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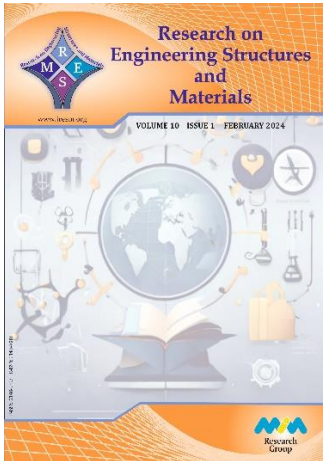
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Online Publication Date: 15 December 2023

URL: <http://www.jresm.org/archive/resm2023.55ma1021rs.html>

DOI: <http://dx.doi.org/10.17515/resm2023.55ma1021rs>

Journal Abbreviation: *Res. Eng. Struct. Mater.*

To cite this article

Ikubanni P, Oki M, Adediran A, Akintola S, Adeleke A, Anyim I, Adesina O, Efenovwe L. Valorized rice husk as green corrosion inhibitor for Al 6061 in 1M HCl. *Res. Eng. Struct. Mater.*, 2024; 10(3): 873-883.

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Research Article

Valorized rice husk as green corrosion inhibitor for Al 6061 in 1M HCl

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Article Info

Abstract

Article history:

Received 21 Oct 2023

Accepted 15 Dec 2023

Keywords:

Corrosion inhibitor;

Rice husk;

Al 6061;

FTIR;

SEM/EDAX;

Gravimetric and

potentiodynamic;

Polarization analyses

This study investigated the inhibition characteristics of rice husk (RH) on corrosion of Aluminum 6061 in 1M hydrochloric acid. Gravimetric analysis and Scanning Electron Microscopy (SEM)/ Energy Dispersive X-ray analysis (EDX) as well as electrochemical studies revealed the potency of RH as a good inhibitor of the corrosion of Al 6061 in 1M HCl. Fourier Transform Infrared Spectroscopy (FTIR) indicated that -OH, C=C, and C=O with signals at (3317.19, 2932.9, 2848.2), (1631.8) and (1105.73, 1030.2, 447.15), respectively in RH were the viable active functional groups which adsorbed on the metal surface to effect reduction of corrosion rates. However, literature suggested the presence of amorphous silica in RH which served as a complimentary corrosion inhibitor. The corrosion current density of Al 6061 was reduced to 3.46×10^{-7} A/cm² at 24 h as against 9.27×10^{-7} A/cm² at the commencement of the potentiodynamic polarization measurements in inhibited 1M HCl. The control specimens exhibited an average corrosion rate of 3.86×10^{-6} A/cm² in HCl solution at the start of this investigation as compared to the corrosion rate of 3.46×10^{-7} A/cm² at 24 h of exposure in 5 mg RH- inhibited HCl. The obtained results gave a corrosion inhibition efficiency of about 90 to 92%.

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1. Introduction

Aluminium alloys are important structural engineering materials and their applications in industries come closely behind those of steel because of their lightweight, relatively high strength, and good corrosion resistance properties [1, 2]. Although highly resistant to atmospheric corrosion, aluminium alloys do undergo serious pitting corrosion in the presence of extraneous ions such as chlorides as may be encountered in process streams [3, 4]. Most of the generally known methods of corrosion control of metals are also suitable for aluminium alloys, however, in process streams as well as in radiators and heat exchangers, it is more appropriate to use inhibitors streams [3 – 6]. Inhibitors are either organic and/or inorganic chemicals that, when added in small quantities to corroding systems, reduce corrosion rates of metals by functioning as anodic, cathodic, or mixed-type inhibitors depending on their molecular structures and some other factors in the

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DOI: <http://dx.doi.org/10.17515/resm2023.55ma1021rs>

Res. Eng. Struct. Mat. Vol. 10 Iss. 3 (2024) 873-883

environment. Misgivings with inhibitors in current usage are their synthetic routes of production, high costs as well as being hazardous which affect both the environment and humans. These amongst others have made searches for low-cost, biodegradable inhibitors from renewable sources inevitable. Hence, researchers have beamed searchlights on extracts from plants such as *Sabdariffa calyx*, castor oil, and agricultural wastes such as rice husks, hydrolyzed chicken feather, walnut leaf, almond fruit leaves, *Jatropha curcas* leaf, *Cascabela thevetia*, and so on, as alternatives [7 – 11].

