



Study the Inhibition effect of Three Newly Synthesized Schiff Base Based Cationic Surfactants on Aluminum Corrosion in 0.5 M HCl Solution

Doaa F. Seyam^a, Salah Eid^{b,a}, A. Y. El-Etre^a, Ahmed H. Tantawy^a

^aChemistry department, Faculty of science, Benha University, Benha 13518, Egypt

^bChemistry department, College of Science and Arts, Alqurayat, Jouf University, KSA



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Abstract

Three novel Schiff base based cationic surfactants, namely, (*E*)-3-(decylideneamino)-*N,N,N*-trimethylpropan-1-aminium methyl sulfate (DSMS), (*E*)-*N*-(3-(decylideneamino)propyl)-*N,N*-dimethylbutan-1-aminium bromide (DSBB) and (*E*)-3-(decylideneamino)-*N*-ethyl-*N,N*-dimethylpropan-1-aminium iodide (DSEI) were synthesized and their chemical structures were assessed. The surface activities of the target prepared surfactants were investigated. Based on gravimetric, gasometric and electrochemical techniques, their inhibition impact on aluminum corrosion in solution with 0.5 M HCl was studied. The effect of raising both temperature and surfactant concentration on the inhibition efficiency was studied and the thermodynamic functions were measured and discussed. Langmuir adsorption isotherm fitted widely with the empirical data.

Keywords: Cationic surfactants; Critical micelle concentration; Corrosion inhibition; Aluminum.

1. Introduction

Because of its low price, low density, and excellent peculiarities, aluminum and its alloys is a major performer in the engineering industry. However, similar to other metals, aluminum is susceptible to corrosion. Consequently, the corrosive behavior of aluminum and its alloys in different media has been investigated. One of the most effective ways to reduce corrosion is to use inhibitors [1, 2].

Surfactant is a sort of corrosion inhibitor that includes both hydrophilic and hydrophobic constituents. The hydrophilic portion, unlike the hydrophobic portion, can interact with aqueous solutions, while the hydrophobic portion repels water. Surfactant molecules adsorb at the water/air interface, reducing surface tension, and after saturating the water/air interface, molecules begin to form aggregates or micelles [3, 4]. Surprisingly, it has been discovered that surfactant molecule adsorption on mineral surfaces is directly related to its ability to accumulate and form micelles, and thus preventing metal corrosion [5-7]. [8] studied the inhibitory effect of the cationic surfactant cetyl trimethylammonium chloride (CTAC) on aluminum in 0.2 M HCl, and found that the inhibitory effect increased with increasing concentration up to 55%. [9] synthesized and tested a novel series of self-assembled nonionic Schiff base amphiphiles as corrosion inhibitors for

aluminum in 4N HCl and the results showed the maximum corrosion inhibition efficiency at 55.28%. [10] evaluated anionic surfactants as corrosion inhibitors for aluminum in 1 M HCl and found that they were effective up to 86.9%. [11] synthesized a polymeric surfactant and tested it as a corrosion inhibitor for aluminum in 0.5 M HCl, with findings showing a maximum inhibition efficiency of 88.7%. [12] tested a series of aniline monomeric surfactants and their analog polymers in 0.5 M HCl solution and these surfactants revealed a maximum value of 71.3%. The authors [6] synthesized amido amine based cationic surfactant (DAEI) for aluminum corrosion inhibition with a maximum IE% (90.07%). [13] studied the corrosion of pure aluminum in 1 M HCl with two synthetic nonionic surfactant compounds having heterocyclic a six-membered ring moiety, and the results revealed inhibition efficiency of up to 95.4%. [14] synthesized and studied nonionic surfactant compounds with a five-membered heterocyclic moiety, and they found that these surfactants had high efficiency, with a maximum of 93.79%. The authors [15] synthesized and examined two amido-amine based cationic surfactants (DABB and DAMS) for aluminum corrosion inhibition with maximum inhibition efficiencies (92.72% and 95.24%).

The goal of our work is the study of the inhibition impact of (*E*)-3-(decylideneamino)-*N,N,N*-

*Corresponding author e-mail: doaaafouadms@gmail.com

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trimethylpropan-1-aminium methyl sulfate (DSMS), (E)-N-(3-(decylideneamino)propyl)-N,N-dimethylbutan-1-aminium bromide (DSBB) and (E)-3-(decylideneamino)-N-ethyl-N,N-dimethylpropan-1-aminium iodide (DSEI) on the aluminum corrosion in 0.5 M HCl solution making use of potentiodynamic polarization, weight loss as well as hydrogen evolution methods. Their influence on the thermodynamic parameters of the corrosion process was also measured and discussed. These surfactants are a modification of amido-amine based cationic surfactants, synthesized and tested by the authors as corrosion inhibitors for aluminum in the same conditions of the present work with excellent results. The impact of this modification on the effectiveness of these surfactants was studied.

2. Experimental

2.1. Materials

The metal used is aluminum 1050, given by the Egyptian Copper Works Company, Egypt, with the components in (w/w %): Cu (0.05 max), Al (99.50 min), Si (0.25 max), Fe (0.4 max), Ti (0.03 max), V (0.05 max), Mn (0.05 max), Mg (0.05 max), and Zn (0.05 max).

Butyl bromide, decanoic acid and Decanal were bought from Acros Organics, Geel, Belgium. HCl (37%) with density of 1.185 g/cm³, Dimethyl sulfate and ethyl iodide were purchased from Sigma-Aldrich Company, St. Louis, USA. Diethylether, acetone, methylene chloride (98%) and triethylamine have superior quality and were bought from EL-NASR Pharmaceutical Chemicals Company, Abu-Zabaal, Cairo, Egypt. The FT-IR spectra are used to identify the structure of the prepared surfactants in potassium bromide on a Thermos Nicolet IS10 FT-IR Spectrophotometer, Thermo Fisher Scientific Inc., Waltham, MA, USA. The measurements of NMR spectra were done via Bruker Avance III, Billerica, Massachusetts, USA, with 400 MHz for ¹H NMR and 100 MHz for ¹³C NMR. Also, tetramethylsilane (TMS) as an internal reference was utilized.

By diluting the measured concentration with the appropriate amount of distilled water, 0.5 M HCl solutions can be produced.

2.2. Synthesis of Novel Schiff Base Based Cationic Surfactants

Into a conical flask, 1.12 mmol of *N*¹,*N*¹-dimethylpropane-1,3-diamine and 1.11 mmol of decanal were mixed in 50 ml ethanol and refluxed for 3 hrs under uninterrupted stirring in the presence triethylamine as appropriate base catalyst. The completion of reaction was detected by TLC. Then, the evaporation of solvent under vacuum was carried out to produce the target viscous liquid. The product was purified via recrystallization process by dissolving it in 20 ml of methanol with stirring and

heated to complete the dissolution process, leave the solution to cool and the precipitate will be formed [yellowish white solid, 85%, (E)-*N*¹-decylidene-*N*³,*N*³-dimethylpropane-1,3-diamine].

