscope

It is possible to support the IE results showing that tri-cationic surfactants resist metal corrosion in an acidic environment by taking images of the metal surface with scanning electron microscopy. Figure 4 shows the surface images of metal coupon surfaces immersed in acid solution with and without inhibitor for 24 h, taken with a SEM device at 1000 times magnification. SEM images were taken on square cut metal coupons with side lengths of 2.0 cm and were taken immediately after corrosion tests performing at room temperature for 24 h. The image in Figure 4a shows the metal surface immersed in the acid solution without inhibitor. It can be seen from the figure that; there is wear on the entire metal surface exposed to the acid environment without inhibitor, and the metal surface becomes indented and rough. This shows that corrosion occurs on the metal surface and corrosion products are formed on the surface. Figure 4b, c show images of metal surfaces immersed for 24 h at room temperature in acid solutions containing inhibitors **4a** and **4b**, respectively. There was no wear and roughness on the metal surfaces exposed to the acid environment with inhibitor. Scratches due to sanding before the test have been seen. This result proves that the inhibitor molecules are adsorbed on the metal surface to resist corrosion in the acidic environment.

Corrosion inhibition mechanism

There are several factors behind the inhibition of the synthesized tri-cationic surfactants against corrosion.

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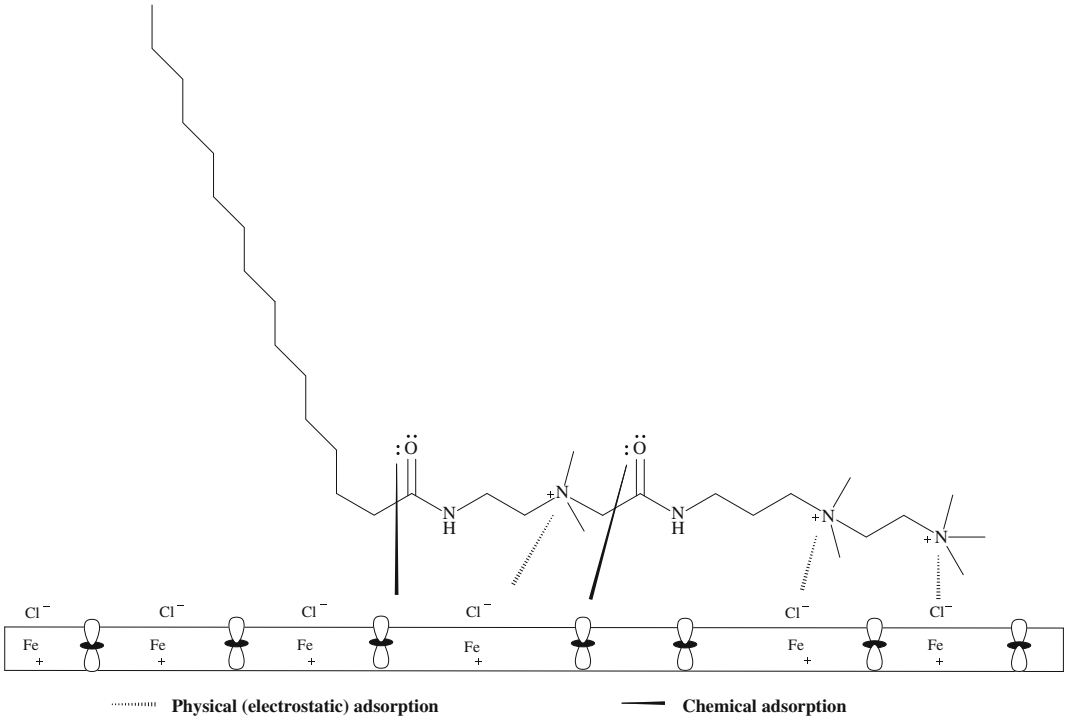