A series of studies by various authors [7-14] on the inhibitive effects of extracts of plants and waste farm produce, on acid corrosion of metals, using different electro chemicals, in conjunction with other techniques, indicated that all the extracts inhibited the corrosion process by virtue of adsorption of their phytochemical constituents on the corroding metal surface. Inhibition efficiencies were shown to improve with a concentration of the active constituents. From such studies, the mechanisms of inhibition showed no generalized behaviour, however, it was noted that inhibition efficiencies, in excess of 90 to 95% in many instances, may be an indication that these extracts are suitable candidates for the formulation of non-toxic, environment-friendly corrosion inhibiting materials.

In the current research, rice husk which forms about 25% of wastes from paddy milling [12] was valorized for use as a corrosion inhibitor for Al 6061 in hydrochloric acid. Valorized rice husks contain amorphous silica [13] and organic species such as cellulose [14] and have been severally employed as inhibitors in various environments [14, 15]. The RH was prepared and functional groups were obtained using Fourier transform infrared (FTIR) spectroscopy. The surfaces of the inhibited substrate and uninhibited substrate in 1 M HCl were characterized using scanning electron microscopy with an attachment of energy dispersive X-ray analyzer (SEM-EDX). Both gravimetric and potentiodynamic polarization techniques were employed for the corrosion studies.

2. Materials and Methods

Spade-like electrodes were made from Al 6061 sheet metal. The electrodes were etched in 10% NaOH, rinsed in water, de-smutted in 50% HNO₃, and given a final rinse in water before drying at room temperature. The corrosion medium was 1M HCl with and without 5 mg of valorized rice husk. All chemicals were laboratory-grade reagents from BDH Chemicals, UK.

2.1. Preparation of Rice Husks and FTIR Analysis

Spade-like electrodes were made from Al 6061 sheet metal. The electrodes were etched in 10% NaOH, rinsed in water, de-smutted in 50% HNO₃, and given a final rinse in water before drying at room temperature. The corrosion medium was 1M HCl with and without 5 mg of valorized rice husk. All chemicals were laboratory-grade reagents from BDH Chemicals, UK.

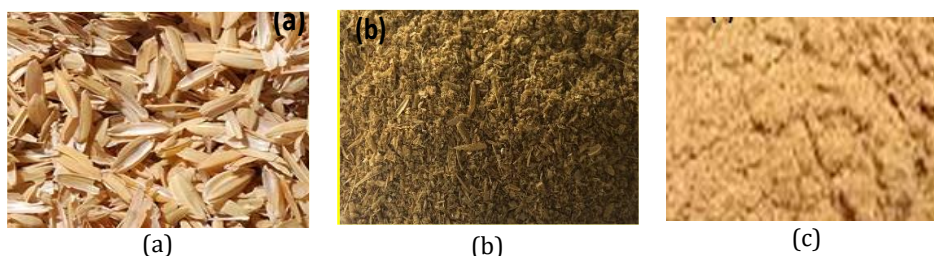


Fig. 1 Rice husk (a) As received (b) Pulverized (c) Powdered

2.2. Gravimetric Measurements

The gravimetric analysis was performed in accordance with NACE recommended practice RP-0775 (NACE, 2005) [16] and ASTM G-31-72 (2004) [17] in which cleaned and weighed Al 6061 electrodes were immersed in 100 ml HCl in a 200 ml beaker in the absence and presence of 5 mg RH for periods ranging from 2 h to 16 h. After each period of immersion, the specimens were rinsed in running water, dried under the fan at room temperature for 30 min, and reweighed. For each immersion period, three different measurements were made and the average of the readings were taken as final measurements. From the gravimetric measurements, corrosion rates were obtained with Eq. (1) and % inhibition efficiency, %IE, was calculated with Eq. (2).

$$\text{Corrosion rate, } CR = \frac{K \cdot W}{A \cdot D \cdot T} \quad (1)$$

where CR is the corrosion rate (mm/year), K is a constant (8.766×10^4), T is the time of exposure (hr), A is the area (cm²), W is the weight loss (mg), and D is the density of Al, (g/cm³).

$$IE \% = (CR - CR_i) / CR \times 100 \quad (2)$$

where CR is the corrosion rate in the absence of inhibitor and CR_i is the corrosion rate in the presence of inhibitor.

