Inhibition efficiency of silver nanoparticles solution on corrosion of mild steel, stainless steel and aluminum in 1.0 M HCl medium

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Abstract

Research work on corrosion behaviour of metals and their alloys continues to attract considerable attention because of the huge cost required to prevent corrosion and replace failed or contaminated components due to corrosion reactions. Different inhibitors have been used to minimize corrosion rate, but addition of nanoparticles has been reported to enhance inhibition efficiency. In this work, the influence of silver nanoparticles (AgNPs) on inhibition of corrosion of mild steel, stainless steel and aluminum in 1.0 M HCl were investigated. Biosynthesized AgNPs were acquired from Laboratory of Industrial Nanobiotechnology, Ladoke Akintola University of Technology Ogbomosho. The particles were hitherto characterized using FTIR, UV-Vis and TEM. Five concentrations of AgNPs solution (0, 5, 10, 15, and 20 µg/ml) were added to 1.0 M HCl. The corrosion inhibition effects of the AgNPs on the metal samples were analyzed using gravimetric (weight loss), potentiodynamic polarization and gasometric (hydrogen gas evolution) approaches. Gravimetric study was conducted within 2000 h of exposure, and the results showed that weight loss increased with exposure time but decreased with increased concentration of AgNPs solution. In addition, inhibition efficiency was enhanced by the addition of AgNPs solution; 52% for mild steel, 70% for stainless steel and 62% for aluminum. Gasometric results revealed that the volume of hydrogen gas evolution reduced with increased exposure time and concentration of AgNPs solution. Furthermore, potentiodynamic polarization results showed that the presence of AgNPs modified the mechanism of anodic dissolution and cathodic hydrogen gas evolution. These results indicated that AgNPs can be incorporated into the existing inhibitors towards minimizing corrosion rate.

Keywords: silver nanoparticles, gravimetric, gasometric, potentiodynamic polarization
1. Introduction

Metals and their alloys remain one of the most versatile engineering materials for structural applications. They are used as integral components of machinery, building construction, bridge trusses, screws, military weapons and automobile components among others. These materials are attractive because of their availability, excellent mechanical properties and ease of fabrication. However, metal alloys are susceptible to corrosion leading to degradation of mechanical properties when exposed to corrosive environment [1,2]. Study of corrosion behaviour in metals has continued to attract considerable attention because a large amount of money is expended on corrosion prevention and maintenance or replacement of failed or contaminated components due to corrosion reactions. Over $1.8 trillion or 3-4% of GDP of most industrialized nations is spent on corrosion related activities [3].

Metallic components come in contact with acids during various industrial processes. For instance, acidic solutions are used during acid pickling, chemical cleaning and processing, ore production, industrial cleaning, acid rescaling, oil-well acidification, and petrochemical processes among others [4]. Most industries prefer to use hydrochloric acid as against other acids (such as nitric acid, sulphuric acid and phosphoric acid) because HCl is more efficient and cost effective [5]. This action makes metallic components susceptible to corrosion during industrial processes. To minimize corrosion of industrial equipment and facilities, appropriate corrosion control mechanism must be adopted.

Several approaches have been deployed to minimize corrosion rates in metals. These include cathodic protection, protective coating, heat treatment, alloying, use of inhibitors and environmental modifications [6]. Among these, the use of corrosion inhibitors (CIs) remains one of the most economical [7] and effective approach [8] to corrosion prevention. According to NACE International [9], a corrosion inhibitor, which is usually a chemical substance, effectively minimizes corrosion rate when added in small concentration to an environment. So whenever an inhibitor is present in the corrosion system at a suitable concentration, corrosion rate is significantly decreased. Corrosion inhibitors lead to the formation of a thin film of passive layer on the surface of the material. This hinders the corrosive substance from penetrating into the metal surface, thereby restraining or minimizing either the oxidation or reduction part of the redox corrosion system or render the dissolved oxygen useless [10].

