

# **Synthesis of Aromatic Structured Di-cationic Surfactants Used as Inhibitors Against Corrosion of Carbon Steel in Acidic Medium**

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**Abstract:** In this study, three aromatic-structured di-cationic surfactants of different carbon chain lengths were synthesized and their inhibition efficiencies against metal corrosion in 1.0 M HCl solution were investigated. Corrosion inhibition efficiencies of di-cationic surfactants, the structures of which were elucidated by spectroscopic methods (FT-IR and NMR), were determined in acidic media by gravimetric method based upon weight loss in metal coupons. Corrosion tests resulted in good inhibition efficiencies (90.40% - 96.85%) against metallic corrosion at different concentrations of inhibitors. By using the weight loss values, corrosion rates were also calculated in inhibited and uninhibited medium. Finally, images of metal surfaces were obtained using scanning electron microscopy to give support to the data on the corrosion inhibition effect.

**Keywords:** Di-cationic surfactants, carbon steel, corrosion inhibitor, acidic medium, SEM.

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## **INTRODUCTION**

Metallic corrosion is the deformation of metals in their environment, which occurs on the surfaces of the metals as a result of chemical and electrochemical attacks (1). The tendency of metals and alloys to turn into stable states of the compound causes metal corrosion leading to serious economic losses. Corrosion in the metals consists of two electrochemical reactions. The metal is oxidized at the anode while the positively charged hydrogen ion from the acidic medium is reduced to  $H_2$  gas at the cathode.

A large number of corrosion types are known in the literature (2). The most common corrosion type occurring in low pH (acidic) environments is called "general corrosion" (3). One of the most commonly used methods to overcome this type of corrosion is the addition of an organic corrosion inhibitor to the corrosive medium. Corrosion inhibitors dissolve or disperse in corrosive media (4) and generally form a protective film layer by adsorbing onto the metal surface. This slows down the corrosion rate by slowing down the rates of either the anode or cathode reactions or both of them.

In recent years, it has been established that cationic surfactants act as the most effective inhibitors against corrosion occurring on iron and steel materials in acidic environments (5-8). In particular, cationic quaternary ammonium surfactants have been reported to play a significant role in preventing corrosion in iron and steel materials in acidic environments (9-12). Therefore, there has been a growing number of studies on the synthesis of di-cationic surfactants containing more than one positive group and their use as corrosion inhibitors in the acidic medium (15-21).

In this study, three aromatic di-cationic surfactants with different carbon chain lengths were synthesized and their structures were elucidated by FT-IR and NMR techniques. The aim of the study was to determine the corrosion inhibition activities of di-cationic surfactants in the acidic corrosive environment by the gravimetric method based on following the weight loss in metal coupons (in accordance with ASTM

NACE / ASTMG31-12a standard method (22)). For this purpose, corrosion tests were carried out using low carbon steel at room temperature for 24 h at various inhibitor concentrations.

## **MATERIALS AND METHODS**

## **Materials and Instrumentation**

Ethyl-(4-dimethylamino)benzoate, corresponding long-carbon-chain amines, ethanol, and other solvents used in the synthesis part of this work were obtained from Merck and Sigma-Aldrich and used without further purification. FT-IR spectra of the synthesized compounds were recorded using Thermo Nicolet 6700 FT-IR spectrometer. NMR spectra ( $^{1}$ H NMR and  $^{13}$ C NMR) of the compounds were obtained in DMSO (dimethylsulfoxide) solvent with Agilent 600 MHz Premium Compact NMR spectrometer. After the corrosion test, images of the metal surface were taken with a Carl Zeiss EVO 40 model scanning electron microscope. The NUVE EV 018 vacuum oven was used to dry metal coupons to a constant weight in air-free environment. Elemental analyses were performed using a LECO CHNS-932 instrument.

## **Synthesis of Compounds**

*Synthesis of 4-(dimethylamino)-Noctylbenzamide (1a):* 2.0 g (10.35 mmol) of ethyl-(4-dimethylamino)benzoate and 1.338 g (10.35 mmol) of octylamine reagents were placed in a 50 mL reaction flask. The flask was heated overnight at 150 °C under reflux without solvent. At the end of the reaction, the flask was cooled down to room temperature. A small amount of DMF was added to the cooling reaction mixture, and then cold water to precipitate the crude product. The crude product, after filtering off under vacuum and drying, was crystallized from a mixture of methanol / water (9:1). The yellow solid product was obtained in 82% yield.

