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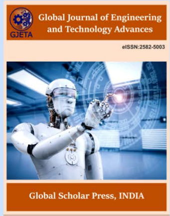

(Research Article)

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Gravimetric and electrochemical studies of the control of aluminum deterioration in HCl using Ukpo clay - polyaniline as inhibitor

Fidelis Ejike Iloamuzor [1], Gordian Onyebuchukwu Mbah[2], Monday Omotioma [2, *] and Dominic Okechukwu Onukwuli [3]

[1] *Material and Energy Technology Department, Projects Development Institute (PRODA), Emene, Enugu, Nigeria.*
[2] *Department of Chemical Engineering, Enugu State University of Science and Technology, Agbani, PMB 01660, Enugu, Nigeria.*
[3] *Department of Chemical Engineering, Nnamdi Azikiwe University, Awka, Anambra State, Nigeria.*

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Abstract

This study presents the use of Ukpo clay- polyaniline as inhibitor to control deterioration of aluminium in HCl. The substance (inhibitor) was characterized using gas chromatography-mass spectrophotometer. Gravimetric (weight loss) and electrochemical (potentiodynamic polarization and electrochemical impedance spectroscopy) techniques were employed in the corrosion control procedure. Analyses of the experimental results showed that Ukpo clay/polyaniline composite contains chemical constituents (2-cyclopropyl-2-methyl-n-(1-cyclopropylethyl), 2-bromo-4-fluorophenyl ester, bis(2-ethylhexyl) phthalate, piperidine-4-carbonitrile) suited for the deterioration control of Al in HCl. For the gravimetric analysis, the corrosion inhibition process was found to be spontaneous, exothermic, and in agreement with physical adsorption mechanism. The activation energy of inhibition was less than the critical values of 80kJ/mol, and Langmuir adsorption isotherm best described the inhibition procedure. The relationship between inhibition efficiency of Ukpo clay - polyaniline composite and factors of inhibitor concentration, temperature and time is a quadratic model. Electrochemical analysis of the study revealed that Ukpo clay/polyaniline composite acted as mixed-type inhibitor. It inhibited both cathodic and anodic reactions. Adding the inhibitor to the HCl medium enhanced the magnitude of the impedance spectrum, which indicates high inhibiting strength of the inhibitor's constituents in impeding the invasion of the electrochemical reaction. Thus, Ukpo clay - polyaniline composite should be applied as a viable corrosion inhibitor of aluminium in hydrochloric acid environment.

Keywords: Ukpo clay; Polyaniline; Inhibitor; Aluminium; HCl medium

1. Introduction

As a metallic material, aluminum is widely used in production and transportation industries. Broad utilization of aluminum has necessitated curiosity in the processing of its ore for better quality product. The quality of processed aluminum depends on the percentage of impurities contained in the processed form. Those impurities alter their surface structure thereby making it susceptible to corrosion. Corrosion of aluminum is a destructive attack of aluminum metal as a result of electrochemical activity between the metal and its environment. Corrosion is caused by oxidation and reduction reactions (Omyma and Shadia 2011). Generally, metals tend to corrode because they struggle to return back to their original state thereby lowering their Gibbs free energy. To avert the reversal back to the original form owed to deterioration or any other distortion on the metallic surface, it is pertinent to carry out corrosion control.

* Corresponding author: Omotioma M

Corrosion control in metals is an imperative activity of environmental, technical, economical and aesthetical importance, (Arthur et al, 2013). There are different ways to control corrosion, but the most efficient method is the use of inhibitors, Kim et al. (2002). Corrosion inhibitors prevent corrosion. Corrosion inhibitors are adsorbed on the metal surface and act by forming a protective layer or barrier to oxygen or moisture. Protective layer is formed as passive film on the metal surface. Numerous researches were carried out using corrosion inhibitors like plant extracts, drugs, ionic liquids, polymer material (polyaniline). The inexpensive is plant extract but the alteration of their efficiency is perturbing, this is because it depends on the location. Polyaniline is cheap, good and efficient corrosion inhibitor, Wu et al (2000), but environmental concern makes it not suitable for industrial application due to its non-biodegradability. Therefore, there is a compelling need to alter their nature by blending it with Ukpo clay material, making the surface to open for better degradation and absorption.