2.3. Scanning Electron Microscopy (SEM) and Energy Dispersive Analysis of X-Ray (EDX)

Spade-like electrodes immersed for 16 h in 1M HCl in the presence of 5 mg of rice husk extract and without the extract were examined in a Scanning Electron Microscope, Model Pro X, with an Energy Dispersive X-ray analyzer (EDX) attached at high vacuum (HV) mode with 20 kV accelerating voltage.

2.4. Electrochemical Measurements

Electrochemical measurements were performed in a conventional three-electrode cell using computer-controlled potentiostat/galvanostat (Autolab PGSTAT 302N). The platinum electrode served as the counter electrode (CE), Ag/AgCl, as the reference electrode (RE), and Al 6061 specimens were employed as working electrodes (WE). The area of WE exposed to the medium was approximately 1 cm². Before each potentiodynamic polarization measurement, the electrode potential was allowed to stabilize while the open circuit potential (OCP) was recorded as a function of time up to about 30 minutes. After this, a steady-state OCP corresponding to the corrosion potential (E_{corr}) of the working electrode was obtained. The potentiodynamic measurements were carried out on the specimens in 1M HCl with and without 5 mg of inhibitor. The potential scans were executed automatically between 200 and -200 mV vs OCP at a rate of 10 mV/s. Fresh HCl solution and Al 6061 samples were used after each potential sweep.

3. Results and Discussion

3.1. FTIR Analysis

The spectrum generated for the rice husk is displayed in Fig. 2, where it can be observed that -OH, C=C, and C=O had signals at (3317.19, 2932.9, 2848.2), (1631.8), and (1105.73, 1030.2, 447.15), were present respectively. These have electron-rich centers that interact with corroding surfaces to stifle corrosion reactions.

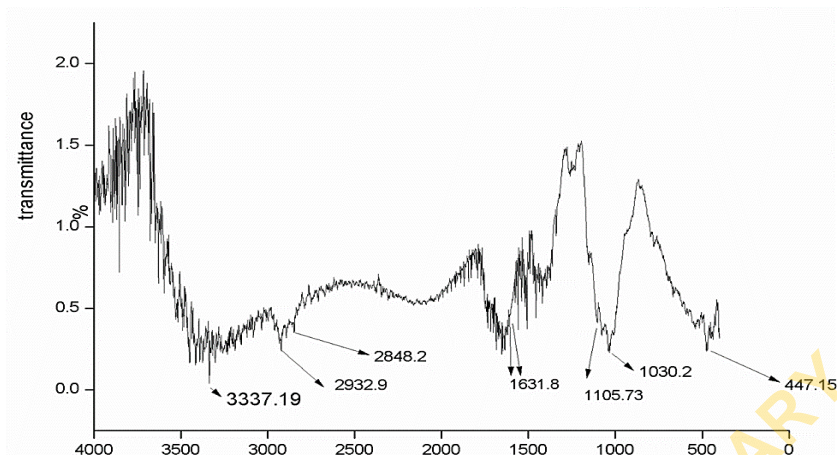


Fig. 2. FTIR spectra for rice husk

However, for C=C, especially when the double bonds are conjugated as in organic moieties found in rice husks, delocalization of electrons occurred in electrolytes leading to interactions with corroding surfaces thereby destabilizing corrosion reactions to reduce corrosion rates. Cellulose in rice husks is a typical reference point with both double bonds and reactive -OH groups. Therefore, such interactions are expected to reduce corrosion while descaling aluminium artefacts and radiators in water cooling systems.

3.2. Gravimetric Analysis

The plot for the corresponding corrosion rates in 1M HCl solution at ambient temperature from gravimetric analyses is displayed in Fig. 3. The weight loss in blank HCl was observed to increase steadily with time from about 0.22 mg at 2 h to about 0.56 mg at 16 h of immersion periods with corresponding corrosion rates of 3.4×10^{-6} and 1.08×10^{-6} mm/y, respectively. On the other hand, the inhibited weight loss commenced from a lowly 0.03 mg at 2 h to about 0.14 mg at 16 h of immersion time with corresponding corrosion rates of 4.63×10^{-7} and 2.7×10^{-7} mm/y, respectively. From the foregoing, an inhibition efficiency of about 92% was observed.

