

# Inhibition Effect of Gold Nanoparticles Embedded in Gloss Paint on Corrosion of Mild Steel in HNO<sub>3</sub> Solution



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**ABSTRACT:** Corrosion is a common and inevitable process in engineering materials, particularly metals. This study investigated the effectiveness of gold nanoparticles (AuNp) as a corrosion inhibitor for mild steel in a 2 M HNO<sub>3</sub> environment. Mixtures of AuNp with gloss paint were prepared at concentrations of 0, 5, 10, 15, and 20 µg/ml to coat the surface of mild steel. The corrosion behaviour was evaluated using gravimetric and gasometric analyses. Weight loss and inhibition efficiency were measured after exposing the samples to the corrosive environment for 24, 48, 72, 96, and 120 hours, while hydrogen evolution was recorded at 10-minute intervals over 0 to 160 minutes of exposure. The findings indicated that the corrosion rate increased with exposure time but decreased as the concentration of AuNp increased. The inhibition efficiency of AuNp decreased over time but increased with higher concentrations, with the 20 µg/ml sample showing the highest inhibition efficiency of 90% at 20 hours. The gasometric analysis further confirmed that higher AuNp concentrations led to reduced hydrogen evolution, demonstrating the potential of AuNp as a corrosion inhibitor for mild steel in HNO<sub>3</sub> solutions. Gravimetric and gasometric analysis showed the inhibitory effect of AuNp in mild steel corrosion in HNO<sub>3</sub>, which can be applied in several engineering industries to inhibit corrosion.

**KEYWORDS:** Corrosion, gasometric analysis, gravimetric analysis, gold nanoparticles, paint coating Embedded Systems

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## I. INTRODUCTION

Corrosion is a natural process that results in the degradation of materials when they interact with their environments. Specifically, metal corrosion is a phenomenon where metals lose their original properties due to chemical reactions with their surrounding medium, such as water, air, or acids (Li *et al.*, 2019). While corrosion is often associated with metallic materials, all material types, including polymers, ceramics, and composites, are susceptible to some form of degradation (Shaw & Kelly, 2006). However, corrosion is most prominent and costly in metals, particularly those used in industries where structural integrity is vital. The impact of corrosion can be severe, leading to loss of efficiency, costly repairs, and even complete failure of vital systems. These failures can be especially hazardous in industries such as transportation, energy, and construction, where the safety of individuals and the environment may be compromised. Moreover, the costs associated with corrosion damage are significant, amounting to billions of dollars globally each year (Al-Mashhadani *et al.*, 2020). In addition to the direct financial costs, corrosion

hinders technological progress, as materials continuously degrade when exposed to environmental factors, preventing them from fulfilling their intended functions (Chopra *et al.*, 2025).

Common forms of corrosion include uniform corrosion, galvanic corrosion, pitting corrosion, crevice corrosion, intergranular corrosion, and microbial corrosion, as shown in Fig. 1 (Dall'Agnol & Moura, 2014). These types of corrosion manifest differently depending on the material, environment, and conditions involved. Among metals, mild steel is particularly vulnerable to corrosion and is one of the most widely used materials in industries, such as construction, machinery, and pipelines (Obot, 2021). Its susceptibility to corrosion can lead to catastrophic failures, especially in harsh environments. This emphasizes the critical need for effective corrosion control methods. There are various methods for managing and preventing corrosion, such as using corrosion-resistant materials, implementing optimal structural designs, and applying cathodic protection. However, one of the most cost-effective and widely used strategies is the application of corrosion inhibitors, chemical substances that can significantly reduce or prevent corrosion when applied in small quantities. These inhibitors can either

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be organic or inorganic. For instance, Jirarungsatian and Prateepasen (2010) explored the use of inorganic passivators like chromates, nitrates, and molybdates, which work by forming a protective layer on metal surfaces, thus preventing corrosion. On the other hand, organic inhibitors such as amines, phosphoric acids, and other compounds have been shown to effectively protect metals in both aqueous and non-aqueous environments (Iacoviello and Cocco, 2007).

In recent years, nanotechnology has emerged as a promising field for enhancing the performance of corrosion inhibitors. Nanomaterials, with their unique properties and high surface-to-volume ratio, have attracted significant attention for their potential in improving the efficiency of corrosion inhibition (Afolalu *et al.*, 2022). Nanotechnology has found applications across various sectors, from medicine to materials science, and it continues to revolutionize industries due to its ability to manipulate materials at the atomic and molecular levels (Malik *et al.*, 2023). Nanoparticles, such as gold nanoparticles (AuNp), have shown particular promise in corrosion inhibition due to their remarkable surface area and enhanced chemical reactivity. Previous studies have demonstrated the potential of nanoparticles in corrosion protection. Jain *et al.* (2020) investigated the use of nanoparticles as corrosion inhibitors and found that nanomaterials exhibit superior corrosion resistance due to their high surface-to-volume ratio. Additionally, Rahal *et al.* (2015) examined the role of sulfur-containing compounds in inhibiting steel corrosion in nitric acid, revealing that corrosion inhibition occurs through a physicochemical adsorption mechanism. However, despite these advancements, there is a gap in the literature regarding the use of gold nanoparticles (AuNp) embedded in gloss paint as a corrosion inhibitor for mild steel. This study aims to fill this gap by exploring the potential of AuNp-based coatings in mitigating the corrosion of mild steel in an  $\text{HNO}_3$  environment. By embedding gold nanoparticles into gloss paint, it is hypothesized that the gold particles will create a protective layer on the steel surface, reducing its exposure to the corrosive medium and improving its resistance to degradation. This research could provide valuable insights into the development of more effective, environmentally friendly and sustainable corrosion inhibitors and contribute to the advancement of corrosion control technologies in industrial applications.

