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Article · August 2023

DOI: 10.14456/easr.2023.44

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Engineering and Applied Science Research 2023;50(5):413-419 Research Article

Engineering and Applied Science Research

https://www.tci-thaijo.org/index.php/easr/index

Published by the Faculty of Engineering, Khon Kaen University, Thailand

Inhibition potential of silver-gold nanoparticles on mild steel in 3.5% NaCl solution

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Received 30 March 2023
Revised 5 July 2023
Accepted 10 August 2023

Abstract

This study investigates the corrosion behaviour of silver-gold nanoparticles as an inhibitor on the degradation of mild steel in 3.5% NaCl (saline environment) using gravimetric analysis and potentiodynamic measurement. The inhibitor Ag-AuNPs was synthesized from Kola nut pod. Five different concentrations of the Ag-AuNPs solution (0, 5, 10, and 20 μg/ml) were added to the saline environment. The gravimetric result showed that inhibition efficiency of 83.33% was the highest at 20 μg/ml of Ag-AuNPs inhibitor concentration. The Tafel polarization result showed that the solution with 20 μg/ml of Ag-AuNPs had the highest inhibition efficiency of 99.465%. At 0 μg/ml of Ag-AuNps, the surface morphologies of the mild steel sample did not show the existence of Ag-AuNps constituent in the saline environment containing the nanoparticles. The outcome showed that the saline environment with 15 and 20 μg/ml of Ag-AuNPs could successfully limit the corrosion of the mild steel.

Keywords: NaCl environment, Saline environment, Mild steel, Gravimetric, *Tafel* polarization, Surface morphology

1. Introduction

Metals and their alloys are the most important material used in engineering activities such as constructions [1]. Steel is an alloy and are majorly used in petrochemical, chemical, transportation, automobile industries, production, and storage industries. Acid solutions are frequently used on metal substrates such as cleaning solution or production of cleaning solution, pickling, descaling, and acidizing operation to ensure the intended purpose of the metal substrate is achieved [2]. The major problem of mild steel in its application is corrosion attack, which leads to structural degradation, equipment shutdown, and loss of valuable product [3].

Corrosion has been a colossal problem to engineering and society, due to its negative impact on society (degradation of metals). Corrosion causes instability of human life and loss of energy. Corrosion is gotten from the Latin word *'corrodere'* which implies 'to attack' or 'to chew away'. It is predicted that corrosion will destroy one-quarter of yearly steel output, which is around 150 million tons per year. This implied a loss of 5 tons every second [4]. Corrosion is a natural process that brings about the gradual deterioration of materials (typically metals) through chemical and/or electrochemical interactions with their surroundings [5, 6]. The idea of rusting of metal (such as car bodies, water pipe, and pipeline) are all gotten from corrosion.

To prolong the work span of metals or eliminate corrosion of metals, different corrosion control, and resistance technique have been developed from various research and experiments carried out over the years [7-11]. These controls and techniques help in the reduction of metal corrosion, which results in reduced failure associated with corrosion. Thus, a sharp reduction in the operation cost [12]. The common methods of controlling corrosion include cathodic protection, use of inhibitors, anodic protection, and coating [7, 13]. Inhibitors tend to gradually reduce the rate of corrosion by forming a protective layer over the material such as metal. With the use of appropriate corrosion inhibitors and correct application, corrosion would be reduced. The use of an inhibitor can significantly reduce the rate of corrosion, therefore improving metal service life [14]. Organic inhibitors are synthetic which makes them not environmentally friendly due to the harmful compounds that are discharged into the environment after use. Moreover, most of the inorganic inhibitors are non-ecologically friendly, and toxic [15]. The utilization of plant-extracted nanoparticles as inhibitors could meaningfully bring improvement to inhibition performance [1]. Nanoparticles are gotten from plants extracts, enzymes, polymers, carbohydrates, and vitamins [16].

