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Annonia Muricata Extract Mediated Green Nanoparticles Synthesis and Corrosion Inhibition of Mild Steel in Sea Water

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***Abstract**—Extract of Annonia muricata leaves were utilized in the synthesis of copper oxide nanoparticles (CuONps). Nanoparticles synthesis was confirmed by a color change of the metal salt solution from blue to greenish brown color. With a visible peak at 784nm and absorbance of 3.850, an absorption spectrum was discovered via UV-VIS spectroscopy. FTIR spectroscopy confirmed and identified functional groups present in the nanoparticles. XRD and the Debye-Scherer's equation confirmed the crystalline nature and average size of nanoparticles to be 7.2nm. The morphology and chemical makeup of the CuONPs were studied using SEM and EDX. The SEM image revealed a complex, irregular morphology of the biosynthesized nanoparticles. Furthermore, the corrosion inhibition potential of the synthesized CuONps on mild steel in seawater was studied. Concentration dependence of the inhibitor was observed with increase in efficiency with an increase in concentration of nanoparticles. Maximum efficiency of 58% was recorded at concentration of 25ml.*

***Keywords—**Annonia Muricata: Corrosion: Copper Nanoparticles: Extracts: Inhibition: Seawater: Synthesis.*

I. Introduction

Biogenic synthesis of nanoparticles presents a cost effective and straightforward approach for the production of non-toxic and highly stable nanoparticles. This process utilizes a wide range of bioactive agents from plants extracts, microbes and even biowaste. The efficiency in reducing the formation of harmful byproducts has made biosynthesis of nanoparticles a promising route for the sustainable and eco-friendly production of nanoparticles. (Altamar *et al,* 2023, Asghar *et al,* 2024). Phytochemicals and biogenic compounds in plants have a dual function of acting as natural reducing and capping agents while also eliminating the use of harmful chemicals associated with conventional nanoparticles synthesis routes. (Rajalakshmi *et al,* 2024) Green synthesis approach eliminates the use of toxic and hazardous chemicals typically employed in conventional physiochemical methods thus minimizing the environmental impact and high operational costs. (Shafqat *et al,* 2023, Salem, 2023). Biogenic synthesis approach offers an environmentally benign and economically viable alternative to chemical reduction by leveraging naturally occurring biomolecules in plant extracts and micro-organisms to facilitate the reduction of metal ions to nanoparticles. Additionally conventional synthesis methods produces heterogeneous nanoparticles with limited applications and often requires sophisticated equipment, rigorous procedures and conditions. The biogenic synthesis technique generates nanoparticles by high-yield, scalable, and environmentally friendly routes. (Shah *et al,* 2025, Gebre and Sendeku, 2019, El-seedi *et al,* 2024, Fares *et al,* 2024) Green synthesis approach leverages specialized enzymes and plants phytochemicals to facilitate the reduction of metal ions and ensures the precise control of morphology and size distribution of the synthesized nanoparticles. While acting as simultaneous capping and reducing agents, significant enhancement in the particles utility is provided by the capping layer. This layer when formed provides long term stability and biocompatibility, facilitating superior interaction with biological systems compared to chemically synthesized nanoparticles. (Millot, 2020, Soliman *et al,* 2022, Patil & Chandrasekaran, 2020, Sebesta *et al,* 2022).

Corrosion inhibition using synthetic chemical inhibitors, while effective, has drawbacks such as toxicity, environmental harm, and high cost, prompting the development and advancement of green nanoparticles derived from plant extracts as promising alternatives to conventional and traditional corrosion control methods. Green synthesized nanoparticles have demonstrated excellent corrosion inhibition properties with minimal hazardous byproducts compared to the conventional chemical inhibitors. This study focuses on the production and characterization of green nanoparticles derived from *Annona muricata* leaf extract for corrosion prevention of mild steel in seawater.

II. Methodology

A detailed systematic methodology employed in the nanoparticles synthesis using leaf extracts of *Annonia muricata* and copper (II) nitrate salts as the precursor under laboratory conditions is presented.

