Research Article

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Inhibition of Corrosion on Mild Steel by Green Synthesized Titanium Dioxide Thin Film

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ABSTRACT

In this work, TiO_2 nanoparticles were prepared from titanium tetra isopropoxide by conventional method and green synthesis using neem (*Azadirachta indica*) leaf extract. The green synthesized TiO_2 was coated on mild steel plates. The morphological features of thin films were characterized by SEM and EDS. The rate of corrosion was assessed by weight loss and salt spray methods in NaCl environment.

Keywords: Green synthesis; Neem leaves; Titanium dioxide; Weight loss method; Salt spray methods.

1. INTRODUCTION

Azadirachta indica (neem) has many phytochemicals (Hossain *et al*. 2013). Neem leaves have been used in the synthesis of nanoparticles. Magnesium oxide nanoparticles were synthesized from neem leaves and extensively characterized (Moorthy *et al*. 2015). Silver nanoparticles were prepared using a reducing agent found in an aqueous extract of neem leaves to reduce silver ions (Joany *et al*. 2015). Narenkumar *et al*. (2018) synthesized silver nanoparticles from $AgNO_3$ using neem leaf extract, which acted as a reducing as well as stabilizing agent. Parthipan *et al*. (2017) used a green inhibitor from neem for preventing corrosion of mild steel.

Green nanomaterial corrosion inhibitors offer a sustainable and environmentally friendly approach against steel corrosion (Alghamdi *et al*. 2023). Experiments on green synthesized nanomaterials and their potential as inhibitors for mild steel corrosion offer sustainable and eco-friendly solutions to combat corrosion related challenges (Sarmah *et al*. 2023). Microbiologically induced corrosion rate is higher while compared to an abiotic system. Several studies have been carried to evaluate the anti-corrosive property of green synthesized NPs. ZnO NPs synthesized using *Ocimum sanctum* were also reported to be effective against mild steel corrosion (Subhasree *et al*. 2020). Yadeta *et al*. (2024) conducted photocatalytic test under solar irradiation for comparing the performance of zinc oxide, copper oxide and nickel oxide nanoparticles synthesized using neem leaves. Zn and Mg plates were coated with green synthesized (*Delonix elata* leaf extract) nickel oxide NPs. The efficiency of the NiO NPs as anti-corrosive agent under the aqueous electrolyte solution was evaluated. The corrosion rate was found to be less in NiO NPs-coated plates when compared to uncoated plates (Sudha *et al*. 2021). Ramamoorthy *et al*. (2022) reported corrosion-resistant ZnO nanoparticles with better photocatalytic behavior. A hybrid epoxy coating material synthesized from ixora leaf showed exceptional corrosion resistance property towards steel in NaCl medium (Veedu *et al*. 2022). Hossain *et al*. (2022) reported arjuna leaf extract as a green corrosion inhibitor for mild steel in HCl.

Pawar *et al*. (2022) reported the green synthesis of metal oxide nanoparticles like calcium oxide, titanium dioxide, iron oxide, manganese oxide, chromic oxide, cobalt oxide, cupric oxide, molybdate oxide, and zinc oxide. They have a wide spectrum of applications.

ZnO NPs have been evaluated for their anti-bacterial and anti-proliferative properties (Beltagi *et al*. 2024). CuO NPs were shown to serve as supercapacitors and electrochemical devices (Ikhioya *et al*. 2024a). Aluminum oxide NPs have applications in optical, solar, and electrochemical storage devices (Ikhioya *et al*. 2024b).

In this paper, green synthesized titanium dioxide solution and pure titanium dioxide solution are prepared. Each solution is applied to the substrate as a nanocoating to study their corrosion inhibition property in mild steel.

2. MATERIAL PROCUREMENT AND PREPARATION

2.1 Sample Preparation

Mild steel was cut into pieces (20×20×3 mm) for testing (Fig. 1). The pieces were first polished with a

linisher polisher and then with sandpapers (400 to 600 grit). These samples were washed with ethanol and acetone to remove unwanted materials such as dirt, grease, rust, and mill scale. These specimens were finally washed with distilled water and kept in an ultrasonic heater maintained at 400°C for 30 minutes.



