**GREEN SYNTHESIS OF COPPER OXIDE NANOPARTICLES (CUO NP) USING *GONGRONEMA LATIFOLIUM* (UTAZI LEAVE) EXTRACT FOR EFFECTIVE PHOTOCATALYTIC DEGRADATION OF DYE.**

**BY**

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**DEPARTMENT OF PURE AND INDUSTRIAL CHEMISTRY**

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**PROJECT SUPERVISOR:**

# TITLE PAGE

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# CERTIFICATION

This is to certify that this research work titled: Synthesis, characterization and application of Zn-doped Fe2O3 nanoparticle for the removal of methylene blue dye from aqueous solution was originally done by Nwodo Emmanuel Chimaobi with registration number 2018/249227, has been approved by the undersigned as having met the standard of the department of Pure and Industrial Chemistry, University of Nigeria, Nsukka and has not been submitted either for diploma, any other if this or in any other university.

……………………………….. ………………………………..

DATE

(PROJECT SUPERVISOR)

……………………………….. ………………………………..

PROF. B. E. EZEMA DATE

(HEAD OF DEPARTMENT)

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EXTERNAL EXAMINER DATE

DEDICATION

This work is dedicated to God Almighty, my parent, my siblings

Acknowlegement

# Abstract

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# CHAPTER ONE

INTRODUCTION

## BACKGROUND STUDY

Green nanotechnology has gained attraction in the synthesis of metallic nanoparticles due to their cost-effectiveness, simple preparation steps, and environmentally-friendly(Nzilu et al., 2023).

In recent years, the field of nanotechnology has seen significant advancements in the synthesis and application of nanomaterials for environmental remediation, with a particular focus on copper oxide nanoparticles (CuO NPs) (Akintelu et al., 2020; Majumdar & Ghosh, 2021; Panhwar et al., 2021). These nanoparticles have unique properties that make them promising candidates for various applications, including in the field of supercapacitors (Majumdar, 2020). The use of green synthesis methods, such as those involving plant extracts, has been highlighted as a cost-effective and environmentally friendly approach to producing CuO NPs(Akintelu et al., 2020; Joshi et al., 2019; Panhwar et al., 2021). These methods have the potential to scale up to large-scale production and have been shown to enhance the toxicity of CuO NPs against microbes, making them effective for environmental remediation (Akintelu et al., 2020; Panhwar et al., 2021).

### 1.1.1 Copper Oxide Nanoparticles (CuO NPs)

Copper oxide nanoparticles (CuO NPs) have shown promise in environmental applications due to their high surface area, catalytic activity, and photodegradation capabilities (Chauhan et al., 2020). These properties make them effective in degrading organic pollutants, as demonstrated in the degradation of dyes (Chauhan et al., 2020). Furthermore, CuO NPs have been explored for their potential in supercapacitor applications, with their unique advantages of low cost, high chemical stability, and remarkable electrochemical performance (Majumdar & Ghosh, 2021). The use of 3D hierarchical nanoporous structures of copper has also been investigated for the degradation of organic pollutants, showing high efficiency and reusability (Yang et al., 2018). The synthesis methods and applications of nanostructured copper oxide semiconductors have been reviewed, highlighting their unique properties and potential in various fields (Zoolfakar et al., 2014).

### 1.1.2 Green Synthesis: A Sustainable Approach

The concept of green synthesis, which utilizes natural sources such as plant extracts as reducing and stabilizing agents, has gained prominence as a sustainable approach to nanoparticle production (Murphy, 2008). This method not only reduces the environmental impact by avoiding toxic chemicals, but also enhances biocompatibility, making the nanoparticles safer for various applications (Jadoun et al., 2021). The use of green renewable resources and energy-efficient synthetic routes further contributes to the eco-friendliness of this approach (Madani et al., 2022). The potential of green synthesis in the preparation of metallic nanoparticles using plant extracts, ascorbic acid, and sodium citrate as green reagents is also highlighted (Adil et al., 2015).

### 1.1.3 *Gongronema latifolium* Extract: A Natural Resource

*Gongronema latifolium,* also known as Utazi Leaves, is a plant with a rich phytochemical composition, including flavonoids, alkaloids, and phenolic compounds (Udoidong et al., 2014).