Fresh and matured *Annonia muricata* (soursop) leaves were harvested and authenticated in the department of Agricultural science, Niger Delta University, Nigeria. All chemicals used were of analytical grades. Mild steel sheet was obtained from Kristorall Global concept Yenagoa, Bayelsa State, Nigeria. The seawater was sourced from the Brass River coastal area in Bayelsa State, Nigeria. Prior to the experiment, all samples were appropriately stored in the Chemical Engineering Laboratory.

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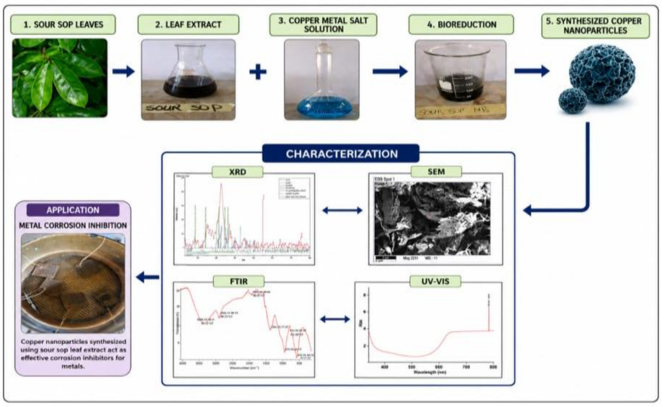

Fig. 1: A schematic diagram of the green synthesis of nanomaterials from *Annonia muricata* leaf extract.

Plant Extract Preparation and Analysis

Fig. 2: Annonia Muricata Leaves

The plant extraction process involves an aqueous-based preparation of plant tissues, which serves as a sustainable source of phytochemicals to modulate metal ion reduction (Iqbal *et al,* 2021). The leaves of *Annonia muricata* were washed under tap water, rinsed with de-ionized water and air dried until crisp. Dried leaves were pulverized into fine powder with a blender and stored in an airtight container before the extraction. 30g of the grinded leaves was weighed into a 500ml beaker containing 250ml of ethanol and left for 48hrs. This mixture was filtered using a mesh sieve to obtain high yield extract filtrate. The filtrate was heated in a water bath evaporating excess ethanol and water leaving the concentrated plant extract. The ethanolic extract of *Annonia muricata* was subjected to phytochemical investigation to assess its bioactive potential. Tannins, saponin, terpenoids, anthraquinone, and cardiac glycosides were all detected using standard qualitative procedures. (Singh *et al.,* 2021, El Golli *et al.,* 2023).

Biosynthesis of Nanoparticles

0.2M aqueous solution of Copper (II) nitrate as the precursor salt was reacted with the *Annonia muricata* leaf extract to produce nanoparticles. This aqueous solution was heated on a magnetic stirrer, with the temperature maintained between 60-70°C. The extract was gradually added to the Copper (II) Nitrate solution, and the mixture was monitored until a distinct color change was observed. This color transformation indicated the successful formation of Copper Nanoparticles in the aqueous solution. (El-Golli *et al,* 2023).

Characterization of Synthesized Nanoparticles

The produced nanoparticles' structural, morphological, and chemical compositions were examined using the methodologies given below:

UV-Visible spectroscopy was used to characterize the synthesized nanoparticles by scanning the absorbance spectra in 340-800nm wavelength range.

Fourier Transform Infrared Spectroscopy (Agilent Technology Cary 630 FTIR) was done to identify the possible biomolecules and their functional groups present in the produced nanoparticle that are responsible for the reduction of Cu^{2+} ions and the capping of the bio reduced Copper nanoparticles. The sample's FTIR spectra was obtained across a range of 450 to 4000 cm^{-1}.