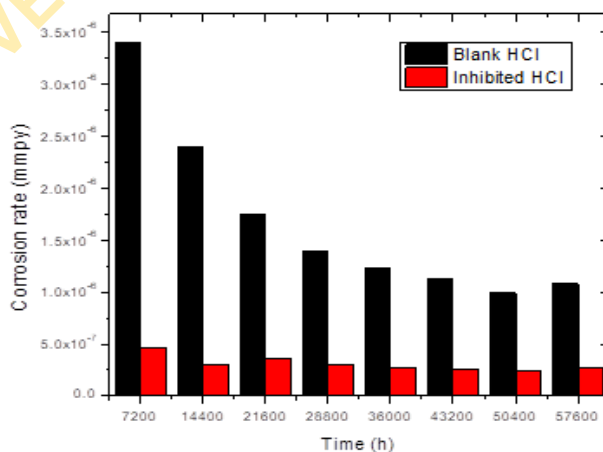


Fig. 3. Corrosion rate versus time in blank and 5 mg inhibited 1M HCl solution

This was expected as adsorption and desorption of inhibiting species occurred with time while the offending species continually interacted with the corroding surface. However, an overall decrease in corrosion rate was observed.

3.3. Scanning Electron Microscopy (SEM)/Energy Dispersive Analysis of X-ray (EDX) Analysis

The scanning electron micrograph of Al6061 specimens immersed in inhibited 1M HCl solution for 16 h is displayed in Fig. 4. It can be observed that there are only two major pits along a scratch line revealed on the surface, which indicated that the inhibitor was effective throughout the exposure period. The EDX revealed a major peak for Al and numerous other elements as displayed in Table 1 along with their percentage occurrences. The other major elements are C at 0.5 %, Si at 1% and Fe at 2.3% as against 0% C, 0.7% Si, and 0.6% Fe in Al6061 [18].

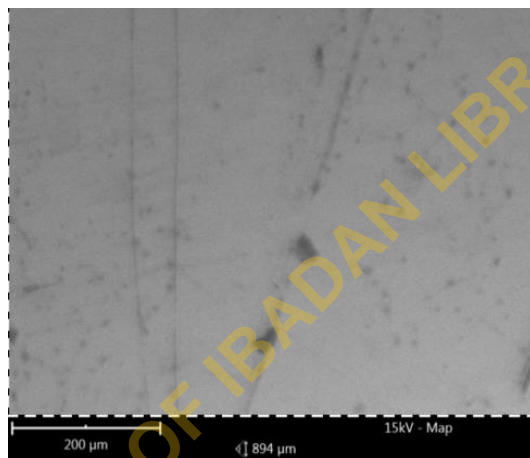


Fig. 4. Scanning electron micrograph of Al 6061 immersed for 16 h in RH inhibited 1M HCl

C is derived from organic components like cellulose in rice husks with active -OH groups at various positions [19], which can adsorb on corroding sites to inhibit corrosion in such areas. The presence of Si at a percentage higher than was expected in Al6061 suggested the interaction of SiO₂ with corroding alloy of interest. Amorphous silica is present in rice husks and is known to be a good corrosion inhibitor separately [14, 15] and in combination with other chemicals [20]. The higher weight percentage of Fe's presence on the inhibited specimen than expected in Al6061 also indicated the interaction of rice husks with the corroding surface. In all, most of the elements detected are also present in rice husks albeit at higher percentages than expected in Al6061.

The scanning electron micrograph of Al 6061 exposed for 16 h in 1M HCl is displayed in Fig. 5, where it can be observed that almost the entire surface was ravaged with pitting corrosion episodes with almost 90 to 95% of the surface covered with pits of various shapes as against 2 to 5% pitting corrosion observed on the inhibited specimen, Fig. 3.

Table 1. Percentage elemental composition of Al6061 exposed to 1 M HCl with 5 mg of rice husks and 1 M HCl only

Element Number	Element Symbol	Al6061 in HCl + 5 mg RH		Al6061 in HCl	
		Atomic Conc.	Weight Conc.	Atomic Conc.	Weight Conc.
13	Al	92.61	88.90	94.68	90.50
26	Fe	1.15	2.29	1.26	2.50
47	Ag	0.43	1.64	0.37	1.41
20	Ca	0.73	1.04	0.77	1.09
41	Nb	0.30	1.01	0.29	0.96
14	Si	1.00	1.00	0.70	0.70
39	Y	0.31	0.99	0.32	1.00
19	K	0.67	0.93	0.54	0.75
16	S	0.61	0.70	0.38	0.44
6	C	1.33	0.57	-	-
22	Ti	0.25	0.42	0.09	0.16
12	Mg	0.44	0.38	0.40	0.34
11	Na	0.16	0.13	0.19	0.15
15	P	0.00	0.00	0.00	0.00