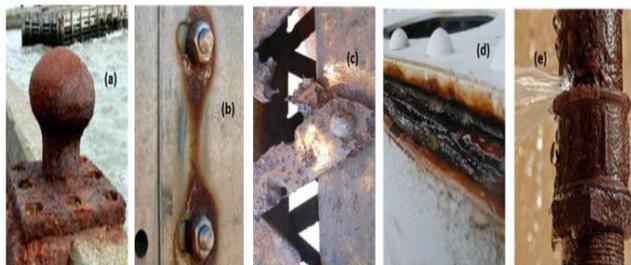


Fig. 1: Forms of corrosion (a) Uniform (b) Galvanic (c) Pitting (d) Crevice (e) Microbial (Dall'Agnol & Moura, 2014)

## II. METHODOLOGY

### A. Materials

The material used in this study was a mild steel plate with a thickness of 2.5 mm. To ensure accurate and reliable results, various laboratory apparatus and materials were employed throughout the study such as Pyrex beakers and test tubes, desiccators, and weighing balance. A desiccator was used to maintain a controlled environment and prevent moisture interference in the samples. A digital weighing balance (Shimadzu Tx 423L) with a precision of 0.001 g was utilized for precise measurement of sample mass before and after exposure to test conditions. Emery papers of grit sizes 80, 120, 220, 400, 600, 800, 1000, and 1500 were employed for surface preparation, ensuring uniformity and consistency in the test specimens. Gloss paint was applied as a protective coating where necessary. The experimental procedure involved the use of an acidic solution ( $\text{HNO}_3$ ) for corrosion testing, while distilled water was used for sample rinsing to eliminate contaminants. Additionally, rope threads were used for handling and suspending the specimens during the immersion process. This combination of carefully selected materials and equipment ensured a controlled and standardized experimental setup for studying the corrosion behaviour of the mild steel samples.

### B. Sample Preparation

Mild steel samples were precisely machined into rectangular coupons with dimensions of 25 mm  $\times$  20 mm to ensure uniformity in the experimental setup according to ASTM G1 standards (Odusote *et al.*, 2024). A 3 mm diameter hole was drilled along the 20 mm seam of each sample using a high-precision drill bit to facilitate suspension in the corrosive medium and enable convenient retrieval after immersion. Surface preparation was carried out by polishing the metal specimens using silicon carbide (SiC) emery papers of varying grit sizes 80 - 1500 to achieve a smooth and uniform surface, thereby minimizing surface defects that could influence corrosion behaviour. The polished samples were then subjected to chemical cleaning by degreasing in absolute ethanol, followed by immersion in acetone to remove residual contaminants. To prevent atmospheric oxidation before testing, the cleaned samples were thoroughly dried with a Dewalt D2614 hand drier at 200°C and stored in a desiccator under controlled conditions as adopted by Odusote *et al.* (2021). To enhance corrosion resistance, the prepared mild steel coupons were coated with a protective layer comprising a mixture of gloss paint and gold nanoparticles. This coating was evenly applied to ensure uniform coverage by painting the coupons and allowed to cure in under room temperature for 6 hours for complete drying and adhesion. The control sample was not coated but was chemically cleaned as reported for other samples. The elemental composition of the mild steel samples was determined using an Optical Emission Spectrometer (OES) to accurately quantify the constituent alloying elements. This analytical technique provided precise compositional data, ensuring the reproducibility and consistency of the experimental results.

### C. Preparation and Characterization of the Gold Nanoparticles

#### 1) Preparation of Gold Nanoparticles

The synthesis and characterization of gold nanoparticles (Fig. 2) were conducted at the Nanoparticles Laboratory, Ladoké Akintola University of Technology (LAUTECH), Ogbomosho, Nigeria. The method employed followed the procedure adopted by Odusote *et al.* (2024). The gold nanoparticles were synthesised by green synthesis from kola nut pods. The procedure involved the addition of 1 ml of supernatant to 1 mM HAuCl<sub>4</sub>. The supernatant is derived from suitable agricultural waste with the capacity of reducing HAuCl<sub>4</sub> salt to metal. The gold nanoparticles were synthesized at varying concentrations and subsequently integrated into a protective coating for mild steel corrosion studies.



Fig. 2: Gold nanoparticles

#### 2) Preparation of Nitric Acid Solution as a Corrosive Medium

A 2 M nitric acid (HNO<sub>3</sub>) solution was prepared to serve as the corrosive medium by dissolving 126 g of HNO<sub>3</sub> in distilled water to obtain a final volume of 1000 mL (1 L). The solution was mixed thoroughly to ensure complete homogenization before use in the immersion experiments.

