

Synthesis and Characterization of Al₂O₃ Nanoparticles Using the Co-precipitation Method as Corrosion Inhibitor

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Abstract

Nanoparticle technology is a promising one for its unique properties, which can be utilised for different applications, for instance, as a corrosion inhibitor. This study investigates the corrosion-inhibition behaviour of Al₂O₃ nanoparticles synthesised by the Co-precipitation method. In this study, 800 ppm Al₂O₃ NPs are used as an inhibitor in a 1M H₂SO₄ solution for the Mild Steel (MS) sample at different times and temperatures. Experimental result shows that Corrosion inhibition efficiency is highest with 59.59% at 30°C after 2 hours, and lowest with 8.22% at 60°C after 12 hours. Corrosion efficiency decreases gradually with time and temperature. Characterisation of Al₂O₃ NP was done by SEM analysis, and the presence of Al₂O₃ NPs on the MS sheet surface was determined by FTIR analysis.

Keywords

Nanoparticles, Co-precipitation, Corrosion, Mild Steel, FTIR, Nanoparticle.

1. Introduction

Nanoparticles (NPs) are extremely small particles, basically ranging from 1 to 100 nm in diameter instead of small size, capturing a very large surface area relative to volume and exhibiting unique chemical, physical and mechanical properties compared to bulk counterparts (Swathy et al., 2025a). Their nano-size enables them to interact more efficiently with other materials and can modify the microstructure of host matrices that can lead to enhanced performance in a variety of applications, including catalysis, electronics and material protection (Yadav et al., 2024a)(Deyab, El-Shamy, et al., 2024a). To synthesize NPs coprecipitation method was used. The coprecipitation method basically involves simple operations, good reproducibility and easy-to-control experimental conditions such as pH, temperature and concentration, which are very necessary parameters to control NPs size, shape, and crystallinity (Mirza et al., 2024)(Mim et al., 2023). Besides, coprecipitation demonstrates high product purity with minimal or no use of organic solvents, reducing contamination risks and environmental impact. Overall, it combines operational simplicity, process control, cost efficiency and eco-friendly behavior, making it a preferred technique for synthesizing Al₂O₃ NPs for corrosion protection and similar applications.

For corrosion resistance, Al₂O₃ NPs play a crucial role by serving both as physical barriers and active agents in coatings or solutions(Yadav et al., 2024b). It can block corrosive reactants such as water, oxygen, chloride ions, sulphate ions and create protective layers, release corrosion-inhibiting ions that form stable, adherent films on metal surfaces (Deyab, El-Shamy, et al., 2024b). The strong surface reactivity of nanoparticles also allows for the generation of chemical passivation at the interface, which slows corrosion rates and increases long-term durability of metals exposed to harsh environments(Deyab, Alghamdi, et al., 2024; Deyab, El-Shamy, et al., 2024b). Despite significant advances in the use of Al₂O₃ nanoparticles for corrosion inhibition, there remain notable gaps in both scientific

understanding and practical deployment. Current literature underscores that while coprecipitation offers a controllable and green route for Al₂O₃ nanoparticle synthesis, the molecular details of its interaction at steel/acid interfaces, particularly under varying operational conditions such as dynamic pH, elevated temperature and fluctuating ion concentrations, are still not fully evaluated (Devi & Ghanbusi, 2024; Deyab, El-Shamy, et al., 2024b; Oreko & Okuma, 2025). The stability, dispersion uniformity, and size-dependent activity of nanoparticles in corrosive media present unresolved challenges, as many studies report a progressive decrease in inhibition efficiency over extended exposure, likely due to nanoparticle aggregation or detachment from the surface (Farahmandjou & Golabiyan, 2019; Goswami et al., 2025). Additionally, the long-term effectiveness and regeneration of Al₂O₃ NPs after multiple corrosion cycles in concentrated acid environments have not been systematically addressed, raising questions about their real-world sustainability and eco-toxicity (El-Hamid et al., 2024; Garba et al., 2025). There is also a lack of comparative data examining coprecipitation-derived Al₂O₃ nanoparticles against other synthesis routes regarding inhibition performances, film stability, and practical cost-effectiveness. The present research aims to fill these gaps by synthesising Al₂O₃ nanoparticles via the coprecipitation method and exploring their corrosion-inhibiting behaviour in mild steel exposed to H₂SO₄ solutions, with a focus on optimising nanoparticle dosage, size control, and exposure duration for maximum protection. Advanced characterisation techniques, including SEM and FTIR, will be employed to evaluate the nano-structural and interfacial mechanisms underlying corrosion resistance, while quantitative tests will determine inhibition efficiency, corrosion rate, and mass loss reduction across varying concentrations and experimental periods (Al-Senani & Al-Saedi, 2022; Basher & Ali, 2022; Busi & Rajkumari, 2019; Xu et al., 2011). By addressing the highlighted barriers, this study aims to establish clear guidelines for the practical application of coprecipitation-synthesised Al₂O₃ nanoparticles as eco-friendly, robust, and scalable corrosion inhibitors, contributing essential insights for future industrial uptake and environmental safety.