Thus, it can be inferred that components of rice husks adsorbed and performed as corrosion inhibitors on Al6061 in 1M HCl solution. Table 1 presents the elemental composition of the uninhibited specimen. It is interesting to note that Si remained at 0.7 wt.% and C remained at 0 wt.% as in Al 6061 as against 1% and 0.57% on the surface of the inhibited specimen. These further suggest interactions of rice husk components with inhibited specimens in reducing pitting corrosion episodes.



Fig. 5. SEM micrograph of Al 6061 exposed for 16 h in 1M HCl without inhibitor

Niobium and Yttrium present in both specimens may be contaminants present in chemicals employed during the various preparations in carrying out the research. However, since their weight concentrations remained almost constant at about 1%, Nb and Y are more likely to be in the substrate Al alloy.

3.4. Electrochemical Analysis

The potentiodynamic curves for Al 6061 in HCl with and without inhibitor are displayed in Fig. 6 and corresponding parameters from the Tafel extrapolations are shown in Table 2, respectively.

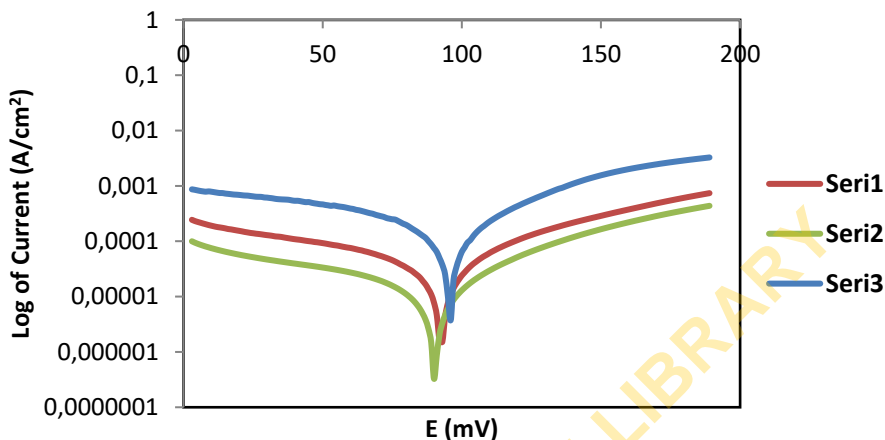
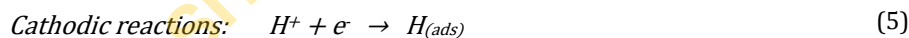
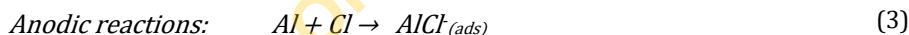


Fig. 6. Potentiodynamic polarization scans of Al 6061 exposed to 1M HCl at 30°C with and without 5 g of RH Inhibitor (Series 1 – OCP/blank; Series 2 – OCP/RH, Series 3 – 24 h/RH)

It has been canvassed in the literature [21,22] that the associated mechanisms with anodic and cathodic reactions for Al as described in Fig. 6 can be written as Eqs. (3-6):



The mechanism of corrosion inhibition of the rice husk on Al substrate in 1M HCl could be described as follows. Corrosion inhibitor causes adsorption on the interface of the metal and 1M HCl aggressive solution. The dissolution of metal and hydrogen gas liberation occurred at the anode and cathode, respectively [23]. The rice husk molecules' adsorption on the surface of the substrate could be attributed to a negatively charged acid anion (Cl^-) from the HCl solution. Hence, the adsorption results in the surface coverage of the metallic material by preventing the corrosion of the substrate [24]. The metal surface protection occurs due to coordinated bond creation between the Al alloy and the rice husk inhibitor molecules [25, 26].

From the foregoing, it is obvious that Al readily reacts in HCl to form the various products observed in Equations 3 and 4. The cathodic polarization curves in the absence and presence of RH appear parallel to each other and the same can be observed for the anodic polarization which suggested that the inhibitor did not change the processes occurring on the electrode surface, it only decreased the rate of reaction to delay corrosion.