#### 3) Synthesis and Preparation of Gold Nanoparticle-Coating Solutions

Gold nanoparticles were synthesized at five different concentrations: 0, 5, 10, 15, and 20 µg/mL based on Equation 1. The synthesis process involved the chemical reduction of gold precursors, ensuring a controlled particle size distribution suitable for coating applications. The prepared gold nanoparticle solutions were characterized using appropriate techniques to confirm their size, dispersion, and stability before incorporation into the coating medium. To investigate the effect of gold nanoparticle concentration on the corrosion resistance of mild steel, ten different coating solutions were prepared by thoroughly mixing gloss paint with varying concentrations of gold nanoparticles. Each mixture was formulated in a separate container to ensure uniform dispersion of the nanoparticles. Two different mild steel samples were coated for each of the five nanoparticle concentrations, making a total of ten coated samples. The mixing ratios for the gold nanoparticle-gloss paint mixtures are detailed in Table 1. The mixtures were stirred continuously to maintain homogeneity before application on the metal substrates.

#### 4) Coating of Mild Steel Samples and Immersion Corrosion Testing

Mild steel samples (25 mm × 20 mm × 2.5 mm) were thoroughly polished with emery papers of varying grit sizes to achieve a smooth surface free from contaminants. The samples were degreased using ethanol, followed by acetone cleaning, and then air-dried in a desiccator to prevent oxidation before coating. The coating was applied by painting the prepared gold nanoparticle-gloss paint mixture onto the surface of the steel coupons. The coated samples were air-dried at room temperature for 6 hours to allow for proper adhesion before exposure to the corrosive medium. The coated and uncoated mild steel samples were then completely immersed in separate beakers containing 2 M HNO<sub>3</sub> solution for different exposure durations of 24, 48, 72, 96, and 120 hours. The beakers were labeled according to the corresponding coating type and immersion duration, and the samples were suspended using rope threads to prevent direct contact with the beaker walls, ensuring uniform exposure. The experiment was conducted at room temperature under controlled laboratory conditions to minimize external interference. After the designated immersion periods, the samples were retrieved, rinsed thoroughly with distilled water, and dried before further analysis. The weight loss of each sample was recorded using a digital weighing balance, allowing for a precise determination of corrosion rates. The corrosion behaviour of the coated and uncoated mild steel samples was then evaluated based on weight loss measurements over time (Equation 2), and the data obtained were analyzed to determine the effect of gold nanoparticle concentration on corrosion resistance.

$$\text{Sample} = \frac{\text{Original concentration}}{\text{Desired concentration}} \times \text{desired Volume} \quad (1)$$

$$\text{Weight loss } (\Delta w) = w_f - w_i \quad (2)$$

where  $w_i$  and  $w_f$  are the weights of the coupon samples before and after the corrosion test.

Table 1. Mixing ratios for gloss paint and gold nanoparticle mixtures.

S/N	Sample	Constituent
1.	Control	0 µgml <sup>-1</sup> Concentration (i.e. 5 ml volume of precursor + gloss paint)
2.	1 <sup>st</sup> Concentration	5 µgml <sup>-1</sup> Concentration (i.e. 5 ml volume of prepared 5 µgml <sup>-1</sup> solution + gloss paint)
3.	2 <sup>nd</sup> Concentration	10 µgml <sup>-1</sup> Concentration (i.e. 5 ml volume of prepared 10 µgml <sup>-1</sup> solution + gloss paint)
4.	3 <sup>rd</sup> concentration	15 µgml <sup>-1</sup> Concentration (i.e. 5 ml volume of prepared 15 µgml <sup>-1</sup> solution + gloss paint)
5.	4 <sup>th</sup> Concentration	20 µgml <sup>-1</sup> Concentration (i.e. 5 ml volume of prepared 20 µgml <sup>-1</sup> solution + gloss paint)

#### D. Gravimetric Measurement of Corrosion Rate

The weight loss measurement was conducted to evaluate the corrosion behaviour of mild steel samples in 2 M nitric acid (HNO<sub>3</sub>) with and without varying concentrations of gold nanoparticles (AuNp). Each metal sample was carefully weighed using a digital analytical balance before immersion in the corrosive medium. The samples were then suspended in separate Pyrex beakers containing 2 M HNO<sub>3</sub> solution to ensure uniform exposure to the acidic environment. To investigate the influence of gold nanoparticles on corrosion resistance, the experiments were carried out in the absence and presence of AuNp at different concentrations (0, 5, 10, 15, and 20 µg/mL). After the designated immersion periods (24, 48, 72, 96, and 120 hours), each sample was carefully retrieved using non-contaminating tools, rinsed thoroughly with distilled water to remove residual acid, and air-dried in a desiccator to prevent moisture interference. Once dried, the samples were weighed again to determine the weight loss due to corrosion (Ikubanni *et al.*, 2022). The weight loss data were systematically recorded for further analysis.

#### E. Calculation of Corrosion Parameters

To quantify the corrosion behaviour, the corrosion rate, surface coverage ( $\theta$ ), and inhibition efficiency (IE%) were calculated using Equations (3) – (5), following the methods adopted by Odusote *et al.* (2024) and Ugi *et al.* (2020).

##### 1) Corrosion Rate (CR) Calculation

The corrosion rate was determined using Equation 3:

$$\text{Corrosion rate (mmpy)} = \frac{87.6 \Delta W}{DAT} \quad (3)$$

##### 2) Surface Coverage ( $\theta$ )

The surface coverage, which indicates the extent of surface protection offered by the AuNp coatings was calculated using Equation 4.

$$\text{Surface Coverage } (\theta) = \frac{(CRB-CRW)}{CRB} \quad (4)$$

##### 3) Inhibition Efficiency (IE%)

The inhibition efficiency (IE%) of the gold nanoparticle coating in reducing corrosion was calculated using Equation 5.

$$\text{Inhibition Efficiency } (\%) = \frac{(CRB-CRW)}{CRB} \times 100 \quad (5)$$

Where D is the density of the coupon, A is the area of the metal sample, T is the exposure time, and CRB and CRW are the corrosion rates in the absence and presence of the gold nanoparticle inhibitor.

