ORIGINAL ARTICLE

Synthesis and Evaluation of 4-Diethyl Amino Benzaldehyde Schiff Base Cationic Amphiphiles as Corrosion Inhibitors for Carbon Steel in Different Acidic Media

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Abstract A novel series of cationic surfactants containing Schiff base groups were synthesized by condensation of fatty amines namely: dodecyl, tetradecyl, hexadecyl and octadecyl amine and 4-diethyl aminobenzaldehyde. The chemical structures of these surfactants were confirmed using elemental analysis, FTIR spectra, ^{1}H-NMR and atomic absorption spectroscopy. Surface properties of the synthesized compounds were determined using surface tension techniques. The results of the surface tension measurements showed good surface behaviors of these compounds in their aqueous solutions. The surface activities were found to be greatly influenced by the chemical structures of the synthesized compounds. The synthesized cationic Schiff bases were evaluated as corrosion inhibitors for carbon steel in different acidic media (HCl and H_2SO_4) at different doses (400, 200, 100, 50, 25 ppm). The corrosion inhibition efficiencies of these inhibitors were calculated using the corrosion rates of the carbon steel in the studied media and found in the acceptable range of the commercially used inhibitors. Also the chemical structure of these inhibitors was found of great influence on their inhibiting efficiency.

Keywords Cationic Schiff base · Surface activity · Adsorption · Micellization · Emulsification · Corrosion rate · Corrosion inhibition efficiency

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Introduction

Schiff bases are one of the important classes of organic compounds which have many interesting properties and extensive applications in biology and materials science [1–3]. The Langmuir–Blodgett films of amphiphilic Schiff base compounds have been investigated and their interesting features revealed [4, 5]. The main properties studied focused on the coordination with transition metal ions, and some organized molecular films of these Schiff base derivatives have been reported. Using the Langmuir–Blodgett technique, it is convenient to form ordered layer structures by the transferring monolayers from water surface-to-solid substrate. This ordered deposition offers potentially greater control over molecular orientation and inter-molecular packing for these materials, which could lead to more and more creations of new types of thin-film devices especially in the field of electronics and optics. Research work on the behavior of Langmuir and LB films has been attracting increasing attention in recent years. The formation of bilayer membranes from single-chain amphiphiles in dilute aqueous solution is mainly determined by the balance between the interactions among the hydrophobic tails and that among the hydrophilic head groups and the molecular geometry as well. Through any of the following ways to enhance the assembly between amphiphiles, namely the introduction of a rigid segment [6], H-bonding [7], protonation of the head groups and coordination [8], stable bilayers are obtainable. Synthesis of novel bilayer-forming amphiphiles are of great interest as the introduction of rigid segments promotes the formation of not only a hydrophobic assembly but also a microenvironment containing functional units [9]. The inhibition of steel corrosion by acids has been previously studied by various researchers using different organic compounds

[10–12]. These compounds in general are adsorbed onto the metal surface blocking the active corrosion sites. Several Schiff bases have been investigated as corrosion inhibitors for various metals and alloys in acidic media. In this study, novel cationic amphiphiles containing Schiff base groups were synthesized from condensation reactions of 4-diethyl aminobenzaldehyde and different fatty amines. Their chemical structures were confirmed using different analytical tools. The surface activity–chemical structure relationship was discussed. The synthesized cationic Schiff bases were evaluated as corrosion inhibitors for carbon steel in different acidic media (HCl and H_2SO_4) at different doses (400, 200, 100, 50, 25 ppm). The corrosion inhibition efficiencies of these inhibitors were calculated using the corrosion rates of the carbon steel in the studied media.

Experimental

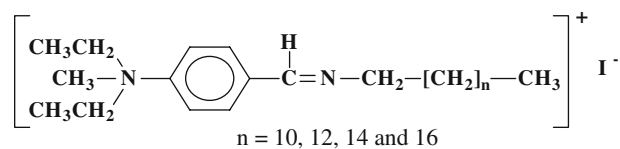### Synthesis of Schiff Bases

A 0.5 mol sample of 4-diethylaminobenzaldehyde was condensed with 0.5 mol samples of fatty amines namely; dodecyl, tetradecyl, hexadecyl or octadecyl amines in the presence of 250 mL of ethyl alcohol as a solvent. The reaction mixture was refluxed for 6 h and then left overnight until the product was precipitated. The product was filtered then washed with petroleum ether and recrystallized twice from ethanol. The final product was dried under vacuum at 40 °C [13]. The chemical structures of the Schiff bases produced are presented in Scheme 1.

