Environmentally-friendly Inhibitor From Waste for Aluminium Corrosion in 0.5M Sulphuric Acid Environment

Omotayo Sanni (✉ tayo.sanni@yahoo.com)

Tshwane University of Technology - eMalahleni Campus <https://orcid.org/0000-0001-6979-795X>

OSI Fayomi

Department of Chemical, Metallurgical and Materials Engineering, Tshwane University of Technology, Pretoria, South Africa

API Popoola

Department of Chemical, Metallurgical and Materials Engineering, Tshwane University of Technology, Pretoria, South Africa

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Abstract

This research aims to identify cheap, effective, and green inhibitors from agricultural waste (coconut shell) as sustainable inhibitors for pure cast aluminium corrosion in 0.5 molar sulphuric acid medium at different concentrations. The inhibitive effect of coconut shell (CS) in 0.5 molar sulphuric acid solutions on aluminium was studied using series of standard corrosion monitoring methods: gravimetric and electrochemical techniques. The nature of adsorption and aluminium surface morphology was conducted with scanning electron microscopy and energy-dispersive X-ray spectroscopy (SEM/EDX) method. The obtained result implies that the CS can serve as an environmentally friendly inhibitor for aluminium corrosion in 0.5 molar H_2SO_4 environments with the highest corrosion inhibition efficiency of 99% at 10 g. The inhibition mechanism was proposed to be physical adsorption and fits the Langmuir adsorption isotherms. The inhibitive effect of CS was proposed to be a mixed inhibition mechanism as shown by the potentiodynamic polarization method. The SEM and EDX observation show that the CS species formed a protective layer on the aluminium surface. The results obtained confirmed that CS is a sustainable inhibitor, which could be used in the industry as an environmentally friendly corrosion inhibitor.

Introduction

Corrosion is a natural process affecting our society daily. Aluminium and its alloys corrosion behavior is interesting research, recently shown by the enormous number of literature outputs. Since aluminium and its alloy is a material of choice for several industrial applications [1, 2]. Due to its attractive properties such as excellent thermal and electrical conductivity, low density, attractive appearance, fairly good resistance to corrosion due to the formed compact, adherent passive film oxide on the surface of the aluminium on exposure to an aqueous solution or air [2, 3]. Nevertheless, the protecting layer is amphoteric and break-up on aluminium exposure to alkalis and strong acids, this makes aluminium and its alloy corrodes. Acid solutions such as acetic, hydrochloric, nitric, sulphuric, and phosphoric acids are used in the aluminum corrosion study since they are the type of acids used frequently for descaling, pickling, and industrial cleaning [4-6]. This process is substantial for the reason that they remove salts deposit, reinforce oil recovery, and unwanted scale formed during industrialization. Unfortunately, the hydrochloric and sulphuric acid used for this purpose leads to the degradation of the metal. The damage related to metallic corrosion leads to high costs for renovation, environmental hazards, and replacement of diverse equipment. In an attempt to mitigate the aluminum corrosion, diverse techniques have been used; chemical inhibition, upgrading materials, process control, and blending of product fluids. Still, an inhibitor is regarded as one of the most cost-effective and convenient means of fighting corrosion, particularly in acid solutions [7-12]. The corrosion inhibitive behavior of aluminium in an acid medium using different organic compounds with polar functions such as sulphur, phosphorous, oxygen, and nitrogen has been documented [13-23]. Several processes in the industry have used inorganic and organic inhibitors for corrosion defense, but due to their toxicity and high cost, research awareness has moved towards the use of efficient, cost-effective, nontoxic, and benign corrosion inhibitors. Green

inhibitors are advantageous because they are biodegradable, low processing cost, absent of toxic compounds and heavy metals [24-26]. The application of waste as an inhibitor has recently attracted substantial awareness because they possess multiple adsorption sites, they are cheap and stable to metallic material in aggressive solutions, and with their functional group, they form complex with metallic ion, and on the surface of the metal, this complex occupies a large area, thus cover and protect the metal surface from corrosive agent in the aggressive solutions. Their inhibitive ability is structurally related to the heteroatoms considered as adsorption centers. Different waste materials have been reported as an effective inhibitor for different metals in different aggressive solutions [27-37]. Nevertheless, at the present, waste from green raw material and their uses as corrosion inhibitors for aluminium alloys are barely reported. Because of this, this research is being taken to develop cheap and eco-friendly corrosion inhibitor with high protection capability. This inhibitor is prepared from environmentally friendly raw material that is: coconut shell (easily available, cheap, and non-toxic). Also, the purpose of this report was to assess the corrosion protective tendency of coconut shells in the Al/CS/Acid system through weight loss and electrochemical techniques. The electrochemical method is suitable to monitor in situ any perturbation by an inhibitor and the metal/corrosion interface electrochemical process plus giving information on the rate of corrosion. Changes in the morphology of the corroding aluminium surface were carried out by microscopy (SEM/EDX).