#### F. Gasometric Measurement of Corrosion Rate

The gasometric method, as adopted by Raghavendra *et al.* (2019), is considered a precise and reliable approach for assessing corrosion. In this method, the evolution of hydrogen gas is measured using a Buckner flask, a burette, a pipette, and a cork to prevent the escape of hydrogen gas, along with a small hose to facilitate the collection of the gas. The volume of hydrogen evolved per minute is recorded, and

from this, the inhibition efficiency (% IE) and surface coverage ( $\theta$ ) can be calculated using Equations 6 and 7.

##### 1) Inhibition Efficiency

Inhibition efficiency (IE) measures the percentage reduction in the corrosion rate due to the presence of the inhibitor. It was calculated using Equation 6.

$$IE (\%) = \left(1 - \frac{V_{Ht}^i}{V_{Ht}^o}\right) \times 100\% \quad (6)$$

##### 2) Surface Coverage

Surface coverage ( $\theta$ ) is related to inhibition efficiency and was calculated using Equation 7.

$$\theta = \left(1 - \frac{V_{Ht}^i}{V_{Ht}^o}\right) \quad (7)$$

where  $V_{Ht}^i$  = the volume evolved at time t for the inhibited solution, and  $V_{Ht}^o$  = is the volume evolved at time t for the uninhibited solution.

##### 3) Experimental Setup and Procedure

This gasometric method was applied to assess the corrosion of mild steel in a 2 M HNO<sub>3</sub> solution, which was chosen due to its aggressive corrosive nature. The corrosion was further studied by introducing an emulsion containing gold nanoparticles (AuNp) at various concentrations: control, 5, 10, 15, and 20 µg/ml. After immersion of the mild steel samples in the acidic solution, hydrogen gas evolution was observed over time. The volume of hydrogen gas evolved was measured and recorded, allowing for the calculation of both the inhibition efficiency (% IE) and surface coverage ( $\theta$ ) for each nanoparticle concentration. These calculations provided a valuable understanding of how the gold nanoparticles influenced the corrosion process.

#### G. Statistical Analysis

Statistical analysis was performed using analysis of variance (ANOVA) to evaluate the significance of the results and to compare the inhibition efficiencies at different nanoparticle concentrations, as discussed by Abd El Wanees *et al.* (2021).

### III. RESULTS AND DISCUSSION

#### A. Compositional Analysis of Mild Steel

The compositional analysis of the mild steel used in this study is presented in Table 2. The results confirm that iron (Fe) is the principal constituent element, comprising 99.54% by weight. This high iron content is consistent with the typical composition of mild steel, reinforcing its classification as a low-carbon steel alloy primarily made of iron. The presence of carbon (C) was found to be 0.21% by weight, which is characteristic of mild steel, as it falls within the typical carbon content range (0.05%–0.25%) for this class of materials. The low carbon content contributes to the mild steel's desirable properties, such as its ductility, machinability, and relatively low strength, making it ideal

for a variety of industrial applications where toughness and formability are crucial. The low carbon content also indicates a higher resistance to brittleness compared to high-carbon steels. In addition to iron and carbon, several trace elements were detected as detailed in Table 2. These trace elements, although present in minor amounts, may have a significant impact on the material's overall properties. The presence of such elements could affect the corrosion behaviour, mechanical strength, and workability of the mild steel, depending on their nature and concentrations. For instance, elements such as manganese (Mn), silicon (Si), sulfur (S), and phosphorus (P), which are commonly found in mild steel, can influence various aspects of its performance. Manganese, for example, improves toughness and hardness, while sulfur and phosphorus can have negative effects, such as reducing ductility or causing the material to become brittle under certain conditions. The observed composition aligns with the expectations for mild steel as outlined by Orhadahwe *et al.* (2020) confirming the suitability of the material for the intended applications in this study. Given its high iron content and low carbon concentration, mild steel is expected to exhibit predictable corrosion behaviour which is important for evaluating the effectiveness of inhibitors in preventing corrosion in acidic environments.

**Table 2.** Compositional analysis of mild steel

Element	Wt. (%)	Element	Wt. (%)
C	0.2100	V	0.0005
Si	0.0010	W	0.0050
Mn	0.1020	Pb	0.0010
P	0.0221	Sn	0.0052
S	0.0052	As	0.0047
Cr	0.0342	Zr	0.0010
Mo	0.0010	Bi	0.0010
Ni	0.0014	Ca	0.0041
Al	0.0134	Ce	0.0010
Co	0.0159	Sb	0.0010
Cu	0.0143	Se	0.00219
Nb	0.0010	Te	0.0024
Ti	0.0002	Ta	0.0070
B	0.00096	La	0.00030
Zn	0.00099	Fe	99.5400