(E)-*N*¹-decylidene-*N*³,*N*³-dimethylpropane-1,3-diamine (1.1 mmol) is dissolved in 5 ml dry acetone and introduced into three flasks separately, 1.5 mmol of ethyl iodide, butyl bromide and dimethyl sulfate were dropped wisely added and dissolved in dry acetone (2 ml), then the solution mixture was refluxed for 72 h. The reaction mixture was finished by evaporating the solvent, then the residue dried under reduced pressure and the product was washed three times using diethyl ether to separate the undesired materials to form a white and pale brown precipitate [(E)-3-(decylideneamino)-*N*,*N*,*N*-trimethylpropan-1-aminium methyl sulfate (DSMS), (E)-*N*-(3-(decylideneamino)propyl)-*N*,*N*-dimethylbutan-1-aminium bromide (DSBB) and (E)-3-(decylideneamino)-*N*-ethyl-*N*,*N*-dimethylpropan-1-aminium iodide (DSEI)] (**Scheme 1**).

3-Decylideneamino-Propyl-trimethyl-ammonium methyl sulphate (DSMS): Yellowish-white viscous liquid, yield = 87%, FT-IR (KBr pellet) cm⁻¹: 2854-2956 (ν_{C-H} of aliphatic fatty chain), 1634 (ν_{C=N} of imine). ¹H NMR-400 MHz, (DMSO-*d*₆): δ ppm: 0.86 (t, 3H, **CH**₃-CH₂), 1.24 (s, fatty chain CH₃-(**CH**₂)₆), 1.62 (m, 2H, **CH**₂-CH₂-C=N), 1.74 (m, 2H, **CH**₂-CH₂-N=C) 2.78 (t, 2H, CH₂-**CH**₂-C=N), 2.87 (t, 2H, CH₂-**CH**₂-N=C), 3.15 (t, 2H, CH₂-**CH**₂-N⁺), 3.41 (s, 9H, (**CH**₃)₃-N⁺), 8.86 (t, 1H, CH₂-**CH**=N). ¹³C NMR-100 MHz, δ_C (ppm) (DMSO-*d*₆): 14.32 [**CH**₃-CH₂], 22.55 [fatty chain, (**CH**₂)₅], 28.23 [**CH**₂-CH₃], 29.38 [**CH**₂-CH₂-C=N], 30.28 [**CH**₂-CH₂-N=C], 31.76 [N=C-**CH**₂], 53.23 [(**CH**₃)₃-N⁺], 62.23 [**CH**₂-N=C], 62.85 [**CH**₂-N⁺], 141.98 [**CH**=N, imines].

2.3. Critical Micelle Concentration and Surface-Active Properties

The measurements of surface tension were done at 298 K through Tensiometer-K6, KRÜSS Company, Hamburg, Germany, with a platinum ring in distilled water. The surfactant solutions were kept at 298 K for one day to maintain equilibrium. Before each measurement, the platinum ring was washed many times with filtered water to eliminate any residue on the metal surface. Three measurements were taken for each sample to ensure repeatability and to calculate an average value. Also, through a digital conductivity/temperature meter, ADWA AD3000 EC/TDS, Szeged, Hungary, at 298 K, the electrical conductivity of the DSMS, DSBB and DSEI surfactants was measured.

2.4. Weight loss measurements

In the weight-loss method, aluminum coupons with size of 2 × 1 × 0.8 cm³ are used. These coupons

were mechanically brushed with various grade emery papers before being rinsed with distilled water, propanone, and distilled water. Prior to adding aluminum samples into the test solution, the weight of the samples is determined. The experiment was replicated three times, with the average weight loss reported each time. The following equation was used to calculate the inhibition efficiency (IE %):

$$I.E. \% = [1 - (\Delta w_{\text{surf}}/\Delta w_{\text{free}})] \times 100. \quad (1)$$

where, Δw_{surf} and Δw_{free} reflect the weight change of aluminum samples per unit area with and without DSMS, DSBB and DSEI surfactants, accordingly. The following equation was used to measure the corrosion rate (CR) in grms.cm⁻².hr⁻¹:

$$CR = (\Delta w/A) \times T. \quad (2)$$

where, Δw is the change in the weight of the specimen in grams, A is the area of specimen in cm² and T is the time spent immersed in hours [16].

2.5. Gasometry

The corrosion reaction progress was determined using volumetric measurements of the evolved hydrogen. The test solution was put in a Büchner flask with the specimen. A rubber bung was used to close the flask. A rubber tube jutting from the flask neck and was attached to the underside of an overturned measuring cylinder hanging over a basin. The distilled water was used to fill both the cylinder and the basin. The hydrogen gas, which is assembled at the top of the inverted cylinder, ultimately displaces the distilled water, and the volume is measured over time. This experiment is replicated three times with and without varying concentrations of the DSMS, DSBB, and DSEI surfactants.

2.6. Electrochemical measurements

The potentiodynamic polarization curves have been determined for the corrosion of aluminum in 0.5 M HCl without and with various concentrations of DSMS, DSBB and DSEI surfactants. The working electrode is made of aluminum 1050 that is encased in Araldite resin, with just 1 cm² of the aluminum electrode visible. The electrode was rubbed with emery papers up to 2500 grade before being degreased with propanone and washed with distilled water. The counter electrode was a platinum wire, and the reference electrode was a saturated calomel electrode. Utilizing Potentiostat PS-6, Meinsberg, Germany, with its PS remote software, the curves of polarization were developed [17-20]. All tests were conducted at scan rate 1 mV/sec.

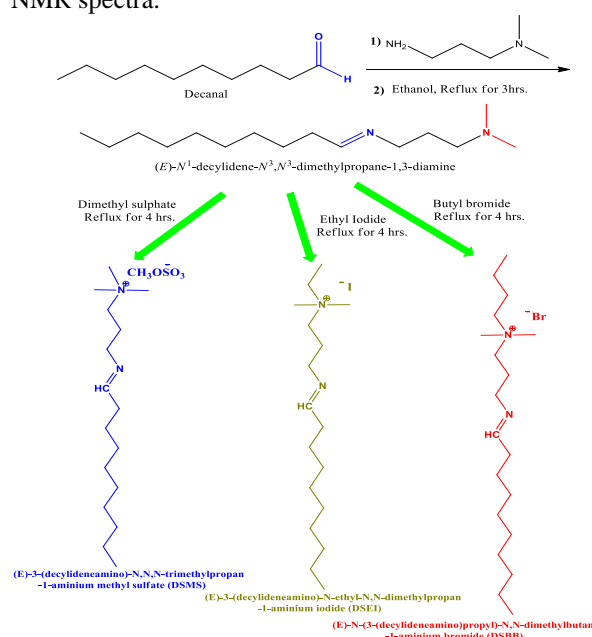
3. Results and discussion

3.1. Chemistry of the synthesized surfactants

As shown in **Scheme 1**, the *N,N,N*-dimethylpropane-1,3-diamine was taken to react with decanal in the presence of triethylamine as appropriate base catalyst in ethanol as a solvent to

form (*E*)-*N*¹-decylidene-*N*³,*N*³-dimethylpropane-1,3-diamine. The Schiff base synthesized compound was reacted with ethyl iodide, butyl bromide and dimethyl sulfate, individually in acetone as solvent to form the target cationic surfactants, (*E*)-3-(decylideneamino)-*N,N,N*-trimethylpropan-1-aminium methyl sulfate (DSMS), (*E*)-*N*-(3-(decylideneamino)propyl)-*N,N*-dimethylbutan-1-aminium bromide (DSBB) and (*E*)-3-(decylideneamino)-*N*-ethyl-*N,N*-dimethylpropan-1-aminium iodide (DSEI) with varied yields and colors.

