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Inhibition efficiency of gold nanoparticles on corrosion of mild steel, stainless steel and aluminium in 1M HCl solution

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ABSTRACT

In this study, the influence of gold nanoparticles (AuNPs) on corrosion behavior of mild steel, aluminium and stainless steel in 1.0 M HCl was investigated. The nanoparticles were previously characterized using FTIR, UV–Vis and TEM. Five concentrations of AuNPs solution (0 μg/ml, 5 μg/ml, 10 μg/ml, 15 μg/ml, 20 μg/ml) were added to 1M HCl. The corrosion rates of the metal samples and inhibition efficiency of the nanoparticles were analyzed using gravimetric (weight loss) and potentiodynamic polarization techniques. After 2000 h of exposure, gravimetric study showed that weight loss was reduced by ~75% translating to ~85% reduction in corrosion rate for the solution containing 20 μg/ml of AuNPs. The equivalent inhibition efficiency was 88%, 98% and 96% for aluminium, mild steel and stainless steel, respectively. Furthermore, potentiodynamic polarization results showed that the presence of AuNPs modified the mechanism of anodic dissolution by the formation of adsorption layer on the surface of the metal samples. These results indicated that AuNPs can be incorporated into existing inhibitors towards minimizing corrosion rate.

Selection and peer-review under responsibility of the scientific committee of the International Symposium on Nanostructured, Nanoengineered and Advanced Materials.

1. Introduction

Metals and their alloys have been identified as the most widely used engineering materials for various structural applications. This is largely due to their desirable mechanical properties, ease of fabrication and availability in the market. Metal alloys have found applications in building constructions, trusses, weapons, automobiles, machineries and many more. However, like other materials, they are prone to corrosion thus bringing about gradual degradation of their mechanical properties when exposed to corrosive environments [1,2]. Studies on corrosion behaviour of metals has attracted a large amount of money aiming at preventing corrosion and maintaining or replacing corroded materials or products. More than $1.8 trillion constituting approximately 3–4% of GDP of developed nations has been expended on corrosion related studies and practices [3]. Similarly, 15%–25% of annual global steel production is estimated to be used for repair or replacement of damages due to corrosion. Approaches to corrosion control in metals include cathodic protection, protective coating, heat treatment, alloying, use of inhibitors and environmental modifications [4,5]. In recent times, corrosion inhibitors (CIs) have been mostly adopted as it is one of the most economical [6,7] and effective approach [8] in corrosion inhibition. Inhibitors, which can either be organic (synthetic) or inorganic (green), perform their functions by forming a thin film of inactive layer on the surface of the material thus hindering the corrosive substance from reaching metal surface. This restrains or minimizes any redox reactions of corrosion systems or render dissolved oxygen useless [9]. The aim of making the world toxic free has shifted the choice of researchers to more environmentally friendly green inhibitors which are biodegradable, non-toxic, safe and easily accessible [6,7]. Due to these, biosynthesis of nanoparticles using plants extracts are more encouraged [1,10–15]. Other studies have shown that plant extract biosynthesized nanoparticles improve inhibition performance and eco-friendliness. They can also be manipulated into forming varieties of sizes with possibility of being scaled up [16,17]. Recently, studies have shown that

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AuNPs can be used as corrosion inhibitors leading to the formation of protective layer on the surface of the metals. Osoh et al. [18] studied the inhibition properties of AuNPs-modified $La_{0.5}Ca_{0.5}$-MnO_3 solution on mild steel for 21 days. A proportionate increase in inhibitory property of the solution with increased concentration of AuNPs was reported. AgNPs, AuNPs and Ag-Au bimetallic nanoparticles have been produced from different biological sources through green synthesis in previous studies [19–26]. This study was designed to evaluate the efficacy of AuNPs as an on mild steel (MS), stainless steel (SS) and aluminium (Al).

2. Materials and methods

2.1. Preparation of gold nanoparticles (AuNPs)

The gold nanoparticles used in this study was prepared at the Laboratory of Industrial Biotechnology as described by Oladipo et al. [22]. 1 ml of crude oil enzyme of A. *niger* L3 (28.69 U/ml) and T. *longibrachiatum* L2 (22.13 U/ml) with a 30 ml of 1 mM $HAuCL_4$ (pH 5.2) solution were used for the synthesis. The characterization was carried out using FTIR, TEM, selected area electron diffraction (SAED) and UV–Vis. Aside colour change, the AuNPs was also confirmed using UV–Vis spectra using spectrophotometer (Cecil, USA).

