**SYNTHESIS, CHARACTERIZATION AND APPLICATION OF Zn-DOPED Fe2O3 NANOPARTICLE FOR THE REMOVAL OF METHYLENE BLUE DYE FROM AQUEOUS SOLUTION**

**BY**

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**2018/249227**

**A PROJECT SUBMITTED IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE AWARD OF B.SC DEGREE IN PURE AND INDUSTRIAL CHEMISTRY**

**DEPARTMENT OF PURE AND INDUSTRIAL CHEMISTRY**

**FACULTY OF PHYSICAL SCIENCE**

**UNIVERSITY OF NIGERIA, NSUKKA**

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**APRIL 2024**

# **TITLE PAGE**

**SYNTHESIS, CHARACTERIZATION AND APPLICATION OF ZN-DOPED** **FE2O3 NANOPARTICLE FOR THE REMOVAL OF METHYLENE BLUE DYE FROM AQUEOUS SOLUTION**

**CERTIFICATION**

This is to certify that this researchwork 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.

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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 of study

Present era has seen rapid advancements in industry and technology but with a disastrous environmental impact. Unscrupulous waste disposal by factories, especially release of toxic organic dyes into the water bodies produce life-threatening effects, the long-term effects are still to be known. Wide investigations are going onto find a solution to this problem through environment-friendly methods(Jack et al., 2015; Xu et al., 2012, p. 2012; Zhong et al., 2006) using environmentally benign materials like TiO2, ZnO, iron oxide, etc (Ashraf et al., 2019; Chen et al., 2017). there has been significant interest in iron oxide nanostructures due to its nontoxicity, biocompatibility, abundance and the desirable optoelectronic properties (Li et al., 2016; Zhang et al., 2012). suitable for applications in biosensing and photocatalysis (Kumar et al., 2019; Manna et al., 2018; Stephen Inbaraj et al., 2012). Iron oxide usually exists in 16 different phases. Of these, the prominent phases are hematite (α-Fe2O3), magnetite (Fe3O4), maghemite (γ-Fe2O3) and wurtzite (FeO). Hematite, the most stable phase of iron oxide is widely used in gas sensing, photovoltaic and photocatalytic applications (Belle et al., 2011; Zhang et al., 2012).

Nanoscience is a branch of science that comprises the study of properties of matter at the nanoscale, and particularly focuses on the unique, size-dependent properties of solid-state material (Mulvaney, 2015). nother proposed definition is that nanomaterials exhibit a specific surface area to volume ratio greater or equal to 60 m2 /cm3 (Kreyling et al., 2010). Nanometer-sized particles (1–100 nm) have attracted considerable interest for a wide variety of applications, ranging from electronics via ceramics to catalysts due to their unique or improved properties, which are primarily determined by size, composition, and structure(Xia et al., 2001). With reduction in size, a greater function of the atoms is at the surface, and promote different interaction with its environment, as compared to the bulk material(Shrestha et al., 2020). the small size also leads to high surface energy, and NPs tend to aggregate, thereby lowering the surface energy. In all applications, the uncontrolled aggregation of NPs can have negative effects and needs to be avoided (Shrestha et al., 2020).

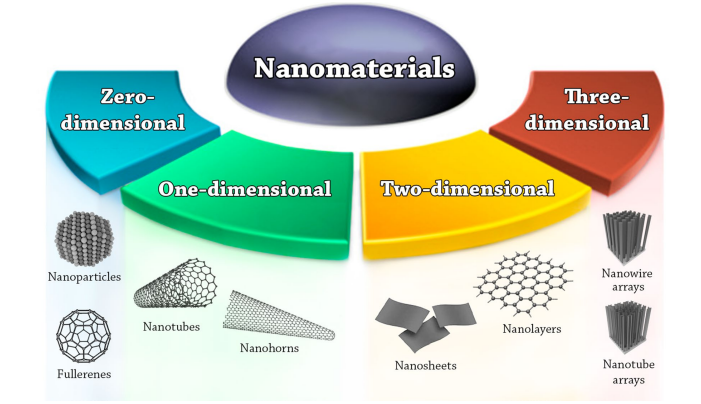


Figure 1: Nanomaterials classification based on dimensionality (Joudeh & Linke, 2022)

NPs as synthesized, tend to be very reactive since their surfaces possess a high density of dangling bonds, and defects. Due to the small grain sizes, the surface energy is high, and processes to reduce the surface energy through assembling of NPs can become dominant (Kamiya et al., 2008).