*Synthesis of 4-(dimethylamino)-Ndecylbenzamide (1b):* The synthesis of compound **1b** was performed with the same procedure used for **1a**, using 2.0 g (10.35 mmol) of ethyl-(4-dimethylamino)benzoate and 1.628 g (10.35 mmol) of decylamine reagents. The yellow solid product was obtained with a yield of 62%.

*Synthesis of 4-(dimethylamino)-Ndodecylbenzamide (1c):* The synthesis of compound **1c** was carried out applying the same procedure used for **1a**, using 2.0 g (10.35 mmol) of ethyl-(4-dimethylamino)benzoate and 1.918 g (10.35 mmol) of dodecylamine reagents. The yellow solid product (yield: 69%) was obtained.

*Synthesis of N,N,N,N',N'-pentamethyl-N'-[4- (octylcarbamoyl)phenyl]ethane-1,2-diaminium dibromide (2a):* 2.0 g (7.23 mmol) of compound **1a**, 1.786 g (7.23 mmol) of (2 bromoethyl)trimethylammonium bromide and 20 mL of ethanol as solvent were placed in a 50 mL reaction flask. The mixture was heated at  $100 °C$ under reflux for 48 h. At the end of the reaction,

ethanol was evaporated under vacuum. The reaction mixture was washed several times with ether and a light brown solid wax was obtained as the product. FT-IR (KBr), vibrational modes, v/cm-1: 3370 (amide N-H), 3013 (aromatic C-H), 2962, 2904 ve 2871 (aliphatic C-H), 1691 (amide C=O). <sup>1</sup>H NMR data (600 MHz, DMSO-d<sub>6</sub>): δ  $(ppm) = 7.74$  (d, 2H, Ar-H), 6.68 (d, 2H, Ar-H), 4.20 (quartet, 2H,  $CH_2$ -CH<sub>2</sub>-NH-CO), 3.31 (t, 2H, Ar-N<sup>+</sup>-CH<sub>2</sub>-CH<sub>2</sub>-N<sup>+</sup>), 3.24 (t, 2H, Ar-N<sup>+</sup>-CH<sub>2</sub>-CH<sub>2</sub>- $N^{+}$ ), 3.10 (s, 6H,  $(C_1H_3)_2-N^{\dagger}-CH_2-CH_2-N^{\dagger}-(CH_3)_3$ ), 2.97 (s, 9H, CH<sub>2</sub>-N<sup>+</sup>-(CH<sub>3</sub>)<sub>3</sub>), 0.83 (t, 3H, CH<sub>3</sub>). <sup>13</sup>C NMR data (150 MHz, DMSO-d<sub>6</sub>): δ (ppm) = 166.32 (HN-CO), 153.63 (Ar-C), 131.12 (Ar-C), 116.48 (Ar-C), 111.21 (Ar-C), 60.05 (-N<sup>+</sup>-CH<sub>2</sub>- $CH_2-N+$ ), 53.96 ( $-N+CH_2-CH_2-N+$  $CH_3$ )<sub>3</sub>), 53.31  $(-({\rm CH}_3)_2-N^+-{\rm CH}_2-{\rm CH}_2-N^+-(\rm CH}_3)_3)$ , 39.27  $(-{\rm CH}_2 NH-CO$ ), 22.51 (CH<sub>3</sub>-CH<sub>2</sub>-), 14.80 (CH<sub>3</sub>-CH<sub>2</sub>-).