Synthesis of Cationic Surfactants of Schiff Base Derivatives

Cationic form of the synthesized Schiff bases were obtained throughout direct interaction between equimolar amounts of the synthesized Schiff bases and methyl iodide in ethanol. The reaction mixture was refluxed for 8 h, and then left overnight for complete precipitation of the cationic quaternary ammonium compounds. The product was filtered and recrystallized for three times from ethanol to produce the pure cationic Schiff base surfactants [14] (Scheme 2).
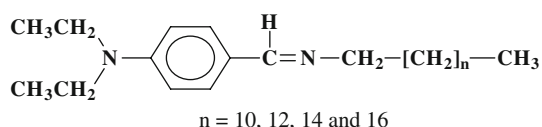
Scheme 1 Chemical structures of the synthesized Schiff bases

Scheme 2 Chemical structures of the synthesized cationic Schiff base amphiphiles

Surface Tension Measurements

Surface tension measurements were obtained using a De-Nouy Tensiometer Kruss K-6 with a platinum ring. Freshly prepared aqueous solutions of Schiff base cationics and their transition metal complexes were used over a concentration range of 10–0.01 mM/L at 25 °C. Apparent surface tensions were measured three times for the sample with a 2-min interval between each reading [15].

Emulsification Efficiency and Interfacial Tension Measurements

Emulsification efficiencies were determined by measuring the time required for separation of 9 mL of pure water from a strongly shaken emulsion of 10 mL (0.1%) surfactant solution and 10 mL of paraffin oil at 25 °C. Interfacial tension measurements were made for the same concentration (0.1%) using paraffin oil as the nonpolar phase at 25 °C [15].

Corrosion Measurement

A weight-loss technique [16] was used to measure the inhibiting efficiency to corrosion of the prepared inhibitors for carbon steel in 2 N HCl and 2 N H_2SO_4 solutions at 25 °C for different periods (24 and 48 h). The experiments were performed with carbon steel specimens having a composition (wt.%): 0.21 C, 0.035 Si, 0.51 Mn, 0.82 P, and the remainder Fe. Each specimen was machined into regular shapes of 18 cm^2 cross-sectional area. The specimens were sequentially abraded with different emery papers, degreased with acetone, washed with distilled water and dried. Corrosive solutions of 2 N HCl and 2 N H_2SO_4 in the absence and presence of the synthesized inhibitors at concentrations of 400, 200, 100, 50 and 25 ppm were prepared with doubly distilled water.

Analyses

Microelemental analyses were done using a Vario Elementar instrument, FTIR spectroscopic analyses were done using a Fourier-transform infrared spectrophotometer, ^{1}H-NMR spectroscopic analyses were done using a Bruker

Table 1 Microelemental analyses of the cationic Schiff bases and their metal complexes

Compound	Molecular weight, g/mol	Carbon		Hydrogen		Nitrogen		Iodide	
		Calculated	Found	Calculated	Found	Calculated	Found	Calculated	Found
SB-12	485.60	59.36	58.62	8.72	8.61	5.77	5.70	26.15	25.83
SB-14	513.64	60.79	60.03	9.03	8.91	5.46	5.39	24.73	24.42
SB-16	541.70	62.08	61.30	9.30	9.19	5.17	5.11	23.44	23.15
SB-18	569.75	63.24	62.45	9.55	9.43	4.92	4.86	22.29	22.01

model DRX-300 NMR spectrometer with TMS as an internal standard and atomic absorption spectroscopy (AAS) was carried out using a Hitachi 180/80 atomic absorption spectrometer.

Results and Discussion

Chemical Structure

Elemental Analyses

The elemental analyses of the synthesized cationic Schiff base amphiphiles showed that the values expected and found of the different elements are very close to each other, indicating the purity of the synthesized compounds (Table 1).

