RESEARCH PAPER

Green Synthesis of Aloe vera -Mediated ZnO Nanoparticles for Photocatalytic Degradation of Cationic and Anionic Dyes

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ABSTRACT

The present study reports the green synthesis of zinc oxide (ZnO) nanoparticles using Aloe barbadensis Miller extract as a sustainable and environmentally friendly approach. The synthesized nanoparticles were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDS) to investigate their structural and morphological properties. XRD analysis confirmed the formation of phase-pure hexagonal wurtzite ZnO nanoparticles with an average crystallite size of 53.78 nm. SEM micrographs revealed the morphological characteristics of the synthesized nanoparticles, while EDS analysis verified the presence of zinc and oxygen as the major constituent elements.

The photocatalytic activity of the green synthesized ZnO nanoparticles was evaluated using methylene blue (MB), a cationic dye, and methyl orange (MO), an anionic dye, under UV irradiation. The effects of operational parameters, including initial dye concentration, catalyst dosage, irradiation time, and pH, on MB degradation were systematically investigated. The degradation efficiency was found to be strongly influenced by these parameters, with enhanced photocatalytic performance observed under optimized conditions. The synthesized ZnO nanoparticles also exhibited remarkable activity toward methyl orange degradation, achieving 95% degradation within 40 min of UV irradiation.

The comparative degradation of cationic and anionic dyes demonstrated the effectiveness of the Aloe vera-mediated ZnO nanoparticles as photocatalysts for the treatment of dye-contaminated wastewater. The high degradation efficiencies obtained for both MB and MO indicate the potential of the synthesized ZnO nanoparticles as sustainable and efficient photocatalytic materials for environmental remediation applications.

Keywords: Green synthesis, ZnO nanoparticles, Photocatalysis, Methylene Blue, Methyl Orange, Wastewater treatment

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1. INTRODUCTION

Increasing industrial and urban activities have accelerated the release of contaminants into aquatic environments, making water pollution one of the most pressing environmental issues worldwide. The continuous discharge of organic and inorganic contaminants into aquatic ecosystems has led to significant deterioration of water quality, creating an urgent need for efficient and sustainable wastewater treatment technologies[1,2,10]. Among various pollutants, synthetic dyes are particularly problematic due to their complex molecular structures, high chemical stability, and resistance to biodegradation[3,7]. Dyes such as Methylene blue (MB) and Methyl orange (MO), widely used in textile, paper, leather, cosmetic, and printing industries, can adversely affect aquatic ecosystems and human health because of their toxic, mutagenic, and carcinogenic nature[3,7]. Therefore, the effective removal of dye pollutants from wastewater remains a significant environmental challenge.

Conventional wastewater treatment methods, including adsorption, coagulation flocculation, biological treatment, membrane filtration, and chemical oxidation, are often associated with limitations such as incomplete pollutant removal, generation of secondary waste, high operational costs, and reduced effectiveness against recalcitrant organic compounds[1,2,10]. Consequently, advanced treatment technologies based on nanomaterials and advanced oxidation processes have attracted considerable attention for environmental remediation applications[1,2,11,18].

Among various nanomaterials, Semiconductor metal oxides are extensively investigated for wastewater remediation because of their favorable catalytic properties, stability, and large reactive surface area[4,8,18]. Semiconductor photocatalysts such as TiO_2, ZnO, Fe_2O_3, SnO_2, and WO_3 can generate electron–hole pairs under light irradiation, producing highly reactive oxygen species capable of degrading organic contaminants into less harmful products[7,8,12-17]. Among these materials, ZnO has received particular attention because of its wide band gap, high exciton binding energy, low toxicity, environmental compatibility, and excellent photocatalytic performance[4,7]. These characteristics make ZnO nanoparticles highly attractive for dye degradation and wastewater treatment applications. Although ZnO nanoparticles can be synthesized using conventional methods such as sol gel, hydrothermal, precipitation, and combustion techniques, these approaches often involve toxic chemicals and high energy consumption[2,8]. To overcome these limitations, green synthesis has emerged as an environmentally friendly alternative that utilizes biological resources as reducing and stabilizing agents[2]. Plant-mediated synthesis is especially attractive due to its simplicity, low cost, scalability, and sustainability. Among various plant extracts, Aloe vera has gained considerable attention because of its rich phytochemical composition, which facilitates nanoparticle formation and stabilization. Aloe vera-mediated ZnO nanoparticles have been reported to exhibit enhanced crystallinity, reduced agglomeration, and improved photocatalytic activity compared with conventionally synthesized counterparts[9].