1.1 Objectives

The main objectives of this study are:

- a. To produce fine-sized Al₂O₃ nanoparticles by the co-precipitation method.
- b. Examine the performance of Al₂O₃ nanoparticle powder as a corrosion inhibitor.

2. Literature Review

(Saadouni et al., n.d.) focuses on Sulfathiazole (STZ), which inhibits mild steel corrosion in 1M HCl by adsorbing on the surface and forming a mixed-type protective film, blocking both anodic and cathodic sites. Its inhibition efficiency reaches almost 94% at optimal concentration, based on experimental and theoretical studies, with adsorption involving physical and chemical interactions following physical and chemical interactions following Langmuir isotherm behaviour (Fouda et al., 2021) synthesized ZnO nanoparticles from *Convolvulus arvensis* leaf extract act as highly efficient, green corrosion inhibitors for carbon steel in 1M HCl, providing over 91% inhibition at room temperature. Their performance is due to adsorption and protective film formation, which blocks both anodic and cathodic reactions. Efficiency increases with higher nanoparticle concentration and is maintained over extended immersion periods, following the Langmuir adsorption isotherm.

(Alao et al., 2022) review that nanoparticles improve corrosion resistance by forming protective films on metal surfaces, reducing corrosion rates through adsorption mechanisms, and acting as effective, eco-friendly inhibitors in various acidic and saline environments. High inhibition efficiencies (80–95%) are achieved with metals like mild steel, aluminium, and copper, especially when using green-synthesized or plant-extract-based nanoparticles. Their small size and large surface area facilitate strong adsorption, making them cost-effective and sustainable alternatives to traditional inhibitors. Metal and metal oxide nanoparticles, such as Al₂O₃, ZnO, TiO₂, and SiO₂, significantly enhance corrosion resistance when incorporated into coatings by improving barrier properties, providing self-healing, and boosting photodegradation resistance. Anjum (Anjum et al., 2020) show that their effectiveness depends on dispersion, concentration, and interaction with the coating matrix, making them promising for advanced anti-corrosion applications in harsh environments. However, optimal performance requires careful control of nanoparticle dispersibility and loading levels. Clinopodium acinos extract has been shown in (Al-Senani, 2020) to effectively inhibit mild steel corrosion in 1M HCl by forming a protective adsorbed layer, increasing efficiency with higher concentrations and lower temperatures. The inhibitor acts as a mixed type, blocking both anodic and cathodic reactions, and its adsorption follows the Langmuir isotherm. This eco-friendly plant-based inhibitor offers a low-cost and sustainable alternative to conventional inhibitors in acidic environments. (Swathy et al., 2025b) this review discusses the use of nanomaterials as sustainable corrosion inhibitors, highlighting their high efficiency, eco-friendly synthesis, and superior protection compared to traditional inhibitors. Green synthesis using plant extracts enhances environmental safety while nanoparticles provide improved adsorption and barrier properties on metal surfaces.