#### B. Gravimetric Analysis: Weight Loss Measurement

The results of the gravimetric analysis, presented in Fig. 3, show that the weight loss of mild steel exposed to a nitric acid (HNO<sub>3</sub>) environment increases over time, with values ranging from 0.025 g at a 20 µg/ml concentration of gold nanoparticles (AuNp) after 24 hours to 0.35 g in the control sample (with no AuNp) after 120 hours of immersion. This data suggests that mild steel undergoes more corrosion with longer exposure to the acidic solution. However, the presence of gold nanoparticles significantly

reduces the corrosion rate. As the concentration of AuNp increases from 5 to 20 µg/ml, the weight loss decreases, indicating that these nanoparticles play a protective role against corrosion in the nitric acid environment. The trend observed in Fig. 3a further supports this, showing that the weight loss of mild steel increases with time, a typical behaviour when metals are exposed to corrosive environments. As the steel interacts with the nitric acid solution, it undergoes oxidation, with iron ions (Fe<sup>2+</sup> and Fe<sup>3+</sup>) being released into the solution, leading to a loss of mass from the surface. This behaviour aligns with the fundamental principles of corrosion, where the metal dissolves over time due to exposure to an aggressive chemical medium like HNO<sub>3</sub>.

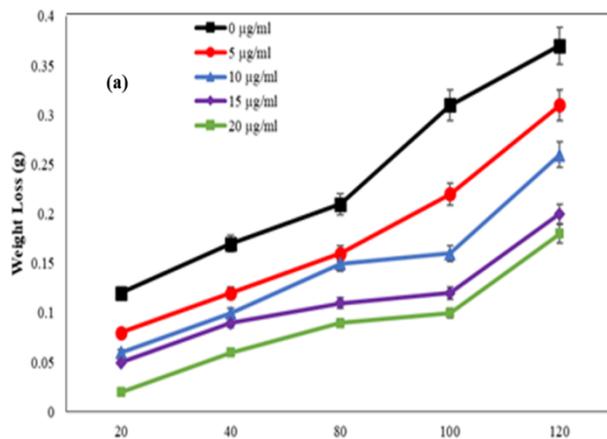
The corrosion rates were consistently higher in the control sample (0 µg/ml AuNp) compared to the samples containing gold nanoparticles as shown in Fig. 3b. The highest corrosion rate was recorded after 20 hours of immersion, with the control sample showing a corrosion rate of 0.10 g/h, whereas the 20 µg/ml AuNp sample exhibited a significantly lower corrosion rate of 0.02 g/h. This indicates that the presence of AuNp substantially slows down the corrosion process, with higher concentrations of nanoparticles leading to even lower corrosion rates. This decrease in corrosion rate over time, as shown in the data, is consistent with the findings of previous studies, such as those by Strebl *et al.* (2020), which suggest that corrosion rates tend to decrease as exposure time increases. This phenomenon is often attributed to the formation of a protective oxide layer on the metal surface, which reduces the metal's susceptibility to further corrosion. In this study, the gradual build-up of such a protective layer is likely enhanced by the gold nanoparticles, which interact with the metal surface and form a protective barrier.

The formation of this passivation layer is particularly important in the context of nitric acid solutions, which contain NO<sub>3</sub><sup>-</sup> ions. These ions contribute to the development of a stable oxide layer on the steel surface, which reduces further oxidation and protects the underlying metal. According to Al-Moubaraki & Al-Rushud (2018), the presence of NO<sub>3</sub><sup>-</sup> ions is critical in this process, as they help to stabilize the protective layer, particularly in acidic environments like the one used in this study. As the corrosion progresses, the concentration of the metal ions in the solution increases, and this oxide layer becomes thicker, ultimately slowing down the corrosion rate. This behaviour is particularly noticeable in Fig. 3a, where weight loss increases with time, but the rate of corrosion decreases as the passivation layer strengthens. One of the most significant findings of the study is the role of gold nanoparticles in reducing corrosion rates. Gold is a noble metal, meaning it is highly resistant to oxidation and corrosion. When gold nanoparticles are introduced into the system, they can adsorb onto the steel surface and form a protective layer, which prevents further corrosion. This passivation effect is particularly pronounced when smaller gold nanoparticles are used. Odusote *et al.* (2024) showed that gold nanoparticles are effective in reducing the corrosion of metals by forming

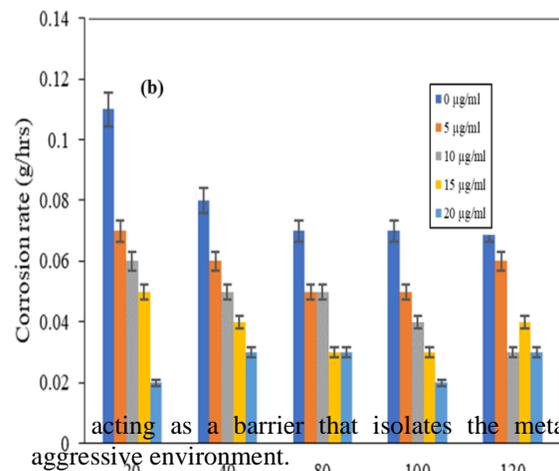
a barrier that inhibits the interaction of corrosive ions with the metal surface.