FIGURE 5 Mechanism of adsorption of the 4b inhibitor on the metal surface

One of them is that these compounds contain three positively charged nitrogen atoms in their structure, which will provide solubility in acidic aqueous medium. Because in order for an organic compound to be used as a corrosion inhibitor, it must first be dissolved in the corrosive environment (Papavinasam, 2000). As second, cationic surfactants must be well adsorbed on the metal surface. This adsorption, which will take place on the surface, forms the basis of corrosion protection. Because in this way, they form a protective film layer on the surface. Synthesized surfactants have the opportunity to be adsorbed on the metal surface both physically (electrostatic interaction) and chemically. Chemical adsorption is realized by the interaction between the unshared electron pairs in the O heteroatom, which is especially in the structure of the compounds, and the empty d orbitals of the Fe atoms in the metal plate (Öztürk et al., 2021). On the other hand, as a result of the oxidation of iron to +2 oxidation state in the anode reaction of corrosion, the positively charged metal surface creates dipoles with the adhesion of Cl^- ions from the acidic environment (Öztürk, 2013; Yıldırım et al., 2013). Physical adsorption occurs as a result of the electrostatic effect of the positively charged nitrogen atoms in the compounds on the metal surface, which has become negative thanks to the dipoles. It is possible to see this physical and chemical adsorption, which causes inhibition against corrosion by allowing tri-cationic surfactants to adsorb to the metal surface, in Figure 5.

One of the factors that protect the metal surface against corrosion in the acidic environment of the tri-cationic surfactants obtained to act as corrosion inhibitors is that they contain a long carbon chain that will keep the corrosive water molecules in the HCl aqueous solution away from the metal surface. This long carbon chain is hydrophobic (does not like water), is oriented perpendicular to the surface as seen in Figure 5, and protects the surface by not bringing water molecules closer to the metal surface. As the carbon chain length in the structure of the surfactant increases and it continues to dissolve in the corrosive environment, the IE also increases. This is why compound **4b** shows higher inhibition.

CONCLUSIONS

The synthesized two tri-cationic surfactants have been exhibit good corrosion inhibition activity in 1.0 M HCl solution. They exhibited a corrosion inhibition comparable to tricationic surfactants synthesized in other studies, by corrosion inhibiting efficiencies of 85.86%–92.88%. Between the two inhibitors, it was determined that the **4b** tri-cationic surfactant, which contains a longer carbon chain (C16) in its structure, protects the metal surface more against corrosion, depending on the carbon chain length they contain. Accordingly, the CRs were lower on the metal surface immersed in the acidic medium containing compound **4b**, since the

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same compound inhibited corrosion more. This confirms the information that the surfactant, which has more hydrophobic properties, protects the metal surface against corrosion by not bringing the acidic aqueous medium closer to the metal surface. As in many other studies, the CMC of the compound (**4b**), which has more hydrophobic character, and the surface tension at this concentration were found to be lower. With this result, we can say that the surfactant with a longer carbon chain has more micelle formation efficiency and therefore, the surfactant property is more dominant. Thanks to the SEM images, the fact that no deformation and corrosion products are formed on the metal surfaces immersed in the acidic environment containing tricationic surfactant for 24 h, has proven to us that these materials show surface activity and anti-corrosion properties.

AUTHOR CONTRIBUTIONS

Serkan Öztürk: Design of tri-cationic surfactants, synthesis of surfactants, corrosion tests via gravimetric measurements, determination of physicochemical parameters, SEM analysis, writing and reviewing of draft manuscript, editing of manuscript. **Nuray Altınölçek**: Synthesis of surfactants, corrosion tests via gravimetric measurements, determination of physicochemical parameters, reviewing of draft manuscript. All authors reviewed the results and approved the final version of the manuscript.

CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

ETHICS STATEMENT

This research falls outside of human or animal studies and institutional ethical approval was not required.

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How to cite this article: Öztürk S, Altınölçek N. Synthesis of tri-cationic surfactants containing two amide functional groups and inhibition effectiveness against metal corrosion in acidic medium. J Surfact Deterg. 2022;25(6):821–32. https://doi.org/10.1002/jsde.12614

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