Experimental Procedure

2.1. Specimen preparation

99.99% pure aluminum specimens employed in this work were cut into samples of $1 \times 1 \times 0.5$ cm dimensions. The samples were mechanically polished with silicon abrasive paper, degrease and rinse with absolute ethanol and distilled water respectively, then dry in air. The 0.5 molar sulphuric acid solution was prepared from 98% sulphuric acid (analytical reagent grade) via distilled water and serve as corrosion throughout the experiment. The chemical structure of CS was shown in Figure 1 which was dissolved in the sulphuric solutions to attain the desired concentrations (2, 4, 6, 8, and 10 g) as the test inhibitor. Afterward, the coupons were degreased in ethanol and cleaned in acetone respectively. The samples were dried and kept in desiccators before use. All the solutions for weight loss and electrochemical tests were prepared using distilled water. Freshly prepared test solutions were used for each experiment. The sample was weighed accurately with four digital analytical electronic balance.

2.2. Weight loss tests

The pre-weighed/ polished aluminium samples were separately immersed in 250mL of 0.5 molar sulphuric acid solution, with and without inhibitor. The weight loss values were established using the difference in the initial and final weight of aluminum sample, precisely following ASTM G-81. The weight was recorded with four digital analytical balance. The sample was immersed with and without CS solution at different concentrations (2, 4, 6, 8, and 10 g) in the 0.5M H_2SO_4 medium. The weight loss after an exact time of exposure was estimated by taking the samples from the solutions, thoroughly wash with

distilled water, dry, and reweigh. This experiment was carried out three times at ambient temperature for each exposure time, and the average value of the loss in weight was documented. From this weight loss value, the surface coverage (θ), corrosion rate (mm/year), and inhibitor efficiency (%) were calculated using equations reported elsewhere [36, 38].

2.3. Electrochemical studies

Potentiodynamic polarization measurement was conducted utilizing an Autolab electrochemical workspace analyzer. Aluminum specimens with 1 cm² exposed area were used for the electrochemical studies. The tests were conducted using three conventional electrode cells assembly with aluminum sample as the working electrode. The reference electrode used was a silver/silver chloride electrode while a platinum electrode serves as the counter electrode. Before the Tafel measurement, the electrode was dipped in the acid solutions at OCP (open circuit potential) for 30min to reach a steady state. A corrosion test was conducted in the blank solution with and without the presence of various inhibitor concentrations. All experiment was carried out in stagnant aerated solution. The anodic and cathodic linear Tafel section curve was extrapolated to E_{corr} (corrosion potentials) to attain the (i_{corr}) corrosion current density. From the polarization curve, the corrosion parameters, specifically, corrosion potential (E_{corr}), cathodic slope (bc), and anodic slope (ba), were estimated.

2.4. Scanning electron microscopy (SEM)

The morphology of the aluminium surface was studied by immersing the aluminium samples in the presence and absence of CS in a 0.5 molar sulphuric acid medium for 168 hours. After 168 hours, the samples were removed, rinse with distilled water, and dried, then, examined with SEM to observe the morphology of the material surface. The morphology of the aluminium surface was conducted via a computer-controlled scanning electron microscope.

2.5. Energy-dispersive x-ray test

The same sample observed by scanning electron microscope was as well characterized by energy-dispersive x-ray spectroscopy (EDX). This test was conducted using the JEOL model scanning electron microscope equipped with the energy-dispersive spectrometer.