X-Ray diffraction (XRD) analysis was done on the synthesized nanoparticles to establish the formation, quality and crystalline structure of the produced nanoparticles using a Rigaku miniflex-600 diffractometer. The Debye-Scherrer equation was used to determine the crystalline size of the produced nanoparticles.

$$\text{Crystallite size} = \frac{K \times \lambda}{FWHM \times \cos\theta}$$

Where K is Scherrer's constant (K=0.94), λ is the X-ray wavelength (1.54056 Å), FWHM is the full width at half maximum of the peaks, and θ is the peak position in the XRD graph. (Mengesha *et al,* 2024)

Scanning Electron Microscopy/Energy Dispersive X-ray Spectroscopy (SEM model Phenom ProX) analyzed the crystalline structure, morphology and elemental composition of the nanoparticles.

III. CORROSION TESTING

Mild steel coupons measuring 4cm by 4cm with 0.35cm holes were polished, degreased with ethanol, and dried to remove dirt scales and rust coatings on the surface. Before the corrosion, the cleaned coupons were weighed with an analytical balance and the weights were noted. The weighed coupons were immersed into a plastic container with 500ml of seawater as the corrosive media. The metal coupons were reweighed at regular intervals of 4 days to monitor weight loss. Corrosion inhibition studies were done using varying concentrations of synthesized nanoparticles solution as inhibitors. For corrosion testing, the weight loss method was performed on both inhibited and uninhibited coupon samples. Corrosion rate (CR) was estimated with the following formula (ASTM, 2012, Malaret, 2022).

$$CR = \frac{(87.6 \times W)}{(D \times A \times T)} \times 10^4 \text{ mm/yr} \qquad (1)$$

Where:
CR = Corrosion rate (mm/yr)
W = Weight loss (g), calculated as W = M_initial - M_final
D = Density of mild steel (7.85 g/cm³)
A = Surface area of the exposed sample (cm²)
T = Exposure time (hours)
87.6 × 10^4 = Unit conversion factor to express corrosion rate in mm/yr.

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The inhibition efficiency (IE%) of the green inhibitor is calculated using the equation (Kreysa & Schutze, 2007):

$$IE\% = \left(\frac{Wo - Wi}{Wo}\right) \times 100 \quad (2)$$

Where:
Wo = Weight loss uninhibited
Wi = Weight loss with inhibitor.

IV. RESULTS AND DISCUSSION

TABLE 1: Results of Phytochemical Analysis

Phytochemicals	Results
Anthraquinone	-
Tannins	+
Saponins	+
Cardiac Glycosides	-
Alkaloids	-
Flavonoids	-
Terpenoids	+

The phytochemical screening of *Annona muricata* (soursop) leaf ethanolic extract revealed the presence of specific bioactive compounds that may contribute to its efficacy as a green corrosion inhibitor. The detected constituents include saponins, tannins, and terpenoids, while alkaloids, flavonoids, cardiac glycosides, and anthraquinone glycosides were absent. The presence of saponins, tannins, and terpenoids in the ethanol extract of *Annona muricata* leaves suggests good potential as a green corrosion inhibitor. These phytochemicals are likely to contribute to adsorption on the metal surface, film formation, and complexation with metal ions, all of which are key mechanisms in mitigating corrosion.

These phytochemicals act as stabilizing agents that dictate the morphology and prevent the agglomeration of the synthesized metallic particles (Kiarashi *et al,* 2024). Furthermore, these bioactive secondary metabolites facilitate the rapid reduction of metal salts at ambient temperature and pressure, providing a highly scalable and cost-effective alternative to traditional energy-intensive fabrication protocols (Keat *et al,* 2015, Kisimba, *et al,* 2023). In addition to these chemical advantages, plant-mediated synthesis offers a streamlined scale-up potential without the requirement for maintaining high-energy input (Khandel *et al,* 2018, Shafiq *et al,* 2021)