Fig. 1: Mild steel specimens

2.2 Green Synthesis

Neem leaves were cleaned with distilled water. Then 40 mL of ethanol was poured on to the leaves and heated by ultrasonication. $BaCl_2$ solution was introduced dropwise. About 40 mL of Na_2SO_4 was added dropwise after 10 minutes. The resultant green precipitate was separated using a Whatman filter paper. Neem solution preparation is represented in Fig. 2.



Fig. 2: Synthesis of pure neem solution

2.3 Synthesis of TiO_2 Solution

The TiO_2 solution was prepared by using titanium (IV) tetraisopropoxide (TTIP), ethanol and acetic acid (CH_3COOH). Titanium (IV) tetraisopropoxide was used as a source for Ti, while acetic acid and ethanol were used as solvents (Fig. 3). Ethanol and acetic acid were taken in a beaker and mixed well using a magnetic stirrer for 20 minutes. Then TTIP was added. TiO_2 thin films were prepared after allowing the solution to age. The composition of the precursor and solvents is represented in Table1.

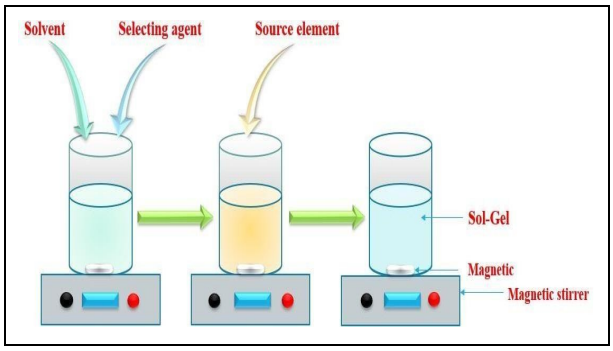


Fig. 3: Solgel composition

Table 1. Chemicals and its compositions

S. No.	Chemical	Chemical (mL)
1.	Ethanol (solvent)	7
2.	Acetic acid (solvent)	3
3.	Titanium tetra isopropoxide (Element source)	2

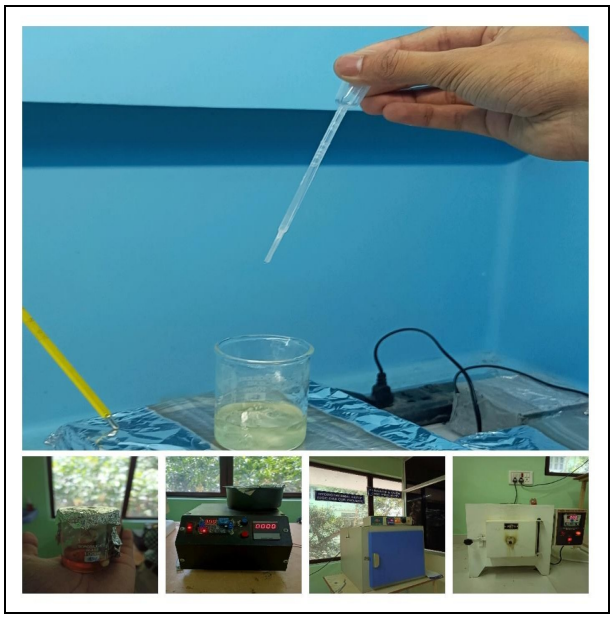


Fig. 4: Experimental approach

2.4 Green Synthesized Titanium Dioxide Solution

The green TiO_2 solution was prepared by mixing few drops of neem solution with titanium dioxide solution using a magnetic stirrer and coated on the substrate (mild steel plates).

3. SPIN COATING DEPOSITION PROCESS

Each nanoparticle solution was coated on mild steel substrates by using spin coaters.

Fig. 4 shows the schematic diagram of the preparation of thin films. Typically, a small volume of coating material was applied dropwise to the centre of the substrate. Using a spin coater, the substrate was rotated quickly to distribute the coating material *via* centrifugal force. The spin coater machine spindle was rotated at 3000 rpm in 30s for uniform nanocoating. Then, the coated steel substrates were annealed at 150°C for 1 hour in a muffle furnace. The coated specimens had better surface and uniformity. These coated samples were investigated by conducting morphological and corrosion studies.