Of the two types of inhibitors-organic and inorganic-the former is synthetic and therefore not eco-friendly because of the toxic products released to the environment after usage [11].
Consequently, the interest of researchers had shifted to environmentally safe and green inhibitors, which are non-lethal, biodegradable and promptly accessible [7]. In this regard, plant extracts have been used with encouraging results [1,11–16]. Further studies have demonstrated that the use of nanoparticles synthesized from plant extract can significantly improve inhibition performance. The use of plant extracts in synthesizing silver nanoparticles is eco-friendly, reduce energy consumption and can be produced in large scale. Recently, researches have shown that silver nanoparticles can serve as anti-corrosion agent inducing formation of protective layer on the surface of the metals. For instance pitting corrosion in low carbon steel (1010) was significantly reduced by addition of AgNPs [6,17]. AgNPs have been produced from different biological sources through green synthesis. In our previous works [18-24], we produced AgNPs, AuNPs and Ag-AuNPs from diverse materials such as cobweb, cola pod, seeds and seed shell, as well as cocoa pods. Nanoparticles synthesized via green or biological approach have shown to have low toxicity with wide variation in size and shape.

In this study, AgNPs were synthesized from kola nut extract, and were used to inhibit corrosion of mild steel, stainless steel and aluminum in 1.0 M HCl using weight loss, potentiodynamic polarization and hydrogen gas evolution methods.

2. Materials and Methods

2.1 Synthesis and Characteristics of Silver Nanoparticles (AgNPs)

Details of the synthesis and characterization process of AgNPs used in this study are reported elsewhere [21] while summary of the process is presented herein. AgNPs were produced from silver salt with kolanut pod extract as reducing agent. The synthesized nanoparticles were confirmed by UV-vis which showed a maximum absorbance wavelength of 431.5 nm falling within the range previously reported for AgNPs [21]. Furthermore, FTIR spectra with strong peaks at 3336.85, 2073.48 and 1639.49 cm⁻¹ indicate that the 9.5% crude protein in the kolanut was responsible for the stabilization of the synthesized AgNPs (Figure 1a). In addition, silver was the predominant element present in the AgNPs solution based on the EDX pattern (Figure 1b), while the magnesium, sulphur, calcium, sodium and carbon were impurities from the pod extracts and are present on the surface of the biosynthesized AgNPs. The well-dispersed nanoparticles were spherical in shape and poly-disperse in nature, with size ranging from 12 to 80 nm (Figure 1c). The silver is crystalline as revealed by the
characteristic ring-like SAED pattern (Figure 1d) which is typical of face-centred cubic crystalline silver [21].

Figure 1. (a) FTIR spectrum (b) energy-dispersive X-ray signal (c) Transmission electron micrograph and (d) selected area electron diffraction pattern of the biosynthesized AgNPs [25]

2.2 Samples Compositions and Preparation

Three different metal alloys (stainless steel, mild steel and aluminum) were selected for this study. The composition of the mild steel was obtained using Olympus Optical Emission Spectroscopy, while those of stainless steel and aluminum were analyzed with Positive Material Identification (PMI) machine following ASTM E1476 standard. The samples were cut into dimensions of 20 mm × 18 mm while 3 mm hole was drilled on each sample to facilitate easy suspension and removal from the corrosion environments. The samples were mechanically polished using emery papers of different grades, degreased in ethanol, cleaned in acetone and then dried and stored in a desiccator to prevent moisture accumulation. Prior to the storage, the weight of each sample was measured using electronic weighing balance.
2.3 Preparation of corrosion environment

The corrosion environment was prepared by mixing AgNPs solution with 1.0 M HCl. The AgNPs solutions were prepared at different concentrations: 0 µg/ml (control), 5, 10, 15 and 20 µg/ml prepared from the 100 µg/ml stock using distilled water. Then the AgNPs solutions were thoroughly mixed with equal volume of HCl solution in 15 different containers (5 containers for each of the metal alloys). The control solution had no AgNPs added.