Photo catalysis is considered one of the most effective advanced oxidation processes for wastewater treatment because it enables efficient degradation of organic pollutants under mild operating conditions[12-17]. Exposure to light excites electrons from the valence band to the conduction band, creating charge carriers that participate in oxidation reduction reactions[13-17]. Despite extensive research on ZnO-based photo catalysts, most studies have focused on the degradation of individual dyes, while comparative investigations involving both cationic and anionic dyes remain limited. Since dye charge significantly influences adsorption behavior, surface interactions, and degradation efficiency, evaluating both dye classes is essential for assessing the practical applicability of photocatalysts in wastewater treatment[9].

Therefore, the present study aims to synthesize ZnO nanoparticles via an environmentally friendly Aloe vera-assisted green synthesis route and evaluate their photocatalytic performance toward Methylene blue (MB) and Methyl orange (MO), representing cationic and anionic dyes, respectively. Particular emphasis is placed on comparing the degradation behaviour of oppositely charged dyes using the same photocatalytic system, thereby providing insight into the influence of dye charge on photocatalytic efficiency. The synthesized nanoparticles were characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDS), while the effects of catalyst dosage, initial dye concentration, irradiation time, and solution pH on photocatalytic activity were systematically investigated. The findings contribute to the development of sustainable ZnO-based photocatalysts for the treatment of dye-contaminated wastewater.

2. MATERIALS AND METHODS

2.1 Materials

Zinc nitrate hexahydrate, ammonia, ethanol, Methylene blue, Methyl orange from standard commercial sources. All chemicals were analytical grade .So employed directly without further purification. Throughout the synthesis, washing, photocatalytic degradation procedures, deionized water was utilized. Aloe barbadensis Miller (Aloe vera) leaves were collected from local vendors.

2.2 Methods

2.2.1 Green Synthesis of ZnO Nanoparticles

A fresh and mature Aloe Vera leaves are harvested from the plant, washed thoroughly with distilled water to remove dust. The outer green skin was peeled out to obtain the clear inner gel approximately 50 g which was taken in 100 ml of deionized water, blended it and warmed the mixture at 80 o C for 1 h to activate the enzymes. The mixture was cooled down to room temperature, filtered over Whatman paper No. 1 filter paper to remove unwanted fibrous material. The clear filtrate was used for synthesis of ZnO nanoparticles.

2.2.2 Preparation of ZnO nano particles

A mixture of Zinc nitrate hexahydrate (18.2g) was dissolved in distilled water (150 ml) and followed by 40 ml of aloe vera extract and stirred solution for 15 min. The pH of the reaction mixture was adjusted to 10-11 by 25% ammonia solution and the mixture was stirred at 80 ^{0}C for 6 h. A mixture was cooled to RT, filtered, washed with water thoroughly and finally by ethanol to obtain off-white solids which were dried under high vacuum at 100 ^{0}C for 12 h and calcined at 600 ^{0}C for 4 h.

2.2.3 Preparation of stock solution

1000 mg/L stock solution of Cationic dye (Methylene blue-MB) and anionic dye (Methyl orange) were prepared in deionized water separately. Aliquots of stock solution were transferred and diluted with deionized water to prepare 10, 20, 30, 40, 50 mg/L standard concentrations of dyes.

2.2.4 Photo catalytic Degradation using green synthesized ZnO

2.2.4.1 Degradation of cationic and anionic dyes

The photocatalytic degradation experiments were carried out at room temperature using green synthesized ZnO nanoparticles as the photo catalyst. A series of experiments were conducted to investigate the effects of initial dye concentration, catalyst dosage, irradiation time, and solution pH on the degradation efficiency of methylene blue (MB). For another batch of experiments in degradation of methyl orange (MO), 20mg/L concentration of MO at different irradiation time and catalyst dosage were studied. For every experiment, 100 mL of dye solution with the desired concentration was transferred into the reaction vessel, followed by the addition of a predetermined amount of ZnO photo catalyst.

The pH of the dye solution was adjusted to the required value using dilute hydrochloric acid (HCl) or sodium hydroxide (NaOH) solutions. Prior to irradiation, the suspension was magnetically stirred in the dark for 30 min to set up adsorption desorption equilibrium between the dye molecules and the photocatalyst surface. This step ensured that any subsequent decrease in dye concentration was primarily due to photocatalytic degradation rather than adsorption.

The photocatalytic reactions were conducted in a closed chamber equipped with a 150 W UV lamp as the irradiation source. During the experiment, aliquots of the reaction mixture were withdrawn at regular intervals of 10 min, including samples collected before irradiation and throughout the irradiation period.