Combining nanotechnology with green chemistry offers promising, effective, and environmentally friendly corrosion control solutions for the future. Alamiery (Alamiery, 2021) investigates 4-ethyl-1-4-oxo-4-phenylbutanoylthiosemicarbazide (EOPT) as a corrosion inhibitor for mild steel in 0.5 M H₂SO₄. EOPT shows up to 88.7% inhibition efficiency by adsorbing on the steel surface via mixed chemisorption and physisorption, following Langmuir isotherm behaviour. Efficiency increases with concentration and immersion time up to 5 hours, reducing corrosion rates significantly. The corrosion inhibition study (Samaila et al., 2024) shows that zinc oxide nanoparticles synthesized by green methods from local leaf extracts inhibit mild steel corrosion in an acidic medium. Smaller nanoparticles (~15.4 nm) have the highest inhibition efficiency (54%) due to better surface coverage, while larger particles show lower efficiency. The inhibition mechanism involves adsorption, forming a protective film on the steel surface, and reducing corrosion rates. Inhibition efficiency depends strongly on particle size, with smaller sizes providing greater surface coverage and better corrosion resistance. (Li et al., n.d.) show that the synthesized OPP compound exhibits excellent corrosion inhibition efficiency (up to 99.5%) on mild steel in mixed 1 M HCl and 0.5 M H₂SO₄ solution at 20°C, maintaining good stability up to 60°C. OPP acts as a mixed inhibitor with predominant anodic protection by adsorbing strongly on the steel surface via chemisorption, confirmed by electrochemical tests, surface analysis, and quantum chemical calculations. The inhibitor effectively reduces corrosion rates and surface damage while showing high reusability and stability at elevated temperatures. The ZnO-NiO-polyaniline (ZnNiOPANE) nanocomposite has been synthesized by (Al-Masoud et al., 2022) and characterized as an effective corrosion inhibitor for mild steel in acidic chloride media. Characterization by SEM, TEM, XRD, FTIR, UV-Vis, XPS, and BET surface area analyses confirmed a heterogeneous composite structure containing crystalline ZnO and NiO particles embedded in an amorphous polyaniline matrix. Electrochemical studies (OCP, PDP, EIS) showed that ZnNiOPANE acts as a mixed-type inhibitor, significantly reducing corrosion current density and increasing polarization resistance, with inhibition efficiency up to 97.89% at 200 mg/L concentration. The adsorption of ZnNiOPANE on the steel surface follows the Langmuir isotherm, involving physicochemical adsorption mechanisms. Density functional theory (DFT) and Monte Carlo simulations supported the experimental findings, showing strong adsorption energy and electron donation capacity of ZnNiOPANE, favouring stable protective layer formation on steel. This nanocomposite outperforms many other polymeric corrosion inhibitors for steel in acidic chloride environments, presenting a promising approach for corrosion protection. These studies collectively demonstrate the potential of green plant extracts and metal oxide nanoparticles as effective, sustainable corrosion inhibitors in acidic environments, providing alternatives to traditional toxic chemicals with high inhibition efficiencies.

In the present study, synthesizing and performance of fine-sized Al₂O₃ NPs as a corrosion inhibitor on MS sheet, by simply mixing with Sulfuric acid solution, is investigated.

3. Methods

3.1 Al₂O₃ Nanoparticle (NP) Synthesis Method

At first, Aluminium nitrate (Al (NO₃)₃.9H₂O) salt, Ammonia solution (NH₄OH, 25% w/w), Ethanol (C₂H₅OH), and distilled water were collected from a local Chemical shop and the Metallurgy lab of CUET. For Al₂O₃NP synthesis, initially, 0.1M, 300mL Aluminium nitrate solution, and 0.5M, 150mL Ammonia solution were prepared with distilled water. Then, the Aluminium nitrate solution was heated at 60°C for 15min. and continuously stirred with a magnetic stirrer for proper dissolving. The initial pH of this solution was 3, and the Ammonia solution was added dropwise by pipette till precipitation of Al(OH)₃ formed, and the pH reached 10. Then, the resulting solution is kept overnight for proper precipitation, and any excess water is removed. In the next step, the precipitation was washed two times with DI water and lastly with ethanol in a centrifuge machine. Finally, the washed precipitate was dried in an oven for 24 hours. at 90°C, calcinated in a muffle furnace for 3hr. at 600°C accordingly (Figure 1- Figure 6).

