

EIS Studies of Anionic Surfactants Inhibiting Effect of AI-2017 Corrosion in HCI Aqueous Solutions

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Abstract: This paper describes the use of the EIS (electrochemical impedance spectroscopy) technique and potentiodynamic polarization in order to study the corrosion inhibition process of aluminium in hydrochloric acid solution. Three anionic surfactants: GOS (gasoil sulfonate), KES (kerosene sulfonate) and HSS (heavy solvent sulfonate) have been synthesized from petroleum fractions and tested during corrosion of aluminium in 1 M HCl solution at 25 °C. The Nyquist diagrams consisted of a capacitive semicircle at high frequencies followed by a well defined inductive loop at low frequency values. The impedance measurements were interpreted according to a suitable equivalent circuit. The results obtained showed that the addition of the surfactants inhibits the hydrochloric acid corrosion of aluminium. The inhibition occurs through adsorption of the surfactant on the metal surface without modifying the mechanism of corrosion process. Potentiodynamic polarization measurements showed that the surfactant acts predominately as cathodic inhibitor. The inhibition efficiency increases with rising of the inhibitor concentration and decreases in the order: GOS > KES > HSS. The corrosion inhibiting mechanism is thought to proceed via an adsorption of the surfactant molecules on the aluminium surface, generating a film and hindering the active sites. Our experimental adsorption data were found to obey the Langmuir adsorption isotherm. Optical images of the treated specimens, revealing the likely formation of a protective film, demonstrated the inhibiting capacity of the petroleum surfactants.

Key words: Aluminium, inhibitor, corrosion, HCl acid, electrochemical impedance spectroscopy.

1. Introduction

Corrosion of Al (aluminum) in hydrochloric acid solution and its control is an active area of research due to its high industrial importance. The control of this corrosion assumes greater significance, particularly by the use of inhibitors due to its easy methodology. The acid inhibitors find extensive applications in pickling, chemical and electrochemical etching of aluminum [1]. The corrosion inhibition of aluminum in acidic solutions is based on organic compounds containing nitrogen, oxygen, sulfur atoms and multiple bonds in the molecules that facilitate adsorption on the aluminum surface [2-10]. The inhibition efficiency of organic compounds is related to their adsorption properties. Adsorption depends on

the nature and the state of the metal surface, on the type of corrosive medium and on the chemical structure of inhibitor [11]. Studies reported that the adsorption of inhibitors mainly depends on some physicochemical properties of the molecule, related to its functional group, to the possible steric effects and electronic density of donor atoms, adsorption is also to depend on the possible interaction of π -orbital's of the inhibitors with d-orbital of the surface atoms which induce greater adsorption of inhibitor molecule on tip the surface of metal leading to the formation of a corrosion protection film [12].

Recently, many surfactants were widely investigated as corrosion inhibitors for various metals and alloys in acidic media. Hence, SDBS (dodecyl benzene sulfonate) was used alone as an AS (anionic surfactant) inhibitor for the corrosion of aluminium and steel [13-16]; or with heterocyclic organic compounds in inhibitive mixture to prevent

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aluminium alloys corrosion [17, 18]. In addition influence of SDS (sodium dodecylsulfate) on aluminium, nickel and lead alloys has been studied [19, 20].

In the present work three new surfactants: GOS (gasoil sulfonate), KES (kerosene sulfonate) and HSS (heavy solvent sulfonate) have been synthesized from petroleum fractions and tested during corrosion of aluminium in 1.0 M HCl solution. The choice of these surfactants as corrosion inhibitors is based on the following reasons: these molecules can be synthesised easily, they contain sulfonate groups as active centres, and they have high solubility in acidic media and are not expensive. The study was conducted using impedance spectroscopy methods and potentiodynamic polarization, complemented with scanning electron microscopy. In addition, the effect of immersion time on the inhibition efficiency and adsorption mechanism has been investigated.