Figure 1 shows how the structure of the synthesized cationic surfactants was clarified, chemically identified, and approved using FT-IR, ¹H, and ¹³C NMR spectra.



Scheme 1. Synthetic route for the preparation of the cationic surfactants (DSMS, DSBB and DSEI).

It is evident from **Figure 1** that the C=N group appears in the composition of the surfactants rather than the CO-NH group in the skeleton of the surfactants (DAEI, DABB and DAMS) previously synthesized by the authors [6, 15]. FT-IR showed 1634 cm⁻¹ ($\nu_{C=N}$ of imine) instead of 3441 cm⁻¹ (ν_{NH} of amide), and 1648 cm⁻¹ ($\nu_{C=O}$ of amide). ¹H NMR-400 MHz, (DMSO-*d*6): δ ppm: 1.62 (m, 2H, CH₂-CH₂-C=N), 1.74 (m, 2H, CH₂-CH₂-N=C), 2.78 (t, 2H, CH₂-CH₂-C=N), 2.87 (t, 2H, CH₂-CH₂-N=C) and 8.86 (t, 1H, CH₂-CH=N) instead of 1.46 (m, 2H, CH₂-CH₂-C=O), 2.08 (m, 2H, CH₂-CH₂-NH) and 2.34 (t, 2H, CH₂-C=O). ¹³C NMR-100 MHz, (DMSO-*d*6): δ ppm: 29.88 [CH₂-CH₂-C=N], 30.28 [CH₂-CH₂-N=C], 31.76 [N=C-CH₂] and 141.98 [CH=N, imines] instead of 29.36 [CH₂-CH₂-CO], 31.72 [CO-CH₂], 35.23 [CH₂-NH] and 173.28 [C=O, carbonyl amide]. In this work, we will study the effect of this change on the efficiency of the surfactants as corrosion inhibitors.

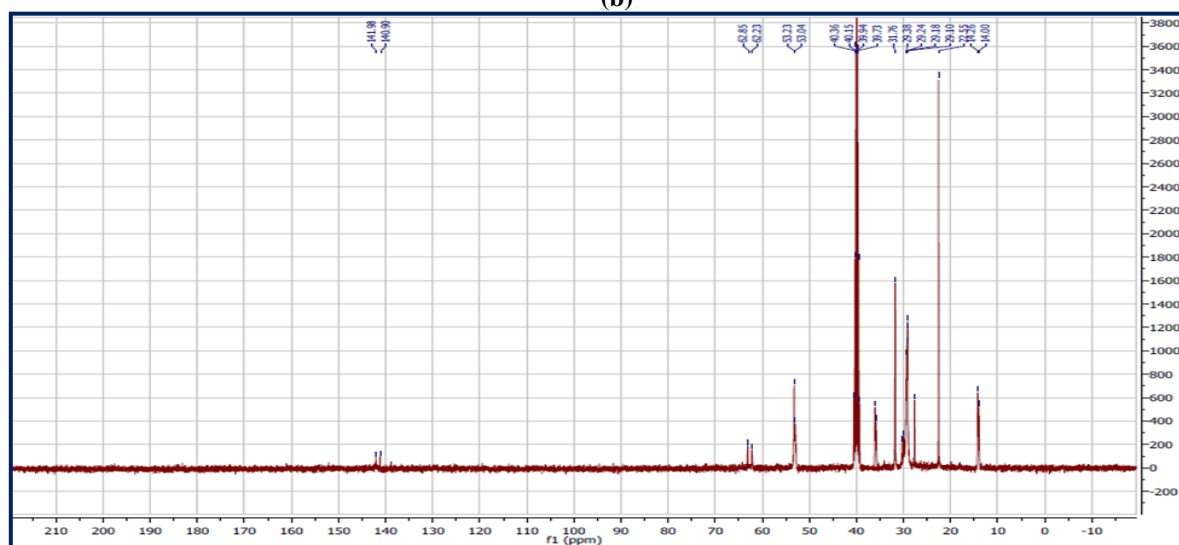
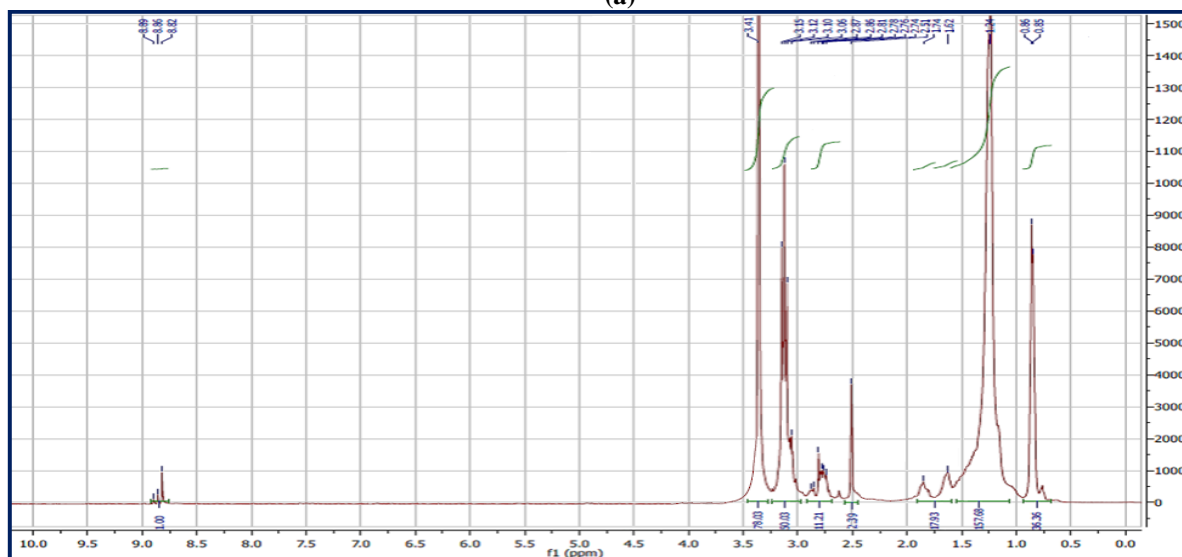
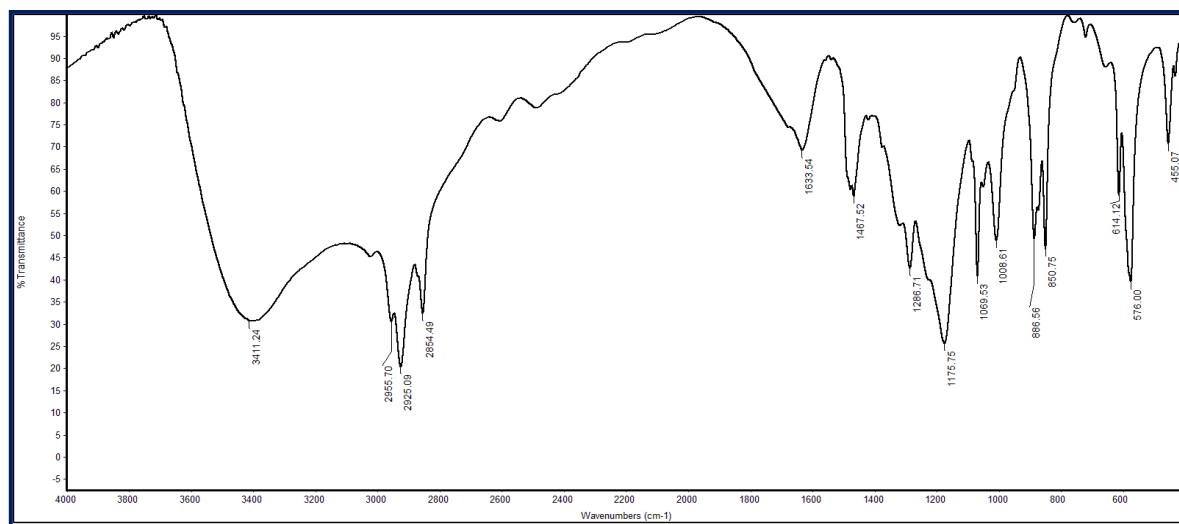