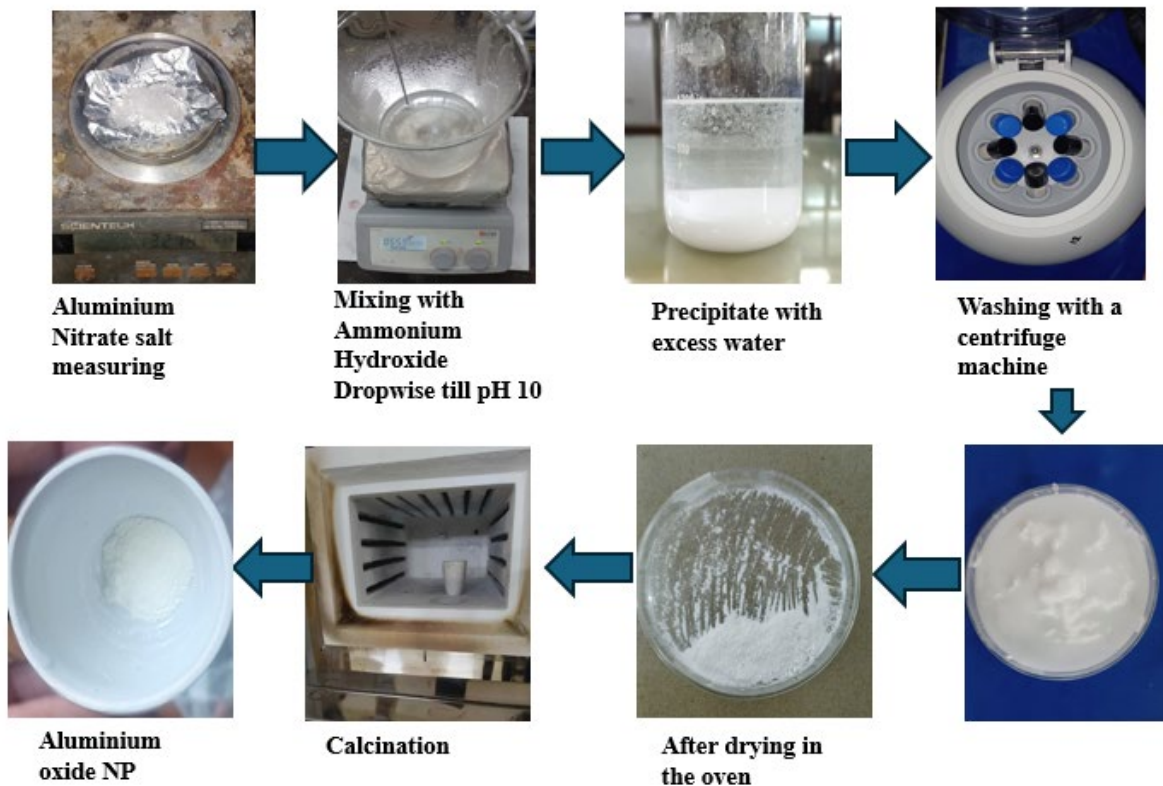


Figure 1. Step-by-step of the co-precipitation method for $\text{Al}_2\text{O}_3\text{NP}$ preparation