2. Materials and Method

2.1. Characterization of the inhibitor (Ukpo clay - polyaniline)

Chemical analysis of Ukpo clay - polyaniline was carried out using gas chromatography-mass spectrophotometer (Agilent model 7890A and 5977B MSD). In the instrumental analysis, the gas chromatography-mass spectrophotometer combined the features of gas chromatography and mass spectrophotometer to recognize different substances within the inhibitor. When the sample of the inhibitor was heated in the gas chromatography-mass spectrophotometer, its molecular constituents were separated into distinct substances. The substances were carried through a column with an inert gas (nitrogen). As the separated substances emerged from the column opening, they flew into the mass spectrophotometer. The mass spectrophotometer identified the chemical constituents in the inhibitor.

2.2. Gravimetric Method of the Corrosion Inhibition Study

Considering one factor at a time, the weight loss method was carried out at different temperatures, time and inhibitor concentrations. According to this method, aluminium samples were separately immersed in 250 ml open beakers containing 200 ml of 1 M HCl (blank). Also, aluminium coupons were separately immersed in 250 ml open beakers containing 200 ml of 1 M HCl with various concentrations of the inhibitor. The variation of weight loss was monitored periodically at various temperatures and immersion time, in the absence and presence of various concentrations of the inhibitors. The experimental readings were employed to determine the degree of surface coverage (Θ) in line with the method used by previous research report (Onukwuli and Omotioma, 2019).

$$\theta = \frac{\omega_0 - \omega_1}{\omega_0} \quad \text{..................(1)}$$

where ω_1 and ω_0 are the weight loss values in presence and absence of the inhibitor, respectively.

The data of the degree of surface coverage (Θ) were fitted into the following adsorption models (Omotioma and Onukwuli, 2016; Anadebe et al, 2018; Onukwuli and Omotioma, 2019):

1. Langmuir isotherm;

$$log\frac{C}{\theta} = \log C - logK \quad \text{.................. (2)}$$

2. Temkin isotherm;

$$\theta = -\frac{2.303logK}{2a} - \frac{2.303logC}{2a} \text{.................. (3)}$$

3. Frumkin isotherm

$$\log\left((C) * \left(\frac{\theta}{1-\theta}\right)\right) = 2.303logK + 2\alpha\theta \text{.................. (4)}$$

4. Flory-Huggins isotherm

$$\log\left(\frac{\theta}{C}\right) = \log K + x\log(1-\theta) \qquad (5)$$

The free energy of adsorption (ΔG_{ads}) was the obtained using Equation (6):

$$\Delta G_{ads} = -2.303RTlog(55.5K) \qquad (6)$$

In this case, C represents the concentration of inhibitors, K is equilibrium constant, θ is the degree of surface coverage, α is the lateral interaction term, x is the size parameter.

R is the gas constant, T is temperature. K values obtained from the isotherms (Langmuir, Frumkin, Temkin and Flory-Huggins isotherms).

Response surface methodology in Design Expert Software was used to design the experiment for the weight loss method. Inhibitor concentration, temperature and time were the considered factors while inhibition efficiency was the expected response of the study, which was determined using Equation (7). The RSM was used to analyze the responses in line with previous reports (Omotioma and Onukwuli, 2016; Anadebe et al, 2018). The ANOVA and graphical analyses of the inhibition efficiencies were carried out. The mathematical models in terms of coded factors were obtained.

$$IE\% = \frac{\omega_0 - \omega_1}{\omega_0} * 100 \qquad (7)$$

The variations of inhibition efficiency with concentration, temperature and time were analyzed using graphs and mathematical models determined by response surface methodology (RSM). The RSM will effectively achieve the optimization by analyzing and modeling the effects of multiple variables and their responses and finally optimizing the process. The analysis of the data will include 3-D graphical representations of the relationships among inhibition efficiency and factors of concentration, temperature and time.

2.3. Electrochemical Studies

Electrochemical (potentiodynamic polarization and electrochemical impedance spectroscopy) studies were carried out according to the method used by Oguzie et al (2010), Ihebrodike et al (2011) and Anadebe et al (2018). Electrochemical tests were conducted using a potentiostat/galvanostat, electrochemical system workstation, with conventional three-eletrode cell. A graphite rod and a saturated calomel electrode (SCE) were used as counter and reference electrodes, respectively. The aluminium fixed in epoxy resin with a surface area of 1 cm^2 exposed to the test solution, served as the working electrode. Electrochemical measurements were carried out in aerated and unstirred solution at the end of 1800 s of immersion, which allowed the open circuit potential (OCP) to attain steady state (Al-Otaibi et al, 2014; Anadebe et al, 2019). Temperature was fixed at 30±1 ^{0}C. Polarization studies were performed in the range of ±250 mV versus corrosion potential at a scan rate of 0.333 mV/s.