Furthermore, research by Jovanovic *et al.* (2018) supports the idea that gold nanoparticles prevent surface oxidation due to gold's inherent corrosion resistance. The passivation effect is more pronounced as the particle size of the gold nanoparticles decreases, as smaller nanoparticles have a higher surface area relative to their volume. This allows for a greater number of nanoparticles to interact with the steel surface, creating a more robust protective layer. In this study, as the concentration of AuNp increased, the corrosion rate continued to decrease, further supporting the idea that gold nanoparticles provide an effective means of corrosion inhibition. In summary, the results of this study provide compelling evidence that gold nanoparticles significantly reduce the corrosion of mild steel in an  $\text{HNO}_3$

environment. The corrosion rates were consistently lower in samples containing AuNp compared to the control sample, with the 20  $\mu\text{g/ml}$  concentration showing the most pronounced protective effect. This behaviour can be attributed to the formation of a passivation layer on the steel surface, facilitated by the presence of gold nanoparticles, which inhibit the corrosion process by preventing direct contact between the steel surface and the corrosive nitric acid. The findings align with previous research on the role of gold nanoparticles in corrosion inhibition and suggest that these nanoparticles may be a promising solution for enhancing the corrosion resistance of metals in acidic environments (Odusote *et al.*, 2024).



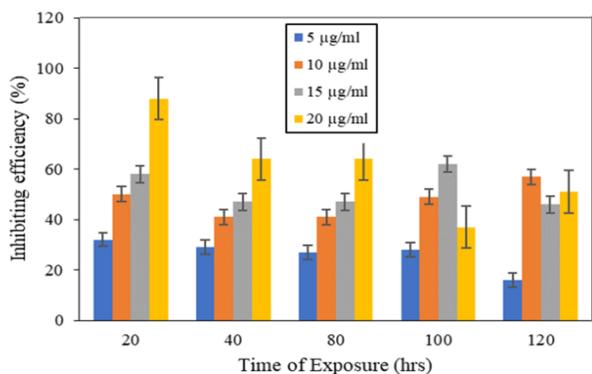
**Fig. 3: Gravimetric Analysis results for mild steel sample (a) weight loss (b) corrosion rate**



acting as a barrier that isolates the metal from the aggressive environment.

Fig. 4 illustrates the inhibition efficiency of the various samples in the study. The inhibition efficiency ranged from 10% for the control sample at 120 hours of immersion to 90% for the 20  $\mu\text{g/ml}$  AuNp sample after 20 hours of exposure. This trend indicates that the presence of gold nanoparticles significantly enhances the corrosion resistance of mild steel in an  $\text{HNO}_3$  environment. However, the inhibition efficiency was observed to decrease with an increase in immersion time, while it improved with an increase in AuNp concentration. These results agree with the findings of Alamiery *et al.* (2021), who reported that the efficiency of inhibitors in acid environments typically decreases over time but increases with the amount of inhibitor present. In this case, as the concentration of gold nanoparticles increased, the inhibition efficiency improved due to the enhanced barrier effect provided by the gold particles. The gold nanoparticles, when absorbed onto the metal surface, form a protective coating that prevents direct contact between the mild steel and the corrosive nitric acid, thus reducing the corrosion rate. This mechanism of action is consistent with the work of Al-Baghdadi *et al.* (2021), who demonstrated that gold particles could effectively create a protective layer on the surface,

However, an interesting observation emerged when the exposure time increased to 100 and 120 hours. At these extended time points, the inhibition efficiency did not follow the same trend, showing a decrease in performance at the highest concentration of the AuNp. This suggests that although the gold nanoparticles initially provide significant corrosion resistance, their effectiveness begins to diminish over time. This could be attributed to the possibility of the protective layer formed by the nanoparticles either becoming degraded or overwhelmed by prolonged exposure to the acidic environment. Given these observations, the study suggests the need for parametric optimization to determine the optimal immersion time and AuNp concentration to achieve the best corrosion inhibition efficiency for mild steel. This is similar to the conclusions of Raghavendra *et al.* (2022) and Morales-Gil *et al.* (2014), who emphasized the importance of optimizing the concentration and exposure time for corrosion inhibitors to achieve maximum protection. It is likely that beyond a certain threshold, the nanoparticle layer may reach a saturation point or be consumed by the corrosive environment, leading to a reduction in inhibition efficiency. Overall, the results highlight the potential of gold nanoparticles as effective corrosion inhibitors, but they also emphasize the need for careful optimization of both concentration and exposure time to ensure sustained effectiveness over longer immersion periods.



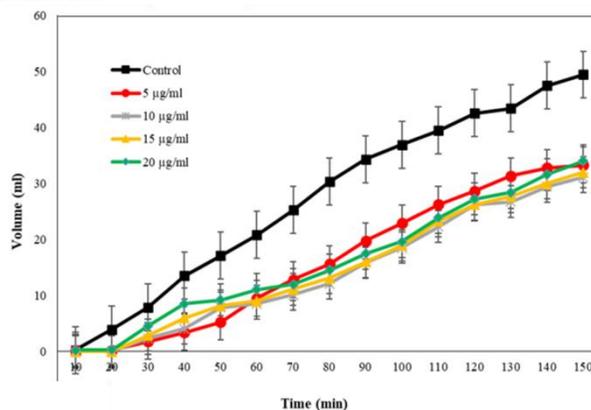
**Figure. 4: Inhibiting efficiency of mild steel in the HNO<sub>3</sub> environment**