Results And Discussion

3.1 Weight loss studies

The process of aluminum corrosion in 0.5M H₂SO₄ solutions, studied by weight loss, for different concentrations (2, 4, 6, 8, and 10g) in the presence and absence of coconut shell immersed for 168 hours are given in Figures 1 and 2. Figure 1 illustrates the variation in the corrosion rate with the time of exposure for Al in 0.5M H₂SO₄ containing various doses of CS. The initial rate of corrosion without the inhibitor in the acid solutions was calculated to be between 0.930319 and 2.100719 mm/yr. Figure 1

shows that the rate of corrosion values reduced with an increase in the concentration of the inhibitor, signifying an increase in the adsorbed molecule number on the surface of the aluminum blocking the acid attack of the active sites and thus protects the metal against corrosion [39-41]. This observation is due to the interaction between the surface of the metal and lone pair electron of the N atom in the heterocyclic ring, also the surface of the metal, and the contact between the p electrons of the aromatic ring [42-44]. However, the presence of the inhibitor in the corrosive solutions tremendously decreased the rate of Al corrosion as the inhibitor concentration increase in the H_2SO_4 solutions. After 168 hours of exposure, the corrosion rate decreased from 0.630216 to 0.483766 and $1.89E-05$ to 0.001768 mm/yr in the solutions with 2 and 10 g of CS. This observation established the formation of barrier layers on the aluminium surface and the adsorption of the inhibitor. The inhibition capacity changes with the different inhibitor concentrations for aluminium in the acidic solutions are shown in Figure 2. The result shows that the inhibition capacity depends on the inhibitor concentration. The protection capacity was found to rise with an increase in the inhibitor concentration. The increased inhibition capacity in the case of the inhibitor studied may be due to the inhibitor adsorbed on the aluminium surface and the increase of the interface coverage. When the CS concentration is increased, more CS compound is adsorbed in the acid/aluminium interface leading to larger surface coverage. This reveals that the CS tested prohibits aluminium corrosion in an acid medium. The maximum efficiency of the inhibitor was estimated as 95.26% and 99.0% at a maximum concentration (8 and 10 g) after 24 hours. The increasing inhibition efficiency (%) with an increase in the inhibitor concentration implies that the inhibition is dependent on the nature and the inhibitor adsorption mode on the metal surface.

3.2 Potentiodynamic polarization studies

The Tafel plots are techniques to determine the rate of corrosion from the polarization curve experiment. The electrochemical data; cathodic Tafel constant (b_c), corrosion potential (E_{corr}), corrosion rate, anodic Tafel constant (b_a), and the corresponding corrosion current density (I_{corr}) for the aluminum corrosion in acidic solutions with the presence and absence of different concentrations (2, 4, 6, 8 and 10 g) of coconut shell at room temperature obtained by Tafel lines extrapolation are illustrated in Table 1. Typical aluminium polarisation curves in the acidic solutions with and without diverse CS concentrations are illustrated in Figure 3. The plot shows that CS affects the anodic and cathodic parts of the curve. This shows that the CS influences both the hydrogen evolution process and aluminium dissolution, implying that the CS functions as a mixed-type inhibitor. There was no considerable shift observed in the corrosion potential (E_{corr}) values with different CS concentrations, implying that CS inhibits the corrosion of aluminium simply by blocking the available surface for the cathodic and anodic process. Table 1 show that the E_{corr} values shifted slightly to positive potentials with an increase in the CS concentrations. This result suggests that the CS is mixed- type in nature. The value of i_{corr} for aluminium in 0.5 M H_2SO_4 is 0.0005076 A and $3.88E-06$ A for the uninhibited and inhibition system with 10g of the investigated inhibitor. The decrease in the values of i_{corr} with the inhibitor reveals that the aluminium is protected against corrosion because of the formed adsorbs film of the inhibitor species on the surface of the metal. The obtained results in Table 1 indicate a decrease in the values of i_{corr} with an increase in the inhibitor

concentration, which is a result of an increase in the blocked fraction of the electrode’s surface due to CS adsorption. The reductions in the corrosion current values support the inhibitor action on both cathodic and anodic reactions. The cathodic and anodic slope indicates that the retardation of the anodic metal dissolution and the cathodic hydrogen reduction was affected with no changes in the dissolution mechanism with and without the presence of CS. Further, the trend of gravimetric results is also in good agreement with the Tafel results. Moreover, in other words, the CS decreases the corrosion surface area with no effect on the corrosion mechanism and only causes inactivation of a part of the surface to the acidic solutions [45].