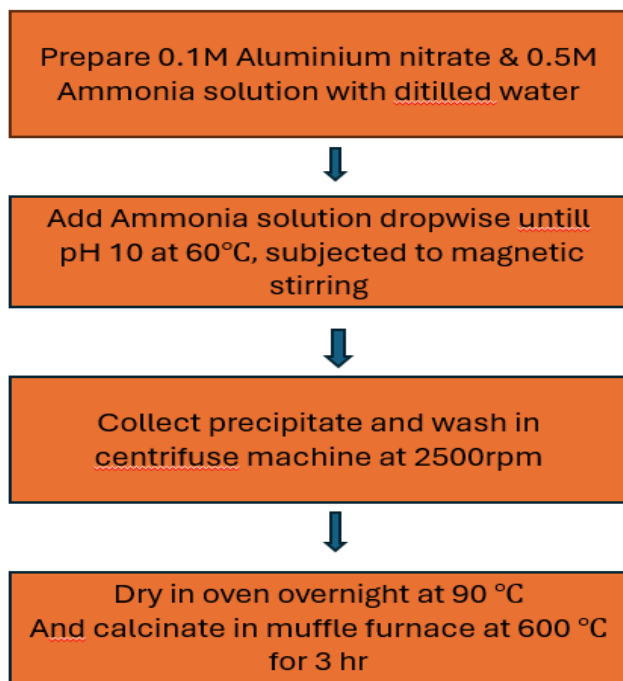


Figure 2. Flowchart of the co-precipitation method for $\text{Al}_2\text{O}_3\text{NP}$ preparation

3.2 Corrosion test

3.2.1 Material preparation

Mild steel samples are regularly cut into small pieces with dimensions $2 \times 2 \times 0.1 \text{ cm}^3$ were used for this investigation. Sulfuric acid (H_2SO_4) 98% annular grade was utilised to prepare the corrosive solution of 1M & 2M. The MS sheet samples were polished with a surface grinder, sandpaper, and washed with methanol and distilled water and weighed.

3.2.2 Weight-loss method

The test was conducted in 1M Sulfuric acid, 50mL solution for 30°C & 60°C , with (800ppm) and without $\text{Al}_2\text{O}_3\text{NP}$ addition. The MS samples were kept in solution for 2-12 hours. After exposure, the samples were dried and reweighed on a digital balance. The weight loss was evaluated by the sample weighting difference before and after exposure to the acidic solution.

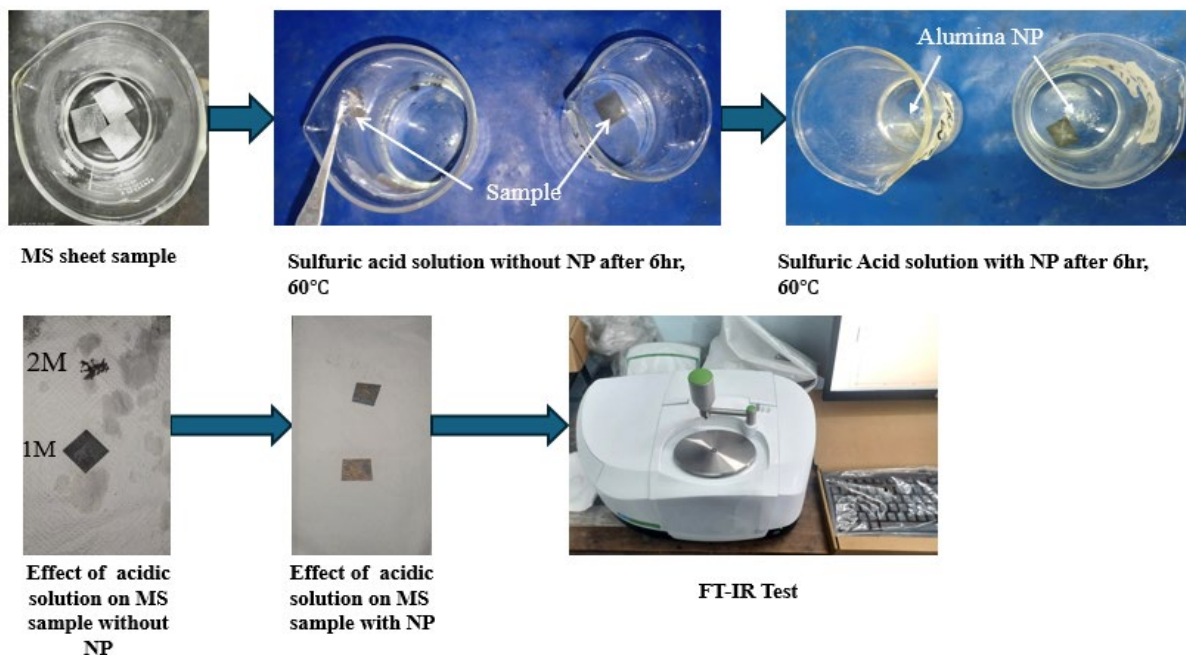


Figure 3. Corrosion Test

3.2.3 Characterization

The Al_2O_3 NPs obtained by the co-precipitation method were characterized using a scanning electron microscope (SEM) and Fourier transform infrared spectroscopy (FTIR). The morphological structure was examined using SEM, and the thin layer of Al_2O_3 nanoparticles formed on the MS steel sample was identified by FTIR.