2. Experimental Conditions

2.1 Materials and Equipment

Three types of commercial petroleum fractions: heavy solvent, kerosene and gasoil were obtained by distillation method from Algerian crude oil. They were used as reactant in sulfonation reaction. Oleum and calcium hydroxide $Ca(OH)_2$ were obtained from Merck. They were used as sulfonating agent and neutralizer, respectively. Experiments are classified into two main steps as traced in Fig. 1. The first step was surfactant production, petroleum fractions were used as base materials for sulfonation process. The sulfonation process was conducted in reaction cell equipped with a mechanical stirrer. The temperature was maintained constant at 40 °C with thermostat. The sulfonation agent was 20% oleum where the major components that can be sulfonated in petroleum fractions are aromatic hydrocarbons (alkyl benzenes) [21]. Two phases were obtained, the oils sulfonated products in the form of boil acid (which are denser) recovered with the unsulfonated oils (namely: the unreacted alkyl benzene and paraffin hydrocarbons) in the higher phase. After separation of two phases, the second step was the neutralisation of the produced sulfonated oils with calcium hydroxide solution.

The aggressive solutions (1 mol dm⁻³ of HCl) were prepared by dilution of analytical grade 37% HCl (Merck) with distilled water. The concentrations of inhibitors were screened from 0.25 to 1 g/L. The working electrode was made aluminium 2017. Its chemical composition was as follows: 0.53% Mg, 0.59% Mn, 0.62% Si, 0.51% Fe, 0.09% Zn, 0.03% Ti, 0.12% Cr, 3.83% Cu, and Al.

For electrochemical measurements, the surface pre-treatment of specimens was carried out by grinding with emery paper of 600-1,200 grit, washed in acetone and distilled water and put into the cell.

2.2 Electrochemical Measurements

The electrochemical measurements were conducted in a conventional three-electrode cell. A platinum disc was used as counter electrode, the reference electrode was a SCE (saturated calomel electrode) and the working electrode was aluminium disc embedded in



Fig. 1 Synthesis of anionic surfactants.

Araldite. The surface area in contact with corrosive solution is A = 0.785 cm². Measurements were potentiostat/galvanostat performed using а (Radiometer-PGZ 301), controlled with Voltamaster 5 software. Before recording the polarization curves, the open circuit potential stability was controlled during 30 min. Anodic and cathodic polarization curves were recorded from -200 to 200 mV and potential scanning rate of 1 mV/s. Inhibition efficiencies were determined from corrosion currents calculated by the Tafel extrapolation method and fitting the curve to the polarization equation as well. EIS diagrams were obtained in the frequency range of 100 kHz to 10 mHz and a sine wave potential amplitude of $\Delta E = 5 \text{ mV}$ after 30 min of immersion.

2.3 SEM (Scanning Electron Microscopy)

Immersion corrosion analysis of aluminium samples in the acidic solutions with and without the optimal concentration of the inhibitor was performed using SEM. Immediately after the corrosion tests, the samples were subjected to SEM studies to know the surface morphology. SEM (JEOL JSM 5300) was used for the experiments.

3. Results and Discussion

3.1 Impedance Measurements (EIS)

Effect of immersion time

Fig. 2 shows the impedance spectra at different immersion time in 1.0 M HCl (Fig. 2A) and in HCl 1 M with 1 g/L of GOS (Fig. 2B). Examinations of the Nyquist plots of EIS diagrams show that the impedance spectra are composed of a large capacitive loop at HF (high frequency) followed by inductive semicircle part in the LF (low frequency) domain. Usually, the HF capacitive loop is related to charge transfer of Al dissolution process and the LF inductive loop is related to the relaxations processes in the adsorbed species on electrode surface. The slightly depressed nature of the semi-circle, which has the centre below the real part-axis, is the characteristic for solid electrodes. Such frequency dispersion has been attributed to the roughness and inhomogeneous of electrode surface [22, 23]. It is worth noting that the profile of the impedance is the same and without inhibitor, suggesting that the Al corrosion occurs with the same mechanism.

It is marked that the impedance diagrams exhibit one capacitive loop and one inductive loop. It is also noted that the increasing of immersion time affects the diameter loops.

Effect of the immersion versus inhibition efficiency graph was plotted in Fig. 3. It is clear that the increase in the immersion time does not affect much on the inhibition efficiency. These results demonstrate that the formation of surface film, and therefore the GOS adsorption, on the electrode surface is stable with immersion time.

The inhibition efficiency is calculated using charge transfer resistance values and the following formula [24]:

$$\eta_z \% = [(R_t - R_{t0})/R_t] \times 100$$
(1)

where R_{t0} and R_t are respective, the charge transfer resistance value in the absence and the presence of the inhibitor.