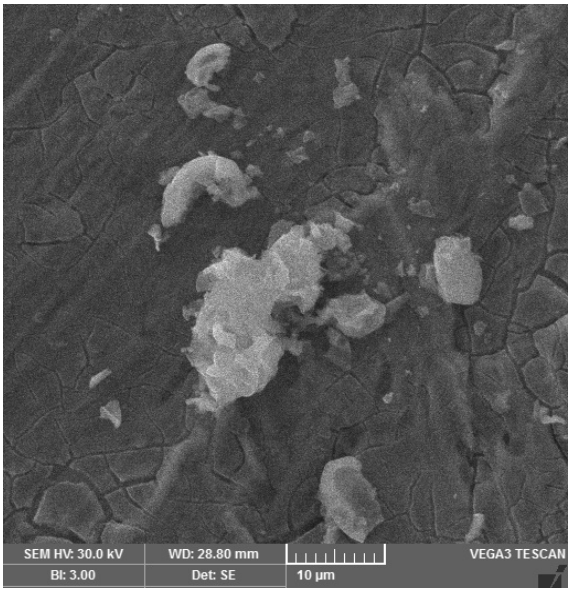


Fig. 5(a): Green synthesized titanium dioxide nano coating

4. RESULTS AND DISCUSSION

4.1 SEM

The SEM test was conducted on the coated mild steel substrates. Fig. 5 a and b show the image of substrate coated with green synthesized TiO_2 and chemically synthesized TiO_2. The smooth surface on the coated substrate suggested a high-quality nanocoating. No significant cracks or voids were observed.

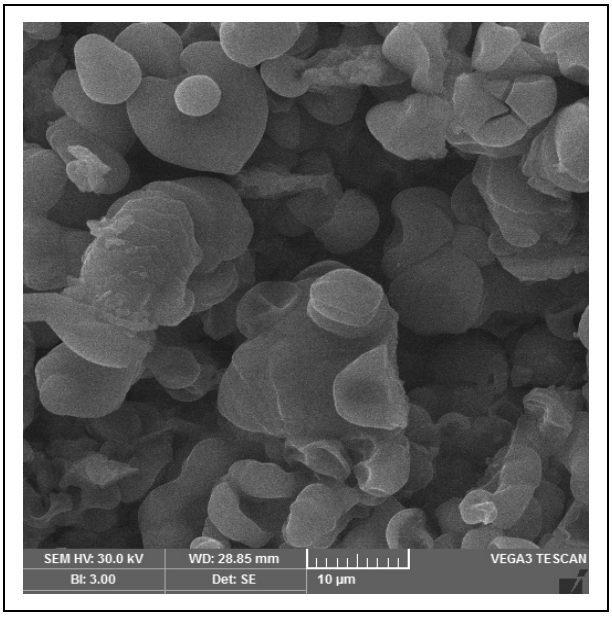


Fig. 5(b): Titanium dioxide nano coating

4.2 Energy Dispersive X-Ray Spectroscopy (EDS)

The elements in the nanocoated samples were identified and quantified using EDS (Fig. 6 a and b). In the case of green synthesized nanocoated substrate, Ti (titanium), O (oxygen), C (carbon), and iron (Fe) were present. Oxygen (O) is a significant peak due to the presence of oxygen compounds present in plants. The minor peaks were for calcium (Ca) and magnesium (Mg), which are essential elements present in all the plants.

Iron (Fe), titanium (Ti), aluminum (Al) and iron (Fe) elements are formed during synthesis depending on the methods and materials used. The titanium element signal is stronger in green synthesized coating when compared to conventional titanium dioxide coating.

4.3 Corrosion Test

Weight loss and salt spray techniques were used for this study.