Figure 1: (a) FT-IR, (b) ^1H and (c) ^{13}C NMR spectrum of DSMS.

3.2. Critical Micelle Concentration and Surface-Active Properties

The CMCs of DSMS, DSBB, and DSEI surfactants were detected using the relation between the surface tension (γ) and $-\log(\text{concentration})$ of the corresponding surfactants, **Figure 2**, and those obtained using the electrical conductivity measurements were close to them.

The surface-active properties of DSMS, DSBB, and DSEI surfactants in **Table 1** were calculated using the following equations [15, 21-24]:

$$\beta = 1 - \alpha \quad (3)$$

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

$$\Gamma_{\text{max}} = -1/2.303nRT [d\gamma/d \log(C)]_T \quad (5)$$

$$A_{\text{min}} = 10^{14}/(\Gamma_{\text{max}} \times N_A) \quad (6)$$

$$\Delta G_{\text{mic}}^{\circ} = (2 - \alpha)RT \ln \text{CMC} \quad (7)$$

$$\Delta G_{\text{ads}}^{\circ} = (2 - \alpha)RT \ln \text{CMC} - 6.023 \times 10^{-1} \pi_{\text{CMC}} A_{\text{min}} \quad (8)$$

where β is the degree of counter-ion binding, α is the result of dividing the slope of the curve's linear section before the CMC by the slope of the curve's linear section after the CMC, π_{CMC} is the effectiveness, γ_{CMC} is the measured surface tension at CMC of DSMS, DSBB and DSEI solutions, and γ_0 is the measured surface tension of the distilled water, Γ_{max} is the maximum surface excess value in mol/m^2 , R is the gas constant ($8.314 \text{ J}\cdot\text{mol}^{-1}\cdot\text{K}^{-1}$), $T = 298 \text{ K}$, n is given 2 for the number of dissolved species, A_{min} is the average area filled by a single molecule's interface at equilibrium in nm^2 , N_A is Avogadro's number ($6.022 \times 10^{23} \text{ mol}^{-1}$), $\Delta G_{\text{ads}}^{\circ}$ is the free energy of adsorption and $\Delta G_{\text{mic}}^{\circ}$ is the free energy of micellization of the corresponding surfactants.

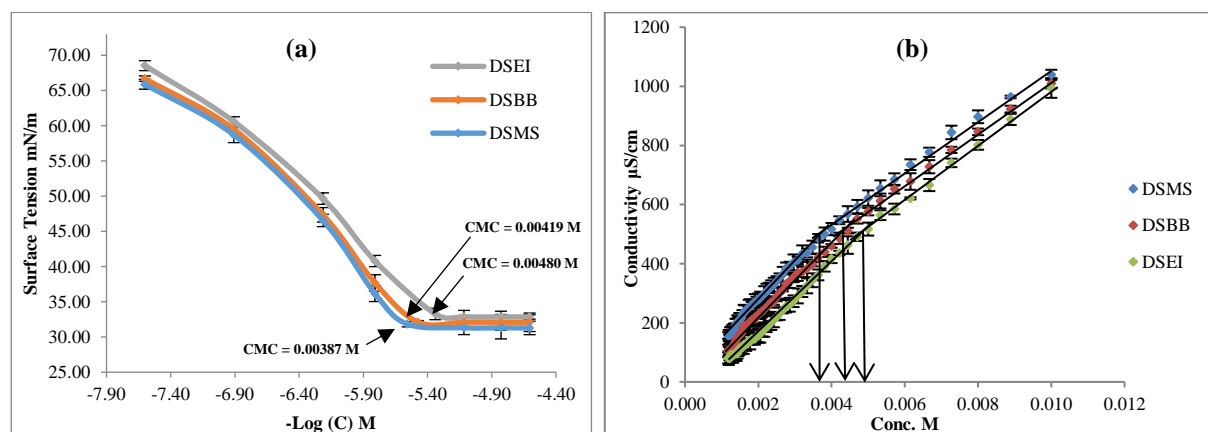


Figure 2: The relation between (a) surface tension and $-\log(\text{concentration})$ of the prepared surfactants and between (b) electrical conductivity and concentration of the prepared surfactants at 298 K (relative standard uncertainties (ur) limits of surface tension and electrical conductivity values are $\pm 5\%$).

Table 1: Surface-active properties of the synthesized cationic surfactants^a.

Surfactant	CMC ₁ , M	CMC ₂ , M	γ_{CMC} , $\text{mN}\cdot\text{m}^{-1}$	π_{CMC} , $\text{mN}\cdot\text{m}^{-1}$	α	β	$\Gamma_{\text{max}} \times 10^{10}$, $\text{mol}\cdot\text{cm}^{-2}$	A_{min} , nm^2	$-\Delta G_{\text{mic}}^{\circ}$, $\text{KJ}\cdot\text{mol}^{-1}$	$-\Delta G_{\text{ads}}^{\circ}$, $\text{KJ}\cdot\text{mol}^{-1}$
DSMS	0.00387	0.00391	31.81	40.99	0.67	0.33	3.44	0.483	18.611	18.623
DSBB	0.00419	0.00438	32.11	40.69	0.7	0.3	3.36	0.494	17.931	17.943
DSEI	0.00480	0.00492	33.27	39.53	0.8	0.2	3.23	0.514	16.141	16.153

CMC₁ values obtained from surface tension measurements and CMC₂ values obtained from conductivity measurements. ^a The relative standard uncertainties (ur) limits of CMC values are $\pm 5\%$.

3.3. Weight loss measurements

In a 0.5 M HCl solution, **Table 2** shows the weight loss of aluminum 1050 and the inhibition performance of different concentrations of DSMS, DSBB, and DSEI surfactants.

With increasing concentrations of the DSMS, DSBB, and DSEI surfactants, weight loss decreases and inhibition efficiency increases, as shown in **Figure S1**. This indicates that the surfactant molecules can adsorb on the aluminum surface, clogging active sites and slowing down the metallic corrosion process [31, 32].

The inhibition efficiency of the DSMS, DSBB, and DSEI surfactants started to wane after 48 hours.