4. Data Collection

For the corrosion test, the weight loss of the Mild steel sheet was observed at regular time intervals, both with and without using nanoparticle powder, and compared.

5. Results and Discussion

5.1 Numerical Results

To calculate Corrosion Rate (CR%), compare the weight loss without and with the inhibitor.

$$CR\% = \frac{\Delta W_{without} - \Delta W_{with}}{\Delta W_{without}} \times 100$$

Here, ΔW =weight loss

Table 1. Corrosion test scenario for MS Sheet in 1M Sulfuric Acid Solution

Temp.	30°C	60°C	30°C	60°C	Corrosion Reduced (%) At 30°C	Corrosion Reduced (%) At 60°C
Time → ↓	Weight Loss of MS without NP		Weight Loss of MS with NP			
2h	0.099 g	0.68 g	0.04 g	0.316 g	59.60	53.53
4h	0.171 g	1.18 g	0.087 g	0.688 g	49.12	41.69
6h	0.293 g	2.025 g	0.168 g	1.387 g	42.66	31.51
8h	0.465 g	3.03 g	0.277 g	2.431 g	40.43	19.77
10h	0.721 g	4.23 g	0.457 g	3.73 g	36.62	11.82
12h	1.006 g	5.35 g	0.683 g	4.91 g	32.11	8.22

5.2 Graphical Results

In Table 1 and Figure 4 results of corrosion rate with time from 2 hours to 12 hours and with varying temperature 30°C and 60°C are depicted. It is seen that the corrosion rate increases with time and temperature. However, using Al₂O₃ nanoparticles in an acid solution significantly reduces the corrosion rate. Another thing to note is that the relative corrosion rate (%) decreases with time at a specific temperature.