2.2. Samples compositions and preparations

Stainless steel, mild steel and aluminium alloys were used in this research. The compositions of the metals were obtained using Olympus Optical Emission Spectroscopy following ASTM E1476 standard. For uniformity and ease of suspension, samples of dimension 20 mm × 18 mm with 3 mm center hole were used for the corrosion study. Prior to suspension in corrosive liquid, they were mechanically polished with emery papers of grade 80, 120, 20 and 320, degreased in ethanol, cleaned in acetone and then dried and stored in a desiccator to prevent moisture accumulation. The weight of each sample was measured prior to the storage using electronic weighing balance.

Table 1a–c shows the elemental composition of the selected metal alloys. Table 1a confirmed the composition of carbon (0.036 wt%) in mild steel sample. The sample in Table 1b is identified as stainless steel with a standard ferric grade having characteristic composition of 14.11 wt% of chromium and 0.51 wt% of nickel. Also, Table 1c shows percentage composition of other minor elements in the selected Al sample characterizing it to be of 1xxx group.

2.3. Preparation of corrosion environment

Four concentrations (5 μg/ml, 10 μg/ml, 15 μg/ml, 20 μg/ml) of AuNPs solutions were prepared by mixing 120 μg/ml of the original AuNPs solution in appropriate volume of deionized water. The prepared solutions were then thoroughly mixed with 1.0 M aqueous solution of HCl in 15 different containers with each selected metal alloys sample having 5 containers. The 0 μg/ml concentration was used as the control.

2.4. Experimental measurements

Three corrosion measurement methods were considered: gravimetric (weight) and electrochemical polarization measurements.

2.4.1. Gravimetric/weight loss measurement study

This method required complete immersion of the metallic alloy samples in the corrosion solution. The 5 containers for the corrosion study are marked for different time steps with the first being 400 h with the next container marked with an increased time of 400 h up to 2000 h. After the first 400 h, the specimen in the first container was carefully removed, sufficiently rinsed in distilled water, dried and weighed. Weight loss was then computed by subtracting the current weight from the initial weight. This was repeated for the other 4 containers for each specimen. Corrosion rate (CR) and inhibition efficiency (IE%) were then computed from the weight loss data acquired using the Eqs. (1) and (2), respectively.

$$CR = 87.6\Delta W/DAT \tag{1}$$

$$\mathrm{Inhibitionefficiency(IE\%)} = \frac{W_o - W}{W_0}x10 \tag{2}$$

where ΔW is the weight loss (g), Density, D in gcm^{-3}, surface area, A of mild steel coupon (cm^2), Time of exposure T (h), weight loss values W_0 and Win absence and presence of applied inhibitors respectively.

2.4.2. Potentiodynamic polarization measurement

AUTOLAB PGSTAT 204N instrument was utilized for the polarization study. The instrument has three electrode cells; working, counter and reference electrodes. Each of the metal samples (10 mm × 10 mm in dimension) was embedded in resin at room temperature. They were then attached to the working electrode and dipped alongside the counter electrode and the reference electrode into the mixture in a crucible. The prepared corrosion environment was used as the running electrolyte. The working electrode was polished with different grades of emery papers until the surface is smooth. Also, the open circuit corrosion potential was carried out for 30 min until a stable value of Ecorr was obtained. The linear polarization study was carried out from cathodic potential of −1250 mV to an anodic potential of +250 mV at a scan rate of 1.0 mVs-1 to determine the current density, corrosion rate and inhibition efficiency (IE). Also, the surface coverage (θ) and inhibitor efficiency (IE%) were computed using Eqs. (3) and (4), respectively.

$$\mathrm{Surfacecoverage}\theta = \frac{I_{corr} - I_{*corr}}{I_{corr}} \tag{3}$$

$$\mathrm{Inhibitionefficiency(\%)} = \frac{I_{corr} - I_{*corr}}{I_{corr}}x100\% \tag{4}$$

where Icorr and Iocorr are corrosion current density in the presence and absence of inhibitor, respectively.

