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Electrochemical Synthesis of Microwave-Assisted Zinc Nanoparticles for Photocatalytic Degradation of Phenolic Compounds

1st Muhammad Farhan Hanafi
Universiti Kuala Lumpur Branch Campus Malaysian Institute of Chemical and Bioengineering Technology (UniKL MICET), Lot 1988 Vendor City, Taboh Naning, 78000 Alor Gajah, Melaka, Malaysia
farhanhanafi@unikl.edu.my

2nd Norzahir Sapawe
Universiti Kuala Lumpur Branch Campus Malaysian Institute of Chemical and Bioengineering Technology (UniKL MICET), Lot 1988 Vendor City, Taboh Naning, 78000 Alor Gajah, Melaka, Malaysia
norzahir@unikl.edu.my

***Abstract*— This study explores the electrochemical synthesis of zinc nanoparticles using microwave-assisted technology for the photodegradation of phenolic compounds in aqueous solutions. The electrochemical method offers a simple, efficient approach for synthesizing zinc nanoparticles with enhanced catalytic properties. The microwave-assisted process significantly improved the nanoparticles' surface area, pore structure, and stability. The synthesized of mesoporous electrogenerated zinc nanoparticles (denoted MsEgZn) demonstrated excellent photocatalytic performance, achieving a 97% degradation of phenol within 60 minutes under optimal conditions. This approach offers a sustainable, cost-effective solution for removing toxic phenolic pollutants from water, with potential applications in environmental remediation.**

Keywords*— **mesoporous zinc nanoparticle catalyst; phenol; photocatalytic degradation; visible light irradiation; wastewater treatment***

I. INTRODUCTION

Phenolic compounds and their derivatives are among the most hazardous pollutants in aquatic environments, originating from a wide range of industrial activities [1-3]. Examples of industrial sources include petrochemical production, pulp and paper manufacturing, pharmaceuticals, textiles, dye industries, plastics, leather tanning, fiber additives, the oil industry, and phenolic resin manufacturing [1,4,5]. These pollutants pose a severe environmental threat due to their toxicity, carcinogenic properties, and resistance to natural degradation processes [1,6,7]. Even at low concentrations, phenolic compounds can disrupt aquatic ecosystems and harm human health when consumed or absorbed indirectly [8-10]. Addressing this issue requires effective and sustainable strategies to degrade and eliminate these pollutants from water sources.

Conventional methods such as adsorption, chemical oxidation, and biological treatment have been widely employed to address phenolic compound contamination [6,9,11,12]. However, these methods often face critical limitations, including high operational costs, the generation of secondary waste, and inefficiencies in treating low concentrations of phenolics. These challenges underscore the need for innovative and eco-friendly technologies capable of providing more effective and sustainable solutions. Among these, semiconductor photocatalysis has gained significant attention as a promising approach for the degradation of organic pollutants, including phenolic compounds, under ambient conditions [11,13,14].

Zinc-based materials have emerged as excellent candidates for photocatalysis due to their favorable properties, such as non-toxicity, high natural abundance, tunable electronic structure, and ability to generate reactive oxygen species (ROS) under light irradiation [12,15]. However, the photocatalytic efficiency of zinc-based materials heavily depends on factors such as particle size, surface area, porosity, and crystalline structure. Researchers have focused on synthesizing zinc nanoparticles with mesoporous structures to enhance their performance. Mesoporous zinc nanoparticles offer a large surface area and a highly accessible network of pores, which facilitate pollutant adsorption and catalytic reactions [16-18].

Among the various synthesis techniques, microwave-assisted synthesis has gained attention for its ability to

produce nanomaterials with uniform size, controlled morphology, and enhanced catalytic activity [19, 20, 21]. Microwave heating provides rapid, energy-efficient processing by delivering uniform heat distribution and minimizing the formation of undesirable by-products. Additionally, it is considered a green and scalable technique, aligning with the principles of sustainable chemistry. Despite these advantages, the application of microwave-synthesized mesoporous zinc nanoparticles for the degradation of phenolic compounds has yet to be extensively explored [21-23].