2.4 Experimental measurement approaches

Three measurement methods; gravimetric, gasometric (hydrogen gas evolution) and potentiodynamic polarization, were considered for the corrosion study.

2.4.1 Gravimetric/weight loss measurement study

For the gravimetric study, the samples were completely immersed in the corrosion solutions for 400, 800, 1200, 1600 and 2000 h. After immersion for the first 400 h, the specimens were carefully removed, rinsed in distilled water, dried and weighed again. The weight loss was obtained by subtracting the current weight from the original weight. The procedure was repeated after 800, 1200, 1600 and 2000 h of immersion in the corrosion environment. The corrosion rate and inhibition efficiency (IE%) were calculated from the weight loss data according to the following equations [11]:

\[
\text{Corrosion rate (g cm}^{-2} \text{h}^{-1}) = \frac{87.6 \Delta W}{DAT} 
\]

\[
\text{Inhibition efficiency (IE%) = } \frac{W_o-W}{W_o} \times 100\% \tag{2}
\]

where \(\Delta W\) is the weight loss(g), D is the density of the coupon g/cm\(^3\), A is the surface area of the mild steel coupon (cm\(^2\)), T is the time of exposure (h) [16], \(W_o\) and W are the weight loss values in the absence and presence of inhibitor respectively [12].

2.4.2 Electrochemical polarization measurement

Electrochemical study was carried out at room temperature using AUTOLAB potentiostat instrument utilizing a conventional three-electrode cell assembly. The metal samples, each of 20 mm\(^2\), were embedded in resins. The embedded sample, platinum electrode and saturated silver/silver chloride were used as the working electrode, counter electrode and reference electrode, respectively. The surface of the working electrode was made smooth by polishing with emery paper of different grits size. The electrolyte used was 1.0 M HCl solution with varying concentrations of inhibitor (AgNPs). The open circuit corrosion potential was carried
out for 30 min until a stable potential was attained. Polarization experiment was carried out by changing the potential of the working electrode and monitoring the current produced as a function of time. Tafel polarization was studied between cathodic potential of -250 mV and an anodic potential of +250 mV with a scan rate of 1.0 mVs⁻¹ to quantify the influence of inhibitor solution on the corrosion behaviour. The corrosion current densities were estimated from the Tafel curves by extrapolating the linear segments of the anodic and cathodic components to corrosion potential axis. The corrosion rate and inhibition efficiency (IE%) are then obtained from the corrosion current densities. The surface coverage (θ) and inhibitor efficiency (IE%) were determined using Eq. (3-4).

\[
\text{Surface coverage } \theta = \frac{I_{corr} - I_{corr}}{I_{corr}} (3)
\]

\[
\text{Inhibitor efficiency } IE \% = \frac{I_{corr} - I_{corr}}{I_{corr}} \times 100\% (4)
\]

Where \(I_{corr}\) and \(I_{corr}\) are corrosion current density in the presence and absence of the inhibitor, respectively.

2.4.3 Gasometric/ Hydrogen gas evolution measurement approach

The procedure for gasometric measurement of hydrogen gas evolution followed Odusote et al [26]. A delivery tube was used to connect the reaction vessel to the burette. Then, 40 ml of HCl and silver nanoparticles solutions of different concentration (5, 10, 15 and 20 μg/ml) were separately introduced into the mylius cell, and the initial volume of air in the burette was recorded. The prepared metal specimens were dropped into the solution and the mylius cell was quickly closed to avoid entry of air. The volume of hydrogen gas evolved due to the corrosion reaction was estimated by the volume of water changed in the burette. The change in volume was recorded every minute in the HCl solution without inhibitor. These procedures were repeated with the HCl solutions diluted with inhibitors [15]. From the volume of hydrogen gas evolved, inhibition efficiency (%IE) was calculated from Eq. (5)[13, 27].