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 aluminium 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

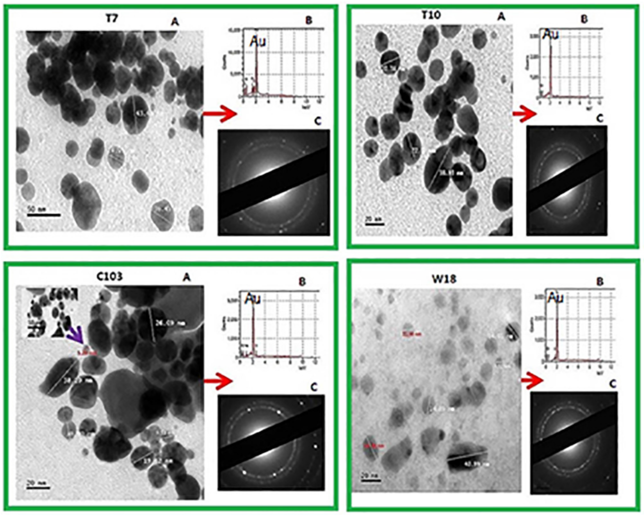

Fig. 1. . The TEM micrographs (a), EDX spectra (b) and SAED patterns (c) of the biosynthesized AuNPs [25].

3. Results and discussion

3.1. Characteristics of the nanoparticles and elemental composition of the metal alloys

AuNPs were spherical in shape and poly-disperse in nature, with size ranging from 18 to 40 nm (Fig. 1a). Gold was the predominant element present in the AuNPs solution based on the EDX pattern (Fig. 1b). The gold is crystalline as revealed by the characteristic ring-like SAED pattern (Fig. 1c) which is typical of crystalline gold [18].

3.2. The dissolution rate

Weight loss variation with respect to time of the samples are shown in Fig. 2. Weight loss in all samples varies inversely with increasing concentration (5, 10, 15, 20 µg/ml) of the AuNPs (inhibitor). Weight loss in mild steel (MS) as compared to other metallic specimens used in this study, is most with loss ranging between 1.0 and 1.5 mg for all concentrations. A control experiment conducted with 0 µg/ml of inhibitor solution was also observed. Weight loss was measured over 2000 h at every 400 h. Most weight loss in each concentration was experienced at the maximum time. Weight loss was minimum at 20 µg/ml and maximum at 5 µg/ml concentration of inhibitor solution for all samples. In mild steel samples, significant increase in weight loss was recorded at each 400 h time step. The loss does not vary with a large margin for each concentration as there exist a very little disparity from one concentration to another. The effect appears approximately the same for the prepared inhibitor concentrations in other samples but the weight loss range in either stainless steel (SS) or aluminium alloy (Al) was less than 1.0 mg at 2000 h. Aluminium specimen however experienced the lowest weight loss as compared to the other two specimens with a maximum loss of 0.7 mg at 2000 h. This observation may be due to the effect of passivation. At the initial stage, there is direct contact between the metallic materials (samples) and the HCl acid solution and thus there are weight loss. This stage is called the active-dissolution phase of passivating metals such as Al, Ni, etc. [27]. However, as the corrosion continues in the presence of the inhibitor, there is formation of thin film through the process of passivation. The presence of thin film layer on the surface of the samples hindered the corrosive environment (HCl acid solution) from reaching the metal surface, thereby drastically reduced the rate of corrosion as shown in Fig. 2.

3.3. Inhibition efficiency (IE%)

Fig. 3 shows the inhibition efficiency (IE) of the metallic samples in HCl solution in absence and presence of AuNPs. There is an increase in the inhibitor efficiency of all three metallic materials after the first 400 h. Formation of protective layer possibly formed due to the transition of metal/solution interface from active dissolution to passive state enhanced IE as reported [28,29]. This

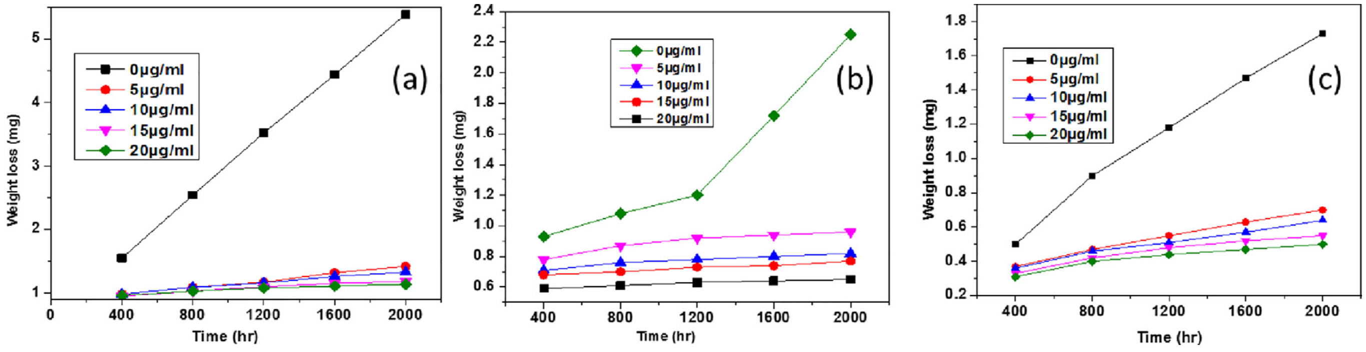

Fig. 2. . Cumulative weight loss for (a) mild steel (b) stainless steel and (c) aluminium samples against time of exposure in 1.0 M HCl medium with and without AuNPs.