Visual examination

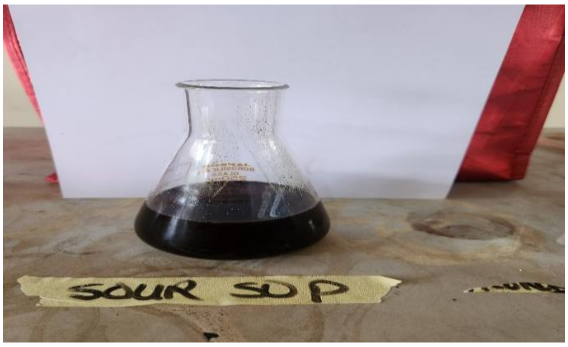

Fig. 3a: *Annonia muricata* leave extract

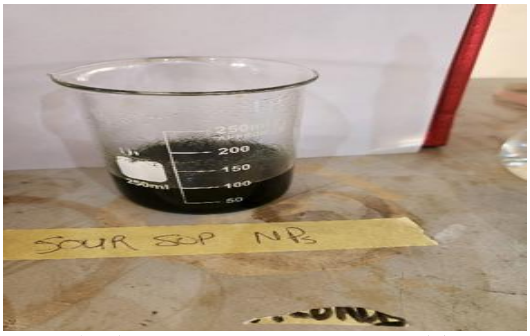

Fig. 3b: *Annonia muricta* extract nanoparticles

Change in color from blue to greenish brown was observed for nanoparticles synthesized from *A. muricata* leaf extracts. This color change is the first indication of nanoparticles synthesis. This color change is attributed to the bio-reduction of copper salts to copper ions in solution.

UV-Visible spectroscopy

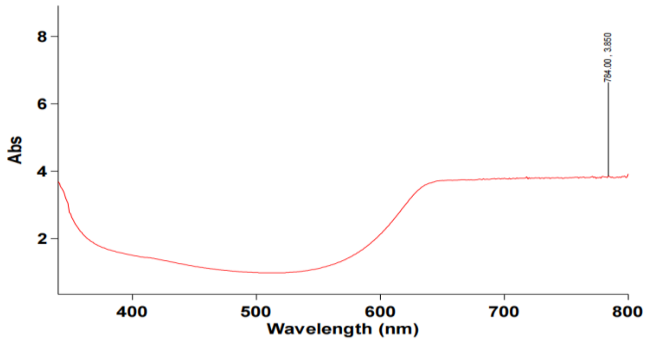

Fig. 4: Uv-visible spectra of Annonia muricata CuONps

The UV-Vis absorption spectrum of *Annona Muricata* synthesized CuONps revealed a distinctive pattern, providing insight into its phytochemical composition. The recorded spectrum showed a gradual decline in absorbance from approximately 350 nm, followed by a significant increase starting around 600 nm, peaking at 784 nm with an absorbance value of 3.850. This pattern suggests the presence of bioactive compounds capable of absorbing both in the ultraviolet and visible regions of the electromagnetic spectrum. The UV-Vis spectrum of *Annona Muricata* synthesized CuNPs showing a sharp rise in absorbance starting at 600nm and culminating at 784nm confirms the presence of diverse bioactive compounds, including highly conjugated phenolic compounds and potential presence of anthocyanin-like pigments. This confirms the formation of stable nanoparticles.

Fourier Transform Infra-Red Spectroscopy

The FTIR spectrum for *Annona muricata* mediated copper oxide nanoparticles (CuONps) reveals several characteristic absorption bands corresponding to functional groups likely involved in the bio-reduction and stabilization of the nanoparticles. FTIR spectrum displayed a number of adsorption peaks reflecting the nature of the nanoparticles. It identifies possible biomolecules in the produced nanoparticles. Observations from the Spectrum reveals a wide peak at

3280cm^{-1} which corresponds to O-H stretching vibrations, this is indicative of the presence of biomolecules of the hydroxyl groups, typical of alcohols and phenols.