3. Results and Discussion

3.1. Characteristics of the inhibitor as determined by the GCMS

Chromatogram of the GCMS is presented in Figure 1. Chemical constituents of the Ukpo clay - polyaniline (UCPC) were revealed as aniline, isobutyl chloroformate, 9,12-octadecadienoic acid, 9,17-octadecadienal, 1h-indene, cyclopropane carboxamide, 2-cyclopropyl-2-methyl-n-(1-cyclopropylethyl), 2-bromo-4-fluorophenyl ester, bis(2-ethylhexyl) phthalate, piperidine-4-carbonitrile, 1,1-diisobutoxy-butane, 3,4-octadiene, 19,19-dimethyl-eicosa-8,11-dienoic acid, 6-methyl-3,4-octadiene and pregnenolone. The nature of the chemical species shows that the UCPC is a potential corrosion inhibitor (Anadebe et al, 2018).

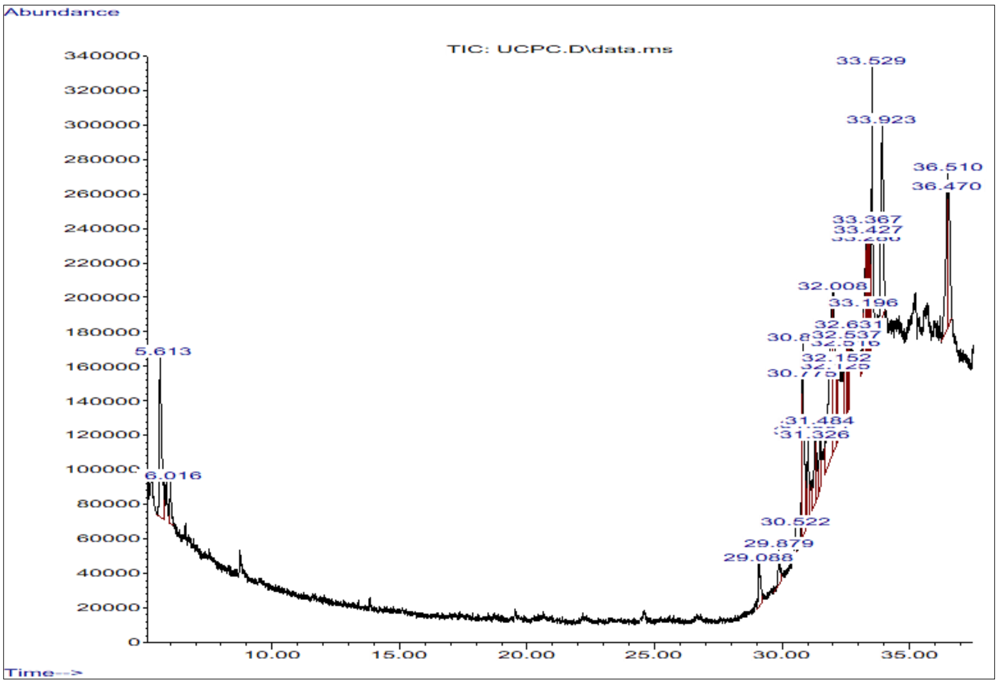


Figure 1 GCMS result of the UCPC

3.2. Adsorption parameters of the corrosion control process

For the corrosion control of aluminium in HCl medium, the parameters of the Langmuir, Temkin, Frumkin and Flory-Huggins isotherms are presented in Table 1. Considering the inhibition process, the adsorption of the Ukpo clay - polyaniline composite (UCPC) at the Al/aggressive HCl interface is the first step in the corrosion control mechanism. Langmuir isotherm gave the highest values of R^2 (very close to one), indicating resilient alignment to Langmuir adsorption isotherm (Vasudha and Shanmuga, 2013; Anadebe et al, 2019). The application of Langmuir isotherm to the adsorption of the UCPC on surface of the metal indicates that there is no chemical reaction between the Al and inhibitor (Deyab, 2015; Onukwuli et al, 2021). For the Frumkin adsorption study, the lateral interaction term (α) gave positive values suggesting attractive tendency of the UCPC on the metal surface (Nwabanne and Okafor, 2011; Udeh et al, 2021).

In the Temkin adsorption consideration, the attractive parameter values (a) were of negative values, indicating that repulsion exists in the adsorption layer (Nabel *et al,* 2012; Omotioma and Onukwuli, 2016). For the Flory-Huggins analysis, the values of the size parameter (x) were of positive values. This showed that the adsorbed UCPC's molecule was massively attached to the metal (Nwabanne and Okafor, 2011; Onukwuli and Omotioma, 2016). The values of ΔG_{ads} are negative and less than the threshold value of -40000 J/mol. It was revealed that adsorption of the UCPC is spontaneous and occurred according to the mechanism of physical adsorption (Aprael et al, 2012; Ihebrodike et al, 2012).