The collected samples were centrifuged at nearly 3000 rpm for 20 minutes to separate the ZnO nanoparticles from the solution. The clear supernatant obtained after centrifugation was analysed using a UV Visible spectrophotometer. The residual concentration of methylene blue and methyl orange was determined by monitoring their characteristic absorption maxima at 664 nm and 464 nm, respectively.The degradation efficiency of dyes was calculated using the following equation:

$$\% \text{ degradation efficiency} = \frac{C_0 - C_t}{C_0} \times 100$$

Eq.1.1

$$\% \text{ degradation efficiency} = \frac{A_0 - A_t}{A_0} \times 100$$

Eq.1.2

Where C_0, C_t are initial concentrations (mg/L) and initial absorption of Dyes and A_0, A_t are represents the dyes concentration (mg/L) and absorption after a certain irradiation time (t).

3. Results and Discussion

3.1. Characterization of green synthesized ZnO

3.1.1 XRD Analysis

The crystalline structure and phase purity of the green synthesized ZnO nanoparticles were investigated using X-ray diffraction (XRD) analysis. The diffraction patterns were recorded using an X-ray diffractometer, Empyrean model equipped with Cu-Kα radiation (λ = 1.5406 Å) over an appropriate 2θ range at room temperature. The XRD spectrum of green synthesized ZnO is shown in Fig.1.The obtained diffraction peaks were compared with standard reference data to confirm the formation of ZnO nanoparticles and to identify any secondary phases or impurities. The average crystallite size of the synthesized nanoparticles was estimated as 53.78nm using the Debye Scherer equation. (Eq.2)

$$D = \frac{0.9\lambda}{\beta \cos\theta}$$

Eq.2

Where D is an average size of crystallite; β is full width at half maximum of the peak in radians; λ is wavelength of X-ray; θ is Bragg's angle.

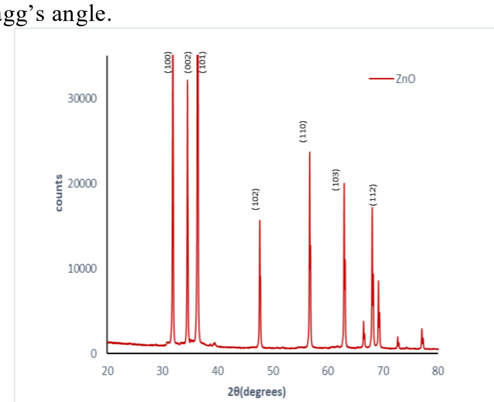

Fig.1.XRD of Green synthesized ZnO

3.2 SEM with EDS Analysis

Scanning electron microscopy (SEM) was utilized to examine the morphology and surface architecture of the green synthesized ZnO nanoparticles using Field Emission Gun Scanning Electron Microscope (FEGSEM) with EDS, JSM7600 F model at 5 kV operating voltage. The obtained micrographs were analysed to assess particle distribution, shape, and agglomeration behavior. (Fig.2)

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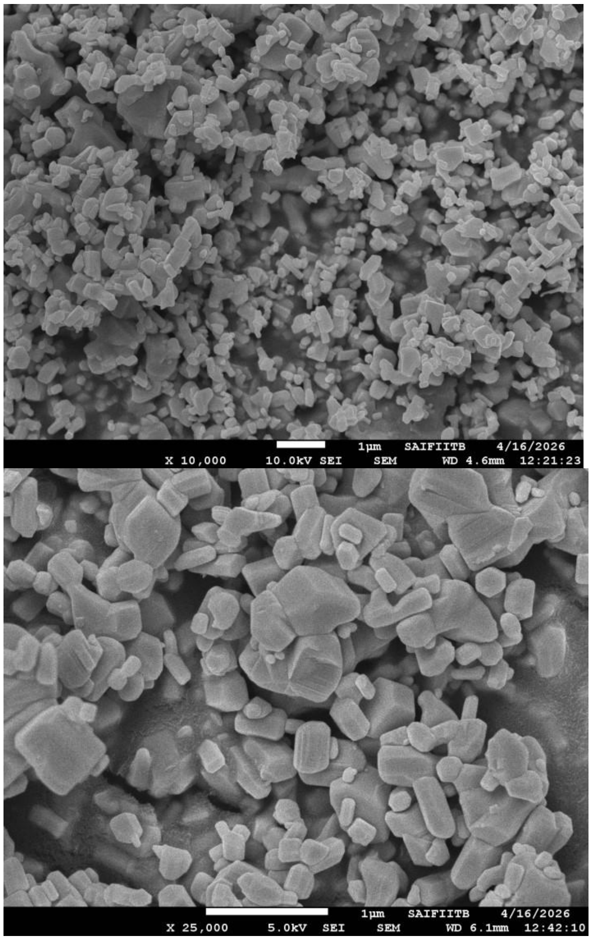