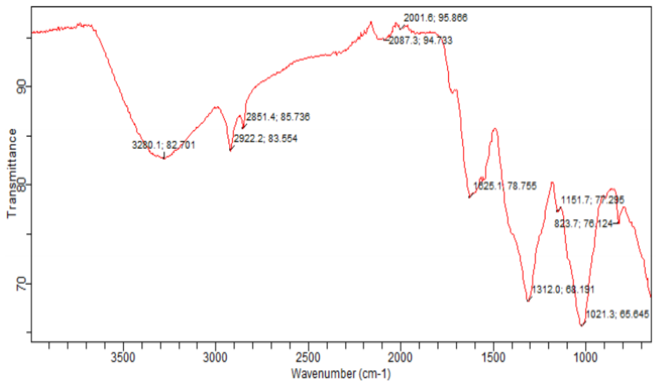

Fig. 5: FTIR Spectroscopy of Annonia Muricata CuONps

Peaks near 2922cm^{-1} are linked to C-H stretching, peaks around 1625cm^{-1} and 1560cm^{-1} usually associated with C=O stretching of conjugated ketones or carbonyl groups, or N–H bending of amines/amides, indicating protein, amino acid, or phenolic constituents, peaks near 1151.7cm^{-1} and 1031.2cm^{-1} correspond to C-O stretching vibrations, found in esters, ethers, or carbohydrate-related compounds. Peaks at approximately 1625cm^{-1} and 1560cm^{-1} are linked to C=O stretching vibrations of conjugated ketones or carboxyl groups, or N-H bending vibrations of amines and amides. Further, the peaks at 1151.7cm^{-1} and 1031.2cm^{-1} are assigned to C–O stretching vibrations. These bands suggest the presence of flavonoids, polysaccharides, or other sugar-based compounds contributing to nanoparticle formation.

X-Ray Diffraction

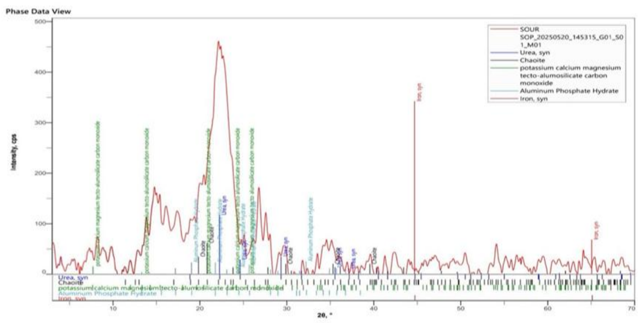

Fig. 6: XRD spectrum of Annonia Muricata mediated CuONPs

XRD patterns of green synthesized CuONps from copper (II) nitrate and *Annonia muricata* leaves extract is presented in Figure 5. XRD analysis on the produced nanoparticles was done to determine and analyze the structure, crystalline nature and size of the nanoparticles. Several intensity peaks observed on the XRD pattern indicates the crystalline nature of the nanoparticles. XRD data from the highest intensity peak with the Debye-Scherer's equation estimated the nanoparticles have an average crystalline size of 7.2nm. The XRD spectrum demonstrates that the produced nanoparticles is of a crystalline nature with no impurities found.

SEM Microscopy

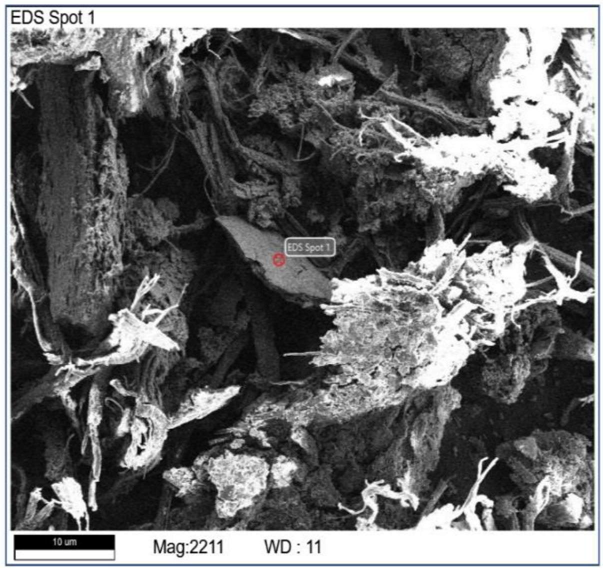

Fig. 7: SEM spectrum of *Annonia Muricata* mediated CuONPs.