Table 1 Adsorption data for the Al in HCl with UCPC

Adsorption Isotherm	Temperature (K)	R^2	K	ΔG_{ads} (J/mol)	Isotherm property	
Langmuir Isotherm	313	0.9921	0.8964	-10168.99		
	323	0.9911	0.7638	-10063.91		
Temkim isotherm	313	0.8899	46967	-38451.97	a	-6.0910
	323	0.9065	20101	-37400.94		-6.6007
Frumkin Isotherm	313	0.9868	0.0343	-1675.58	α	2.3662
	323	0.9694	0.0540	-2948.08		2.2321
Flory-Huggins Isotherm	313	0.6618	6.3782	-15276.25	x	0.8615
	323	0.7845	7.3047	-16128.60		1.5875

3.3. Response surface methodology (RSM) presentation

Table 2 shows the experimental results of the RSM. Maximum value of inhibitor efficiency was recorded as 89.97% for Ukpo clay - polyaniline (UCPC). It showed that it is suitable for controlling corrosion of Al in HCl medium. Results of the interactive effects of inhibitor concentration (0.5 g/L – 0.9g/L), temperature (303k – 323k) and time (1hr – 5hrs) on the IE revealed that highest values of the IE were obtained at the mid points of the considered factors. This observation suggests that relationships among concentration, temperature and time are parabolic in nature (Ezeugo et al, 2018; Anadebe et al; 2018; Udeh et al, 2021). Fit summary of the inhibition efficiency (Ƞ) is presented in Table 3. Linear, two factor interaction (2FI), quadratic and cubic models were examined. Quadratic model was chosen based on sequential p-value (< 0.0001), adjusted and predicted R^2 values. The predicted R^2 is in reasonable agreement with the adjusted R^2. Table 4 presents the ANOVA of quadratic model of the IE for Al in HCl with UCPC. The model F-value of 237.28 indicates that the model is significant. There is only a 0.01% possibility that an F-value as large as 237.28 could arise as a result of noise (Omotioma and Onukwuli, 2016). P-values less than 0.0500 designate model terms are sufficiently significant.

Table 2 UCPC's efficiency as determined by RSM

Std	**NO.**	**A: Inhibitor conc. g/L. IC**	**B: Temperature K. T**	**C: Time hr t**	**Response 1 Inh. efficiency %**
17	1	0.7	313	4	89.97
6	2	0.9	303	5	75.01
4	3	0.9	323	3	58.02
3	4	0.5	323	3	43.99
1	5	0.5	303	3	57.11
10	6	0.9	313	4	84.19
5	7	0.5	303	5	60.04
18	8	0.7	313	4	89.97
12	9	0.7	323	4	74.85
13	10	0.7	313	3	81.29
15	11	0.7	313	4	89.97
2	12	0.9	303	3	66.12
14	13	0.7	313	5	87.80
16	14	0.7	313	4	89.97
11	15	0.7	303	4	83.64
9	16	0.5	313	4	74.77
8	17	0.9	323	5	72.96
7	18	0.5	323	5	50.01
19	19	0.7	313	4	89.97
20	20	0.7	313	4	89.97

Table 3 Fitness of the Ƞ-model Of UCPC

Source	Sequential p-value	Adjusted R^2	Predicted R^2	
Linear	0.3069	0.0461	-0.4689	
2FI	0.9711	-0.1535	-5.1400	
Quadratic	< 0.0001	0.9911	0.9629	Suggested
Cubic	0.0236	0.9972	-0.0893	Aliased

Table 4 ANOVA of Ƞ- model of UCPC with UCPC

Model	Sum- of Squares	Df	Mean Square	F-value	p-value	
Model	4184.28	9	464.92	237.28	< 0.0001	Significant
A-Inhibitor concentration	495.33	1	495.33	252.81	< 0.0001	
B-Temperature	177.16	1	177.16	90.42	< 0.0001	
C-Time	154.37	1	154.37	78.79	< 0.0001	
AB	21.13	1	21.13	10.78	0.0082	
AC	27.68	1	27.68	14.13	0.0037	
BC	10.44	1	10.44	5.33	0.0436	
A^2 IC	367.03	1	367.03	187.32	< 0.0001	
B^2 T	382.11	1	382.11	195.02	< 0.0001	
C^2 t	115.75	1	115.75	59.08	< 0.0001	
Residual	19.59	10	1.96			
Pure Error	0.0000	5	0.0000			
Cor Total	4203.87	19				
Std. Dev.	1.40		R^2		0.9953	
X'	75.48		Adjusted R^2		0.9911	
C.V. %	1.85		Predicted R^2		0.9629	
			Adeq Precision		46.3857	