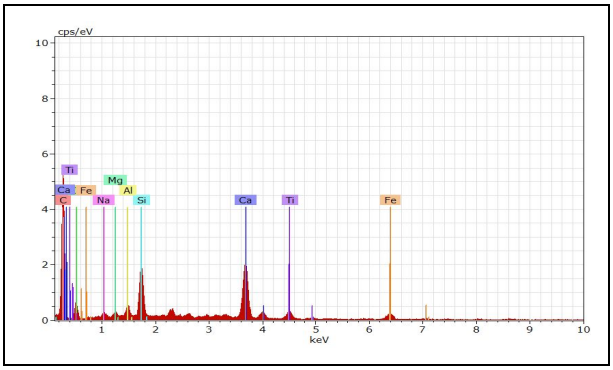


Fig. 6(a): EDS for green synthesized titanium dioxide nano coating

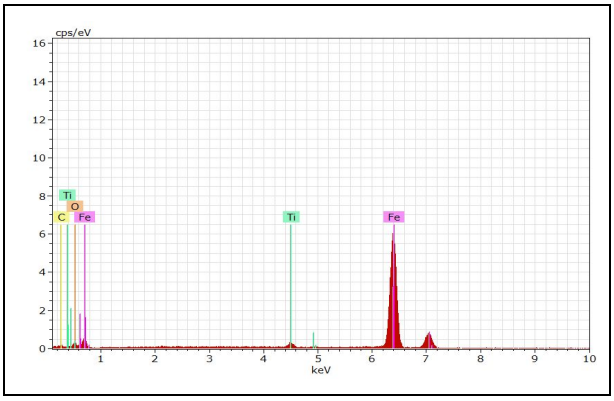


Fig. 6(b): EDS for titanium dioxide nano coating

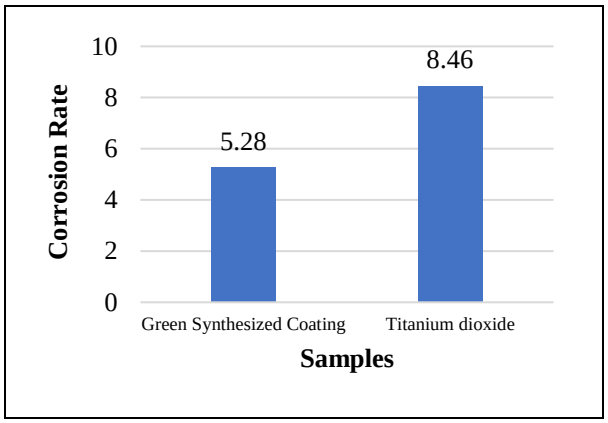


Fig. 7: Corrosion rate by weight loss method

Table 2. Salt spray testing condition

S. No	Test Condition	Requirements	Actual
1	Chamber Temperature	35+2°C	34.2-35.36°C
2	pH of Solution	6.5 to 7.2	6.8 – 7.1
3	Air Pressure	12 to 18 psi	15 psi

4.3.1 Weight Loss Method

In this method, the pre-weighed samples were dipped into 3.5% NaCl solution, i.e., sea water condition, for 2 days. Then, the samples were removed from the tank and weighed. This procedure was used to measure the corrosion rate for both coatings.

The corrosion rate was found to be less in green synthesized coatings compared to titanium dioxide coatings (Fig. 7). Thus, the green synthesized nanocoated mild steel substrate showed a minimum corrosion rate.

4.3.2 Salt Spray Test

In the salt spray method, the samples were tested in a sodium chloride environment. The test was conducted in 3.5% wt. NaCl at 35°C. The time duration for the test was 48 hours. The parameters considered for testing the samples are shown in Table 2. The initial weight of the coated substrate was calculated before exposure to the salt spray. Rust was observed at the end of 24 hours. Red rust increased at the end of 48 hours of testing.

The green synthesized nanomaterial coated substrate had less rust when compared to the normal titanium dioxide coating. Initial and post-exposure weights were calculated, which indicate the amount of material lost due to corrosion (Fig. 8).

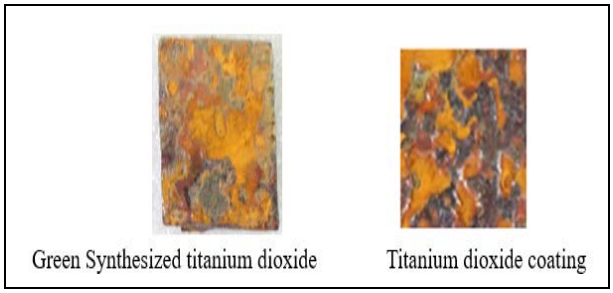


Fig. 8: Salt spray test

5. CONCLUSION

Green synthesized (from neem) and conventionally synthesized TiO_2 solutions were coated on the mild steel substrate to form a thin film. The coated substrate had a smooth surface without any cracks or voids. Weight loss method and salt spray methods showed that the green synthesized TiO_2 coating on a mild steel substrate has better corrosion resistance than the conventional TiO_2 thin film coating.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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