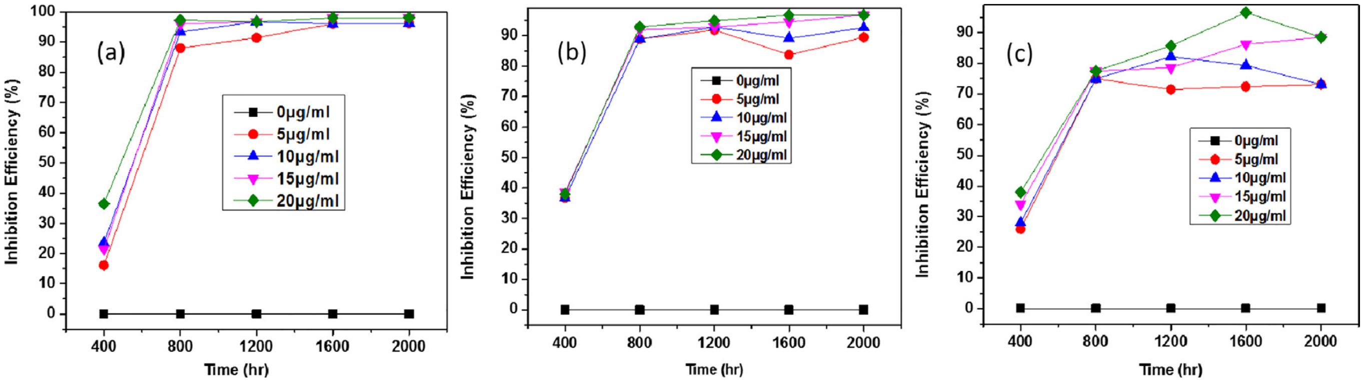

Fig. 3. . Inhibition efficiency for (a) mild steel (b) stainless steel and (c) aluminium samples against time of exposure in HCl solution with and without AuNPs.

explains that the nanoparticles reduce dissolution but was not sufficient to retard the reactive surface of the different metal samples from the inhibition solution. It is noted that the impacts of exposure time became less significant due of the presence of barrier between the metal surface and the inhibitor [10].

3.4. Corrosion rates

Fig. 4 shows the corrosion rates of the samples after exposure to the corrosive solutions. Aluminium samples, for the test duration, have relatively higher corrosion rate than other samples with exceptions noticed earlier in the experiment at 400 h. An unexpected rise in corrosion rate at 15 µg/ml and 800 h of Al samples was observed. Likewise, corrosion rates were minimum for all samples at 20 µg/ml and at maximum concentrations, the surfaces are fully covered by the inhibition film making a complete monolayer on steel surface which resulted in optimum inhibition efficiency in HCl solution. This also validates the claim by other researcher [30]. Also, 5 and 10 µg/ml concentration of mild steel samples at 400 and 800 h have approximately the same corrosion rate although the rate appears higher at 400 h as compared to 800 h. However, inherent corrosion resistance of stainless steel and aluminium also bring about the reduction in their corrosion rates which very likely due to quick formation of passive layer as a result of the presence of large amount of Cr and Al in stainless steel and aluminium samples, respectively.