### AIM AND OBJECTIVE

#### 1.2.1 AIM OF STUDY

The aim of this study is to explore the efficiency of Zn-doped iron oxide nanoparticles in removing methylene blue from aqueous solutions.

#### 1.2.2 SPECIFIC OBJECTIVES OF STUDY

The specific objectives of this study are as follows:

* Synthesizing Zn-doped Fe2O3 nanoparticles using the co-precipitation method.
* Characterizing the Zn-doped Fe2O3 nanoparticles through various techniques including X-ray diffraction (XRD), Ultraviolet Spectroscopy, and Fourier Transform Infrared Spectroscopy (FTIR).
* Assessing the efficacy of Zn-doped Fe2O3 nanoparticles in methylene blue dye removal via adsorption experiments.
* Investigating the impact of experimental parameters such as initial methylene blue concentration and contact time on the adsorption capacity of Zn-doped Fe2O3 nanoparticles.

### Justification and significance of the study

This work is justified for several reasons:

* Environmental pollution is a major global concern and industrial wastewater is a significant contributor to this problem. Hence, the study has the potential to mitigate this problem and improve the sustainability of industrial process (Estrada et al., 2022).
* Conventional methods of waste water treatment are often costly, energy intensive, and generate large amounts of sludge thereby necessitating Nanoparticles adsorbents like this produced from incorporating Goethite (Fe3O4) doped with cerium nanoparticle offers a more sustainable, eco-friendly and cost effective alternative because of its easy reusability and regenerability (Bethi & Sonawane, 2018).
* The result of this study has practical application in industries that produce wastewater containing dyes, such as textile, paper and leather industries. The use of this efficient and effective nanoparticle adsorbent could help these companies to meet environmental regulations and reduce their environmental impact (Mbarek et al., 2022).
* Being an area of active research, this study could also have broader implication for the development of new material and technologies for environmental application(Kumari et al., 2019)

# CHAPTER TWO

## LITERATURE REVIEW

Many research have explored the use of various nanomaterials for the removal of methylene blue dye from aqueous solutions. Saini et al., (2018) synthesized silver silica-coated magnetite (Fe3O4@Ag/SiO2) nanospheres using the sonication method and evaluated their performance as nanoadsorbents for removing Methylene Blue in batch mode experiments. Physical characteristics of these nanospheres were studied using XRD, SEM, EDX, TEM, and FTIR techniques. The Fe3O4@Ag/SiO2 nanospheres effectively removed 99.6% of Methylene Blue from aqueous solutions at pH 7. A proposed mechanism for the adsorption of Methylene Blue onto Fe3O4@Ag/SiO2 was presented. Adsorption equilibrium and kinetics were analyzed for experimental data, revealing that the removal process followed the Langmuir isotherm with a maximum monolayer adsorption capacity of 128.5 mg/g. Experimental kinetic data fitted well to Pseudo-second-order and Intraparticle diffusion models. Thermodynamic parameters, including ΔG0, ΔS0, and ΔH0, indicated spontaneous, endothermic, and feasible adsorption of Methylene Blue under the studied conditions. The Fe3O4@Ag/SiO2 nanospheres were found to be regeneratable and reusable for five successive cycles.

Arasteh Nodeh et al. (2019) synthesized spherical α-Fe2O3 nanoparticles (NPs) using Forced Hydrolysis and Reflux Condensation (FHRC) and supported them on silica sand via the Solid-State Dispersion (SSD) method. Characterization of the silica and α-Fe2O3/SiO2 catalyst involved Fourier-Transform InfraRed (FT-IR) spectroscopy, Scanning Electron Microscopy (SEM) imaging, X-Ray Diffraction (XRD) analysis, and Brunauer, Emmet, and Teller (BET) surface area measurements. The supported α-Fe2O3/SiO2 nanocatalyst, with an average crystallite size of 27.5 nm, was used for photocatalytic removal of Methylene Blue (MB) from aqueous solutions under Ultra-Violet (UV) light. Optimization of effective parameters for MB degradation employed the single-variable method, determining optimal conditions at pH=11, initial MB concentration=10 ppm, and catalyst mass=1.0 g. Under these conditions, the degradation efficiency reached 97.32%.