Figure 1: Structure of flavonoids

Source from (Dias et al., 2021)



Figure 2: Simple substituted phenol compounds

Source from (Mamari, 2021)

These compounds contribute to the plant's antioxidant, antibacterial, and anti-inflammatory properties (Adekanle & Omozokpia, 2015). The plant has a wide range of nutritional and ethnomedical uses in different tropical African communities, and its extracts have potential applications in herbal formulations, food preservation, alcoholic fermentation, drug discovery, and allelopathy (Morebise, 2015). Furthermore, the methanol extract of Gongronema latifolium has been found to possess significant anti-hyperglycemic and antioxidant activities (Chidi, 2017)

### **1.1.4 SYNERGY OF CUO NPS AND UTAZI EXTRACT**

The combination of CuO NPs and Utazi extract, as explored by Raizada (2020), has shown potential in addressing environmental challenges, particularly in the photocatalytic degradation of dyes in wastewater(Raizada et al., 2020). This approach is in line with the broader trend of using nanoscale metal oxide materials for water treatment, as discussed by Raizada et al. (2020). The enhanced photodegradation activity of CuO NPs, as demonstrated by Rasheed, further supports the potential of this combination. The use of CuO NPs in the purification of dye-polluted water, as highlighted by Rafique et al. (2020), also underscores the effectiveness of this approach. Saghir (2020) further emphasizes the importance of efficient adsorbents in wastewater treatment, which could potentially be complemented by the CuO NPs and Utazi extract combination(Saghir et al., 2020).

## **1.2 AIM AND OBJECTIVES**

Aim: The aim of this research paper is to explore the green synthesis of copper oxide nanoparticles (CuO NPs) using *Gongronema latifolium* (Utazi Leave) extract and investigate their effectiveness as a photocatalyst for the degradation of dyes in wastewater treatment.

Objectives:

1. To synthesize CuO nanoparticles using *Gongronema latifolium* extract via a green synthesis method.
2. To characterize the synthesized CuO nanoparticles using techniques such as TEM, SEM, XRD, and FTIR to confirm their size, morphology, crystallinity, and chemical composition.
3. To evaluate the photocatalytic activity of the synthesized CuO nanoparticles for the degradation of a selected dye under UV or visible light irradiation.
4. To compare the photocatalytic efficiency of CuO nanoparticles synthesized with *Gongronema latifolium* extract with that of conventional methods or other green synthesis approaches.
5. To investigate the potential mechanisms involved in the photocatalytic degradation process, such as the generation of reactive oxygen species (ROS) and the adsorption of dye molecules onto CuO nanoparticle surfaces.
6. To discuss the implications of the findings in terms of the application of green-synthesized CuO nanoparticles for environmentally friendly wastewater treatment strategies.

## **1.3 JUSTIFICATION AND SIGNIFICANCE**

The justification for this research lies in addressing critical environmental and technological challenges. Traditional methods of dye degradation in wastewater are often energy-intensive, chemically hazardous, and environmentally detrimental. Green synthesis of copper oxide nanoparticles (CuO NPs) using plant extracts offers a sustainable alternative with reduced environmental impact. *Gongronema latifolium* extract, known for its bioactive compounds, can serve as a green reducing and stabilizing agent for CuO NP synthesis (Nzilu et al., 2023). This approach aligns with the principles of green chemistry by minimizing waste generation and utilizing renewable resources.

Significance:

1. Environmental Sustainability: The use of green-synthesized CuO NPs represents a step towards sustainable wastewater treatment. It reduces the reliance on chemical methods that contribute to pollution and resource depletion (US EPA, 2015).
2. Cost-Effectiveness: Utilizing plant extracts for nanoparticle synthesis can be cost-effective compared to conventional methods involving expensive precursors and energy-intensive processes.
3. Efficient Photocatalysis: CuO NPs have shown promising photocatalytic activity in degrading organic pollutants. Investigating their efficacy using *Gongronema latifolium* extract adds to the knowledge base of eco-friendly photocatalytic materials(Sibhatu et al., 2022).
4. Biocompatibility: Green-synthesized CuO NPs are expected to be biocompatible and less toxic, making them suitable for applications in water treatment without adverse effects on aquatic life or human health(Ranjha et al., 2022).
5. Potential for Scalability: Green synthesis methods are scalable and adaptable for large-scale production, offering practical solutions for industrial wastewater treatment plants(Gao et al., 2022).
6. Contribution to Nanotechnology: The study contributes to advancing the field of vnanotechnology by exploring innovative approaches for nanoparticle synthesis and their application in environmental remediation.

In summary, this research is significant as it combines principles of green chemistry, nanotechnology, and environmental stewardship to develop a sustainable solution for dye degradation in wastewater, with broader implications for eco-friendly technologies in various industries

# CHAPTER TWO

## LITERATURE REVIEW

*Gongronema latifolium* Benth belongs to the family *Asclepiadaceae*. It is an edible nutritional/ medicinal plant mostly found in the rain forest zones in Nigeria and other tropical African countries (Chattopadhyah, 1999; Hutchinson & Dalziel, 1931). The plant produces white latex and yellow flowers (Hutchinson & Dalziel, 1931) and can be propagated by seed or stem cuttings (Edim et al., 2012). *G. latifolium* is known by the Ikales of Ondo State of Nigeria as Iteji (Morebise et al., 2006; Morebise & Fafunso, 1998). The Igbos call the plant Utazi, the Efik/ Ibibio call it Utasi while the Yorubas call it Arokeke (Edim et al., 2012). To the Akan-Asantes of Ghana, G. latifolium is known as Kurutu Nsurogya; the Serers of Senegal call it Gasub while to the Kissis of Sierra Leone it is known as Ndodo-Polole (Edim et al., 2012).