*Synthesis of N,N,N,N',N'-pentamethyl-N'-[4- (decylcarbamoyl)phenyl]ethane-1,2-diaminium dibromide (2b):* The synthesis of compound **2b** was performed with the same procedure used for **2a**, using 1.65 g (5.42 mmol) of compound **1b**, 1.338 g (5.42 mmol) of (2 bromoethyl)trimethylammonium bromide and 20 mL of ethanol as solvent. A light brown solid wax product was obtained. FT-IR (KBr), vibrational  $\frac{1}{2}$  modes, v/cm<sup>-1</sup>: 3372 (amide N-H), 3015 (aromatic C-H), 2950, 2917 ve 2847 (aliphatic C-H), 1691 (amide C=O). <sup>1</sup>H NMR data (600 MHz, DMSO-d<sub>6</sub>): δ (ppm) = 7.74 (d, 2H, Ar-H), 6.69 (d, 2H, Ar-H), 4.20 (quartet, 2H,  $CH_2-CH_2-NH-$ CO), 3.32 (t, 2H, Ar-N<sup>+</sup>-CH2-CH2-N<sup>+</sup>), 3.25 (t, 2H,  $Ar-N+-CH_{2}-CH_{2}-N^{+}),$  3.10 (s, 6H,  $(CH_{3})_{2}-N^{+}-CH_{2} CH_2-N^+-(CH_3)_3$ , 2.97 (s, 9H,  $CH_2-N^+-(CH_3)_3$ ), 0.83 (t, 3H, CH<sub>3</sub>). <sup>13</sup>C NMR data (150 MHz, DMSO $d_6$ ): δ (ppm) = 166.32 (HN-CO), 153.64 (Ar-C), 131.11 (Ar-C), 116.48 (Ar-C), 111.21 (Ar-C), 60.04  $(-N^+\text{-}CH_2\text{-}CH_2-N^+)$ , 53.95  $(-N^+\text{-}CH_2\text{-}CH_2-N^+$ -  $(-(CH_3)_3)$ , 53.31  $(-(CH_3)_2-N^+\text{-}CH_2\text{-}CH_2-N^+$ 53.31  $(-(CH<sub>3</sub>)<sub>2</sub>-N<sup>+</sup>-CH<sub>2</sub>-CH<sub>2</sub>-N<sup>+</sup> (CH<sub>3</sub>)<sub>3</sub>$ ), 39.25 (-CH<sub>2</sub>-NH-CO), 22.53 (CH<sub>3</sub>-CH<sub>2</sub>-), 14.80 (CH<sub>3</sub>-CH<sub>2</sub>-).

*Synthesis of N,N,N,N',N'-pentamethyl-N'-[4- (dodecylcarbamoyl)phenyl]ethane-1,2-*

*diaminium dibromide (2c):* The synthesis of compound **2c** was performed with the same procedure used for **2a**, using 0.30 g (0.90 mmol) of compound **1c**, 0.223 g (0.90 mmol) of (2 bromoethyl)trimethylammonium bromide and 20 mL of ethanol as solvent. FT-IR (KBr), vibrational modes, v/cm-1: 3411 (amide N-H), 3015 (aromatic C-H), 2950, 2917 ve 2848 (aliphatic C-H), 1691 (amide C=O). <sup>1</sup>H NMR data (600 MHz,  $DMSO-d<sub>6</sub>)$ : δ (ppm) = 7.73 (d, 2H, Ar-H), 6.69 (d, 2H, Ar-H), 4.20 (quartet, 2H, CH<sub>2</sub>-CH<sub>2</sub>-NH-CO), 3.31 (t, 2H, Ar-N<sup>+</sup>-CH<sub>2</sub>-CH<sub>2</sub>-N<sup>+</sup>), 3.24 (t, 2H, Ar-N<sup>+</sup>-CH<sub>2</sub>-CH<sub>2</sub>-N<sup>+</sup>), 3.10 (s, 6H,  $(CH_3)_2-N^{\dagger}$ -CH<sub>2</sub>- $CH_2-N^+-(CH_3)_3$ , 2.97 (s, 9H,  $CH_2-N^+-(CH_3)_3$ ), 0.83 (t, 3H,  $\overline{CH_3}$ ). <sup>13</sup>C NMR data (150 MHz, DMSO $d_6$ ): δ (ppm) = 166.30 (HN-CO), 153.62 (Ar-C), 131.11 (Ar-C), 116.48 (Ar-C), 111.21 (Ar-C), 60.05 (-N+- $CH_2$ -CH<sub>2</sub>-N+-), 53.95 (-N+-CH<sub>2</sub>-CH<sub>2</sub>- $N^+$ -(CH<sub>3</sub>)<sub>3</sub>), 53.32 (-(CH<sub>3</sub>)<sub>2</sub>-N<sup>+</sup>-CH<sub>2</sub>-CH<sub>2</sub>-N<sup>+</sup>-

 $(CH<sub>3</sub>)<sub>3</sub>$ ), 39.25 (-CH<sub>2</sub>-NH-CO), 22.53 (CH<sub>3</sub>-CH<sub>2</sub>-), 14.80 (CH<sub>3</sub>-CH<sub>2</sub>-).