Among the nanoparticles, silver and gold nanoparticles can be used to prevent corrosion by forming a protective layer on the surface of the metal. Silver nanoparticles have been produced using plant extracts, and other different biological source materials e.g., cola pod, cocoa pods, etc. through the means of green synthesis [1]. In industrial application, nanoparticles are mostly applied in the

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doi: 10.14456/easr.2023.44

protection of metals against corrosion in different environment [17, 18]. Since corrosion causes irreversible destruction or damage to materials as well as it is very expensive occurrence, different techniques have been utilized to ameliorate corrosion rate. These methods include corrosion inhibitors, pH change, surface coating, potential by cathodic and anodic reaction, and environmental modification [19-21]. The utilization of inhibitors (organic and inorganic), a prominent option, help to protect the metals for chemical disintegration through the activation of protective film on the surface of the metal as well as corrosion rate control. Some studies and review articles have summarized the useability and various mechanism of adsorption of the inhibitors on substrates. They are found to have high inhibition efficiency [22-25]. Nanomaterials are considered viable as corrosion inhibitors because of their surface-to-volume ratio with regards to their conventional macroscopic materials [26]. The active sites leading to corrosion on the metal surface are blocked with the utilization of nanomaterials or nano-compounds. These materials are eco-friendly and environmentally degradable. The chemical, mechanical and optical properties of materials are improved because of nanoparticle coatings [27].

Nanoparticles have been used as corrosion inhibitor on some metals such as mild steel, medium carbon steel, aluminium, and stainless steel [28]. It is a fact that there is higher reactivity of silver nanoparticles against aqueous acid environment. Atta et al. [29] utilized silver nanoparticle to prevent carbon steel alloys against corrosion in an aqueous acidic solution of HCl. The study revealed that the coated silver nanoparticle utilized acted as a mixed-type inhibitor with great potential to inhibit further corrosion of the metal sample. The beneficial potency of silica nanoparticle suspension as corrosion inhibitors of copper substrates was determined in a study by Lee et al. [30]. The utilization of silica nanoparticles significantly silica nanoparticles significantly lowered the corrosion rates for about 4 times mostly when 10% of the surface was under the covering of the silica nanoparticles. The mechanism of inhibition was attributed to nanoparticle precipitate on the surface of the copper substrate forming nano-fins, which prevented negatively charged ions from reaching the substrate surface. Hence, metal dissolution rate was reduced. Green corrosion inhibitor obtained from gum Arabic nanoparticles (GA-NPs) was used to inhibit corrosion of steel reinforcement of concrete in a carbon dioxide environment. The study reported that the introduction of 3% GA-NPs inhibitor into the concrete prevented further corrosion through inhibitor molecule adsorption over the surface of the steel reinforcement. This resulted into formation of protective layers. The GA-NPs have great potential to inhibit corrosion as well as improve resistance to carbonization of the concentration matrix [31]. More so, the anti-corrosion performance of cerium oxide (CeO_2) nanoparticles which were introduced in waterborne binders for direct to metal coatings was assessed by Gonzalez et al. [32]. An outstanding performance of anti-corrosion features of CeO_2 nanoparticles was reported with high-resistance to corrosion when immersed in NaCl water solution for several days. The anticorrosive tendencies of copper nanoparticles synthesized from the leaf, stem, and root of potato were assessed on two different duplex stainless steel in H_2SO_4 environment by Olaseinde et al. [33]. The nanoparticles inhibition efficiency increases with its concentration. However, the copper nanoparticles from the roots have the highest corrosion resistance than that of leaves and stems in the corrosive media.

Extensive works are limited on the use of Ag and Au nanoparticles synthesized from kola pod as hybrid composite inhibitor for mild steel in a saline environment. It is important to state that the inhibitor in this present study is also a green sustainable inhibitor. A waste biomass, Kola nut pod, was extracted to obtain the Ag and Au nanoparticles. These two were later mixed together at a balanced ratio. This Ag-AuNPs is also cost-effective and highly needed in corrosion control. The inhibition is superior because the combination of two different inhibitors will help to complement each other for better performance. Hence, the utilization of this waste material helps to sustain the environment by reducing environmental pollution. Also, the extracted nanoparticles would help to ameliorate corrosion of metals in a saline environment. There is no study done on the hybrid usage of Ag-Au nanoparticles (Ag-AuNPs) as environmental-friendly corrosion inhibitor for mild steel in NaCl media. Hence, this present study examined the potential effectiveness as well as the inhibitive properties of hybrid composite of Ag-AuNPs obtained from Kola nut pods (a biomass waste) on mild steel when subjected to saline environment. Various concentrations of the hybrid composite nanoparticles were introduced into the saline environment before the mild steel was immersed, while measurements were obtained through the gravimetric method and potentiodynamic polarization technique. The surfaces of substrates after the corrosion experiments were examined using the scanning electron microscopy with energy dispersive spectroscopy attachment.