This could be due to inadequate or disordered protective molecules on the metal surface, resulting in localized corrosion, or it could be due to increased desorption and decreased adsorption [31, 33].

The order of surfactant inhibition efficiencies is (DSMS > DSBB > DSEI) and (DAMS > DABB > DAEI), and the order is (DSMS > DAMS), (DSBB > DABB), and (DSEI > DAEI) when comparing Schiff base based cationic surfactants to amido-amine based cationic surfactants. The surface-active properties determined in the previous section predicted both orders.

Table 2: Weight loss-Time relation of aluminum 1050 in 0.5 M HCl solution with and without different concentrations of DSMS, DSBB and DSEI at 298 K and different exposure times, hrsa.

Surfactant	Time hrs.	Free	0.0001 M		0.001 M		0.002 M		0.003 M		0.0035 M	
		Δw , g.	Δw , g.	IE %	Δw , g.	IE %	Δw , g.	IE %	Δw , g.	IE %	Δw , g.	IE %
DSMS	17	0.0205	0.0156	23.90	0.0089	56.59	0.0058	71.71	0.0042	79.51	0.0016	92.20
	24	0.0355	0.0258	27.32	0.012	66.20	0.0065	81.69	0.0053	85.07	0.0019	94.65
	41	0.0725	0.048	33.79	0.0182	74.90	0.0092	87.31	0.0059	91.86	0.0029	96.00
	48	0.0855	0.0562	34.27	0.0191	77.66	0.0094	89.01	0.0061	92.87	0.0031	96.37
	65	0.1285	0.0848	34.01	0.0288	77.59	0.0142	88.95	0.0092	92.84	0.0047	96.34
	72	0.1395	0.0931	33.26	0.0315	77.42	0.0156	88.82	0.0103	92.62	0.0052	96.27
DSBB	17	0.0205	0.0161	21.46	0.0112	45.37	0.0102	50.24	0.0052	74.63	0.0017	91.71
	24	0.0355	0.0273	23.10	0.0173	51.27	0.0133	62.54	0.0063	82.25	0.0028	92.11
	41	0.0725	0.0505	30.34	0.0243	66.48	0.0142	80.41	0.0072	90.07	0.0041	94.34
	48	0.0855	0.058	32.16	0.0276	67.72	0.0152	82.22	0.0087	89.82	0.0045	94.74
	65	0.1285	0.0866	32.61	0.0422	67.16	0.0231	82.02	0.0132	89.73	0.0068	94.71
	72	0.1395	0.0952	31.76	0.0461	66.95	0.0251	82.01	0.0144	89.68	0.0075	94.62
DSEI	17	0.0205	0.0189	7.80	0.0162	20.98	0.0127	38.05	0.0101	50.73	0.0045	78.05
	24	0.0355	0.0319	10.14	0.0244	31.27	0.0163	54.08	0.0138	61.13	0.0052	85.35
	41	0.0725	0.064	11.72	0.04	44.83	0.0192	73.52	0.0148	79.59	0.0076	89.52
	48	0.0855	0.0735	14.04	0.0416	51.35	0.0261	69.47	0.0151	82.34	0.0081	90.53
	65	0.1285	0.1111	13.54	0.0628	51.13	0.0398	69.03	0.0231	82.02	0.0123	90.43
	72	0.1395	0.1217	12.76	0.0682	51.11	0.0433	68.96	0.0258	81.51	0.0137	90.18

^a The relative standard uncertainties (u_r) limits of Δw values are $\pm 2\%$.

3.4. Effect of temperature

After 48 hours, **Figure S2** depicts the temperature effect on aluminum in 0.5 M HCl without and with 0.0035 M of DSMS, DSBB, and DSEI surfactants. The weight loss of aluminum with the DSMS, DSBB, and DSEI surfactants is smaller than that in free 0.5 M HCl solution at different temperatures, as shown in the figure. This indicates that the surfactants DSMS, DSBB, and DSEI are effective inhibitors. Since the temperature rise has an inverse relationship with the inhibition efficiency, it's likely that the temperature rise causes desorption of adsorbed DSMS, DSBB, and DSEI molecules from the aluminum surface [34].

After 48 hours of immersion in 0.5 M HCl solutions, the activation energy E_a^* , activation enthalpy ΔH^* , and activation entropy ΔS^* for aluminum corrosion in the absence and presence of 0.0035 M of DSMS, DSBB, and DSEI surfactants were calculated using the Arrhenius equation [35]:

$$CR = A \times \exp(-E_a^*/RT) \quad (9)$$

and transition-state equation [36]:

$$CR = (RT/N_A h) \exp(\Delta S^*/R) \exp(-\Delta H^*/RT) \quad (10)$$

where, CR is the corrosion rate of aluminum, A is the frequency factor, h is plank's constant. Plots of $\log(CR)$ versus $1/T$ and $\log(CR/T)$ versus $1/T$ gave straight lines with slope of $-E_a^*/2.303R$ and $-\Delta H^*/2.303R$, respectively as shown in **Figure 3**.

Table 3 shows that adding DSMS, DSBB, and DSEI surfactants increases the activation energy value (E_a^*), suggesting that the DSMS, DSBB, and

DSEI molecules adsorb on the aluminum surface and form a barrier film. Charge and mass transfer is disrupted, resulting in a decreased corrosion rate [37]. In other words, in the presence of DSMS, DSBB, and DSEI surfactants, the HCl molecules must overcome a higher energy barrier in order to collide and corrode the metal [38].

Table 3 also indicates that the activation enthalpy values (ΔH^*) are positive, suggesting that the endothermic corrosion of aluminum in 0.5 M HCl is occurring. The presence of DSMS, DSBB, and DSEI surfactants, on the other hand, increases the activation enthalpy values, suggesting a higher inhibition efficiency since the energy barrier increases with the addition of DSMS, DSBB, and DSEI surfactants, making the corrosion process much more difficult [39].

In the presence of DSMS, DSBB, and DSEI surfactants, the lower negative value of (ΔS^*) suggests an increase in disorder when moving from reactant to activated complex at the metal surface, as well as an increase in solvent entropy [40, 41].

The order of inhibition efficiency of the tested surfactants as gathered from the increase in the activation energy (E_a^*), the enthalpy of activation (ΔH^*) and the entropy of activation (ΔS^*) values is: (DSMS > DSBB > DSEI) and (DAMS > DABB > DAEI), and (DSMS > DAMS), (DSBB > DABB) and (DSEI > DAEI). This is expected from the weight loss results and the surface-active properties.