FTIR Spectroscopy

The synthesized Schiff bases showed a complete disappearance of the absorption band at 1,734 cm^{-1} corresponding to the carbonyl group and the appearance of a new band at 1,658 cm^{-1} corresponding to a –C=N– group. The IR spectra of the synthesized cationic Schiff base amphiphiles showed new absorption bands at 3,130 and 1,400 cm^{-1} corresponding to a [N$^+$] group (vibration and elongation, respectively). Bands at 2,980 and 2,870 cm^{-1} corresponding to CH$_2$ and CH$_3$, respectively, and 940–980 cm^{-1} corresponding to a benzene nucleus appeared in all of the synthesized compounds indicating the presence of all function groups expected in the synthesized compounds.

^{1}H-NMR Spectroscopy

The ^{1}H-NMR spectra of the synthesized compounds showed a characteristic triplet signal at 0.792 ppm corresponded to methyl groups, a multiple signal at 1.028 ppm (CH$_2$) with an integrated number of hydrogen protons for the alkyl chain, a triplet signal at 2.423 ppm, 4H for methylene groups in the tertiary amine group and multiple signals at 7.585 ppm of the phenyl protons.

Surface Properties of Quaternary Ammonium Cationic Schiff Bases

The relation between the surface tension and the concentration of the synthesized cationic Schiff bases surfactants is represented in Fig. 1. The relation is typically found for cationic surfactants in their solutions [17]. The critical micelle concentration (CMC) values of the synthesized amphiphiles are gradually decreased by increasing the length of the alkyl chains attached to the synthesized molecules (Fig. 2). The lowest CMC value was observed at 0.73 mM/L for the surfactant containing a hexadecyl chain. While, the highest value was observed at 2.63 mM/L for the dodecyl chain derivative at 25 °C. The octadecyl derivative showed an exceptional higher value at 1.26 mM/L at 25 °C. That could be due to the geometrical conformation occurred in the alkyl chain. This conformation (coiling) causes a compactness in SB-18 molecules which decreases their actual volume. Hence, the molecules act as
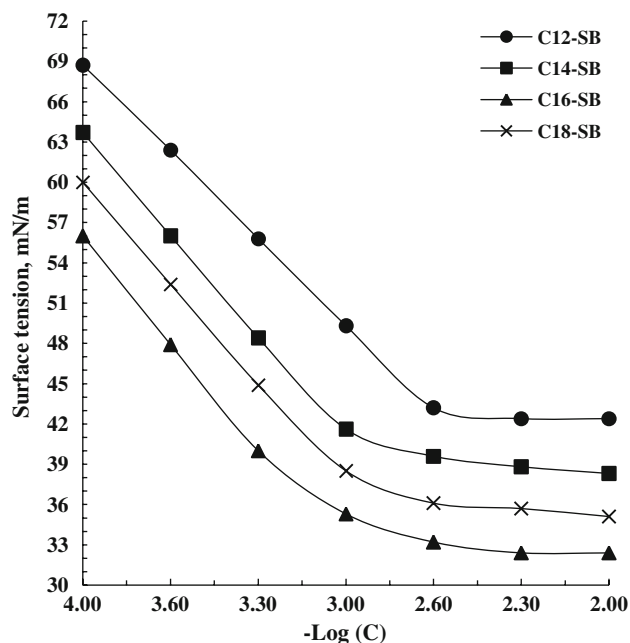
Fig. 1 Variation of surface tension against concentration of the synthesized Schiff base cationics at 25 °C
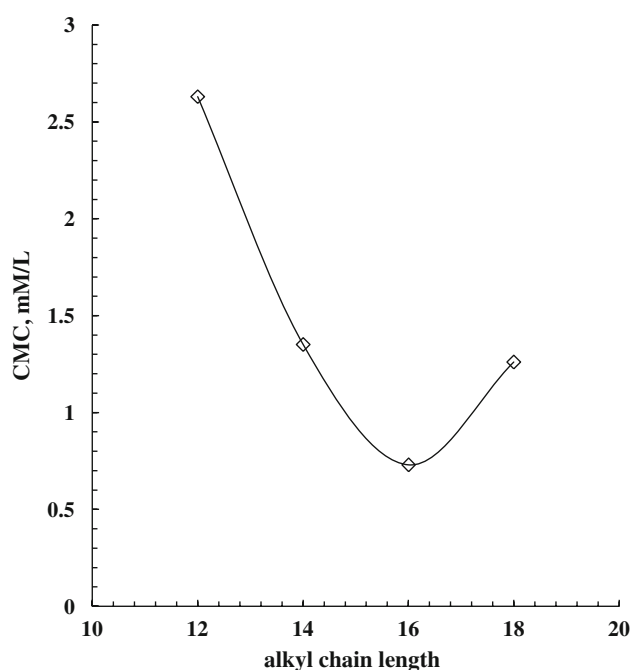
Fig. 2 Effect of alkyl chain length on CMC values of the synthesized surfactants