Table 1 Corrosion parameter for pure aluminium in 0.5 M H₂SO₄ solutions by potentiodynamic polarization method in the presence and absence of CS

Solution (g)	Parameter					
	ba (V/dec)	bc (V/dec)	icorr (A)	Ecorr (V)	Corrosion rate (mm/year)	Polarization resistance (Ω)
0	0.45356	0.07773	0.0005076	-0.3668	0.66574	56.78
2	1.45100	0.10867	0.0004055	-0.3806	4.71170	108.29
4	0.06091	0.04638	0.0001348	-0.3997	1.56670	84.814
6	0.04102	0.02394	6.41E-05	-0.3916	0.07873	102.44
8	0.06455	0.04444	3.31E-05	-0.9753	0.38478	345.18
10	0.05437	0.03762	3.88E-06	-0.5904	0.00497	2489.50

3.3. Adsorption considerations

The surface of the metal in aqueous solutions is covered always with an adsorbed water molecule. As a result, the inhibitor molecules adsorption from aqueous solutions is a quasi replacement process [46]. The inhibitor molecule adsorption is mostly dependent on the electronic characteristic of the surface of the metal, nature, and charge of the surface of the metal, the solvent adsorption, electrochemical potentials at the solution interface, ionic species, and temperature. The adsorption isotherms describe the adsorption behavior of organic compounds to have a better understanding of the mechanisms of adsorption. The commonly used adsorption isotherm is the Temkin, Langmuir, Freundlich, and Frumkin isotherm. If basic adsorption performance for the studied inhibitor on the pure aluminium is assumed, a direct connection between the surface coverage and the inhibition ability of the inhibitor should occur.

Assuming the efficiency of the inhibitor is mainly attributed to the blocking influence of the adsorbed molecules, the surface coverage value was estimated from the inhibition efficiency obtained from gravimetric tests. To get the best isotherm fit, the degrees of surface coverage was estimated for different CS concentration from the gravimetric data. The adsorption performance of CS fits best with the Langmuir adsorption isotherm. The Langmuir isotherms are ideal isotherms for chemical or physical adsorption without contact between the adsorbent and the adsorbate. The Langmuir assumptions relate the adsorbate concentration in the bulk of the electrolyte to the (θ) degree of surface coverage. From Figure 4, a linear plot behavior was attained for the graph of concentration/degree of surface coverage vs. CS concentration. The regression coefficient R^2 value obtains from the plot is incredibly approximately one. This is due to the relationship between the species adsorbed on the surface of the aluminium and those between the organic molecule adsorbed on the cathodic and anodic metal area play significant roles in this process. A similar result was reported by [47], which proposed that the inhibitor covers the cathodic and anodic areas via general adsorption which follows the Langmuir isotherms. The Langmuir adsorption isotherm applicability to the CS adsorb on the aluminum proves the multi-molecular adsorption layer formed where there is no interaction between the adsorbent and the adsorbate.

3.4. Scanning electron microscopy (SEM)

This analysis was carried out on polished aluminum surfaces depicted for 168 h in 0.5M H_2SO_4 solution in the presence and absence of CS, as presented in Figure 5. By comparing the SEM micrographs, the SEM image in the absence of inhibitor (Figure 5a) shows that the aluminium surface is strongly damaged, which results in a heterogeneous and rough surface related to free acid offensive without the inhibitor. On the other hand, the solution inhibited (Figure 5b) reveals smooth surface morphology after adding the inhibitor due to the formation of a CS protective layer on the surface of the aluminium thereby, inhibiting the rate of corrosion. This confirms that the CS inhibited the aluminum corrosion through the adsorption of the inhibitor molecule on the metal surface. The surface of the aluminum in aerobic environments is covered always with aluminum oxide and the isoelectric point of the aluminum oxide is pH 9 [48]. In the 0.5 molar sulphuric acid solutions where pH <0, the aluminum oxide surface has a positive charge, resulting to electrostatic attraction of the negatively charged ion.

3.6. Energy-dispersion x-ray spectroscopy (EDX)

The EDX spectrum was employed in determining the element present on the aluminum surface after 168 hours of exposure in the presence and absence of CS in 0.5M H_2SO_4 solutions. Figure 6 demonstrates the aluminum EDX analysis without CS [Figure 6a] with 10 g of CS [Figure 6b]. The analysis of EDX reveals that O and Al were identified. The spectra additionally reveal lines showing the presence of N and C. The reduction in the oxygen peak intensity spectrum in the presence of CS solution [Figure 6 b] verifies the constraint of deposited oxygen for the corrosion process with CS. The unanticipated peak of Si and Mn could be CS constituent and the S peak with low intensity may be obtained from the corrosive 0.5M H_2SO_4 solutions. These also indicate that the N, O, and C atoms percentage with the CS reduces as compared to the aluminum sample EDX without CS. This data showed that the C, N, Al, and O atoms

cover up the aluminium surface. This result shows the formed protective film on the aluminum surface. The variation in the atom values in the presence and absence of CS aids in understanding the changes that occur due to the protective layer formed on the aluminium surface.