#### **Corrosion Test Based on Gravimetric Measurement**

*Metal and its properties used in corrosion tests:*  Metal plates used in the corrosion tests in acidic media were prepared from low-carbon cold-rolled steel. The steel material used conforms to the norm of DIN EN 10130 (23) with the content composition of 0.07% (C), 0.35% (Mn), 0.015% (P), 0.015% (S) and the rest is Fe. The metal plates were cut in rectangular shape with the thickness of 0.1 cm, the width of 2.2 cm and the length of 5.0 cm.

*Preparation of metal plates before corrosion test:* The metal plates of 0.1 cm thickness, 2.2 cm width and 5.0 cm length, were immersed in 15% HCl solution for a short time to clean the rust stains and oxide layer. The plates were then cleaned with distilled water and briefly stored in acetone. Plates removed from acetone were dried in a vacuum oven until constant weight and the mass of the metal plate was recorded before testing.

## **Corrosion test in acidic environment**

Corrosion tests in 1.0 M HCl solution were carried out following the relevant standard method (22). Before testing, 1.0 M HCl solution was freshly prepared. The di-cationic surfactant solutions were prepared in concentrations of 10, 25, 50, 100 and 250 ppm in 100 mL of 1.0 M HCl medium. These solutions were poured into 150-mL sealed glass bottles. The coupons were kept in the solutions without stirring for 24 h at room temperature. Control tests were performed in the same way without the inhibitors. After 24 h test period, metal plates were removed from the corrosive medium and were rinsed with distilled water. The plates were then cleaned with acetone, dried in a vacuum oven and the mass of the metals were measured after testing.

*Calculation of percent inhibition efficiencies and corrosion rates:* The percent inhibition efficiencies (% IE) of the aromatic structured di-cationic surfactants tested were calculated by the formula given below:

IE % = 
$$
\frac{W_0 - W}{W_0} \times 100
$$
 (Eq. 1)

IE % is the percentage inhibition efficiency,  $W_0$ and W represent the weight losses of the coupon in the absence and presence of an inhibitor in the same environment, respectively. However, using the weight loss values, the corrosion rates in the uninhibited and inhibited corrosive media were calculated by the following formula (24, 25):

$$
CR = \frac{\Delta W}{At}
$$
 (Eq. 2)

CR is the corrosion rate; ΔW is the weight loss (mg); A is the surface area of the coupon  $\text{(cm}^2)$ and t is the time of immersion (h).

### **SEM analysis**

The SEM image of the metal surface was taken on the metal coupons that were square-shaped with the thickness of 0.1 cm, the width of 2.0 cm and the length of 2.0 cm. After performing the corrosion tests in the acidic medium, the metal coupons were washed with acetone and then dried in a vacuum oven. Subsequently, the SEM images of the metal surfaces were obtained, using scanning electron microscope under high vacuum at 1000x magnification.

## **RESULTS AND DISCUSSION**

#### **Synthesis of di-cationic surfactants**

The synthetic scheme of the aromatic-structured di-cationic surfactants that act as corrosion inhibitors in the acidic environment are shown in Figure 1.



**Figure 1.** Synthesis scheme of the aromatic di-cationic surfactants.

As can be seen in Figure 1, the synthesis of organic surfactants were carried out in two steps. In the first step, the ethyl-(4 dimethylamino)benzoate compound was reacted with the corresponding long carbon chain amine compound in a solvent-free environment to give compounds **1a-c**. The compounds obtained in the first step were reacted in ethanol with the (2 bromoethyl)trimethylammonium bromide in the second step to reach the target products **2a-c** dicationic surfactants. The elemental analysis results of the synthesized **2a-c** compounds and some other physical properties are given in Tables 1 and 2, respectively.