It is worth mentioning that the double layer capacitance, C_{dl} value is affected by imperfections of the surface. To take into account this effect, usually a CPE (constant phase element) is introduced in the EC [22] and the value of the capacitance C_{dl} can be calculated from the following relation [23]:

$$C_{dl} = Q(2\pi f_{max})^{\alpha-1}$$
(2)

where Q and α are the constant phase element coefficients, f_{max} represents the frequency at which the imaginary part of the impedance reaches its maximum value.

In Table 1 the data show clearly the decrease of R_t comparing with that in blank solution (without inhibitor) indicating that Al corrosion rate is lower when GOS is present in the acidic solution. However, the C_{dl} values decrease in presence of GOS, indicating



Fig. 2 (A) Nyquist plots of the impedance diagrams measured on aluminium at different immersion time in acidic solution 1 M HCl.



(B) Nyquist plots of the impedance diagrams measured on aluminium at different immersion time in acidic solution 1 M HCl and 1 g/L of GOS.

that the surface coverage by the adsorbed inhibitor increases, consequently the surfactant is efficient. In addition, the decrease in C_{dl} , may be results from a decrease of dielectric constant and/or an increase in the electrical double layer thickness, due to the

inhibitor adsorption at the metal/solution interface [25].

Fitting procedure

In order to simulate the effect of the presence of inhibitor in 1 M HCl solution, the impedance spectra



Fig. 3 Variation of inhibition efficiency in acidic solution 1 M HCl on aluminium with different immersion time.

Table 1 Impedance measurements and inhibition efficiencies for Al in 1 M HCl in the absence and the presence of GOS at different immersion time, at 25 °C.

Time (hour)	Blank (HCl 1 M)		Inhibitor (GOS)		Inhibition efficiency (η_z)
	$R_t (\Omega \cdot cm^2)$	$C_{dl} (\mu F \cdot cm^{-2})$	$R_t (\Omega \cdot cm^2)$	$C_{dl} (\mu F \cdot cm^{-2})$	
0.5	5.56	360.6	41.38	61.3	86.56
1	4.39	445.1	25.27	75.2	82.62
1.5	3.21	674.4	17.90	160.2	82.06
2	2.27	993.0	11.31	273.9	79.98

were fitted with an EC (equivalent circuit). Fig. 4 shows the corresponding circuit which consists of the following elements: a solution resistance R_s related to the conductivity of the electrolytic solution, the charge transfer resistance, R_{ct} linked to the reaction rate, and the CPE (constant phase element) Q linked to the capacity of double layer charge Cd, for the high frequency loop. For the low frequency part of the impedance the characteristic parameters are introduced in the EC. Usually when some parts of diagrams are located in the negative imaginary part of the Nyquist plot it means that adsorption/desorption process of certain intermediates species are occurred in the reaction and the response exhibits an inductive relaxation type [5]. To take into account this process in the fitting procedure, a resistance R and an induction L are introduced in the EC.

The fitting procedure has been performed using commercial software ZSimpWin 3.21 (EChem Software) based on the use of equivalent circuits and CPEs. For the different diagrams, the equivalent circuit presented in Fig. 4 was tested. An example of detailed numeric results of the fittings is presented in Table 2, for some values. Despite of the great number of adjustable parameters, many of them have been obtained with a reasonable statistical determination (for instance the standard error is less than 20 % for R_s , R_{ct} and Q).



Fig. 4 Equivalent circuit used for the fitting procedure of the experimental EIS data. R_s is the solution resistance, R_{ct} is the charge transfer resistance, Q is the CPE element, R is the adsorption resistance and L is the adsorption coil.

Table 2 Some results of the fitting of the impedance data obtained for Al at different immersion time in 1 M HCl with and without 1 g/L of GOS, at 25 °C.