2. Materials and methods

2.1 Steel sample preparation

The mild steel was obtained from Alloy Global Steel LTD., Surulere Ilorin, Kwara State. The samples were prepared according to the ASTM G1-03 (2017) and ASTM E3-11 (2017) guidelines. The mild steel was cut into the dimension of 25 mm by 20 mm and a diameter hole of 1 mm was drilled on the mild steel for gravimetric test using Guillotine and hand drilling machine, respectively. For the metal sample to have a smooth and polished surface, emery paper of various grades (#220, #320, #400, #800, and #1200) were used. After, the samples were degreased using ethanol and cleansed with acetone, and then rinsed with distilled water. Furthermore, to prevent outside air particles from getting to the metal surface, they were dried and kept in a desiccator. The elemental composition of the metal samples was carried out in Midwal Engineering Limited, Lagos State according to ASTM E415-21 (2021) standard using the Spark Atomic Emission Spectroscopy (Spectromaxx LMF06 Spectrometer Machine, Serial Number: 15007384).

2.2 Preparation and characterization of silver-gold nanoparticles

Silver-Gold nanoparticles was obtained from kola nut pod according to the synthesis method used by Asafa et al. [1]. The concentration of the nanoparticles was 250 µg/ml. The nanoparticles obtained serve as the inhibition source to prevent further corrosion growth on the substrates in the saline environment.

2.3 Preparation of 3.5% NaCl and the corrosion environment

NaCl was purchased from Larry Kunle Nig. Enterprises, Ilorin, Nigeria. Kwara State. The corrosion environment solution was obtained by dissolving 35.42 g NaCl in 1000 ml of distilled water to obtain 3.5% NaCl. Eq. (1) shows the weight percentage (weight calculation).

$$\%\,Weight = \left[\frac{mass\ of\ solute\ (g)}{volume\ of\ solution\ (ml)}\right] \times 100 \tag{1}$$

The silver and gold nanoparticles obtained from Nanotechnology Laboratory of Ladoke Akintola University of Technology (LAUTECH), Ogbomoso, Nigeria was prepared alongside with the 3.5% NaCl solution. With this, the saline-inhibitor environment at equal volume in 5 different concentrations (0 µg/ml (Control), 5, 10, 15, and 20 µg/ml) were obtained. The different concentrations were prepared using the Ag-Au nanoparticles alongside with the 3.5% NaCl gotten from 250 µg/ml stocks gotten from distilled (deionized water).

2.4 Corrosion experimentation

In this study, two different corrosion measurement approaches involving the weight loss (Gravimetric analysis) and potentiodynamic polarization were employed.

2.4.1 Gravimetric analysis

The mild steel was measured using a digital weighing balance (HX 302) after a rope (thread) was inserted or tied round the hole drilled on the metal. The obtained weight was recorded as the initial weight (W_1) of the sample. The mild steel sample was immersed into the solution prepared earlier which contains the saline environment and the bimetallic alloy silver and gold nanoparticles and was studied. The experimental period was between 1 and 14 days at 2 days interval. The test was carried out according to the ASTM G1-03 (2017) standards. After the metal was removed from the environment, corrosive particles were formed on the mild steel sample which were later cleaned with regards to ASTM G1-03 standards (2017). Then, the samples were dried and reweighed to obtain the final weight (W_2). The weight loss was determined using the variation between the final weight and the initial weight. The experiments were carried out in duplicates and the average of the values was used. The corrosion rate (CR), inhibition efficiency (IE), and weight loss were evaluated using Eqs. (2 – 4), respectively [15].

$$\mathrm{CR\ (mmpy)} = \frac{87.6\times(W_1-W_2)}{DAT} \tag{2}$$

$$\mathrm{IE\ (\%)} = \frac{CR_B-CR_W}{CR_B} \times 100\% \tag{3}$$

$$\text{Weight loss} = \frac{W_1-W_2}{W_1} \times 100 \tag{4}$$

where W_1 and W_2 are the initial weight (g) and final weight (g), D is the density of the coupon (g/cm^3), A is the coupon area (cm^2), T is the exposure time (h), and, CR_B and CR_W are the corrosion rate in absence and presence of inhibitor, respectively.