3.4. Mathematical model in terms of coded factors

Mathematical model in terms of coded factors for Al in HCl with Ukpo clay - polyaniline composite (UCPC) is presented in Equation 1.The highest power of the variables was 2, which showed that the model is a quadratic equation. The model in terms of coded factors is useful in making predictions about the response for given levels of each factor. It is used in identifying the relative impact of the factors by comparing the factor coefficients (Onukwuli, et al, et al, 2021). The inhibition efficiency is a function of the inhibitor concentration (C, g/L), temperature (T, K) and time (t, hr). The positive signs in the mathematical model signified a synergistic effect, while the negative signs signified an antagonistic effect (Omotioma and Onukwuli, 2017).

$$\eta = +90.40 + 7.04A - 4.21B + 3.93C + 1.63AB + 1.86AC + 1.14BC - 11.55A^2 - 11.79B^2 - 6.49C^2 \quad \text{.........(1)}$$

3.5. Graphical analyses of the results for the Al in HCl

Figures 2-5 show the graphical analysis for Al in the inhibited HCl. In Figure 2, plot of predicted versus actual inhibition efficiency of Ukpo clay - polyaniline composite (UCPC) showed a linear graph. The points clustered along the line of best fit, which is an indication that the obtained model can adequately describe the efficiency of the corrosion control process. This observation is in line with previous research reports (Omotioma and Onukwuli, 2016; Udeh et al, 2021). Figure 3 shows the interactive effect of UCPC concentration and temperature on the inhibition efficiency, where the inhibition efficiency increased with increase in inhibitor concentration till it got to the peak of the parabolic curve. Similar trend was observed in the relationship between inhibition efficiency and the factors of inhibitor concentration and time (Figure 4). In Figure 5, the inhibition efficiency decreased with increase in temperature, but increased with increase in time. It revealed a peak showing optimum inhibition efficiency of 88.54%.

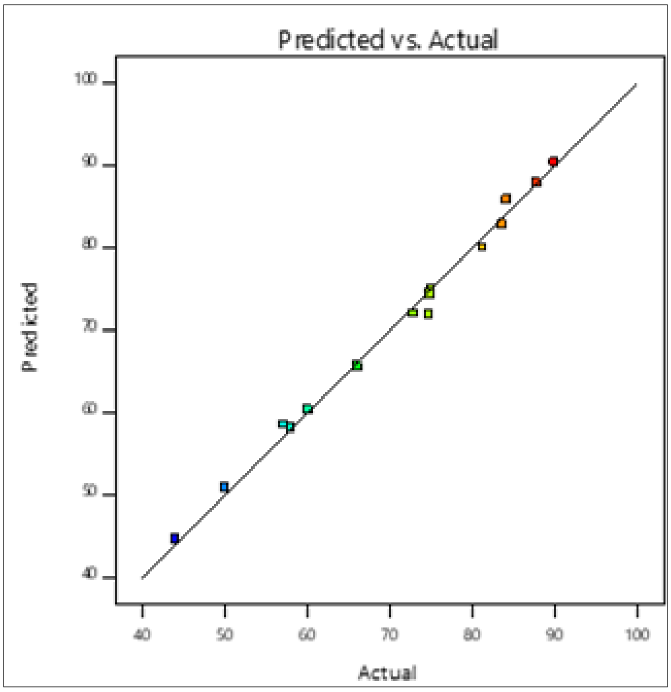


Figure 2 Predicted η against actual η of UCPC

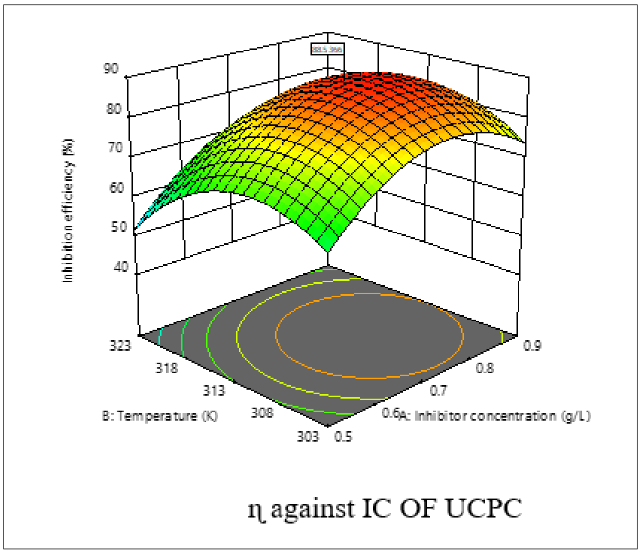


Figure 3 Inh. efficiency versus inhibitor conc. and temp. for Al in HCl with UCPC