Mai et al., (2020) study focused on synthesizing nano Fe2O3 particles through the combustion of a gel made from polyvinyl alcohol (PVA) and tartaric acid (TA) for degrading methyl blue (MB) in aqueous solutions via photocatalysis. Factors affecting Fe2O3 formation, such as solution pH, gel formation temperature, TA/PVA mole ratio, and calcination temperature, were investigated. The structure and morphology of the Fe2O3 particles were characterized using Differential Thermal Analysis, X-Ray Diffraction, and Field Emission Scanning Electron Microscopy. The results revealed that the synthesized Fe2O3 particles were single-phase with an average grain size smaller than 60 nm. Under visible light irradiation, Fe2O3 catalysts exhibited a high photocatalytic capacity for decomposing MB, with identified intermediates from the photocatalytic degradation process.

El Messaoudi et al. (2022) conducted batch adsorption experiments using oxalic acid-modified jujube shells of Ziziphus lotus (OA-JS)/ZnFe2O4(OA-JS@ZnFe2O4) nanocomposite to remove Congo red (CR) and methylene blue (MB) from aqueous solutions. Characterization of OA-JS@ZnFe2O4 was done using Fourier transform infrared (FTIR), Brunauer Emmett Teller, thermogravimetric analysis–differential scanning calorimetry, scanning electron microscope, transmission electron microscope, and X-ray diffraction techniques.The equilibrium data fit well with the Langmuir isotherm, determining maximum adsorption capacities of 980.39 mg/g for CR and 476.18 mg/g for MB. The sorption kinetics were best described by the pseudo-second-order model. Thermodynamic analysis indicated that CR and MB adsorption on OA-JS@ZnFe2O4 is spontaneous and endothermic. The recycled OA-JS@ZnFe2O4 maintained high removal efficiencies of 82.08% for CR and 76.59% for MB after seven cycles, demonstrating its excellent potential as an adsorbent for dye removal from wastewaters.

Nassar et al., (2016) synthesized iron carbonate nanospheres through hydrothermal treatment of iron sulfate, ascorbic acid, and ammonium carbonate solutions. These were then converted to pure α-Fe2O3 nanoparticles via thermal decomposition at varying temperatures. Characterization involved XRD, FE-SEM, HR-TEM, FT-IR, BET, zeta potential, and thermal analysis. The adsorption capacity of α-Fe2O3 for reactive red 195 (RR195) dye was evaluated, showing excellent adsorption efficiency of 98.77% in 10 minutes. Increasing the surface area of α-Fe2O3 nanoparticles from 107.7 to 165.6 m2/g enhanced the adsorption capacity from 4.7 to 20.5 mg/g. Langmuir isotherm model fitting indicated good agreement with the adsorption data, while thermodynamic parameters suggested an endothermic and spontaneous adsorption process for RR195 dye on the hematite adsorbent.

Abkenar et al., (2015) synthesized tannic acid-modified superparamagnetic Fe3O4 nanoparticles (Fe3O4-TAN) and used them as adsorbents for removing methylene blue (MB) from water solutions. Characterization of the magnetic nanoparticles (MNPs) was conducted using Fourier transform infrared (FT-IR), transmission electron microscopy (TEM), thermogravimetric analysis (TGA), and X-ray diffraction (XRD). The study investigated the effects of pH, contact time, dye concentration, and temperature on adsorption, with the Langmuir adsorption model fitting well to the experimental data with a maximum adsorption capacity of 90.9 mg/g at pH 10.5.

Adsorption kinetics indicated equilibrium reached after 25 minutes, and thermodynamic parameters were evaluated. The MNPs could be easily separated from water using an external magnetic field with high efficiency. The desorption process of the adsorbed dyes was also explored in the study.

# CHAPTER THREE

## MATERIALS AND METHODS

### 3.1 REAGENT USED

1. Potassium hydroxide (KOH)

2. Ferric nitrate (Fe(NO3)₃)

3. Distilled water

4. Methylene blue dye

5. Hydrochloric acid (HCl)

6. Sodium Hydroxide (NaOH)

7. pH buffer

### 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

### 3.3 SYNTHESIS OF ZN-DOPED FE2O3 NANOPARTICLE USING CO-PRECIPITATION METHOD

The synthesis technique began with the production of a 1 M ferric nitrate (Fe(NO3)₃) solution (50 mL). Zinc precursor solution, which was zinc nitrate (Zn(NO3)₂), was then added in a pre-determined stoichiometric ratio to achieve the necessary zinc doping level. The combined solution was then subjected to controlled addition of a 4 M potassium hydroxide (KOH) solution delivered dropwise under steady and quick stirring to achieve homogenous mixing and prevent particle agglomeration. The addition proceeded until the solution achieved the optimum pH of 13-14, which remains critical for goethite production. To encourage the creation of smaller nanoparticles, the stirring speed was concurrently raised while the KOH droplet size was decreased. This method boosts the shear forces acting on the growing particles, ultimately leading to a finer particle size distribution.