Time/min	30	60	90	120	
R _s /cm ²	2.95	2.99	2.90	2.94	
St. er./%	1.55	1.324	1.51	1.98	
R_{ct}/cm^2	41.38	25.27	17.9	11.34	
St. er./%	1.68	2.02	2.53	2.47	
$Q/\mu F \cdot cm^{-2}$	61.3	75.2	160.2	273.9	
St. er./%	8.44	12.52	14.58	13.57	
n	0.921	0.902	0.912	0.909	
St. er./%	3.01	1.83	2.63	2.85	
L/H·cm ²	438.9	319.7	171.8	167.1	
St. er./%	8.289	8.69	6.57	8.65	
R/cm ²	39.57	19.19	7.98	7.56	
St. er./%	10.26	10.95	8.74	12.28	

The term St. er., corresponds to the standard error, in %, for the each fitted parameter.

Effect of inhibitor concentration

In order to get more information about the corrosion inhibition phenomenon, EIS (electrochemical impedance spectroscopic) measurements have been carried out for aluminium, for various concentrations of inhibitors in 1 M HCl solution at 25 °C (Figs. 5A-5C).

In Table 3 the data show clearly that the R_t values increase with an increase of concentration of inhibitor indicating that Al corrosion rate is lower when high quantity of inhibitor is present in the acidic solution. Consequently the inhibition effects of the surfactant are more efficient.

According to Table 3, the inhibition efficiency decreases in the order: GOS > KES > HSS. This result shows that the increase of the length of hydrophobic

parts of the molecule (the increasing of the number of carbon atoms) induces more inhibition efficiency. From this we can consider that GOS ($R = C_{12} - C_{24}$) was the best inhibitor. It can be explained that the presence of the electron donating group on the surfactant such as hydrophobic chain increases the electron density on the sulfonate group, increasing the bond strength between the molecule and the metal surface [26].

The functional groups and inhibitor structure play important roles during the adsorption process. Due to the presence of some active sites, such as aromatic rings, heteroatom, and alkyl groups, the surfactant may act as adsorption inhibitors, and their corrosion inhibition efficiencies are directly proportional to the amount of adsorbed inhibitor [27, 28]. Surfactant adsorbs



Fig. 5 (A) Nyquist plots of the impedance diagrams measured on aluminium in acidic solution 1 M HCl at different concentrations of HSS.



(B) Nyquist plots of the impedance diagrams measured on aluminium in acidic solution 1 M HCl at different concentrations of KES.



(C) Nyquist plots of the impedance diagrams measured on aluminium in acidic solution 1 M HCl at different concentrations of GOS.

Table 3 Impedance measurements and inhibition efficiencies for Al in 1 M HCl in the absence and the presence of HSS, KES and GOS at different bulk concentrations at 25 °C.

Inhibitor	Concentration (g/L)	$R_t (\Omega \cdot cm^2)$	Inhibition efficiency (η_z)	Surface coverage (⊖)	
Blank	0				
HSS	0.25	8.36	33.4	0.334	
	0.5	11.12	50.0	0.500	
	1.0	15.97	65.1	0.651	
KES	0.25	12.56	55.7	0.557	
	0.5	24.89	77.6	0.776	
	1.0	27.15	79.5	0.795	
GOS	0.25	16.88	67.0	0.670	
	0.5	29.39	81.0	0.810	
	1.0	41.38	86.5	0.865	

on the aluminium surface with both, the polar head and non-polar hydrocarbon chain interaction with the metal surface. In fact, the inhibitive action of surfactant in acid solution results from the adsorption of the negatively charged SO_3^- species to the positively charged electrode, forming a barrier layer by hydrocarbon chain on the Al surface [29].

3.2 Polarization Measurements

The variations of the corrosion potential (Ecorr) as functions of time and GOS concentration, are presented in Fig. 6. It can be noticed that the potential in the absence of inhibitor was higher than in the presence, and rose with time. Systematic dependence of the potential on the inhibitor concentration was observed; yet, a stable value of -599 mV/SCE was attained after 30 min of immersion. Ecorr in the absence of inhibitor was -582 mV/SCE after this time. Thus, it appears that the corrosion potentials E_{corr} (open-circuit potential value) are shifted to more negative values.

Fig. 7 shows the polarization curves of aluminium simple in acidic solution measured without/with various concentrations of GOS. The corresponding corrosion data are given in Table 4. The data clearly show that the addition of surfactants enhances mainly



Fig. 6 The variations of the corrosion potential (Ecorr) as functions of time and GOS concentration.



Fig. 7 Potentiodynamic polarization curves of aluminium in acidic solution 1 M HCl in the absence and the presence of various concentrations of GOS.