### C. Gasometric corrosion measurement

Fig. 5 presents the rate of hydrogen gas evolution for the various samples over time. As expected, the volume of hydrogen gas evolved increased with the immersion time of the mild steel in the HNO<sub>3</sub> solution. The volume of hydrogen gas evolved ranged from 0 ml (for the 20 µg/ml AuNp sample) to 49.3 ml in the control sample, indicating a significant difference in the corrosion behaviour between the coated and uncoated samples. The highest rates of hydrogen gas evolution were observed in the control sample (without AuNp), which exhibited a rapid release of gas. Notably, it took about 20 minutes for the control sample to start releasing a noticeable volume of hydrogen gas. In contrast, the samples containing AuNp took about 30 minutes to exhibit noticeable hydrogen gas evolution. This delay in hydrogen gas evolution for the samples with AuNp suggests that the presence of gold nanoparticles effectively delays the onset of corrosion. This finding is consistent with the study by The *et al.* (2000), which reported that the presence of certain inhibitors, such as metal nanoparticles, can delay the initiation of the corrosion process by providing a protective layer on the metal surface.

The gold nanoparticles act as an impermeable and impenetrable barrier on the surface of the mild steel. This coating prevents the aggressive interaction between the metal surface and the corrosive nitric acid solution, thereby inhibiting the corrosion process and delaying hydrogen gas evolution. The formation of such a protective layer has been well-documented in previous studies, such as that of Sharma *et al.* (2022), who highlighted the ability of nanoparticle coatings to prevent the initiation of corrosion by physically isolating the metal from its corrosive environment. Additionally, the study revealed a reduction in the rate of hydrogen gas evolution with an increase in the volume of AuNp. This suggests that as the concentration of AuNp increased, the protective effect also intensified, resulting in a lower rate of hydrogen gas production. This supports the hypothesis that gold nanoparticles act as effective corrosion inhibitors. As the concentration of AuNp increased from 5 to 20 µg/ml, the inhibition efficiency improved, as the nanoparticles formed thicker and more effective protective layers on the metal surface.

The ability of AuNp to reduce hydrogen gas evolution and inhibit corrosion can be attributed to the protective layers formed by the gold nanoparticles. These layers, often created through emulsion paints mixed with AuNp, act as a partition between the steel surface and the corrosive HNO<sub>3</sub> solution. By preventing direct contact between the metal and the corrosive medium, these layers effectively impede the aggressive attack of the nitric acid on the steel surface. This mechanism of protection is consistent with the findings of Gawad *et al.* (2021), who reported that such nanoparticle coatings significantly reduced the corrosion rates of metals exposed to acidic environments. In conclusion, the results from the hydrogen gas evolution study confirm that gold nanoparticles are highly effective in delaying the onset of corrosion and reducing the rate of hydrogen gas evolution in mild steel exposed to HNO<sub>3</sub>. The protective layers formed by the nanoparticles act as barriers, preventing the aggressive attack of the acid and reducing the metal's exposure to corrosion. These findings further affirm the potential of AuNp coatings as suitable inhibitors for corrosion control in harsh acidic environments.



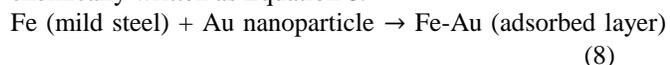
**Fig. 5: Gasometric measurement of hydrogen gas evolution in various concentrations**

### D. Mechanism of Corrosion Inhibition of AuNPs on the substrate

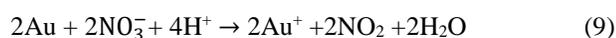
The mechanism of AuNp's corrosion inhibition on the mild steel sample in the HNO<sub>3</sub> acidic medium is a complete chemical process. The inhibitive film which contains Fe<sup>2+</sup> cation complex compounds and the corrosion inhibitor (AuNp) used lowered the rate of corrosion. This is an oxidative process for the corrosion inhibition. Furthermore, the development of products of high molecular polymeric structure on the substrate's surface as well as in the corrosive medium led to the blockage of oxygen which hampers further degradation of the substrate. The AuNp adsorbs on the substrate to block the active sites that could lead to corrosion. The corrosion inhibition of the mild steel substrate in the corrosive environment can also be attributed to the adsorption of the AuNp on the substrate's surface, resulting in a negatively charged anion from the HNO<sub>3</sub> solution. The AuNp catalyzes the NO<sub>3</sub><sup>-</sup> ions reduction, thereby reducing the corrosive tendency of the acidic environment. Through

oxygen heteroatom and  $\pi$ - $\pi$  interactions, this phenomenon could lead to the AuNp forming a strong bond with the metal surface. As a result, the formation of a coordinate bond between the corrosion inhibitor (AuNp) and the mild steel (substrate) leads to the inhibition of corrosion on the metal's surface. This corroborates the mechanism highlighted by previous studies (Ikubanni *et al.*, 2023; Vorobyova *et al.*, 2023).

These mechanisms could be represented with the chemical equations as shown in Equations 8 – 11. The adsorption of Au nanoparticles on mild steel surfaces could be chemically written as Equation 8.



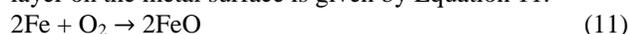
The reaction of the  $\text{NO}_3^-$  ions reduction by the corrosion inhibitor (AuNp) could be expressed as Equation 9.



The overall oxidation of Fe by  $\text{HNO}_3$  is given as Equation 10.



The reaction for the formation of protective oxide (film) layer on the metal surface is given by Equation 11.