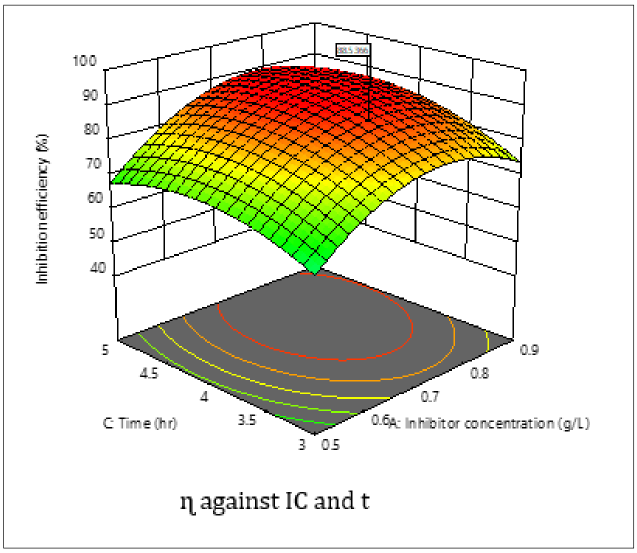


Figure 4 Inh. efficiency versus inh. conc. and temp. for Al in HCl with UCPC

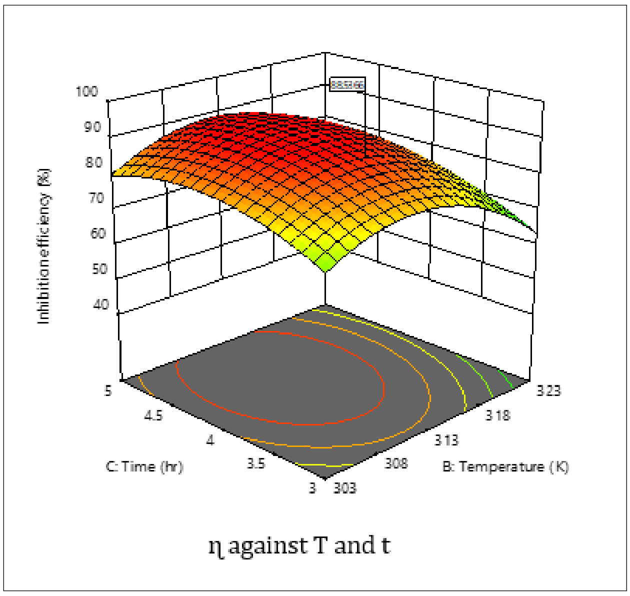


Figure 5 Inh. efficiency versus temp. and time for Al in HCl with UCPC

3.6. Results Validation

Results validation is as expressed in Table 5. It contains optimum conditions of the concentration, temperature and time and the corresponding predicted (optimum) and experimental inhibition efficiencies. Experimental result was compared with the predicted one using statistical tool of percentage deviation. Experimental value was very close to that of the predicted one. It is less than critical value of 5% that determines the acceptability of the generated model. It shows that RSM was appropriate for optimizing the corrosion control process (Omotioma and Onukwuli, 2015; Udeh et al, 2021). Also, it confirmed that the obtained model adequately predicted the experimental data.