they if they were shorter than the hexadecyl derivative (SB-16) and longer than the tetradecyl derivative (SB-14; Table 2). The effectiveness values (π_{cmc}) also increased gradually according to the hydrophobic chain lengths. The highest depression in the surface tension values at CMC was recorded for the hexadecyl derivative (SB-16) at 44 mN/m. The octadecyl derivative (SB-18) showed an π_{cmc} value at 35 mN/m. This could be explained by the conformation factor. The efficiency values (Pc_{20}) of the targeted surfactants (Table 2) showed their good tendency to modify the surface activity of their solutions at considerably low concentrations. The maximum surface excess (Γ_{max}) values describe the accumulation extent of surfactant molecules at the solution interface. Increases in the Γ_{max} values indicates that the water molecules exert a high repulsion force on the surfactant molecules, hence the later escape to the interface therefore Γ_{max} values increased. Increasing Γ_{max} values indicates that the molecules wouldn't undergo to the bulk of their solutions to form micelles at lower concentrations, which coincides with the

high CMC values of SB-12 derivative. Longer chain lengths showed lower Γ_{max} values (Table 2). The minimum surface area occupied by each molecule at the interface (A_{min}) is greatly influenced by the number of repeated methylene groups in the hydrophobic chains. Increasing the hydrophobic chain length increases the area occupied by each molecule at the interface. The maximum area occupied by individual molecule at the interface was found for SB-16 derivative which equals to 27.05 Å^2. SB-18 derivative showed smaller A_{min} than the SB-16 derivative due to the coiling which had occurred in the hydrophobic chain [18].

Thermodynamic Aspects

The thermodynamic characteristics of the synthesized cationic Schiff base amphiphiles were studied using the calculated values of adsorption and micellization free energies ΔG_{ads}, ΔG_{mic}. These data were calculated using the thermodynamic Eqs. 1, 2, [19] (Table 2).

$$\Delta G_{mic} = -2.303 RT \log(CMC) \tag{1}$$

$$\Delta G_{ads} = \Delta G_{mic} - (0.006 \times \pi_{cmc} \times A_{min}) \tag{2}$$

The micellization free energies of the targeted surfactants in their aqueous media showed negative sign, that indicates the spontaneity of the micellization process. The driving force of micelles formation is the repulsion occurring between the hydrophobic chains and the polar medium. In addition, the stabilization occurring for the micelles formed through the attraction forces between the positively charged head groups and the water molecules. The association of the counter ions (I^-) in the attracted system increases the stability of the formed micelles. Obviously, increasing the hydrophobic chain length increases the negativity of ΔG_{mic} values. This reinforces the idea of increasing the micellization tendency by increasing the number of methylene groups along the hydrophobic chains. The gradual increase in the negativity of ΔG_{mic} agrees with the change sequence of the critical micelle concentration values (Table 2). On the other hand, the adsorption free energy change of the studied surfactants showed an exact response as ΔG_{mic} by increasing the hydrophobic chain lengths.

Table 2 Surface, thermodynamic properties, interfacial tension and emulsification efficiency of the synthesized cationic Schiff bases at 25 °C

Compound	CMC (mM/L)	π_{cmc} (mN/m)	Pc_{20} (mM/L)	Γ_{max} ($\times 10^{-11}$ Mol K^{-1} cm^{-1})	A_{min} (Å^2)	ΔG_{ads} (kJ/Mole)	ΔG_{mic} (kJ/Mole)	IT (mN/m)	Emulsification efficiency (s)
SB-12	2.63	27	0.71	7.08	24.25	−8.19	−8.15	12	50
SB-14	1.35	31	0.39	6.48	25.63	−9.11	−9.07	5.5	275
SB-16	0.73	44	0.14	6.14	27.05	−9.99	−9.92	3	665
SB-18	1.26	35	0.24	6.31	26.35	−9.23	−9.17	4	620