Figure 3 The Gongronema latifolium plant

Fekri et al. (2022) conducted a study on the efficacy of photocatalytic and sonocatalytic processes using copper oxide nanoparticles (CuO NPs) synthesized from Peganum harmala seed extract for removing reactive blue 5 dye and organic compounds from textile effluent. Structural confirmation of CuO NPs was done using SEM, TEM, XRD, EDX, and FTIR techniques. Batch experiments were performed to analyze factors affecting dye removal efficiency, including contact time, pH, NPs dosage, and initial dye concentration. The results showed that the photocatalytic process achieved a higher degradation percentage (98.42%) compared to the sonocatalytic process (76.16%). Dye removal efficiency was insignificant in dark conditions. Optimal conditions for maximum dye removal were found with 0.15 g of CuO NPs and dye concentrations of 40 mg/L (photocatalytic) and 60 mg/L (sonocatalytic). Kinetic data followed a pseudo-second-order model with a high correlation coefficient (>0.99) for both processes. Isotherm studies favored the Langmuir model to describe CuO NPs' adsorptive behavior in dark conditions. GC-MS analysis showed over 87% degradation efficiency in removing organic compounds via the photocatalytic process.

Singh et al. (2019) conducted a study on the preparation of copper oxide nanoparticles (CuO NPs) using Psidium guajava leaf extract as a reducing and capping agent. The CuO NPs, characterized as mono-dispersed and spherical with an average size of 2-6 nm and a BET surface area of 52.6 m2/g, demonstrated excellent degradation efficiency for industrial dyes Nile blue (NB) and reactive yellow 160 (RY160). The removal rates were 93% for NB and 81% for RY160 in 120 min, with apparent rate constants of 0.023 and 0.014 min-1, respectively. The CuO catalyst showed reusability for photocatalytic dye degradation over five cycles and had low detection limits (LOD) of 4 and 9 mg/L for NB and RY160, respectively. The study suggests that biogenic CuO NPs are a promising option for water purification from industrial dye contamination due to their high efficiency, reusability, and green synthesis approach.

## **NUTRITIONAL USES OF *GONGRONEMA LATIFOLIUM***

The leaves of G. latifolium are used as vegetables in preparation of soups to which they add a bitter-sweet flavor (Iwu, 1988; Morebise & Fafunso, 1998). The leaves are also sometimes used to spice locally brewed beer (Edim et al., 2012). The soft stem is used as chewing stick in Sierra Leone (Mosango, 2015).

## **ETHNOMEDICAL USES OF *GONGRONEMA LATIFOLIUM***

There have been reports of various uses of Gongronema latifolium in folklore medicine by different ethnic groups. (Morebise & Fafunso, 1998) reported that the leaves of this plant are used by the Ikales of Ondo State of Nigeria to treat malaria, nausea and anorexia. Edet et al also reported that the leaf extract of G. latifolium is commonly used by the Efik and Quas tribes of Cross River state of Nigeria to treat malaria, diabetes, hypertension and constipation (Edet et al., 2011).

Mosango reported that *G. latifolium* is used in some West African communities to treat cough, intestinal worms, dysentery, dyspepsia and malaria. He reported that in Sierra Leone, an infusion or decoction of the stems with lime juice is taken to treat colic and stomach aches, while in Senegal and Ghana, *G. latifolium* leaves are rubbed on joints of children to help them walk while the boiled fruits of this plant are eaten as a laxative (Mosango, 2015). Essien et al reported that G. latifolium is used to treat cough in Nigeria (Edet et al., 2011). Asthmatic patients can chew the fresh leaves of G. latifolium to relieve wheezing while a cold maceration of the roots of the plant can be consumed as treatment for asthma (Essien et al., 2007; Mosango, 2015). Mosango also reported the use of this plant in some communities to treat viral hepatitis, bilharzia and other microbial infections(Mosango, 2015).

Iwu and Oliver-Bever reported that the leaves of G. latifolium are used in some local communities as a vermifuge and stomachic (Iwu, 1988; Oliver-Bever, 1986). Owu et al reported that the leaves are also used to treat dyspepsia in some local communities (Owu et al., 2012). Essien et al. reported that the leaves of G. latifolium are used to treat fowl cough in Nigeria(Essien et al., 2007).