The SEM image shows a complex, irregular morphology typical of biosynthesized nanoparticles. The particles appear to be aggregated with rough, uneven surfaces, characteristic of biogenic synthesis routes where plant biomolecules serve as reducing and capping agents. This morphology can influence the surface area, reactivity, and stability of the nanoparticles, although the image scale suggests nanoscale features, a precise size distribution would require image analysis. The morphology and elemental composition align with previous studies on plant-mediated CuONPs, highlighting the role of biomolecules in controlling size, shape, and surface chemistry.

Using *Ocimum lamiifolium hochst ex.benth* and *Withhana sonmifera (L) dunal* leaf extract for synthesis of CuONps nanoparticles via the green synthesis routes, Mengesha *et al,* 2024, reported synthesis of nanoparticles with similar irregular distribution of spherical nanoparticles. With a rough surface reported for *W. Somnifera* extract CuONps nanoparticles. Similar aggregation of synthesized nanoparticles was also reported by Hyba *et al,* 2023 using aqueous extract of *Moringa oleifera* leaves to synthesize CuONps using the green synthesis process. Hemashree *et al,* 2022, observed similar aggregation and agglomeration of plant extract synthesized nanopartciles using leaf extract of *Phyllanthus niruri.* Nanoparticles were spherical in shape with visible irregular agglomeration in the morphological features. The visible aggregation reported in this study and others is attributed to the self-aggregation of small nuclear particles to form larger particles. Noorafsha *et al,* 2022.

EDX Spectrum

Figure 8 shows the EDX spectrum of *Annonia muricata* mediated copper nanoparticles. The EDX spectrum shows a nanoparticle containing oxygen, copper and silica ions. It also contains calcium, chloride and potassium in minute quantities. The EDX spectrum confirms the presence of elemental signal of copper nanoparticles.

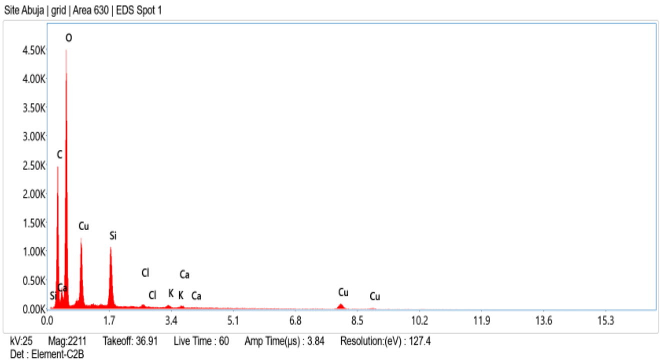

Fig. 8: EDX spectrum of Annonia Muricata mediated CuONps

The elemental composition of the EDX spectrum indicates a predominant peak for copper (Cu), confirming the presence of CuONps. The high intensity of Cu peaks suggests a successful reduction of copper ions to metallic copper peaks for other elements such as carbon (C), oxygen (O), and possibly residual plant biomolecules are also observed, indicating organic capping agents from *Annona muricata* synthesized CuONps, this implies that the presence of organic elements (C and O) aligns with biosynthesis mechanisms where plant phytochemicals (flavonoids, phenolics) stabilize the nanoparticles. This capping prevents agglomeration and enhances biocompatibility. The high carbon content is possibly from several biomolecules present in the plant extract acting as capping and reducing agents in the synthesis process.

V. Corrosion Study

Metal coupons were subjected to corrosion uninhibited by immersion in seawater (saltwater). The rate of corrosion was monitored and recorded over a 20 day period. To study the effects of extract nanoparticles on the rate of corrosion, the pre-corroded metal coupons were subjected to inhibition studies by application of varying concentration of extract nanoparticles modifying the corrosive media. Results of the corrosion rate study before and after inhibition with extract nanoparticles is presented in table 2.