From Table 2, the difference in corrosion potentials in the presence and absence of inhibitor is not up to 85 mV suggesting that the inhibitor acted as a mixed type inhibitor. Thus, both anodic and cathodic reactions were slowed down to lower the corrosion rates of the specimens in inhibited HCl. Moreover, it is evident from Table 2, that the corrosion rate was reduced from $3.8 \times 10^{-6} \text{ A/cm}^2$ to $3.46 \times 10^{-7} \text{ A/cm}^2$ at 24 h of exposure in the presence of an inhibitor. The % inhibition from these readings was calculated to be about 90% which approximates the values obtained from gravimetric analyses further indicated that RH was an efficient inhibitor capable of strong adsorption on the surface of corroding Al6061

Table 2. Polarization parameters for specimens at attainment of OCP and at 24 h in HCl

Samples	Corrosion rate, CR (mm/y)	Corrosion current density, I _{corr} (A/cm ²)	Corrosion potential, Cp (V)	Polarization resistance Rp (Ω)	Cathodic constant B _c (V/dec)	Anodic constant B _a (V/dec)
@OCP/blank	0.044831	3.86E-06	-0.89735	106.97	0.002653	0.001481
@OCP/RH	0.010774	9.27E-07	-0.92046	548.82	0.002334	0.002353
@24h/RH	0.004022	3.46E-07	-0.9351	1289.4	0.001631	0.00278

Further examination of Table 2 in conjunction with Fig. 6 reveals that the values of Ba and Bc were altered on adding inhibitor to the corroding systems which is more prominent with Bc at 24 h exposure in the inhibited system. This implied that the cathodic hydrogen evolution reaction in the acidic system was impacted more by the adsorption of inhibitor moieties [27].

Various studies [3, 8, 15, 25, 28] on the corrosion inhibition performances of plants' extracts have shown that "x-ray electron spectroscopy (XPS), scanning electron microscopy (SEM), and Fourier transform infrared spectroscopy (FTIR) surface study techniques have all supported the existence of protective layers on the numerous metals studied. These extracts operated as mixed-type inhibitors, according to PDP data. The current research revealed significant findings in the preparation of original, environment-friendly, sustainable inhibitors with high inhibitory power commensurate with findings from the literature.

4. Conclusions

This study is very important to the field of materials with regard to the development of eco-friendly and non-toxic corrosion inhibitors. The green inhibition tendencies of valorized rice husk on Al6061 substrate in 1M hydrochloric acid environment were investigated. Fourier Transform Infra-Red spectroscopy revealed the availability of functional groups, -OH, C=C, and C=O in RH with peaks at various wavelengths of 3317.19, 2932.9, 2848.2, 1631.8, 105.73, 1030.2 and 447.15. These functional groups were variously employed to inhibit corrosion on the Al 6061 substrate. The functional groups adsorbed on the corroding substrate and reduced the pitting corrosion episodes to between 90 and 92%.

The micrographs revealed that the density of pits on the surface of the substrates exposed in an uninhibited environment was reduced from about 100/mm² to about 2/mm² on the metal surface exposed to the inhibited 1M hydrochloric acid.

The rice husk performed as a mixed-type inhibitor as the difference in the corrosion potentials of the inhibited and uninhibited specimens was less than 85 mV. The corrosion rate of Al6061 was reduced from 3.8×10^{-6} A/cm² in blank hydrochloric acid to 3.46×10^{-7} A/cm² at 24 h of exposure in rice husk-inhibited hydrochloric acid solution. Thus, rice husk performed as an effective corrosion inhibitor for Al 6061 in 1M hydrochloric acid solution with an inhibition efficiency of between 90 and 92%.

The Tafel constants B_a and B_c differed slightly in values which suggested that the electrochemical mechanisms for the corrosion of Al 6061 remained the same in the presence and absence of inhibitor in the aggressive hydrochloric acid over the immersion periods employed in this investigation. The anodic and cathodic corrosion reactions were only slowed down to effect reduction in corrosion rates, without any change in mechanisms. This is an important study to material scientists and engineers based on the promising results obtained.

Acknowledgment

The authors acknowledge the use of facilities at Landmark University, Omu-Aran, Kwara State, Nigeria.

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