2.4.2 Tafel polarization procedure

The potentiodynamic polarization was performed at room temperature with an AUTO LAB potentiostat device and a standard three-electrode cell assembly. Each sample of 10 mm by 10 mm and thickness of 1 mm was placed inside a container that contains resins. The working electrode's surface was smoothed by polishing using emery paper of varying face sizes. The electrolyte was a 3.5% NaCl solution with various concentrations of inhibitors (Ag-AuNPs). The open-circuit polarization was performed for 30 minutes or till a stable potential was achieved. Polarization equipment was tested by varying the working electrode’s voltage and measuring the current generated with respect to time. At a 1 ms^{-1} scan rate, the polarization potential from cathode to anode ranged from -250 mV to +250 mV to assess the effect of inhibitor solution on corrosion behaviour. The corrosion current densities were calculated from the *Tafel* curves using the linear segments extension of the corrosion potential of the anodic and cathodic components. The surface coverage (θ) and inhibitor efficiency (IE%) were calculated using Eqs. (5) and (6) adapted from [15].

$$\text{Surface coverage } (\theta) = \left(\frac{\mathrm{Icorr} - \mathrm{Icorr}^*}{\mathrm{Icorr}}\right) \tag{5}$$

$$\text{Inhibitor Efficiency (IE \%)} = \left(\frac{\mathrm{Icorr} - \mathrm{Icorr}^*}{\mathrm{Icorr}}\right) \times 100 \tag{6}$$

where Icorr and Icorr^* are the corrosion current density in the presence and absence of inhibitors.

2.5 Surface morphology

The morphological examination of the mild steel sample exposed to corrosion without an inhibitor and with an inhibitor of 20 µg/ml was carried out using scanning electron microscopy (SEM). The morphological study was conducted at the Nigeria Geological Survey Agency (NGSA), Kaduna, Kaduna State, with Phenom-World SEM & EDS Set-Up. The mild steel samples were prepared for examination according to ASTM G1-07 standard (2017).

3. Results and discussion

3.1 Elemental constituent of the substrate (mild steel)

Table 1 shows the result of the compositional analysis of the mild steel samples carried out using Spark atom emission spectroscopy and the positive material identification machine. It can be observed that mild steel has 99.6% of iron content and 0.0590% of carbon content. These confirm that the metal is a low carbon steel, which is commonly referred to as mild steel. The utilization of low carbon steel is limited in aqueous environments except the aqueous environment has corrosion inhibitor inclusion. In some cases, increasing the thickness can result in low-carbon steels having adequate service lives in aqueous streams [34]. Ferrite is a body-centered cubic crystal which is the make-up of most mild steel. Mild steels are more machinable compared to other carbon and alloyed steels owing to being the softest phase of steel [35]. The iron content and carbon content of the mild steel used in this study are greater than the ones in the study by Sundjono et al. [36]. Although, the materials used in both studies are low carbon steel, the difference of the carbon and iron contents could result in different corrosion behaviour and corrosion rate.

Table 1 Elemental constituents of the substrate (metal sample) in wt.%

Elements	**Fe**	**C**	**Si**	**Mn**	**Al**	**Cu**	**Ni**	**P**	**S**	**Ti**
(%)	99.600	0.0590	0.0142	0.0864	0.0185	0.0110	0.0065	0.0431	0.0072	0.0015
Elements	**Ca**	**Se**	**Mo**	**La**	**B**	**Za**	**Sb**	**As**		
(%)	0.0015	0.0020	0.0010	0.00049	0.0014	0.0010	0.0044	0.0034		
Elements	**V**	**W**	**Te**	**Ta**	**Nb**	**Sn**	**Zr**	**Bi**		
(%)	0.0025	0.0050	0.0042	0.0300	0.0010	0.0236	0.0010	0.0011		

3.2 Influence of inhibitor concentration on weight loss

Figure 1 displays the weight loss for corrosion of mild steel with time in 3.5 g NaCl solution containing various concentrations of Ag-Au nanoparticles. It was noticed that the weight loss of the sample increased with time but reduced with increase in the concentration of the inhibitors. The weight loss of mild steel at room temperature indicated that the sample underwent poor corrosion resistance. The control sample showed more weight loss compared to the samples in the saline-inhibition environment. This is an indication that the weight loss has a strong relationship with concentration of inhibitor and time of exposure. This assertion was in agreement with the study by Asafa et al. [1]. The result gotten through corrosion process of mild steel in NaCl may be attributed to the presence of dissolved oxygen. The presence of chloride ions (aggressive ions) present in the saline environment plays a vital role in the corrosion process. The rate of corrosion of mild steel was controlled by the presence of Ag-Au nanoparticles in the environment. It was noticed that the corrosion rate of the mild steel increased between day two and day four as shown in Figure 2. However, there was a significant decrease in corrosion rate from day 2 to day 14 as the nanoparticles concentration was increased. This observation is in variation with the result obtained by Sundjono et al. [36]. It was reported that there was corrosion rate increase till day 8 due to the absence of nanoparticles.