**Table 2.** Some physical properties of the synthesized di-cationic surfactants.



The structures of the synthesized **2a-c** surface active compounds were determined by FT-IR, <sup>1</sup>H NMR, and <sup>13</sup>C NMR spectroscopic methods. FT-IR

and NMR spectra ( ${}^{1}$ H and  ${}^{13}$ C NMR) of compound **2b** are given in Figures 2 and 3, respectively.





**Figure 3.** <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra of compound **2b.**

The FT-IR spectra of (**2b**) showed absorption bands at 3372 cm<sup>-1</sup> (amide N-H), at 3015 cm<sup>-1</sup> (aromatic C-H), at 2950, 2917, 2847 cm-1 (aliphatic C-H) and at  $1691$  cm<sup>-1</sup> (amide C=O). In the <sup>1</sup>H NMR spectrum of **2b** (Figure 3), there is a doublet peak at 7.74 ppm for the two hydrogen atoms which are connected to aromatic carbon labeled with a, a doublet peak at 6.69 ppm for the two hydrogen atoms of aromatic carbon labeled with b, a triplet peak at 3.32 ppm for the two hydrogen atoms of the  $(Ar-N^+-CH_2-CH_2-N^+)$ group, a singlet peak at 3.10 ppm originating from the six hydrogen atoms of the  $(CH_3)_2-N^+$ - $CH_2-CH_2-N^{\dagger}-(CH_3)_3$  group and a singlet peak at 2.97 ppm ascribed to the nine hydrogen atoms of the  $CH_2-N^+$ -( $CH_3$ )<sub>3</sub>. The triplet peak at 3.32 ppm in the spectrum proved that the di-cationic surfactant (**2b**) was successfully synthesized.

#### **Corrosion test results in acidic media**

The corrosion test data of the synthesized **2a-c** aromatic structured di-cationic surfactants in 1.0 M HCl are listed in Table 3. The corrosion rate and inhibition efficiency values given here were calculated from the weight loss values determined for the metal coupons after the corrosion tests for 24 h.

It can be seen from Table 3 that the values of weight loss and corrosion rate of the metal coupons in uninhibited medium (0 ppm) are high, while they are quite low in the inhibited medium (10, 25, 50, 100 and 250 ppm). Due to the large differences in weight loss and corrosion rate between inhibited and uninhibited media, the inhibition efficiencies of the synthesized compounds became high. The plots of corrosion rate and inhibition efficiency calculated at different concentrations, versus inhibitor concentration are shown in Figure 4.



**Table 3.** Weight loss, corrosion rate and corrosion inhibition efficiencies (IE %*)* calculated for different concentrations of compounds **2a-c** in 1.0 M HCl medium for 24 h at room temperature.

**\*** Mean of the two measured values.



Figure 4. Plots of corrosion rate and inhibition efficiency against inhibitor concentration from weight loss measurements after immersed in 1.0 M HCl.

#### **SEM analysis results**

Surface analysis by scanning electron microscopy is of great importance for observing the morphological changes on metal surfaces. Through examination of these morphological changes, the interactions between the organic molecules and the metal surface can be explained. The 1000x magnified surface images of the metal coupon surfaces immersed to inhibitor-free acid solution and to acid solution with inhibitor for 24 h, are shown in Figure 5.

It may be clearly seen in Figure 5 that the metal surface exposed to acidic environment without inhibitor is exposed to wear and the surface

becomes rough. In this environment containing cationic surfactants **2a**, **2b** and **2c**, there is no wear and roughness on the metal surfaces that have been left for 24 h, and it appears to have a smoother appearance. The scratch marks on the metal surfaces are caused by the sanding process made before the test. This proves that the inhibitor molecules are adsorbed onto the metal surface and protect the metal surface against corrosion. Hence, the SEM images of the metal surface obtained support the high inhibition activity results shown in Table 3.



**Figure 5.** SEM images of the metal coupon surfaces, which were kept in a 1.0 M HCl medium without inhibitor and with 100 ppm inhibitor for 24 h.