\[
\text{Inhibition efficiency (IE\%) } = \left(\frac{V_{H0} - V_{H1}}{V_{H0}}\right) \times 100\% (5)
\]

where \(V_{H0}\) and \(V_{H1}\) are volume of hydrogen gas evolved in the absence and presence of the inhibitors respectively.
3. Results and Discussion

3.1 Elemental compositions of the metal alloys

The elemental compositions of the metal alloy samples are presented in Table 1. The percentage of carbon in the steel sample (Table 1a) indicates that it is a low carbon steel i.e. mild steel, while the stainless steel belongs to the standard ferritic grades containing 14.11% chromium with 0.51% Ni (Table 1b) [2]. The aluminum sample belongs to 1xxx group with some additional minor elements (Table 1c) [28].

| Table 1a. Elemental composition of mild steel sample in wt% |
|-----------------------------------------------------------|
| Elements | Fe  | C  | Mn | Ta  | P | S  | Cr | Cu |
| Composition | 99.00 | 0.036 | 0.76 | 0.07 | 0.011 | 0.009 | 0.03 | 0.012 |

| Table 1b. Elemental composition of stainless steel sample in wt% |
|---------------------------------------------------------------|
| Elements | Fe  | Cr  | V  | Mn | Ni | Zn | Cr  | Cu  | Mo |
| Composition | 75.10 | 14.11 | 0.093 | 9.51 | 0.51 | 0.02 | 0.03 | 0.69 | 0.005 |

| Table 1c. Elemental composition of aluminum sample in wt% |
|---------------------------------------------------------|
| Elements | Al  | Si  | V  | Mn | Fe | Zn | Cr  | Cu  | Sn |
| Composition | 99.22 | 0.08 | 0.024 | 0.013 | 0.054 | 0.02 | 0.03 | 0.057 | 0.01 |

3.2 Weight loss against time of exposure

The weight losses as a function of time for all the samples are presented in Figure 2. It was observed that the weight loss after the first 400 h of exposure was higher than the subsequent 400 hrs, which may be due to direct contact between the metal surface and environment. However, the rate of materials dissolution reduced after 400 h of exposure possibly due to formation of passive layers on the metal surface, thereby acting as a barrier between the environment and the surface. For all the samples, the weight loss reduced with increased concentration of the inhibitor/AgNPs solution, implying that the presence of AgNPs in the solution enhanced the formation of passive layers on the metal surface and thus improve the inhibition efficiency. This inhibiting property may be due to the presence of some active constituents such as 9.5 wt% of crude protein and the richness in alkaloids, caffeine (2.8%), theobromine (0.05%), nicotine and kolutine [29]. Mild steel, stainless-steel and aluminum lost 2.24 g, 1.71 g and 1.45 g, respectively after 2000 h of exposure when no inhibitor was
added to the HCl solution. The dissolved mass was reduced to 1.34 g, 1.24 g and 0.77 g, respectively when 5 µg/ml of AgNPs solution was added. At the highest concentration of AgNPs (20 µg/ml), the total dissolved masses were 1.09 g, 0.51 g and 0.58 g, respectively. This indicates that the weight loss strongly depends on the concentration of the inhibitor and exposure time.

![Figure 2](image)

**Figure 2.** Cumulative weight loss for (a) mild steel (b) stainless steel and (c) aluminum samples against time of exposure in 1.0 M HCl medium with and without AgNPs.

The stability of the inhibition behaviour of the extract was evaluated by the trend of the inhibition efficiency (IE) as a function of time (Figure 3). Inhibition efficiency increased with increased exposure time and concentration of AgNPs solution. The increase in IE may be due to the formation of a protective film attributed to the transition of the metal/solution interface from an active dissolution state to a passive state [26]. This implies that the concentration of the nanoparticle solution reduces dissolution of metallic ions but not sufficient enough to fully inhibit the reactive surface of the different metal alloys from the aggressive environment of the acidic medium. They could serve as a viable inhibitor for
metal alloys in HCl solution. However, once adherent film (barrier) was formed on the surface, the effect of the exposure time became less significant because of the difficulty experienced by the corrosion agents in passing through the formed barrier [11].