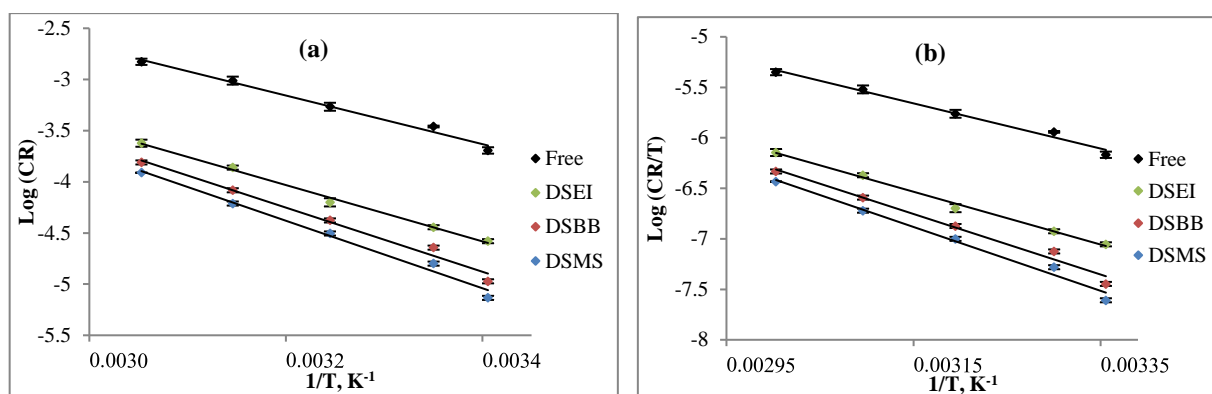


Figure 3: (a) Arrhenius plot and (b) transition state plot for the dissolution of aluminum 1050 in 0.5 M HCl in the presence and absence of 0.0035 M of DSMS, DSBB & DSEI after 48 hours (relative standard uncertainty (ur) limit of CR values is $\pm 3\%$).

Table 3: Activation parameters of the dissolution of aluminum 1050 in 0.5 M HCl in the presence and absence of 0.0035 M of DSMS, DSBB & DSEI after 48 hours^a.

System	E_a^* (kJ.mol ⁻¹)	ΔH^* (kJ.mol ⁻¹)	$-\Delta S^*$ (J.mol ⁻¹ .K ⁻¹)
Free acid	45.48	42.86	170.94
Acid + DSMS	63.16	60.54	138.66
Acid + DSBB	60.02	57.40	146.10
Acid + DSEI	57.89	55.27	148.92

^a The relative standard uncertainties (u_r) limit of CR values, from which E_a^* , ΔH^* and ΔS^* are calculated, is $\pm 3\%$.

3.5. Gasometry

Table 4 displays the amounts of hydrogen gas generated and the resulting inhibition efficiencies during the corrosion of aluminum in 0.5 M HCl solution at 298 K with and without different concentrations of DSMS, DSBB, and DSEI surfactants. The following equation was used to measure the inhibition effectiveness [42]:

$$I.E. \% = [1 - (CR_{surf}/CR_{free})] \times 100 \quad (11)$$

where, CR_{surf} and CR_{free} are the corrosion rate in (ml/cm².hr) of aluminum in inhibited and uninhibited solutions, respectively.

Table 4 shows that as surfactant concentration is increased, the inhibition efficiency of DSMS, DSBB, and DSEI surfactants increases. The results are consistent with the above.

Table 4: Hydrogen volume evolved (ΔV , in ml) during the corrosion of aluminum 1050 in 0.5 M HCl solution with and without different concentrations of DSMS, DSBB and DSEI at 298 K and different exposure times, hrs^a.

System	Time hrs.	Free		0.0001 M		0.001 M		0.002 M		0.003 M		0.0035 M	
		ΔV , ml	IE %	ΔV , ml	IE %	ΔV , ml	IE %	ΔV , ml	IE %	ΔV , ml	IE %	ΔV , ml	IE %
DSMS	17	55	20.00	44	26	52.73	17	69.09	13	76.36	6	89.09	
	24	96	25.00	72	34	64.58	19	80.21	16	83.33	7	92.71	
	41	196	32.65	132	51	73.98	26	86.73	18	90.82	9	95.41	
	48	232	33.62	154	53	77.16	27	88.36	18	92.24	10	95.69	
	65	349	32.09	237	85	75.64	45	87.11	31	91.12	19	94.56	
	72	379	31.66	259	94	75.20	51	86.54	36	90.50	23	93.93	
DSBB	17	55	18.18	45	32	41.82	29	47.27	16	70.91	6	89.09	
	24	96	20.83	76	49	48.96	38	60.42	19	80.21	9	90.63	
	41	196	29.08	139	68	65.31	40	79.59	21	89.29	13	93.37	
	48	232	31.47	159	76	67.24	43	81.47	25	89.22	14	93.97	
	65	349	30.66	242	121	65.33	69	80.23	42	87.97	25	92.84	
	72	379	30.08	265	134	64.64	77	79.68	48	87.34	29	92.35	
DSEI	17	55	3.64	53	46	16.36	36	34.55	29	47.27	14	74.55	
	24	96	8.33	88	68	29.17	46	52.08	39	59.38	16	83.33	
	41	196	10.71	175	110	43.88	54	72.45	42	78.57	22	88.78	
	48	232	13.36	201	115	50.43	72	68.97	43	81.47	24	89.66	
	65	349	12.32	306	177	49.28	115	67.05	69	80.23	40	88.54	
	72	379	12.40	332	194	48.81	126	66.75	79	79.16	46	87.86	

^a The relative standard uncertainties (u_r) limits of ΔV values are $\pm 5\%$.

3.6. Potentiodynamic polarization

At a scan rate of 1 mV/sec, in the absence and presence of varying concentrations of DSMS, DSBB, and DSEI surfactants, the potentiodynamic polarization curves of aluminum in 0.5 M HCl solution are shown in **Figure S3**. **Table 5** lists the relevant electrochemical parameters. To determine the inhibition efficiencies and surface coverages, the following formulas were applied [32, 43]:

$$I.E. \% = [1 - (I_{\text{surf}}/I_{\text{free}})] \times 100 \quad (12)$$

$$\theta = [1 - (I_{\text{surf}}/I_{\text{free}})] \quad (13)$$

where, I_{free} and I_{surf} are the corrosion current densities without and with DSMS, DSBB and DSEI surfactants, accordingly. The table below shows that DSMS, DSBB, and DSEI surfactants minimize corrosion current density (I_{corr}), resulting in a lower corrosion rate (CR) and a higher percentage inhibition efficiency (IE%).

The order of inhibition efficiency calculated from the current densities is in a good agreement with those calculated from weight loss and gasometry techniques.

When the concentration of DSMS, DSBB, and DSEI surfactants is increased, the values of anodic Tafel slopes (β_a) and cathodic Tafel slopes (β_c) change slightly, reflecting that the mechanisms of hydrogen evolution (cathodic reactions) and aluminum dissolution (anodic reactions) seem to be almost unaffected during the inhibition process [44], DSMS, DSBB, and DSEI surfactants may also have an effect on both anodic and cathodic reactions, making them mixed form inhibitors [45].

The use of DSMS, DSBB, and DSEI surfactants did not change the corrosion potential (E_{corr}) significantly, implying that DSMS, DSBB, and DSEI surfactants function as mixed form inhibitors. [46].

3.7. Adsorption isotherm

The surface coverage values (θ) for differing DSMS, DSBB, and DSEI surfactant concentrations were estimated through using weight loss results in order to select the optimal adsorption isotherm for these surfactants. The closest match was for the Langmuir adsorption isotherm, based on the following formula [47]:

$$C/\theta = (1/K) + C \quad (14)$$

where, K is the adsorption equilibrium constant and C is the molar concentration of surfactant.

Figure 4 illustrates the plotting of C/θ versus C , which gives straight lines with nearly unit slopes for DSMS, DSBB, and DSEI surfactants and an intercept equal to $1/K$, assuming that the adsorption is the Langmuir adsorption isotherm.