TABLE 2: Corrosion rates and inhibition efficiency of *Annonia muricata* extract nanoparticle

Inhibitor Conc (ml)	CR Before Inhibition (mm/yr)	CR After Inhibition (mm/yr)	Efficiency (%)
5	0.6814	0.6348	44%
10	0.7338	0.6407	47%
15	0.6989	0.4659	55%
20	0.6290	0.5242	56%
25	0.6640	0.4659	58%

For Uninhibited metal coupons a gradual weight loss was recorded due to the corrosive action of the salt water of the metal surface. Introduction of the *Annonia muricata* extracts nanoparticles saw a decline in corrosion rate. This is indicative of an effective nanoparticle inhibition of the corrosion mechanism of the seawater on the metal surface. Recorded rates of corrosion and inhibition efficiency was observed to increase with increase in concentration with a maximum obtained at a concentration of 25ml and inhibition efficiency of 58%.

Nanoparticle inhibition on metal surface is usually via adsorption on the metal surface. The characteristic high surface area of nanoparticles makes adsorption effective. (Jian *et al*, 2020). Plant extracts contain organic chemicals and heteroatoms that are loaded onto the produced nanoparticles throughout the synthesis process. The high surface area of the nanoparticles combined with presence of these organic compounds and heteroatoms significantly increases the metal adsorption and ultimately the adsorption efficiency. (Dariva and Galio, 2014, Alghamdi, 2023).

Similar results were recorded by Surendhiran *et al,* 2021 in the synthesis of CuONps using *Moringa oleifera* leaves extract as corrosion inhibitors. Using the spin coating method, the green CuONPs were coated on mild steel as corrosion inhibitors in 3.5% NaCl solution. The CuONPs achieved an inhibition efficiency of 56% evidenced from electrochemical measurements. (Surendhiran *et al.* 2021). Bhaswar *et al,* 2023, reported on the inhibitive behavior of *Jacaranda mimosaefolia* flower extract nanoparticles as corrosion inhibitors. Inhibition efficiency of 28% was reported with the application of extract nanoparticles as inhibitor on AISI 1018 carbon steel in HCL medium. Devodoss *et al,* 2023, applied a green mediated CuNPs from *Murraya koenigii* leaves as corrosion inhibitor on dental alloy Ti-6Ai-4N. Plant extract nanoparticles showed corrosion inhibition with inhibition efficiencies of 58.15% and 26.6%.

VI. Conclusion

Green synthesized nanoparticles are a promising anti-corrosion alternative to conventional corrosion inhibitors. Green metallic nanoparticles inhibit corrosion by forming a protective barrier on the metal surface that prevents the ingress of corrosive media into the metal surface.

The *Annona muricata* leaf extract is rich in phytochemicals and biomolecules responsible reduction of Cu ions to CuONps. Results from Uv-vis, SEM, XRD and FTIR confirmed the successful synthesis of the plant extract nanoparticles from reduction of the precursor metal salts into stable nanoparticles.

The results from FTIR confirmed the presence of biomolecules functional groups with the capability to act as both reducing and stabilizing agent in the nanoparticle synthesis process. XRD data together with the Scherer's equation estimated the average crystalline size of the nanoparticle to be 7.2nm. Inhibition studies confirmed successful corrosion inhibition by the CuONps in seawater with the highest inhibition efficiency of 58%. This results confirms successful adsorption of nanoparticles molecules onto the metal surface.

The weight losses recorded was dependent on the time of exposure and the concentration of *Annona Muricata* synthesized CuONPs i.e weight loss increases with increase

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in time (days) and decreases with increase in inhibitor concentration.

These results validate the role of *A. muricata* leaf extract in the green synthesis of CuONPs, with phytochemicals and biomolecules providing bio-reduction and stabilization roles. The combination of structural, optical, and morphological evidence supports the potential of these biosynthesized CuONPs for scientific engineering applications.

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