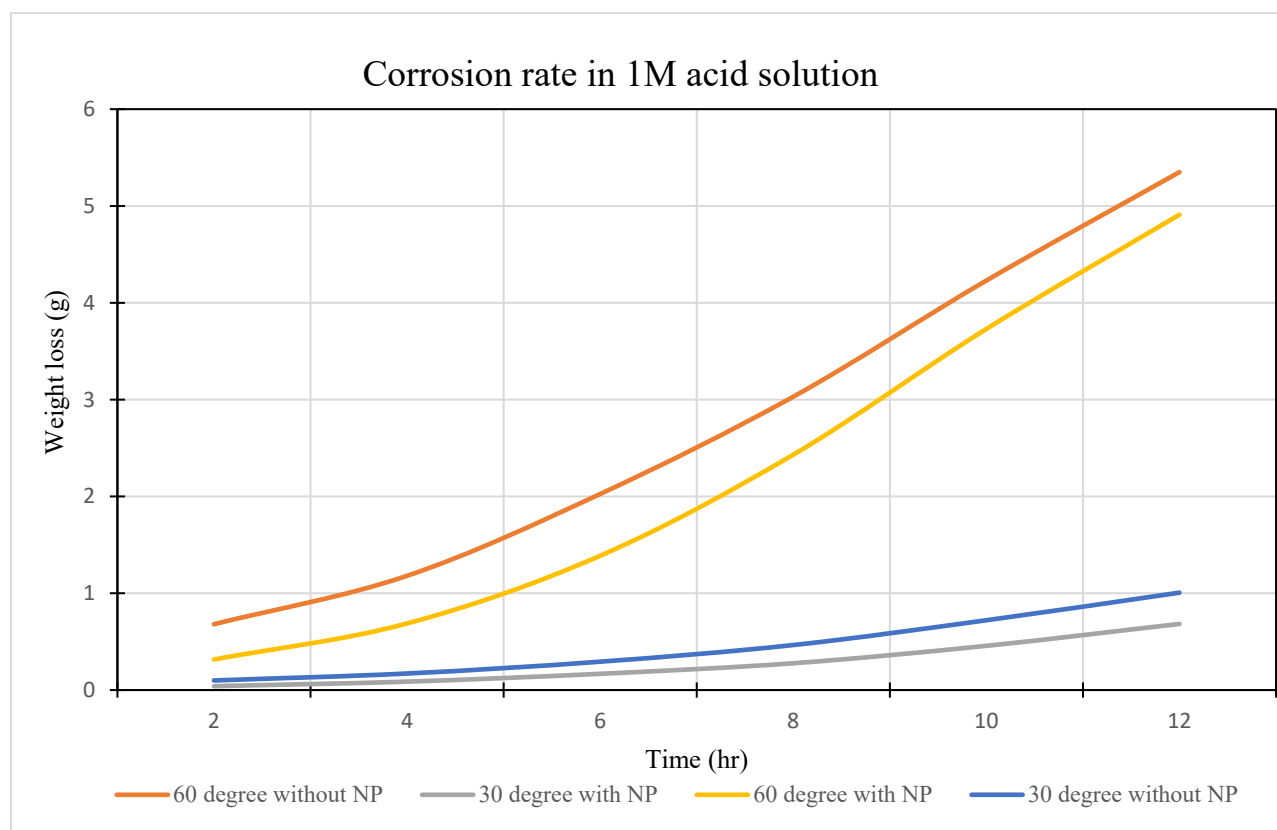


Figure 4. Graphical Representation of Corrosion Rate

5.2.1 Particle size and SEM analysis

The production of nanoparticles is depicted in Figure 5, which shows SEM images of aluminium oxide materials. SEM provides information about the material's surface structure, including size, shape, and morphology, which characterise its nanoscale morphology. It is evident from SEM images that these particles are uniformly distributed

across the surface and have spherical shapes; in topical top-view of SEM micrographs, aggregation of nanoparticles and the formation of nanoclusters have been observed. Increasing the molar concentration of nanoparticles can alter their size (Nabi et al., 2020). The Al₂O₃NPs size is 16-33nm, depicted in the SEM image.