Fig. 3 Effect of the
hydrophobic chain length on the
inhibition efficiency of the
synthesized inhibitors at
different doses
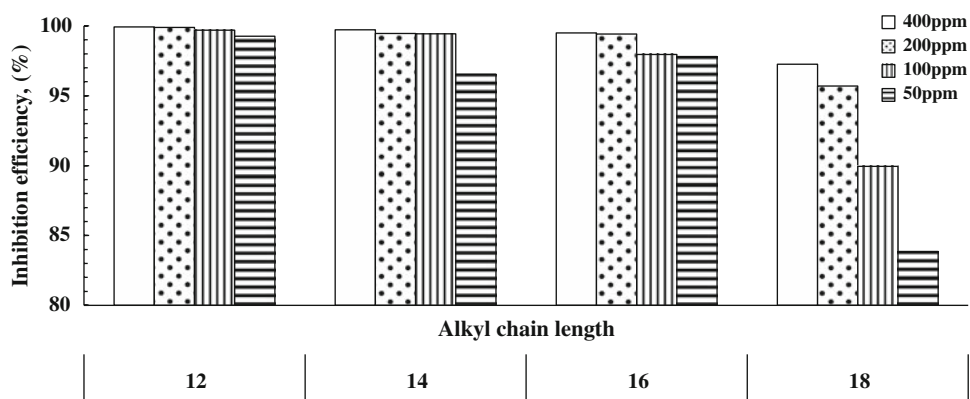
The negativity of both ΔG_{ads} and ΔG_{mic} indicates that the two processes occurred spontaneously. The closeness between the two values (ΔG_{mic} and ΔG_{ads}) reveals the equilibrium between the two phases of the surfactant molecules (adsorbed and micellized phase). Also, the slight increase of ΔG_{ads} values showed that these molecules tend to adsorb at the air–water interface till complete surface coverage is reached, then, the micelles formed. Hence, the micellization and adsorption processes are governed by the thermodynamic aspects. The chemical structure of these molecules is the main factor influencing their thermodynamic aspects. The equivalence between adsorption and micellization tendencies qualifies these surfactants as being applicable in the interfacial applications including emulsification, corrosion inhibition, and biocidal application [20]. In addition, in micellar applications including solubilization, drug delivery and drug carrier purposes.

Interfacial Tension and Emulsification Efficiency

Interfacial tension (IT) values of the synthesized cationic Schiff bases were measured between their aqueous solutions and paraffin oil at 25 °C (Table 2). Table 2 shows higher IT values for the dodecyl derivatives (SB-12; 12 mN/m). Increasing the hydrophobic chain length decreases the interfacial tension considerably. On the other hand, the emulsification efficiency of the surfactants in oil–water or nonpolar–polar systems is a reflection of the ability of surfactant molecules to locate at the boundary lines between the two phases. Hence, the most adsorbed surfactant molecules at the interface are the most powerful emulsifying agents for the emulsions. The emulsification efficiency in this study was measured as the time required for separation of 9 mL of pure water from the emulsion formed from 10 mL of surfactant solution and 10 mL of paraffin oil. Increasing the time required for separation of the desired amount of water from the emulsified system indicates the stability of that emulsion, and vice versa. Table 2 shows that derivatives containing shorter hydrophobic chains exhibit low emulsification efficiencies.

Corrosion Inhibition Efficiency

The calculations of the corrosion rate in millimeter per year (mpy) were made using the following formula:

$$\text{Corrosion rate (mpy)} = (KW)/(ATD)$$

where, K is a constant (3.45×10^6), T is time of exposure in hours, A is the area in cm^2, W is the mass loss (g), D is the density of the steel used.

While, the corrosion inhibition efficiency of the synthesized corrosion inhibitors was calculated from the corrosion rate data as follows:

$$\text{Efficiency } (\eta) = [(Cr - Cr')/Cr] \times 100$$

where, η is the corrosion inhibition efficiency, Cr is the corrosion rate in the absence of inhibitors, Cr' is the corrosion rate in the presence of inhibitors.