Fig.2.SEM images of Green synthesized ZnO

Elemental analysis was subsequently performed using energy-dispersive X-ray spectroscopy (EDS), which confirmed the predominant presence of zinc and oxygen in the sample, indicating the successful formation of ZnO nanoparticles with minimal impurity content. (Fig.3)

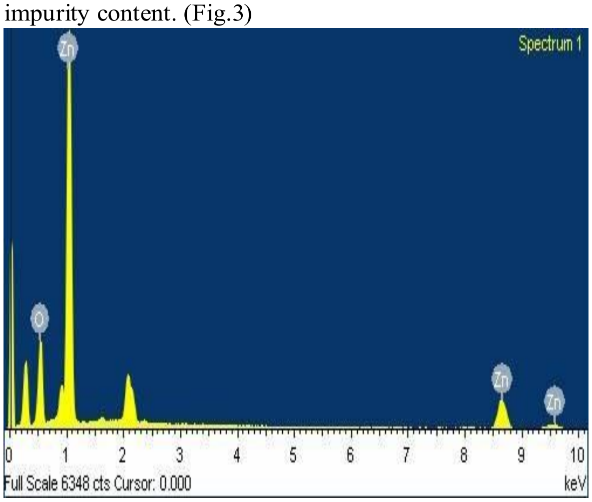
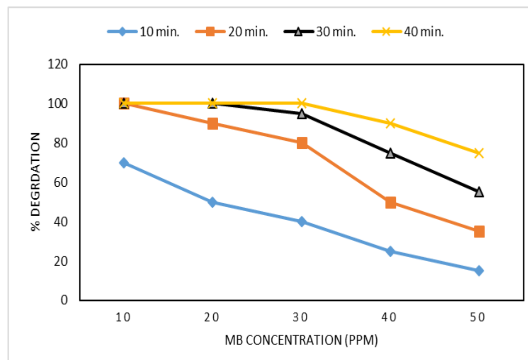


Fig.3.EDS of green synthesized ZnO

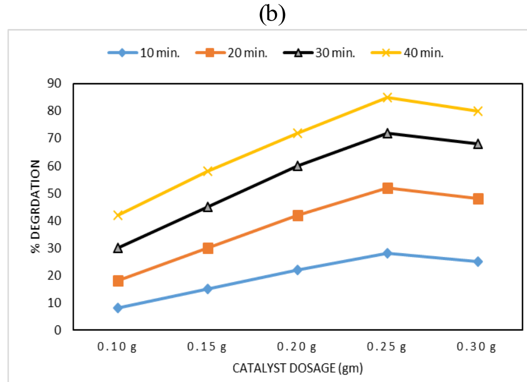
3.3 Photocatalytic Degradation of Methylene Blue: Effect of Dye concentration, Catalyst dosage and pH

The photocatalytic activity of the green synthesized ZnO nanoparticles was evaluated on cationic dye (MB) under UV irradiation. To investigate the effect of the initial dye concentration, MB solutions with concentrations ranging from 10 to 50 ppm were treated in the presence of a fixed synthesized ZnO dosage of 0.25 g. During the degradation process, aliquots were withdrawn at 10 min intervals up to 40 min and analysed using a UV Visible spectrophotometer. The degradation efficiency was found to vary with the initial dye concentration, with lower concentrations generally exhibiting higher degradation percentages due to the enhanced availability of active sites and improved penetration of UV light into the reaction medium. Fig.4(a)

(a)



(b)



(c)

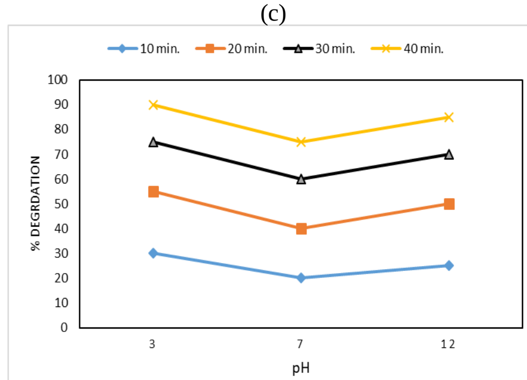


Fig.4.Percentage degradation with respect to (a) MB concentration (b) ZnO catalyst dosage (c) pH at constant intervals of time