In recent years, in the field of nanotechnology, metal and metal oxide nanoparticles have been the most developed materials due to their modified and adjustable morphology, physicochemical properties, small size, and enormous surface area (Gusmão et al., 2013; Khan et al., 2020). They find various applications such as agricultural research, pharmaceuticals, biological, and environmental. Among the various metal and metal oxide nanoparticles, copper oxide (CuO) nanoparticles have a wide and direct band gap of 1.2–2.1 eV, are a member of group II-VI of p-type semiconductors and monoclinic structures. They have become a potential candidate for environmental applications (Koffyberg & Benko, 1982; Weldegebrieal, 2020). Additionally, they exhibit low toxicity, high chemical and thermal stability, compatibility, easy synthesis route, variable morphology at the nanoscale, a high specific surface area, improved oxygen adsorption capacity, and low cost (Arunkumar et al., 2019; B S, 2016; Vinothkumar et al., 2019). In recent years, many well-defined CuO nanostructures have been synthesized, such as nanospheres (Xu et al., 2005), nanoflowers (Yu & Zhang, 2009), micromachines (Yang et al., 2013), nano leaves (Liang & Zhu, 2004), nanorods (Yu & Zhang, 2009), nanotubes (Cao et al., 2003), nanosheets (Jang & Kim, 2009), and nanorings (Liu et al., 2007) to improve the surface reactivity and properties of nanoparticles. Hence, researchers are attempting to develop clean, non-toxic and cost-effective methods that totally reduce the use of hazardous chemicals (Muthuvel et al., 2020).

# **CHAPTER THREE**

### **MATERIAL AND METHODS**

## **3.1 MATERIAL AND REAGENT USED**

1. Extract from *Gongronema Latifolium*
2. Copper nitrate trihydrate (95% Cu(NO3)2.3H2O)
3. Sodium hydroxide (99% NaOH)
4. Ethanol (97%)
5. Distilled water

## **3.2 APPARATUS AND EQUIPMENT**

1. Magnetic stirrer
2. Magnetic bar
3. pH meter
4. Thermometer
5. Electric blender
6. Oven
7. Furnace
8. Glass rods
9. Crucibles
10. Plastic bottles
11. Beakers
12. Conical flasks
13. Volumetric flasks
14. Spatula
15. Dropper
16. Paper tape
17. Whatman no 42 filter papers
18. Hand gloves
19. Nose masks
20. Measuring Cylinder

## 3.4 PREPARATION OF THE LEAF EXTRACT

Fresh leaves of *Gongronema Latifolium* were collected from Nsukka community in Nigeria. 10g of the leaves were thoroughly washed with distilled water to remove any dust, after which it was cut into small pieces and added into 100ml of water in a beaker and heated at 800C on an electric heater for 2 hours. After being cooled at room temperature, it was filtered using whatmann filter paper to extract the filtrate. The extract was stored in a container in a refrigerator until use.



Figure 4: Gongronema latifolium leaves: (a) in the farm (b) washed and grinded



**Figure 5: Filteration process of the leaves extract**

## 3.5 GREEN SYNTHESIS OF COPPER OXIDE NANOPARTICLES

CuO nanoparticles were synthesized by using 0.1M Cu(NO3)2.3H2O. 100ml of the copper nitrates was taken in a 250ml Erlenmeyer Flask and 25ml of the *Gongronema Latifolium* extract was added slowly to reduce copper ions to its copper oxide nanoparticles. Then, 10ml of 2M NaOH solution was added to adjust pH to 11 while stirring it constantly. The solution was stirred continuously at 800C for 2 hours.

 

Figure 6: Copper solution colour change at different time

The blue coloured solution turned green immediately and after about 2 hours, a dark brown precipitate formed indicated that all the copper ions have been reduced and CuO nanoparticles have been formed. The obtained precipitate was centrifugated at 10,000 rpm for 10mins and washed several times using distilled water and ethanol for removal of impurities, and the sample was dried at 800c for 2hours.



Figure 7: (a) separation after 24hrs (b) grinding process of the GSCu-NPs after drying and annealing (c) green synthesized GSCu-NPs

Preparation of 100ml of Cu(NO3)2.3H2O

Mass = 2.42g of Cu(NO3)2.3H2O

Preparation of 10ml of NaOH

Mass = 0.8g NaOH

## 3.5 Characterization and Analysis

UV

Ultraviolet–visible spectroscopy or ultraviolet-visible spectrophotometry (UV-Vis or UV/Vis) refers to absorption spectroscopy or reflectance spectroscopy in the ultraviolet-visible spectral region. This means it uses light in the visible and adjacent (near-UV and near-infrared (NIR)) ranges. The absorption or reflectance in the visible range directly affects the perceived color of the chemicals involved. In this region of the electromagnetic spectrum, molecules undergo electronic transitions(Sahoo et al., 2012).

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