Figure 3 represents the variation of the corrosion inhibition efficiencies of the synthesized cationic inhibitors by increasing the hydrophobic chain length. It is clear that increasing the alkyl chain length from 12 to 14, 16 and 18 carbon atoms decreases the inhibition efficiency of the inhibitors. This effect is inverted in several studies [21, 22], but agreed with our previous work on some Schiff base derivatives [13]. The inhibition of the corrosion processes by the cationic inhibitors in acidic media revealed the adsorption of these inhibitors at the metal surface. The inhibition efficiency was increased by increasing the surface concentration of the inhibitors at the metal surface. This decrease in the inhibition efficiency was attributed to two reasons. The first is the gradual decrease in the critical micelle concentration values (CMC) by increasing the hydrophobic chain lengths. The decrease in CMC values indicates the high tendency of these amphiphiles towards micellization. Hence, the adsorbed molecules at the interface will decrease. The second reason is the surface concentration, i.e., the concentration of the inhibitor molecules at the interface was decreased by increasing the hydrophobic chain length (Table 2). Hence, the uncovered area of the metal surface will increase. The two reasons are

responsible for increasing the corrosion rate and consequently the corrosion inhibition efficiency will decrease.

In spite of the corrosion inhibition efficiency being decreased by increasing the hydrophobic chain length, their values were still high and ranged between 99.92 and 97.25% at 400 ppm and 99.88 and 95.69% at 200 ppm. In addition, the corrosion rates ranged between 0.01 and 0.09 mpy at 400 and 200 ppm for 24 h. While, at 48 h, the efficiency was ranged between 98.33 and 94.77% at 400 ppm and 98.11–94.4% at 200 ppm with a corrosion rates ranged between 0.26 and 0.85 mpy at 400 and 200 ppm. These values are considered to be in the acceptable range in the relatively high acidic concentrations (2 N HCl and H_2SO_4). The high values of the corrosion inhibition efficiencies of the synthesized inhibitors can be seenin Table 3. The high corrosion inhibition efficiencies of the synthesized inhibitors are revealed by their chemical structure. The presence of the positively charged (N^+) group and unsaturated imine group in addition to the π-electron cloud on the benzene ring facilitates the adsorption of these inhibitors at the metal surface. That adsorption

presence of inhibitors and *CR* is the corrosion rate in the absence of inhibitors. The decrease in the inhibitor efficiency at lower doses can be attributed to the decrease in the surface coverage values (θ) of the metal surface by inhibitor molecules at a lower dose (200 ppm). While, at the higher dose (400 ppm) the surface coverage of the metal by inhibitor molecules increased, hence, the corrosion inhibition efficiencies increased (Tables 3, 4).

The Schiff base derivatives are characterized by their strong tendency towards complexation with the transition metal ions. Also, the complexation tendency is increased by introducing several function groups to their chemical structures. In the case of the targeted inhibitors, the quaternary ammonium group $[(CH_3CH_2)_2N^+CH_3]$ increases their complexation tendency extremely. The complexation between the metal ions and the Schiff bases is by the donation of the lone pairs of electrons of the nitrogen atoms in the azomethine groups (–N=C–) and the vacant *d*-orbital of the metal ions [1, 9, 20].

Complexation reaction between the synthesized compounds and the Fe^{2+} ions
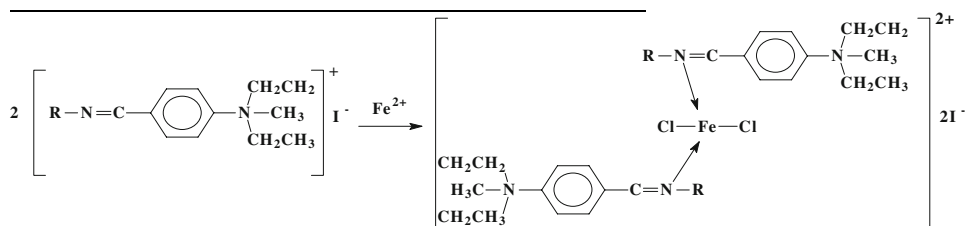
occurred on the positive and negative centers on the metal surface which decreases the electrochemical dissolution of the metal to ions in the solution (Eqs. 3 and 4).