3.7 Corrosion inhibition mechanism

The corrosion inhibition process by adsorption in acid-metal solutions interface may take place by the adsorb neutral molecule onto the aluminium surface through interaction between the vacant d-orbital of Fe and unpaired electron of heteroatom, involving the water molecule displacement from the surface of the metal; and vacant d-orbital of the Fe atom and donor-acceptor interaction between pi-electron of an aromatic ring. The likely corrosion inhibition mechanism could be explained based on the adsorption procedure and the elemental structure present in the CS. The compound contains a heteroatom such as N and O, which supports their adsorptive tendency on the surface of the aluminum. The corrosion inhibitive effectiveness of this molecule could be ascribed to its group and may also be owing to the presence of pi electron in the ring. The protonated CS adsorb via an electrostatic interface between the negatively charged metal surface and the positively charged molecules. From the obtained result, the inhibition efficiency of CS in H_2SO_4 solutions could be clarified as in acidic aqueous solution, the CS exists in the form of the cation (protonated CS) or as a neutral molecule. In general, two forms of adsorption can be considered for the mechanism of corrosion inhibition. The neutral CS could be adsorbed on the surface of the metal via the mechanism of chemisorption which involves the disarticulation of water molecules from the surface of the metal and the electrons sharing between iron and oxygen atoms. The CS molecule can also be adsorbed on the metal surface base on donor-acceptor interaction between vacant d orbital of iron and pi electrons of the heterocycle. Conversely, the protonated CS could be adsorbed via electrostatic interactions between the previously adsorbed sulfate ion and positive molecules. This complex was adsorbed on the surface of the aluminium through Van der Waal force to form a protective layer to protect the surface of the aluminium from corroding. The corrosion inhibition mechanism is normally considered to be due to the surface of the metal. This phenomenon tends to obey diverse performance for the CS studied, alloy or metal, corrosive environment, the inhibitor concentration and acid/alkaline, solubility, temperature, and so on. The organic molecule in CS becomes physisorbed on the aluminium surface, which forms a defensive layer and therefore enables the anticorrosive performance [49]. In this study, the CS is established to behave as a good inhibitor for aluminium corrosion. From the findings above the maximum inhibition capacity (~ 70 and 99%) was attained at 2g and 10g inhibitor concentration. The inhibitor efficiency value reported for non-toxic corrosion inhibitors in the literature is almost between 70% and 95% . This denotes that the coconut shell based on agricultural wastes, with inhibition effectiveness of 70 to 99% , can be regarded as an effective inhibitor for aluminium corrosion control in $0.5\text{ M H}_2\text{SO}_4$ solutions. Also, agricultural waste is considerably available and the method of inhibitor preparation is cost-effective and eco-friendly making it suitable to be efficiently applied as a sustainable inhibitor.

Conclusions

CS was found to be an excellent inhibitor for corrosion of aluminum alloy in 0.5 molar sulphuric acid solutions, which is biodegradable, cheap, non-toxic, and readily available. The weight loss technique illustrates that inhibition performance increased with an increase in CS concentration, however, reduced with increasing exposure time. The interaction mode between the CS and aluminium is described as physisorption and follows the Langmuir adsorption isotherm. The Tafel polarization plots illustrate that the CS acts as a mixed-type inhibitor. The obtained results show that corrosion inhibitions occur via the adsorption process. The surface morphology result confirms the formation of a protective layer on the surface of the aluminium in the 0.5M H₂SO₄ solution. Hence, CS can be utilized in industries for the mitigation of the corrosion process as a sustainable green corrosion inhibitor.

Declarations

Due to technical limitations, Declarations is not available for this version.

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Figures

Figure 1

Corrosion Rate by Weight-Loss Measurement

Figure 2

Sulphuric acid corrosion inhibition efficiency of the inhibitor for 168 hours at different inhibitor concentrations

Figure 3

Potentiodynamic polarization curve for pure aluminium in 0.5 M H₂SO₄ Solutions in the presence and absence of CS

Figure 4

Langmuir Isotherm for the adsorption of coconut shell on the aluminium alloy surface in 0.5 M H₂SO₄ solution.

Figure 5

SEM images of the aluminum sample: (a) aluminium alloy after immersion in H₂SO₄ for 168 h, (b) aluminium alloy after immersion in H₂SO₄ containing 10 g CS for 168 h.

Figure 6

Elemental compositions of aluminum electrodes (a) aluminium alloy after immersion in H_2SO_4 for 168 h, (b) aluminium alloy after immersion in H_2SO_4 containing 10 g CS for 168 h.