3.5. Polarization studies

Although the start and finish potentials for all the samples were −1250 mV and +250 mV, respectively, however, corrosion occurred within the potential ranges of −740 mV to −210 mV, −730 mV to −200 mV and −880 mV to −350 mV for mild steel, stainless steel and aluminum, respectively. Tafel polarization curves were used to visualize the corrosion behavior in the metallic samples as shown in Fig. 5. Tafel polarization parameters; β_a *and* β_c (anodic and cathodic Tafel slopes, respectively), I_{corr} (corrosion current density) were obtained via extrapolating of the linear segments of the curves to the E_{corr} (corrosion potential). Inhibition efficiency and other parameters are presented in Table 2. The results in Table 2 also revealed that when inhibitors are used, there exists a reduction in I_{corr} which brought about a significant impact at both the anodic and cathodic reactions. This explains that the use of AuNPs in solution retarded both the anodic metallic dissolution as well as cathodic hydrogen evolution. At higher concentrations, the decrease in value of I_{corr} can be said to have occurred due to the adsorption of inhibitor molecules over the metallic surfaces. Displacement direction of E_{corr} can be used to determine the inhibitor types. When E_{corr} is greater than 85 mV, this inhibitor can then be categorized as mixed type and as either of anodic or cathodic when E_{corr} is less than 85 mV [30]. However, results obtained in this experiment are generally lower than 85 mV indicating that the inhibitors act as anodic or cathodic type. Also, the shift in β_c and β_a values as shown in Table 2 indicates that adsorption of AuNPs modifies the mechanism of anodic dissolution and the cathodic hydrogen evolution. With gold nanoparticles of the same concentration, stainless steel has the lowest corrosion rate for all the three samples. Accordingly, corrosion rate in mild steel reduced from 19.6 to 5.04 mm/yr (74% reduction) while stainless steel and aluminum reduced by 98% and 91%, respectively for gold nanoparticles of 20 µg/ml. This implies that addition of AuNPs has greater influence on stainless steel than the other two samples.

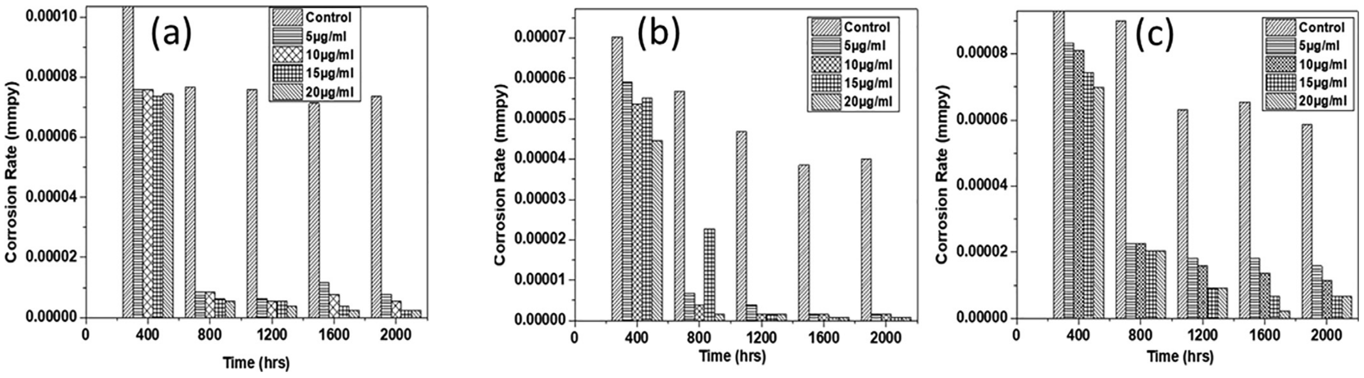

Fig. 4. Corrosion rate of (a) mild steel (b) stainless steel and (c) aluminium samples against time of exposure in acidic medium with and without AuNPs.