Figure 3. Inhibition efficiency for (a) mild steel (b) stainless steel and (c) aluminum samples against time of exposure in acidic medium with and without AgNPs

The corrosion rates of the alloys exposed to HCl solution are plotted in Figure 4. For all the alloys, the corrosion rates decreased with higher concentration of AgNPs solution. However, inherent corrosion resistance of stainless steel and aluminum also contribute to the decrease in their corrosion rates attributed to the quick formation of passive layer enhanced by the large amount of Cr and Al in the stainless steel and aluminum, respectively. It also connotes adsorption of AgNPs on the surfaces of metal samples which thereby reduce the active surface area [14].
Figure 4. Corrosion rate of (a) mild steel (b) stainless steel and (c) aluminum samples against time of exposure in acidic medium with and without AgNPs.

Figure 5 shows the surface coverage of the samples after immersion in the corrosive medium. The surface area coverage is a measure of the fraction of the surface covered by the adsorbed molecules of AgNPs solution. The adsorption process creates a thin layer separating the metal surface from the corrosive medium thereby acting as a barrier between them. This implies higher inhibition with increased fraction of metal surface area covered by the adsorbed molecules. According to Figure 5, the surface area coverage increases with concentration of AgNPs and time of exposure.
3.3 Potentiodynamic Polarization Studies

The potentiodynamic polarization curves for corrosion behavior of the mild steel, stainless steel and aluminum in 1.0 M HCl are shown in Figure 6. The values of Tafel polarization parameters including the anodic and cathodic Tafel slopes ($\beta_a$ and $\beta_c$ respectively) and corrosion current density ($i_{corr}$) were obtained by extrapolating the linear segments of the curves to the corrosion potential ($E_{corr}$). The inhibition efficiency along with other parameters are given in Table 2. It is observed that the inhibitors exerted significant effect on both anodic and cathodic reactions indicating that addition of AgNPs retarded both anodic metallic dissolution and cathodic hydrogen gas evolution. This subsequently reduces the value of corrosion current density ($i_{corr}$).

The decrease in the values of $i_{corr}$ in the presence of inhibitors particularly at higher concentrations is largely due to the fact that inhibitor molecules are adsorbed on the surface of the metals. The direction of displacement in the value of $E_{corr}$ determines the category of the inhibitor (anodic, cathodic or mixed type). If the displacement for the inhibited specimen is more than 85 mV as compared to the $E_{corr}$ value of uninhibited
specimen then the inhibitor can be categorized as anodic or cathodic type and mixed type if otherwise. In this study, the values of $E_{\text{corr}}$ is generally lower than 85 mV for mild steel and aluminum while higher than 85 mV for stainless steel (Table 2). This indicates that the inhibitor acts as mixed type for the former metal samples and anodic/cathodic type for the later. The results depicted in Table 2 showed that the values of $\beta_c$ are more affected as compared to the values of $\beta_a$ indicating that the inhibitor acts as cathodic type inhibitors, that is, it has pronounced influence on the cathodic corrosion process for the stainless steel [5].

The values of inhibition efficiency (IE) obtained from the Tafel polarization experiments (Table 2) were higher than those obtained from both weight loss and gasometric experiments (Figures 2 and 7). This behaviour suggests that the IE depends on the method of measurement. Nevertheless, the increase in IE with increased concentration of AgNPs indicates a significant reduction in corrosion rates.