The influence of catalyst loading on the photocatalytic degradation efficiency was observed by varying the ZnO dosage from 0.10 to 0.30 g while maintaining the MB concentration at 10 ppm. Samples were collected every 10 min during the 40 min irradiation period and analysed spectrophotometrically. An increase in degradation efficiency was observed with increasing catalyst dosage up to an optimum level, which can be attributed to the larger number of photo catalytically active sites and greater generation of reactive oxygen species. At higher catalyst loadings, the improvement in degradation efficiency became less pronounced due to possible particle aggregation and reduced light penetration caused by excessive catalyst concentration. Fig.4(b).

The effect of pH on the photocatalytic degradation of methylene blue was also investigated. In these experiments, the ZnO dosage was fixed at 0.25 g and the initial dye concentration was maintained at 10 ppm. The pH of the solution was adjusted to the desired value prior to irradiation. Aliquots were collected at every 10 min intervals to maintain the uniformity for all the batch of experiments up to 40 min. and analyzed using UV Visible spectroscopy to determine the residual dye concentration. The degradation efficiency was strongly influenced by the pH of the reaction medium, indicating that the surface properties of ZnO nanoparticles and the formation of reactive oxidative species are dependent on solution pH.Fig. 4(c).

The results obtained from these studies demonstrate that the photocatalytic degradation of methylene blue is governed by several operational parameters, including initial dye concentration, catalyst loading, and solution pH. Optimization of these factors is essential for achieving maximum degradation efficiency and improving the overall photocatalytic performance of the Aloe vera-mediated ZnO nanoparticles.

3.4 Photocatalytic Degradation of Methyl Orange

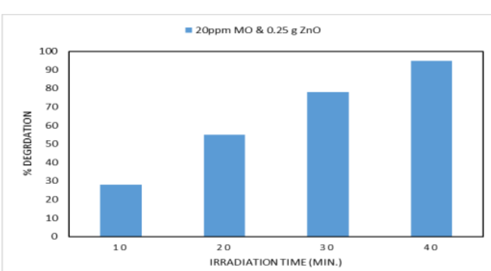


Fig.5 Percentage degradation of MO (20 ppm) with 0.25 g ZnO catalyst at various irradiation time.

The photocatalytic degradation of anionic dye (MO) was investigated using green synthesized ZnO nano catalyst. The experiments were executed at the dye concentration of 20 ppm with 0.25g catalyst loading. The degradation efficiency increased steadily with irradiation time, indicating the effective photocatalytic activity of the synthesized ZnO nanoparticles after 40 min. of UV exposure.Fig.5.

The efficacy in degradation within a relatively short irradiation period demonstrates the potential of Aloe vera-mediated ZnO nanoparticles as efficient photo catalysts for the treatment of dye-containing wastewater. The observations further confirm the applicability of the green synthesized ZnO nanoparticles for the removal of both cationic and anionic dye pollutants.

4. CONCLUSION

Green synthesized ZnO nanoparticles were successfully prepared using Aloe vera extract through an environmentally friendly and sustainable approach. Structural characterization by X-ray diffraction (XRD) confirmed the formation of highly crystalline hexagonal wurtzite ZnO nanoparticles with an average crystallite size of 53.78 nm. The morphology and elemental composition of the synthesized nanoparticles were further verified by scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS), confirming the successful formation of ZnO nanoparticles.

The synthesized ZnO nanoparticles exhibited excellent photocatalytic performance toward the degradation of both methylene blue (MB) and methyl orange (MO) under UV irradiation. The degradation efficiency of MB was strongly influenced by operational parameters such as dye concentration, catalyst dosage, irradiation time, and solution pH, highlighting the importance of process optimization for achieving enhanced photocatalytic activity. The photocatalyst also demonstrated remarkable efficiency toward the degradation of the anionic dye MO, achieving 95% degradation within 40 min of irradiation.

The comparative degradation study of cationic and anionic dyes revealed the effectiveness of Aloe vera-mediated ZnO nanoparticles for the treatment of dye-contaminated wastewater. The high photocatalytic activity can be attributed to the crystalline nature of ZnO, efficient generation of reactive oxygen species, and the availability of active surface sites that promote pollutant degradation. Overall, the findings demonstrate that green synthesized ZnO nanoparticles are sustainable, cost-effective, and efficient photocatalytic materials with significant potential for environmental remediation and wastewater treatment applications.

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