Table 5 Validation of result of the inhibition efficiency

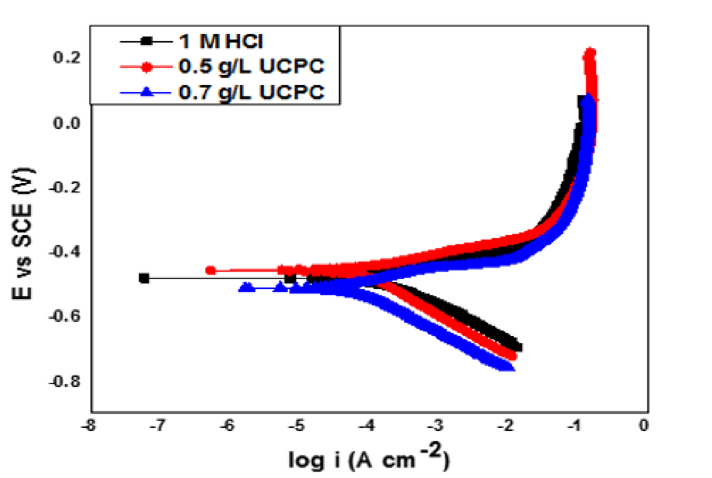
Inh. conc. (g/L). η	Temp. (K)	TIME (HR)	Predicted IE (%)	EXPERIMENTAL IE (%)	PERCENTAGE DEVIATION (%)
0.72	312.55	3.60	88.54	87.18	1.56

3.7. Electrochemical Results

Potentiodynamic and electrochemical impedance spectroscopy results of corrosion inhibition of aluminium in HCl medium using Ukpo clay - polyaniline (UCPC) are presented in Figures (6) and (7) respectively. In the results, the inhibitor was denoted as; Ukpo clay polyaniline (UCPC). Potentiodynamic polarization results revealed the specific effects of the inhibitors on the anodic and cathodic corrosion reactions (Oguzie et al, 2010; Torres et al, 2011; Ezeugo et al, 2018). The curves showed that the anodic and cathodic reactions were inhibited by the UCPC. The observation corroborates with the reports that mixed-type inhibitors are useful in controlling anodic and cathodic types of corrosion (Al-Otaibi et al, 2014; Omotioma and Onukwuli, 2016).

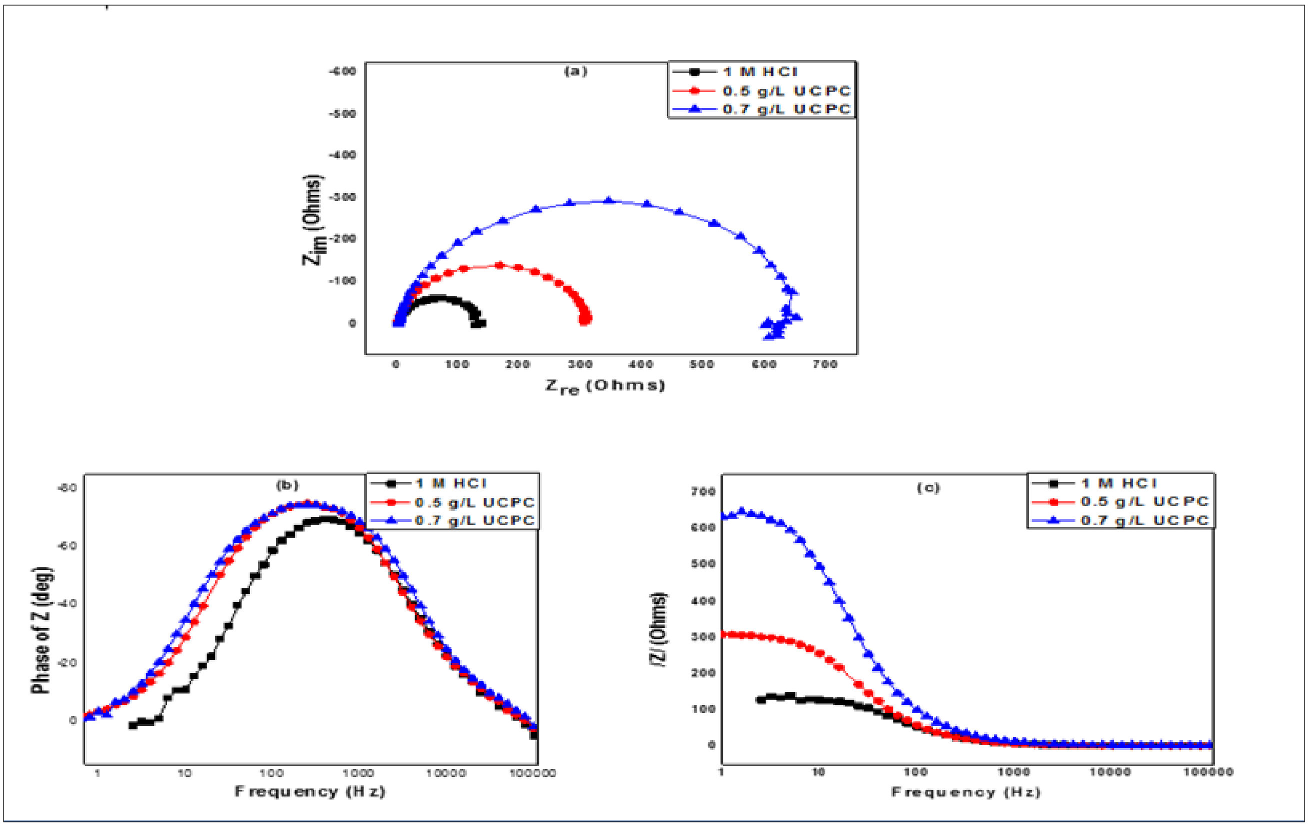
Electrochemical impedance spectra of Al in HCl solution in the absence and presence of the inhibitor are in three categories (Figure 7); Nyquist, Bode phase angle and Bode mag plots. In each of the plots, trend curves of uninhibited and inhibited HCl with 0.5g/L and 0.7g/L UCPC were displayed. The impedance spectra explained the semicircles in the complex plane. The displayed semi-circle is an indication that there is a charge transfer process occurring with charge transfer resistance in parallel with the interfacial capacitance (Anadebe et al, 2018; Udeh et al, 2021). The semicircles were depressed into real axis of the Nyquist plot as a result of the roughness of the metal surface. Revealed dispersing effect can be described by power law dependent capacity (regarded as constant phase element) (khaled, 2003; Anadebe et al, 2018; Onukwuli et al, 2021).