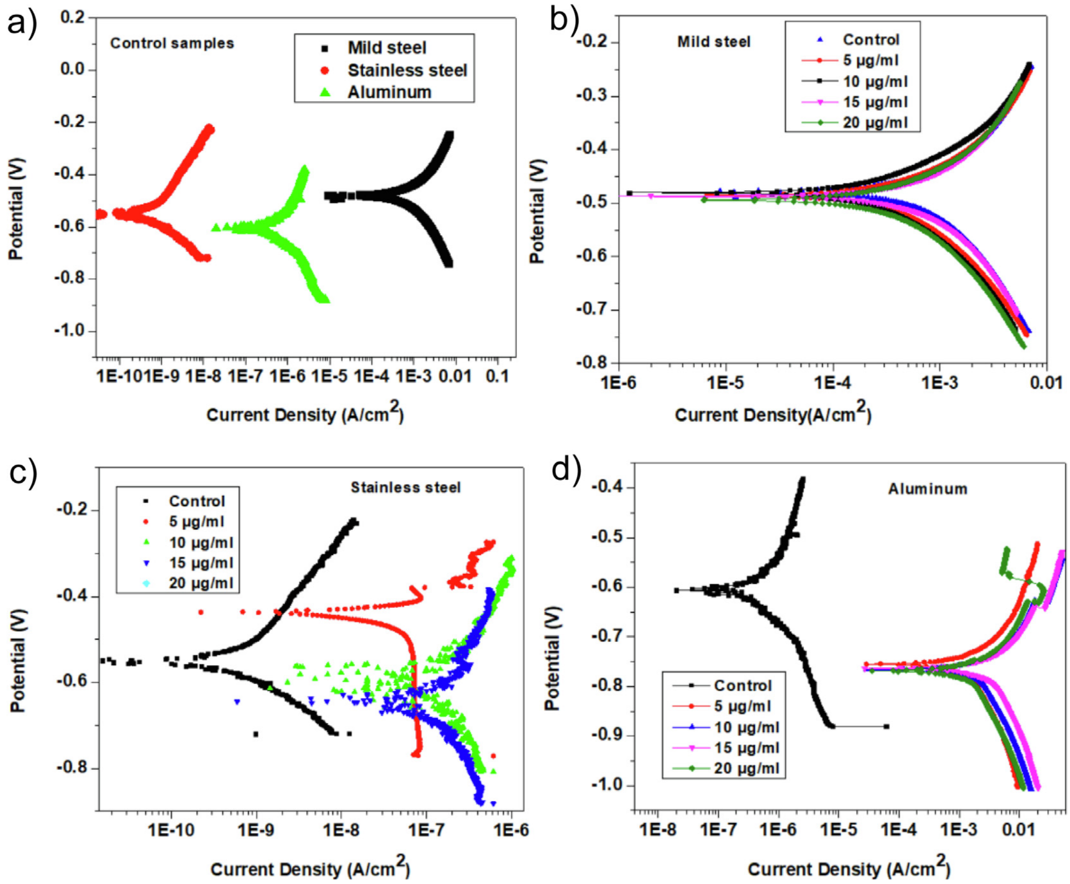

Fig. 5. Tafel Polarization curve for (a) control samples (b) mild steel (c) stainless steel and (d) aluminium for immersed in HCl solution with and without AuNPs.

Table 2
Corrosion parameters of samples with and without nanoparticles.

	Conc. (μg/ml)	$-E_{corr}$ (mV)	I_{corr} (mA/cm)	Tafel slope (mV)		CR (mm/y)
				β_a	β_c	
Mild steel	**Blank**	478.281	16.90	276.12	578.158	19.615
	5.00	485.455	14.51	126.531	184.091	15.2341
	10.00	480.161	12.85	122.592	155.219	13.3071
	15.00	486.549	8.03	175.995	225.544	9.8983
	20.00	494.058	3.12	134.23	190.428	5.0394
Stainless steel	**Blank**	550.577	0.038	93.913	100.308	0.0044
	5.00	415.837	0.012	16.845	194.56	0.0027
	10.00	595.297	0.014	142.199	170.824	0.00010
	15.00	642.746	0.009	149.159	290.166	0.00003
	20.00	487.566	0.005	342.314	342.034	0.000066
Aluminium	**Blank**	604.089	12.47	335.711	695.894	171.23
	5.00	775.009	8.416	178.352	2.837	48.06
	10.00	765.935	5.109	154.971	689.449	35.61
	15.00	763.762	3.979	194.431	608.68	27.73
	20.00	768.795	1.173	137.422	521.597	14.54

4. Conclusion

This study confirmed the inhibition efficacy of AuNPs-modified corrosive environments on mild steel (MS), stainless steel (SS) and aluminum alloy in 1.0 M HCl solution modified with gold nanoparticles. Gravimetric study showed inhibition efficiencies were 88%, 98% and 96% for MS, SS and Al, respectively for solution containing gold nanoparticles of 20 μg/ml. In addition, polarization study indicated that corrosion rates reduced by 74%, 98% and 91% in MS, SS and Al, respectively for the same concentration implying that addition of AuNPs has greater influence on stainless steel than the other two samples. Polarization results also indicated that the presence of AuNPs modified the mechanism of anodic dissolution by the formation of adsorption layer on the surface of the metal samples.

CRediT authorship contribution statement

J.K. Odusote: Supervision, Conceptualization, Methodology, Software. **T.B. Asafa:** Data curation, Writing - original draft. **J.G. Oseni:** Visualization, Investigation. **A.A. Adeleke:** Supervision, Writing - review & editing. **A.A. Adediran:** Writing - review & editing. **R.A. Yahya:** Writing - review & editing. **J.M. Abdul:** Software, Validation. **S.A. Adedayo:** Visualization, Investigation.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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