Figure 6. Tafel Polarization curve for (a) control samples (b) mild steel (c) stainless steel and (d) aluminum for immersed in acidic medium with and without AgNPs
Table 2. Corrosion parameter for the samples in presence and absence of nanoparticles

| Metals  | Concentration (µg/ml) | $E_{corr}$ (mv) | $I_{corr}$ (mA) | $B_a$ (mV) | $\beta_c$ (mV) | CR (mmpy) | IE (%) |
|---------|-----------------------|-----------------|-----------------|------------|---------------|-----------|--------|
| Control | -459.51               | -2.14           | 88.88           | -2.14      | 24.89         | 0         |        |
| 5       | -484.04               | -306.35         | 73.25           | 131.25     | 13.55         | 99.29     |        |
| Mild Steel 10 | -490.65             | -819.37         | 91.57           | 164.66     | 9.507         | 99.73     |        |
| 15      | -487.46               | -418.7          | 88.92           | 132.38     | 4.85          | 99.48     |        |
| 20      | -491.50               | -531.92         | 83.65           | 132.71     | 1.98          | 99.59     |        |
| Control | -72.84                | -2.43           | 48.21           | 316.19     | 0.000283      | 0         |        |
| 5       | -548.94               | -21.73          | 221.18          | 259.68     | 0.000252      | 88.78     |        |
| Stainless Steel 10 | -581.17            | -132.73         | 505.33          | 394.93     | 0.000154      | 98.16     |        |
| 15      | -497.01               | -62.33          | 131.08          | 24.24      | 0.00072       | 96.08     |        |
| 20      | -323.23               | -260.22         | 91.59           | 93.58      | 0.0003        | 99.06     |        |
| Control | -760.62               | -2.92           | 124.33          | 166.73     | 31.858        | 0         |        |
| 5       | -766.32               | -4.99           | 192.98          | 332.647    | 24.369        | 41.42     |        |
| Aluminum 10 | -427.72             | -31.73          | 525.35          | 427.05     | 20.876        | 90.78     |        |
| 15      | -763.24               | -5.14           | 245.85          | 272.32     | 15.95         | 93.07     |        |
| 20      | -702.98               | -2.79           | 63.32           | 806.35     | 0.345         | 96.10     |        |

3.4 Hydrogen gas evolution measurement

For all samples, the volumes of evolved hydrogen gas increased with time (Figure 7). This was observed to vary with the concentration of AgNPs solution as well as the metal alloys. The continuous rise in the volume of hydrogen gas is attributed to the time required for surface passivation given that the maximum time used for the hydrogen gas evolution study was still below the minimum 400 h used for the weight loss experiment. It is expected, however, that as surface passivation began, the rate of metal dissolution would retard. At higher concentrations, hydrogen evolution was slightly delayed. Hydrogen gas evolution in stainless steel and aluminum commenced a few minutes after the samples were inserted in the corrosion environment. However, evolution was more rapid in aluminum sample compared to stainless steel sample. Generally, the rate of evolution of hydrogen gas decreased with increased concentration as previously observed by Odusote and Ajayi [11]. The highest volume of hydrogen gas evolved in the blank solution suggested that there is no adsorption layer to inhibit both the cathodic and anodic reactions.
Figure 7. Inhibition efficiency for hydrogen gas evolution study of (a) mild steel (b) stainless steel and (c) aluminum samples against time of exposure in acidic medium with and without AgNPs.

4 Conclusion

Silver nanoparticles solutions, produced via green chemistry approach, have been shown to enhance inhibition of mild steel, stainless steel and aluminum in 1.0 M HCl solution. The results obtained from gravimetric, potentiodynamic polarization and gasometric measurements showed that AgNPs solution possesses significant corrosion inhibition properties for mild steel, stainless steel and aluminum in 1.0 M HCl by influencing the mechanism of anodic dissolution and cathodic hydrogen gas evolution via adsorption which was considered to be spontaneous, exothermic and physisorption. These results indicated that AgNPs can be incorporated into existing inhibitors towards minimizing corrosion rate.
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