With longer exposure times and higher Ag-Au nanoparticles concentrations, the inhibition efficiency increased (Figure 3). However, once the formation of adherent (barrier) film on the surface of the coupon is achieved, the influence of time of exposure significantly reduces the corrosion rate owing to the challenges faced by the agents of corrosion in penetrating the barrier developed [1, 15]. The change from an active dissolving state to a passive state of the metal/solution interface may have caused a protective coating to form on the substrate; hence, causing the increase in inhibition efficiency. This implies that the concentration of the nanoparticle solution decreases corrosion but is insufficient to completely impede the reactive surface of the substrate in the saline environment.

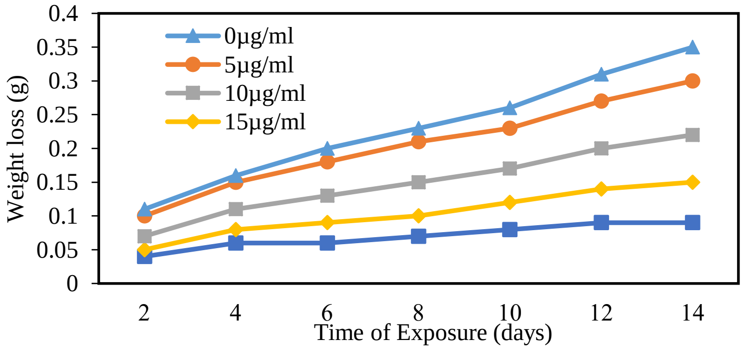

Figure 1 Weight loss vs. exposure time of the corrosion of substrate in the presence and absence of Ag-Au nanoparticle in 3.5% NaCl

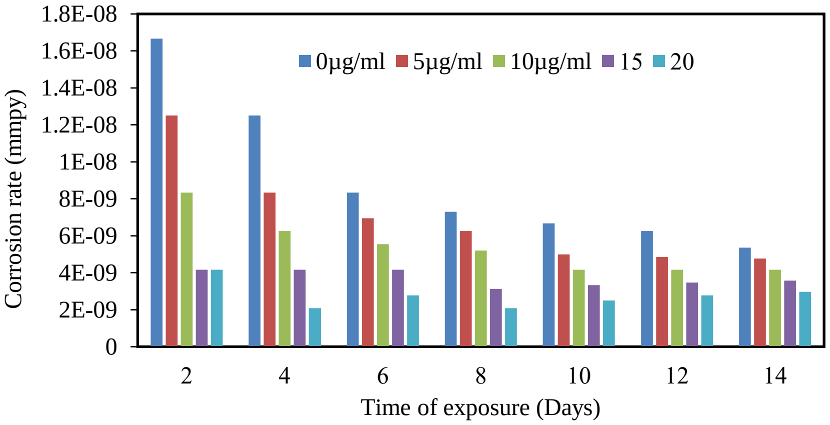

Figure 2 Corrosion rate of substrate in the presence and absence of nanoparticles against time of exposure

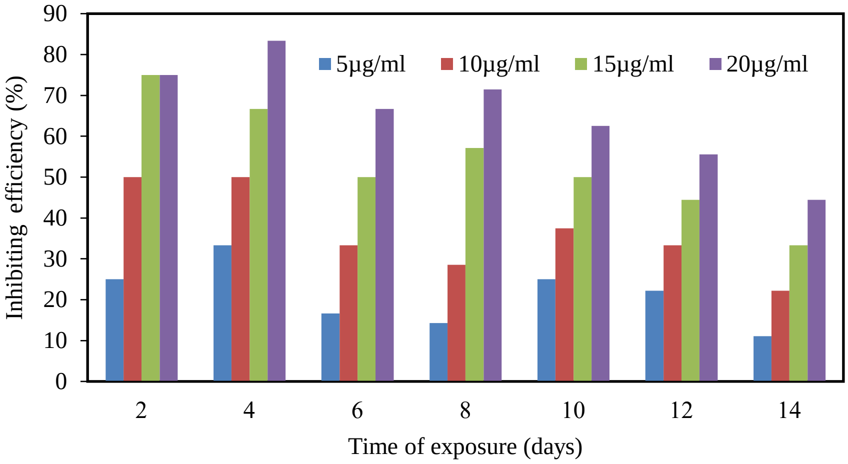

Figure 3 Inhibiting efficiency of substrate in the presence and absence of nanoparticles against time of exposure