Table 4 Kinetic parameters (corrosion current density, I_{corr} and Tafel slope, b_c) and inhibition efficiency determined from polarization curves of Al in 1 M HCl in the absence and the presence of GOS at different concentrations at 25 °C.

C (g/L)	E _{corr} vs. SCE (mV)	I_{corr} ($\mu A \cdot cm^{-2}$)	b_c (mV·dec ⁻¹)	Inhibition efficiency (η_p)
Blank	-582	254.9	296	_
0.25	-593	83.3	252	67.3
0.5	-598	47.1	209	81.5
1	-599	33.3	200	86.9

the cathodic overpotential. At the same time, the corrosion potentials, E_{corr} (open-circuit potential value) are shifted to more negative values. These results indicate that the inhibitor used here, have the same effect and act predominately as cathodic inhibitors. This effect may be related to the potential at the cathodic sites which is more positive than that at the anodic sites, and consequently the adsorption of the anionic surfactants is much more likely at the cathodic sites [30]. The cathodic branch of polarization curves give rise to parallel Tafel lines, which indicate that HER (hydrogen evolution reaction) is under activation control and that the addition of the surfactants does not modify the HER mechanism [31, 32].

The corrosion efficiency $(\eta_p\%)$ was calculated from polarization measurements according to the relation [33]:

$$\eta_{\rm p}\% = [(I_0 - I)/I_0] \times 100$$
 (3)

where I_0 and I are respective, the corrosion current measured without and with inhibitors.

Adsorption isotherms

In order to check the adsorption mode of the

various inhibitors during Al corrosion in acidic solution, the obtained data have tested using various adsorption isotherms.

As explained above, the inhibition power of inhibitor is related mainly to its adsorption capacity surface. aluminium The commensurate onto adsorption mode and its model isotherm would provide an insight into the interactions types between adsorption inhibitor molecules and the aluminium surface. The value of the metal surface coverage extent (θ), calculated from the impedance measurements, was used for establishing the corresponding adsorption isotherm. Fig. 8 depicts the variations of C_{inb}/θ as a function of C_{inb} (the inhibiting concentration) in the range of the concentrations studied. A linear dependence was found, with a high correlation coefficient, $R^2 = 0.99$, indicating that the three inhibitors absorption during Al corrosion in acidic solution is a Langmuir isotherm type governed by Eq. (4) [25]:

$$C_{inb}/\theta = C_{inb} + 1/K_{ads}$$
(4)

where K_{ads} is the adsorption-desorption equilibrium constant.



Fig. 8 The linearized Langmuir adsorption isotherm for the adsorption of inhibitors onto aluminium in acidic solution 1 M HCl.



Fig. 9 SEM images of aluminium samples: (A) after polishing; (B) after 1 h of immersion in 1 M HCl in the absence of inhibitor; (C) after 1 h of immersion in 1 M HCl in the presence of 1 g/L of GOS.

3.4 SEM

SEM images of aluminium samples are shown in Fig. 9. Fig. 9B indicates a strong corrosion attack after immersing the aluminium sample into 1 M HCl for 1 h. A high density of corrosion pits can be observed, in contrast, only a few pits are observed in SEM images of samples tested, under the same experimental conditions, in the presence of 1 g/L of GOS (Fig. 9C). This result is in agreement with the electrochemical experiments.

4. Conclusions

The following conclusions may be drawn from the study: The inhibiting efficiency increases with increasing of the inhibitor concentration and depends on the substituent of the anionic surfactants derivatives according to order GOS > KES > HSS. Impedance spectra consist of a large capacitive loop at high frequencies followed by a small inductive one at low frequency values. The high frequency capacitive loop is usually related to the charge transfer of the corrosion process and the double layer behavior, and the inductive loop may be attributed to the relaxation or related to stabilization of layer by adsorbed intermediate products of the corrosion reaction on the electrode surface involving inhibitor molecules. The electrochemical measurements showed that these surfactants act predominantly as cathodic inhibitor without modifying the mechanism of hydrogen evolution reaction. The high corrosion inhibition effect is obtained with GOS and increases with the inhibitor concentration. The adsorption of the inhibitors molecules on the aluminium surface obeys Langmuir adsorption isotherm.

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