#### **The mechanism of corrosion inhibition**

The first step in the mechanism of action to retard or prevent corrosion is the adsorption of surfactant molecules onto the metal surface. The adsorption process depends on the nature and surface load of the metal used, the chemical structure of the surface active substance, the nature of both the surface active substance and the corrosive environment (26). In the structure of the synthesized aromatic structured di-cationic surfactants, there are two positively charged nitrogen atoms acting as the hydrophilic group. This hydrophilic group leads both to dissolution of the compounds in the aqueous acid environment and adsorption to the metal surface by electrostatic forces between the positively

charged nitrogen atom and the induced negative charges on the metal surface (27). The induced negative charge on the metal surface is provided by the formation of dipoles on the surface. These dipoles arise as a result of attachment to the positively charged metal surface of negatively charged halogen ions from the acidic medium by electrostatic attraction (13, 14). Due to the dipoles formed on the surface, the aromatic surface-active di-cationic surfactants can be easily attached to the metal surface which becomes negatively charged. Therefore, a protective monomolecular layer is formed against the corrosive nature of the acidic environment on the surface of the carbon steel.

Besides the physisorption process on the metal surface due to the electrostatic attraction force, the chemisorption process also plays an important role on the corrosion inhibition in the acidic environment. Chemisorption occurs via the interaction of the benzene ring and the N and O heteroatoms in the structure of the surface active materials with the iron atoms on the metal surface separately. Non-bonding electrons in the p orbital of the heteroatoms (N and O) and πelectrons in the aromatic benzene ring are involved in chemisorption. Due to the donoracceptor interactions between these electrons and the free d orbital in the Fe atoms on the metal surface, the surfactant molecules adsorbed onto the metal surface with coordinated covalent bonds (28). The adsorption of the synthesized inhibitors to the metal surface is favoured by these coordinated covalent bonds and provides a stronger protection against corrosion.

The long alkyl chain containing amide functional group of the synthesized aromatic structured dicationic surfactants constitutes the hydrophobic part of the di-cationic surfactant. This part is oriented perpendicular to the metal surface so as not to bring the water molecules coming from the corrosive environment closer to the surface.

These hydrophobic groups, oriented perpendicular to the metal surface, form nonpolar interactions originating from van der Waals attraction forces. Thus, double layered or multilayered protective layers are formed which act as a barrier between the metal surface and the acidic corrosive medium (13). It is known that non-polar interactions between the long alkyl chains in the molecule increase in parallel with the concentration of surfactant (14). Therefore, as the concentration increases, the inhibition efficiency of the surface active compound against corrosion also increases. In Table 3, a slight increase in the inhibition efficiency value with an increase in the concentration of compound **2b** in the 1.0 M HCl medium is a result of this situation. On the other hand, the inhibition efficencies of the compounds **2a** and **2c**, in particular, does not increase markedly with the increase in the concentration, which means that the di-cationic surfactants reach the critical micelle concentration and are in the form of micelles at the interface of the metal in the working concentrations. The possible orientations and inhibition mechanisms of the compounds **2a**, **2b** and **2c** on the metal surface are shown in Figures 6 and 7, respectively.



**Figure 6.** The possible orientations and inhibition mechanism of the compound **2b**.

## **CONCLUSIONS**

Newly synthesized three aromatic di-cationic surfactants having different carbon chain lengths act as inhibitors against corrosion of the carbon steel in 1.0 M HCl solution. In the light of the data obtained in the study, the following conclusions can be drawn:

(1) All the synthesized aromatic structured dicationic surfactants exhibited good inhibition activities under working concentrations. (2) The inhibitory efficiency of compound **2b**

increased with the increasing of the concentration.

(3) The inhibitory efficiencies of the compounds **2a** and **2c** increased slightly up to a certain concentration, and then decreased afterwards.

(4) Physisorption seemed to be more effective than chemisorption on the corrosion behavior of the synthesized aromatic structured dicationic surfactants.

(5) Corrosion inhibition activities of the synthesized inhibitors in acidic medium were supported by surface imaging technique (SEM).



**Figure 7.** The possible orientations and inhibition mechanism of the compounds **2a** and **2c**.

## **CONFLICT OF INTEREST**

The authors declare no conflict of interest.

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