3.3 Analysis of potentiodynamic polarization (PDP)

Table 2 shows the PDP parameters of corrosion inhibition of the low carbon steel in 3.5% NaCl in the various pre-determined concentrations of Ag-Au nanoparticles. Polarization measurements are useful for determining the impact of an addition on either the anodic or cathodic partial reactions as well as tracking the development and processes of these partial reactions. The Tafel slopes (βa and βc), resistance polarization, and inhibition efficiency and corrosion current density (Icorr) were determined using the linear segment extrapolation obtained from the corrosion potential curves, as shown in Figure 4. The fact that the inhibitors had an important consequence on both the cathodic and anodic reactions suggests that the addition of Ag-Au NPs slowed the dissolution of the anodic metallic materials. As a result, there is a reduction in the value of corrosion current density (Icorr). The inhibitor molecules adsorption on the substrate's surface is a major factor in the decrease in Icorr values in the presence of inhibitors, especially at higher concentrations. The maximum value of inhibition efficiency (%IE) for mild steel was 99.47% in the environment mixture of 3.5% NaCl and 15 µg/ml inhibition concentration of Ag-Au nanoparticles. It was noticed that the RP (resistance polarization) value was increased from 40.3 ohms (blank) to 2034.8 ohms (15 µg/ml) and later reduced to 1566.4 ohms at 20 µg/ml inhibition concentration for mild steel from blank to 20 µg/ml inhibition concentration. In classifying the inhibitor if it was anodic, cathodic or mixed-type, it has been established that with Ecorr value less than 85 mV, the classification can be anodic or cathodic. However, when Ecorr is more than 85 mV, it should be regarded as mixed type inhibitor [37]. Hence, the inhibitor utilized in this study is a mixed-type inhibitor because the Ecorr value is above 85 mV. The study of Asafa et al. [1] reported this same observation, where the Ag NPs used as inhibitor on mild steel was classified as a mixed type, while the classification was anodic or cathodic on aluminium and stainless steel.

Table 2 PDP parameters of the specimen in different concentrations of Ag-Au nanoparticle in 3.5% NaCl

Concentration (µg/ml)	Ecorr, conc (V)	Ecorr, obs (V)	Icorr (mA)	Ba	βc	Corrosion rate (mmpy)	RP (Ω)	IE (%)
Control	-0.750	-0.749	0.249	0.028	0.135	2.891	40.301	0
5	-0.885	-0.886	0.118	0.027	0.078	1.375	286.95	52.61
10	-0.889	-0.890	0.014	0.022	0.026	0.159	379.39	94.50
15	-0.814	-0.812	0.001	0.022	0.009	0.015	2034.8	99.47
20	-0.904	-0.904	0.022	0.011	0.027	0.025	1566.4	91.20

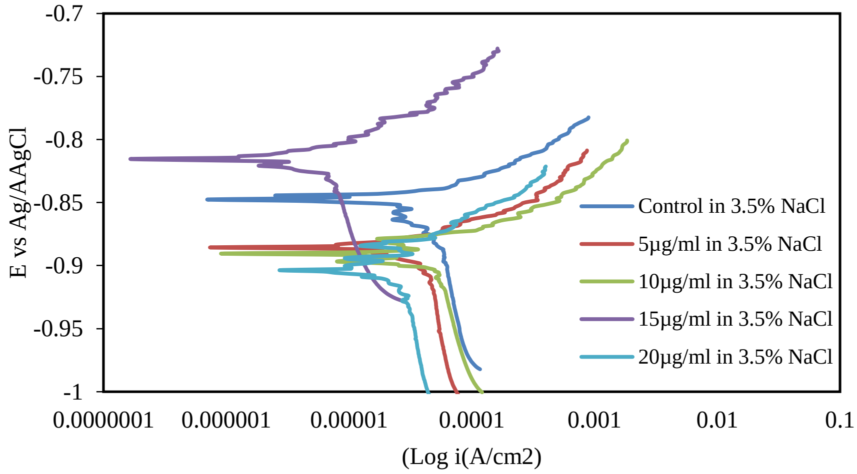

Figure 4 Polarization curve of the substrate in the absence and presence of concentration of Ag-Au nanoparticles in NaCl