Figure 2: Colour change after 10 minutes of stirring continuously

After 10 minutes of continuous stirring, an additional 50 mL of the 4 M KOH solution was added to further elevate the solution's alkalinity and encourage full precipitation of the zinc-doped iron oxyhydroxides. This leads in the production of a well-defined red-brown precipitate. The succeeding steps paralleled the undoped synthesis. The precipitate was diluted tenfold with double-distilled water, followed by transfer to an oven for age at 70-75 °C for 72 hours. This procedure enhances the crystallization and maturation of the zinc-doped iron oxide nanoparticles. Following the aging period, the final product was obtained by a series of washing operations (five to six times) using double-distilled water to eliminate contaminants and ensure the cleanliness of the nanoparticles. Finally, the washed precipitate was oven-dried at a low temperature (50-55 °C) to remove any residual moisture. The resultant powder constitutes the zinc-doped iron oxide nanoparticles, ready for further characterization and application testing.

### 3.4 PREPARATION OF STOCK SOLUTION OF METHYLENE BLUE DYE

100 ppm of methylene blue dye was prepared by adding 0.025g of methylene blue into 250 cm3 of water using the equation below.

Where;

Mass of MB = 0.025 g

Volume of solution = 0.25 L

Stock concentration (ppm) = 100 ppm

## 3.5 ADSORPTION STUDIES

Batch adsorption experiments were conducted to evaluate the impact of initial concentration and contact time on the removal of methylene blue (MB). All adsorption experiments were conducted at room temperature. A stock solution of methylene blue dye was prepared by dissolving 0.025 g of powdered methylene blue in 250 cm³ of water, resulting in a concentration of 100 ppm (mg/L). Required concentrations for the experiments were achieved by diluting the stock solution with distilled water using the equation C1V1 = C2V2.

The effects of contact time (ranging from 10 to 120 minutes) and initial concentration (ranging from 5 to 50 mg/L) on the removal of methylene blue were investigated. Each experiment was carried out by placing the contents in a beaker on a magnetic stirrer rotating at 180 rpm. After the specified contact time, samples were filtered using Whatman filter paper (40 µm size), and the residual concentration of methylene blue in the filtrate was measured to determine the adsorption capacity and removal efficiency.

### 3.5.1 DETERMINATION OF THE EFFECT OF INITIAL CONCENTRATION

Methylene blue solutions with concentrations of 5 ppm, 10 ppm, 15 ppm, 20 ppm, 25 ppm, and 50 ppm were prepared, each adjusted to a pH of 9. These solutions, totaling 10 mL each, were transferred into separate 100 mL beakers. To each beaker, 0.04 g of the adsorbent material was added. The mixtures were then stirred using a magnetic stirrer at a constant speed for 10 minutes to ensure thorough mixing and interaction between the adsorbent and methylene blue.

After allowing the mixtures to equilibrate for a few minutes, they were filtered to separate the adsorbent from the solution. The resulting filtrates were then analyzed for percentage absorbance using a UV-Vis spectrophotometer at a wavelength of 664 nm. This measurement was carried out to assess the adsorption capacity of the adsorbent for different concentrations of methylene blue under the specified pH conditions.

### 3.5.2 DETERMINATION OF THE EFFECT OF CONTACT TIME

A 10 ppm methylene blue solution, with pH adjusted to 9, was dispensed into 100 mL beakers. Subsequently, 0.04 g of the adsorbent material was introduced into each beaker. The contact duration for each experiment was set at intervals of 10 minutes, specifically at 10 min, 30 min, 60 min, 90 min, and 120 min. Upon completion of each contact period, the mixtures underwent filtration, and the percentage absorbance of the resulting filtrates was determined using a UV-Vis spectrophotometer at a wavelength of 664 nm.

### 3.5.3 CALCULATION OF PERCENTAGE REMOVAL AND ADSORPTION CAPACITY

The methylene dye percentage, %R was measured by applying the equation below;

(1)

Where:

= initial concentration of the liquid phase of the dye in (mg/L)

= equilibrium concentration of the liquid phase of dye in (mg/L)

The adsorption capacity is given as:

(1)

Where:

(mg/g) = adsorption capacity

= initial concentration of the liquid phase of the dye in (mg/L)

= equilibrium concentration of the liquid phase of the dye in (mg/L)

V(L) = volume of the solution used for the adsorption

M (g) = the mass of the adsorbent used

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