The following equation describes the relationship between K , the adsorption equilibrium constant, and $\Delta G^{\circ}_{\text{ads}}$, the adsorption standard free energy [48]:

$$K = (1/55.5) \exp(-\Delta G^{\circ}_{\text{ads}}/RT) \quad (15)$$

where the molar concentration of H_2O is represented by the number (55.5). The values of K and $\Delta G^{\circ}_{\text{ads}}$ for DSMS, DSBB and DSEI are (3.333×10^3 and -30.055 kJ/mol), (2.500×10^3 and -29.342 kJ/mol) and (1.667×10^3 and -28.338 kJ/mol). The adsorption process spontaneity of the DSMS, DSBB, and DSEI surfactants on the aluminum surface is demonstrated by the negative values of $\Delta G^{\circ}_{\text{ads}}$ obtained, which are compatible with those achieved from the surface-active properties. The physically adsorption activity is indicated by the small values of $\Delta G^{\circ}_{\text{ads}}$ obtained [6, 35, 49].

Table 5: Corrosion parameters obtained from potentiostatic polarization measurements of aluminum 1050 in 0.5 M HCl and different concentrations of DSMS, DSBB and DSEI at 298 K and scan rate 1 mV/sec.

System	Conc. M	$-\beta_c$ mV.dec ⁻¹	β_a mV.dec ⁻¹	CR mm/Jear	I.E. %	θ	I_{Corr} mA	$-E_{\text{Corr}}$ mV
Free	0.0000	62.9	15.07	4.91	-	-	0.23	743.07
DSMS	0.0001	52.98	12.78	2.92	40.5	0.41	0.13	749.6
	0.0010	34.39	13.77	1.04	78.89	0.79	0.05	733.85
	0.0020	24.39	14.46	0.48	90.2	0.9	0.02	752.16
	0.0030	35.13	9.78	0.29	94.19	0.94	0.01	734.02
	0.0035	33.86	21.74	0.1	98.06	0.98	0.004	734.9
DSBB	0.0001	83.82	11.18	2.95	39.9	0.4	0.14	742.5
	0.0010	53.84	9.44	1.43	70.8	0.71	0.07	741.22
	0.0020	36.38	11.05	0.79	83.87	0.84	0.04	750.97
	0.0030	31.93	27.69	0.48	90.25	0.9	0.02	787.9
	0.0035	30.83	23.7	0.3	93.91	0.94	0.01	778.14
DSEI	0.0001	47.96	17.29	3.86	21.38	0.21	0.18	746.7
	0.0010	42.69	13.8	2.19	55.48	0.55	0.1	749.6
	0.0020	47.79	11.53	0.96	80.37	0.8	0.04	742.22
	0.0030	41.87	13.17	0.78	84.17	0.84	0.04	748.91
	0.0035	39	11.21	0.43	91.33	0.91	0.02	744.12

By comparing the values of $\Delta G^{\circ}_{\text{ads}}$ measured from surface active parameters for the air/liquid interface with those computed for the metal/liquid interface by using Langmuir adsorption isotherm, we notice that $\Delta G^{\circ}_{\text{ads}}$ values for the metal/liquid interface are relatively higher. That might be due to the electrostatic attraction with the metal substrate, which varies depending on steric influences, causing some collision paths to be disfavored or preferred, and even the shape and size of the adsorbent molecules, as well as the water adsorption/desorption equilibrium, all of which contribute to the overall adsorption free energy change [50].

The order of the negative values of $\Delta G^{\circ}_{\text{ads}}$ of the surfactants is found to be: (DSMS > DSBB > DSEI) and (DAMS > DABB > DAEI), and (DSMS > DAMS), (DSBB > DABB) and (DSEI > DAEI). This is expected from those calculated from the surface-active properties.

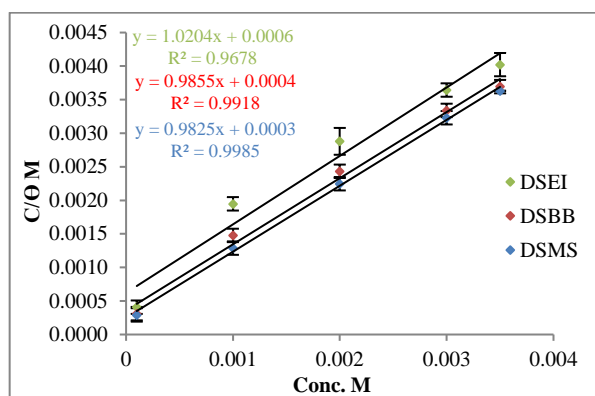


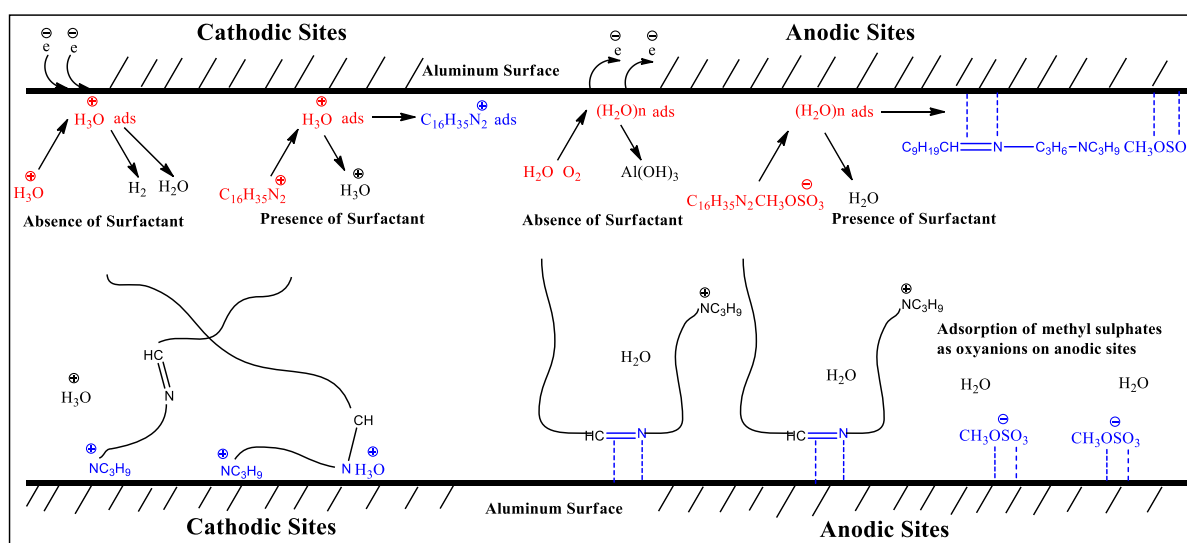
Figure 4: Langmuir adsorption isotherm for DSMS, DSBB & DSEI on aluminum 1050 in 0.5 M HCl at 298 K (relative standard uncertainty (ur) limit of θ values is $\pm 3\%$).