3.4 Surface morphology using SEM/EDS

After the 14 days, the metal samples exposed to the environments with inhibitor concentrations of 0 µg/ml and 20 µg/ml were subjected to surface examination using the SEM/EDS technique. The SEM images for 0 µg/ml shown in Figure 5a revealed the presence of a badly corroded, rough surface with observable pits, fissures, and gullies all over the surface. Figure 5b shows the SEM image of sample exposed to 20 µg/ml. The micrographs showed less corroded surfaces with relatively smooth surface and minute observable pits. This is owing to the inhibitive potential present in the Ag-Au NPs obtained from Kola nut pod to prevent growth of corrosive agents on the coupon. This further coincides with the observation made during the corrosion measurements made using the gravimetric and Tafel polarization methodologies that the 20 µg/ml inhibitor exhibits low corrosion rate, and high inhibition efficiency.

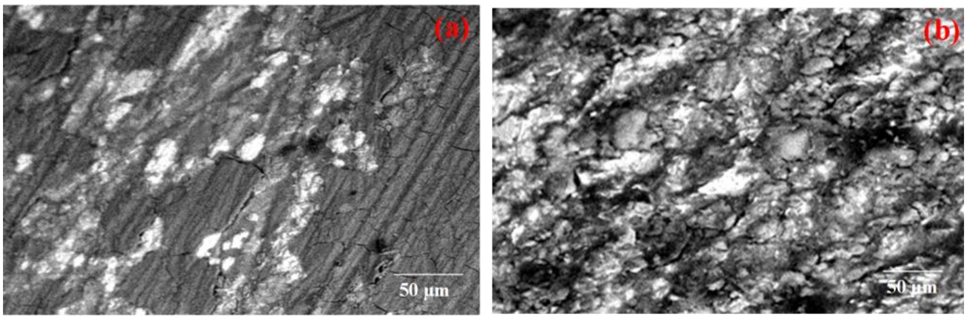

Figure 5 SEM images of samples at (a) 50 µm for the metal sample with 0 µg/ml (b) 50 µm for the metal sample with 20 µg/ml

3.5 Corrosion inhibition mechanism of Ag-AuNPs on mild steel

The corrosion inhibition mechanism of Ag-AuNPs on the substrate in NaCl environment involves a chemical process. Corrosion rate is lowered because of a protective layer formed which comprise Fe^{2+} cation complex compounds as well as the extracted nanoparticles of Ag-Au. In addition, the link to access oxygen is blocked through the formation of high molecular polymeric structure products on the surface of the metal and in the corrosion environment. The polymeric structure is facilitated by the availability of Fe^{2+} cation in the environment [38]. The Ag-AuNPs adsorption on the surface of the substrate could be attributed to a negatively charged acid anion (Cl^-) from the NaCl solution, which inhibits the corrosion of the mild steel in the environment. With this, there is a large likeliness of the Ag-AuNPs to be strongly bonded to metal surfaces via oxygen heteroatom and π-π interactions. Hence, corrosion inhibition of the surface of the metal occurs owing to coordinate bond creation between the Ag-AuNPs and the substrate.

4. Conclusions

In this study, silver-gold nanoparticles synthesized from Kola nut pod was utilized as green corrosion inhibitor of mild steel in saline environment at various nanoparticle concentrations. The introduction of gold-silver nanoparticles solution through green chemistry technique has revealed improvement in the resistance of mild steel to corrosion in 3.5% NaCl. The gravimetric (weight loss) and potentiodynamic polarization measurement indicated that Ag-AuNPs solution exhibit a very strong corrosion inhibition properties on low carbon steel in 3.5% NaCl. The substrate sample passivated in the NaCl environment in the presence and absence of Ag-AuNPs. The rate of passivation increased as the quantity of Ag-Au nanoparticle increased in the solution. The rate of corrosion also decreased as the concentration of Ag-Au nanoparticle in NaCl environment increased. The inhibition efficiency of Ag-Au nanoparticle in NaCl environment is dependent on the concentration as it increased with increasing concentration.

5. Acknowledgements

The authors appreciate the efforts of the laboratory assistant during the experimentation phase of this study.

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