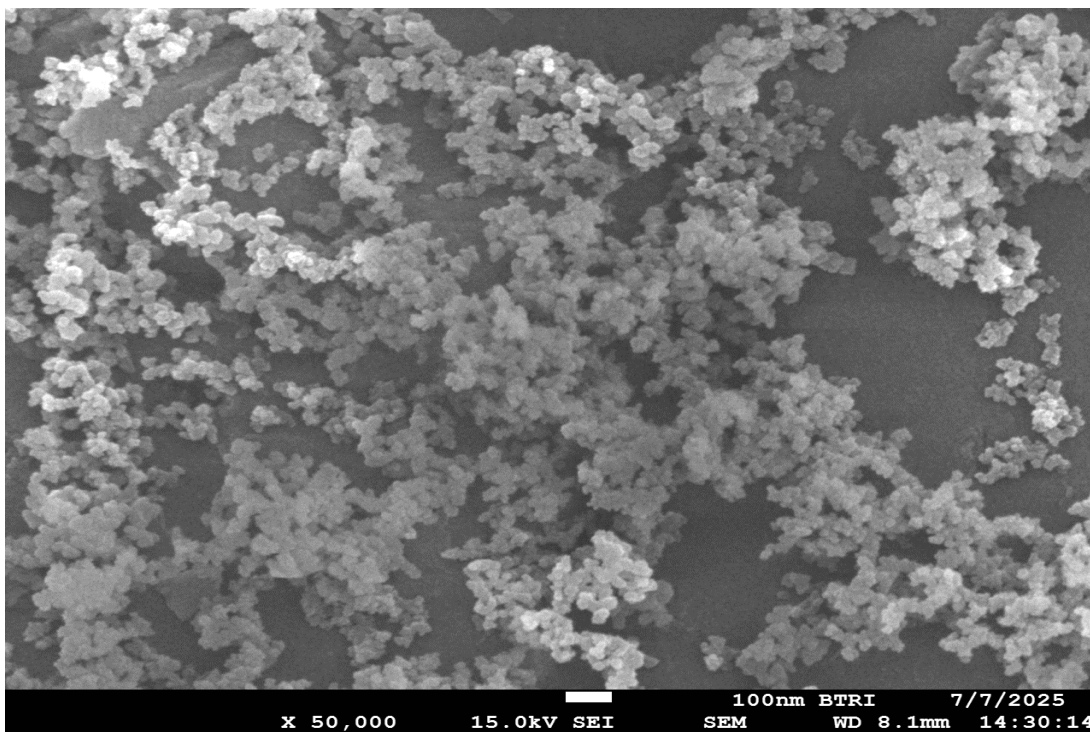


Figure 5. SEM image of Al₂O₃ Nanoparticles

5.2.2 FTIR Analysis

To evaluate the absorption of IR radiation by a sample, FT-IR spectroscopy is convenient. It contains the relation between absorption bands (vibrational bands) and the chemical substances in the sample (Rabu et al., 2020). The FT-IR spectrum analysis of aluminium oxide nanoparticles, formed as a corrosion inhibition layer on the MS sheet surface, is shown in Figure 6. The highest peaks obtained at 3295.27cm⁻¹ and 1069.81cm⁻¹ are related to O-H and C=O stretching. The possible cause of forming C=O bond is due to the reaction of ammonia from the precursor NH₄OH (Rabu et al., 2020). Also, peaks at 400-700 cm⁻¹ ensure the Al-O-Al bond for γ phase alumina.

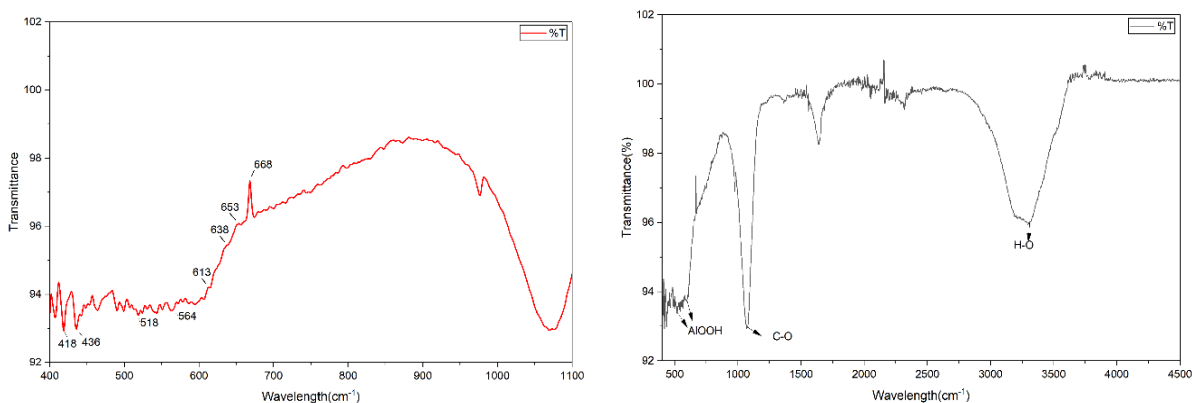


Figure 6. FT-IR spectrum of Al₂O₃ NPs

6. Conclusion

This article examined the effect of Aluminium oxide nanoparticles as a corrosion inhibitor of MS sheet. The synthesis of Al₂O₃ NP is done by the co-precipitation method, which is easy, cost-effective and suitable for mass production. The characterisation test SEM confirms the presence of Al₂O₃ nanoparticles and their size in nanoscales an average of 16-33 nm. The FTIR analysis ensures the presence of γ alumina nanoparticles on the MS sheet surface, which works as a corrosion inhibitor. 800ppm Al₂O₃ NPs mixed in Sulfuric acid solution reduces the metal corrosion rate considerably. Further investigation can be done by varying the nanoparticle and its amount, and at the mass level, to gain more efficiency.

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