$$Fe \rightarrow Fe^{++} + 2e \qquad (3)$$

$$2H^+ + 2e \rightarrow H_2 \qquad (4)$$

Figure 4 represents the effect of the different inhibitor doses on the corrosion inhibition efficiency of the synthesized inhibitors. At higher inhibitor doses (400 ppm) the corrosion inhibition efficiency was found to have its maximum values. In contrast, decreasing the inhibitor dose decreases the inhibition efficiency considerably. The ratio of surface coverage (θ) of the metal by inhibitor molecules was calculated at different doses using the formula:

$$\theta = (CR - CR')/CR$$

where, θ is the ratio of the area of metal surface covered by the inhibitor molecules, CR' is the corrosion rate in the

This facilitates the complexation process between the dissociated Fe^{2+} ions from the slow corrosion reaction and the adsorbed Schiff base inhibitors. Hence, the adsorbed inhibitors on the metal surface decreased with time and the corrosion rate increased. Consequently, the corrosion inhibition efficiency decreases by increasing the contact time between the inhibitor molecules and the metal ions as shown in Fig. 5.

HCl acid produces only one proton while H_2SO_4 acid produces two protons upon their dissociation in the solutions. Hence, the attacking power of H_2SO_4 is double of that in HCl as shown in Eqs. 5, 6, and 7.

$$HCl \rightarrow H^+ + Cl^- \qquad (5)$$

$$H_2SO_4 \rightarrow H^+ + HSO_4^- \qquad (6)$$

$$HSO_4^- \rightarrow H^+ + SO_4^- \qquad (7)$$

It is clear from Fig. 6 that the corrosion inhibition efficiencies of the synthesized inhibitors in the H_2SO_4 acidic

Table 3 Corrosion rates (CR), surface coverage (θ) and corrosion inhibition efficiencies (Eff.) of the synthesized inhibitors in different acidic media for 24 h at 25 °C

| Inhibitor	2 N HCl						2 N H$_2$SO$_4$					
	400 ppm			200 ppm			400 ppm			200 ppm		
	CR	θ	Eff.	CR	θ	Eff.	CR	θ	Eff.	CR	θ	Eff.
SB-12	0.013	0.999	99.92	0.018	0.998	99.88	0.19	0.987	98.74	0.31	0.979	97.96
SB-14	0.044	0.997	99.71	0.084	0.994	99.45	0.21	0.986	98.62	0.54	0.965	96.5
SB-16	0.08	0.994	99.49	0.09	0.994	99.41	0.4	0.974	97.41	0.58	0.962	96.22
SB-18	0.08	0.972	97.25	0.09	0.956	95.69	0.43	0.972	97.21	1.18	0.922	92.28

Fig. 4 Effect of the inhibitor dose on the inhibition efficiency of the synthesized inhibitors
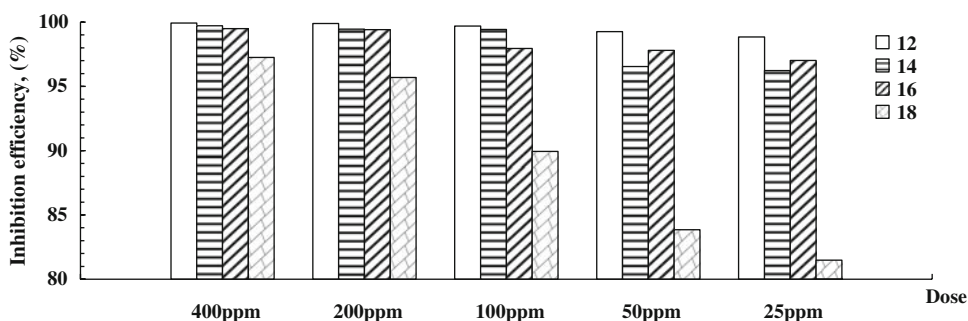
Table 4 Corrosion rates (CR), surface coverage (θ) and corrosion inhibition efficiencies (Eff.) of the synthesized inhibitors in different acidic media for 48 h at 25 °C

| Inhibitor	2 N HCl						2 N H$_2$SO$_4$					
	400 ppm			200 ppm			400 ppm			200 ppm		
	CR	θ	Eff.	CR	θ	Eff.	CR	θ	Eff.	CR	θ	Eff.
SB-12	0.26	0.983	98.33	0.29	0.981	98.11	0.45	0.970	97.05	0.51	0.966	96.64
SB-14	0.48	0.968	96.84	0.50	0.967	96.70	0.82	0.946	94.62	1.67	0.885	88.46
SB-16	0.53	0.965	96.56	0.53	0.965	96.53	1.00	0.934	93.44	2.10	0.863	86.27
SB-18	0.80	0.948	94.77	0.85	0.944	94.46	1.41	0.907	90.77	4.76	0.695	69.50