The developed FeO is the protective oxide (film) layer that inhibits further disintegration of the metal surface in the acidic solution.

Hence, with the adsorbed layer of Fe-Au in the  $\text{HNO}_3$  solution, corrosion is inhibited depending on the concentration of the inhibitor in the corrosive medium. As earlier stated, the higher the concentration of the inhibitor, the lower the corrosion rate.

#### E. Statistical Analysis

The impact of coating mild steel with gold nanoparticles embedded in gloss paint was evaluated statistically using Analysis of Variance (ANOVA). The data collected from the gasometric analysis was used for this statistical evaluation. The results of the analysis are presented in Table 3 and Table 4. Table 3 indicates that the overall average time for hydrogen gas evolution was 80 minutes across all samples. Specifically, the control sample (without any gold nanoparticles) exhibited the longest gas evolution time, with an average of 27.34 minutes. However, as the concentration of gold nanoparticles in the gloss paint increased, the time taken for gas evolution significantly decreased. At a concentration of 10  $\mu\text{g/ml}$ , the gas evolution time dropped to 14.31 minutes. This suggests that the presence of gold nanoparticles accelerates the corrosion process to some extent compared to the control sample, likely due to changes in the interaction dynamics between the steel surface and the acidic environment.

A further increase in the volume of gold nanoparticles led to an increase in the hydrogen gas evolution time. This pattern suggests that while a moderate concentration of gold nanoparticles reduces the time for gas evolution, higher

concentrations may improve the protective properties of the coating, possibly due to the formation of a more effective barrier on the steel surface, which slows down the corrosion process. Table 4 presents the results of the ANOVA test, where the F-value of 21.68508 was calculated. This F-value is greater than the critical F-value of 2.323126. This statistical comparison demonstrates that the addition of gold nanoparticles to the gloss paint has a significant impact on the corrosion resistance of mild steel. The high F-value indicates that the variation in the gas evolution times is not due to random chance but rather is a result of the influence of gold nanoparticle concentration on the corrosion behaviour of mild steel. This finding is in agreement with the conclusions of Basker *et al.* (2016), who also found that nanoparticle coatings significantly enhance the corrosion resistance of metals. The statistical analysis confirms that incorporating gold nanoparticles into gloss paint significantly alters the corrosion dynamics of mild steel. This demonstrates the potential of gold nanoparticles as an effective means to enhance the durability and resistance of metals in corrosive environments.

**Table 3.** Statistical Summary of the Analysis

Groups	Count	Sum	Average	Variance
<b>Time (min)</b>	15	1200	80	2000
<b>Control</b>	15	410.1	27.34	261.9726
<b>5 <math>\mu\text{g/ml}</math></b>	15	242	16.13333	156.1024
<b>10 <math>\mu\text{g/ml}</math></b>	15	214.6	14.30667	117.265
<b>15 <math>\mu\text{g/ml}</math></b>	15	226.2	15.08	118.2574
<b>20 <math>\mu\text{g/ml}</math></b>	15	240.4	16.02667	121.2364

**Table 4.** ANOVA statistics

Source of Variation	SS	df	MS	F	P-value	F crit
<b>Between Groups</b>	50143.75	5	10028.8	21.69	7.063E-14	2.32
<b>Within Groups</b>	38847.67	84	462.5			
<b>Total</b>	88991.42	89				

*Df-Degree of freedom; SS-Sum of squares; MS-Mean squares; F-Fischer.*

## IV. CONCLUSION

This study investigated the effect of gold nanoparticles (AuNp) embedded in gloss paint on the corrosion inhibition efficiency of mild steel in an HNO<sub>3</sub> solution. Chemical composition analysis of the steel revealed that it contained 99.7 wt% Fe, 0.2100 wt% C, and other trace elements, confirming the sample as mild steel. Gravimetric (weight-loss) analysis demonstrated that the mild steel underwent significant corrosion in the HNO<sub>3</sub> solution, but the corrosion decreased as the concentration of AuNp increased. This indicates that gold nanoparticles are effective inhibitors for mild steel in acidic environments, with higher concentrations providing better corrosion resistance. Both gravimetric and gasometric analyses confirmed the effectiveness of gold nanoparticles in inhibiting corrosion. The corrosion rate decreased due to the increased inhibition efficiency, while the volume of hydrogen gas evolved was reduced. However, corrosion resistance improved at certain concentrations of AuNp (5 and 10 µg/ml), while it decreased at higher concentrations (15 and 20 µg/ml), suggesting the need for process optimization to determine the optimal concentration. ANOVA analysis of the gasometric data showed that incorporating gold nanoparticles into gloss paint had a significant effect on the corrosion resistance of mild steel in the HNO<sub>3</sub> solution, further confirming the potential of AuNp coatings as effective corrosion inhibitors

#### AUTHOR CONTRIBUTIONS

**A.A. Adeleke:** conceptualization, methodology, analysis, original draft, and revision of the manuscript. **J.K. Odusote:** Methodology, analysis, manuscript writing – original draft. **M.S. Lawal:** Methodology, analysis, manuscript writing – original draft. **P.P. Ikubanni:** Analysis, manuscript writing – review and editing. **T.A. Orhadahwe:** analysis, manuscript writing – original draft and revision. **T. Ogedengbe:** manuscript writing – original draft and revision. **N.E. Alubankudi:** manuscript writing – original draft and revision

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