3.8. Mechanism of Inhibition

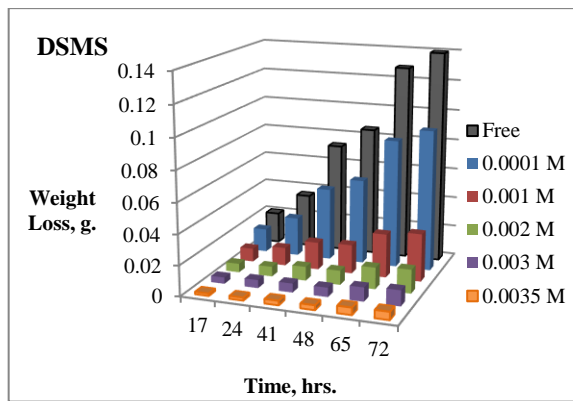
The inhibition mechanism of the studied synthesized DSMS, DSBB, and DSEI surfactants becomes more possibly related to the adsorption of the surfactant molecules on both anodic and cathodic sites of the aluminum surface. The surfactants are forced energetically to escape from the bulk water to its interface, whether it be with air or metal, due to the so-called hydrophobic interaction. The phenomenon is similar to that which occurs during the production of micelles. The functional groups' adsorption (the anchoring groups), such as C=N and the CH_3OSO_3 groups are anchored on the positive sites of aluminum substrate. Also, the electrostatic attraction between the cationic part (the ammonium group) of the surfactant molecules and/or the protonated surfactant molecules and the negatively charged areas on the aluminum surface could be to contribute. In both cases, the adsorbed surfactant molecules form a barrier on the aluminum surface that covers it from the aggressive medium (HCl solution), resulting in a reduced corrosion rate, **Scheme 2** [51].

4. Conclusions

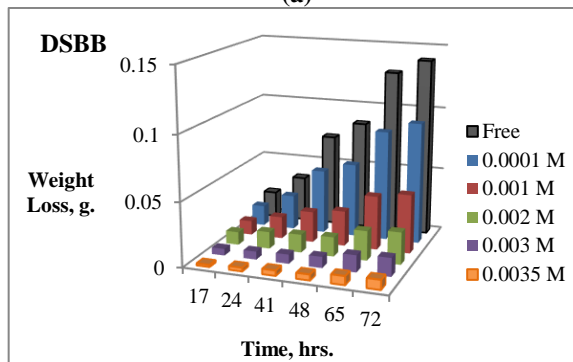
DSMS, DSBB and DSEI, three novel Schiff base based cationic surfactants, were synthesized and showed good surface-active properties. They also showed higher values of $-\Delta G^{\circ}_{\text{ads}}$, ΔS^* , ΔH^* and E^*_a which seem to be in support of reducing corrosion. Through adsorption of their molecules on the metal surface, these surfactants displayed the capability to develop a protection layer against corrosive acid environments at varying temperatures. The adsorption process appears to be spontaneous and physical based on the negative small values of $-\Delta G^{\circ}_{\text{ads}}$. The corrosion potential values did not vary significantly, meaning that these surfactants are mixed form inhibitors.



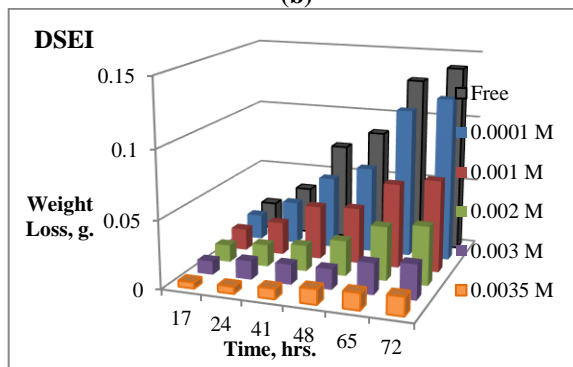
Scheme 2. Adsorption mechanism of cationic surfactants (DSMS, DSBB and DSEI) on aluminum surface.



(a)



(b)



(c)

Figure S1: Weight loss-time curves for the corrosion of aluminum 1050 in 0.5 M HCl in absence and presence of different concentrations of DSMS, DSBB and DSEI.

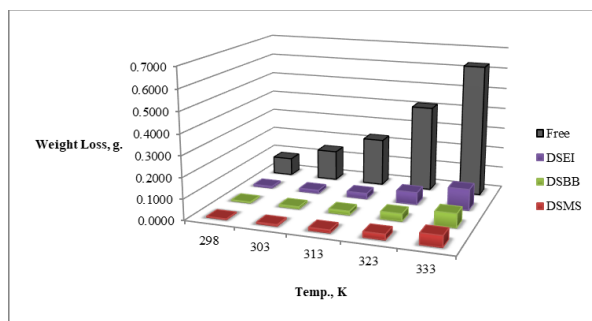
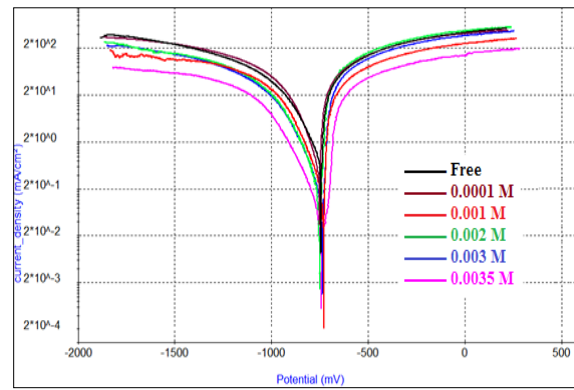
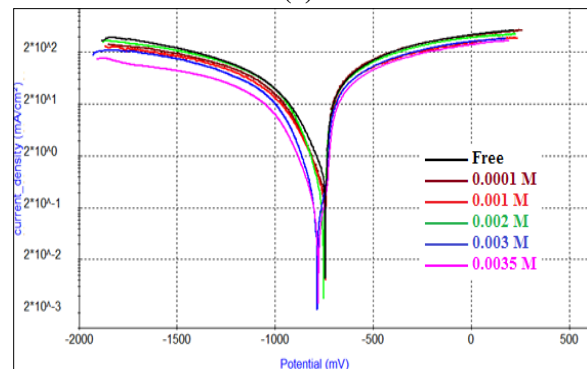


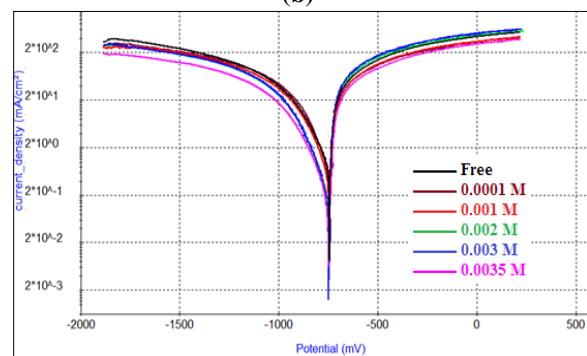
Figure S2: Weight loss-temperature curves for the corrosion of aluminum 1050 in 0.5 M HCl in the presence and absence of 0.0035 M of DSMS, DSBB & DSEI after 48 hours.



(a)



(b)



(c)

Figure S3: Anodic and cathodic polarization curves for aluminum 1050 in 0.5 M HCl solution and different concentrations of (a) DSMS and (b) DSBB and (c) DSEI, at 298 K and scan rate of 1 mV/sec.

5. Conflicts of interest

There are no conflicts to declare.

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