Fig. 5 Effect of time exposure on the inhibition efficiency of the synthesized inhibitors at 200 ppm
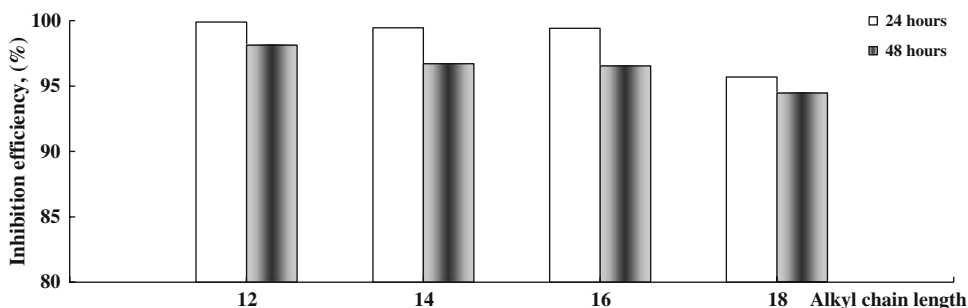
medium are lower than those in the HCl medium. This effect can be attributed to the protonating power of the two corrosive acids. Increasing the protonating power of the acid increases its aggressive action on the metal, which lowers the influence of the inhibitors on the protection process. As a result, the inhibition efficiency decreases.

In conclusion, the synthesized cationic Schiff base amphiphiles are characterized by high corrosion inhibition efficiency for carbon steel in HCl and H$_2$SO$_4$ media. Also, their inhibition efficiencies as corrosion inhibitors were in the acceptable range referring to the corrosion rate which ranged between 0.26 and 0.85 mpy. Hence, these inhibitors

Fig. 6 Effect of corrosive medium on the inhibition efficiency of the synthesized inhibitors at 200 ppm
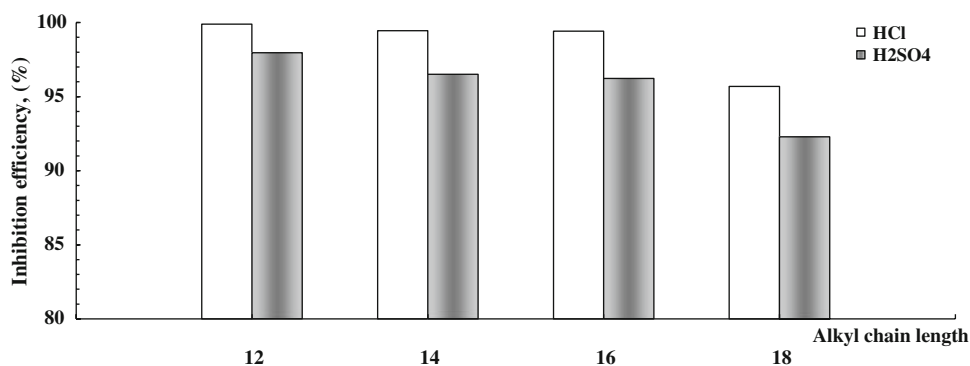
can be considered as efficient additives for metal processing in acidic media and also for short circle petroleum industries (short circle processes are the processes which take place in relatively short time as acid cleaning of the pipeline from scale which take about 4 h of washing the pipelines with acids to remove the precipitated scale).

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Mohamad F. Zaki is an assistant professor in the Petrochemicals department of the Egyptian Petroleum Research Institute. He is interested in surfactants applications, mainly corrosion and biological activity. He received his Ph.D. for a thesis on the field of surfactant applications from Ain Shams University, Cairo, Egypt (2005).

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