In Table 6, values of solution resistance (R_s), charge transfer resistance (R_{ct}), number of electron transferred (N), corrosion potential (E_{corr}), corrosion current density (I_{corr}) and inhibition efficiency (IE) were presented. R_{ct} increased with increase in inhibitor concentration. This is due to the adhesion of film layer on the Al surface (Anadebe et al, 2018). Maximum inhibition efficiency of UCPC was recorded as 82.8%. Inhibition efficiency increased with increase in inhibitor concentration. The mechanism of action of the inhibitor is a function of the electron density and polarizability of the molecular constituents of the inhibitor (Anadebe et al, 2019; Udeh etal, 2021). Addition of the inhibitor to the HCl medium enhanced the magnitude of the impedance spectrum, which signifies high inhibiting strength of the inhibitor's constituents in impeding the invasion of the electrochemical reaction.



PDP GRAPH

Figure 6 Potentiodynamic curves of mild steel in 1 M HCl in the absence and presence of UCPC



EI= Electrochemical impedance

Figure 7 Spectra of the EI: (a) Nyquist and (b) Bode phase angle and (c) Bode modulus plots in 1 M HCl environment in the absence and presence of UCPC

Table 6 Electrochemical parameters for aluminum in 1 M HCl in the absence and presence of UCPC

MEDIUM	E_{corr}	I_{corr}	IE (%)	R_{ct} (ohms)	N	IE (%)
1M HCl	-456.5	206.9		104.7	0.88	
0.5g/L UCPC	-423.9	48.5	76.6	305.4	0.89	65.7
0.7g/L UCPC	-522.4	28.6	86.2	609.7	0.89	82.8

The following conclusions were drawn from the results of the study;

Ukpo clay/polyaniline composite (UCPC) contains chemical constituents (2-cyclopropyl-2-methyl-n-(1-cyclopropylethyl), 2-bromo-4-fluorophenyl ester, bis(2-ethylhexyl) phthalate, piperidine-4-carbonitrile) suitable for the corrosion control of Al in HCl medium.

Gravimetric analysis of the study revealed that inhibition efficiency of the inhibitor is a function of inhibitor concentration, temperature and time. The inhibition efficiency increased with increase in inhibitor concentration and time but decreased with increase in temperature.

Langmuir adsorption isotherm best described the corrosion inhibition process. The corrosion inhibition process was spontaneous, exothermic and occurred in accordance with physical adsorption process. The activation energy of the inhibitor was less than the critical values of 80kJ/mol.

The relationship between inhibition efficiency and factors of inhibitor concentration, temperature and time is a quadratic model. The mathematical model showed that inhibition efficiency is a function of interactive effects of inhibitor concentration, temperature and time.

The electrochemical analysis of the study revealed that Ukpo clay/polyaniline composite acted as mixed-type inhibitors. It inhibited both cathodic and anodic reactions. Addition of the inhibitor to the HCl medium enhanced the magnitude of the impedance spectrum, which signifies high inhibiting strength of the inhibitor's constituents in impeding the invasion of the electrochemical reaction.

Compliance with ethical standards

Disclosure of conflict of interest

No conflict of interest to be disclosed.

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