This study reports the successful synthesis of mesoporous zinc nanoparticles using a microwave-assisted method and investigates their photocatalytic performance in degrading phenolic compounds under simulated environmental conditions. The nanoparticles were characterized using various techniques to determine their structural, morphological, and optical properties. These analyses provided insights into the material's suitability for photocatalytic applications, particularly for addressing water pollution challenges.

The photocatalytic activity of the synthesized nanoparticles was evaluated by monitoring the degradation of phenolic pollutants under visible light irradiation. The experimental setup simulated real-world environmental conditions to assess the practical applicability of the materials. The degradation efficiency and reaction kinetics were analyzed to determine the nanoparticles' effectiveness in removing phenolic compounds from water.

The results of this study demonstrate the potential of microwave-synthesized mesoporous zinc nanoparticles (MsEgZn) as a cost-effective and environmentally friendly solution for water pollution remediation. The findings highlight the significance of structural and morphological optimization in enhancing photocatalytic performance. This work contributes to the broader goal of developing advanced materials for sustainable wastewater treatment.

In conclusion, the integration of microwave-assisted synthesis and mesoporous zinc nanoparticles offers a promising approach to tackling the persistent challenge of phenolic compound contamination in aquatic environments. By addressing key limitations of conventional methods, this study lays the groundwork for further exploration and application of advanced photocatalytic materials in environmental remediation efforts.

II. METHOD

Chemicals and Materials

The chemicals utilized in this study include *N*,*N*-dimethylformamide (DMF), procured from Merck; naphthalene and tetraethylammonium bromide solution, obtained from Fluka; and acetone and methanol, purchased from HmbG Chemical. Sodium hydroxide (NaOH), hydrochloric acid (HCl), and phenol were sourced from QReCTM. Additionally, a platinum (Pt) plate and a zinc (Zn) plate cell, both of >99.9% purity, were acquired from Nilaco Metal, Japan. All chemicals used in this study were of analytical grade and were applied without further purification to ensure consistency and reliability in experimental outcomes. Deionised water served as the base medium for all solution preparations. The pH of the solutions was meticulously adjusted using 0.1 M HCl and 0.1 M NaOH solutions, ensuring precise control for experimental accuracy and reproducibility.

Instrumentation

The catalysts were synthesized using an electrolysis process, adhering to a previously established protocol [24-26]. For the electrolysis procedure, a GW Instek power supply, model GPS 3030 DO, was employed to deliver a stable and adjustable power output. Microwave irradiation was applied during the synthesis process using a Samsung Microwave, model ME711K Black 20L, capable of delivering wave power ranging from 0 to 900 watts. Following the synthesis, the catalysts were oven-dried overnight at controlled conditions to ensure proper dehydration and stability. Subsequently, the dried catalysts underwent calcination in a muffle furnace (B180 Nabertherm) to enhance their structural and catalytic properties by improving crystallinity and removing residual organic matter. The effectiveness of the catalysts was assessed by monitoring the degradation of phenol, with absorbance measurements taken using a UV–Visible Spectrophotometer (Shimadzu UV–1900i model). This instrument provided precise and reliable data on phenol degradation, ensuring the accuracy of the photocatalytic performance evaluation.

Measurement and Evaluation of Photocatalytic Activity

The photocatalytic activity of the synthesized catalysts was evaluated by testing their ability to degrade phenol in aqueous solutions. For each experiment, 0.250 g of the catalyst was dispersed into 200 mL of a 10 mg L^{-1} phenol solution. Prior to light exposure, the system was kept under dark conditions for 1 hour to achieve adsorption-desorption equilibrium between the catalyst surface and phenol molecules. Photocatalytic degradation was conducted at room temperature for 2 hours under continuous stirring, using a standard fluorescent light source (Philip TLD 36W/865, lifespan 15,000 hours, color temperature 6500 K, and emission range 420–520 nm). During the reaction, aliquots of 2.5 mL were collected at specific time intervals. These samples were immediately centrifuged using a Hettich Zentrifugen Micro 120 centrifuge operating at 75,000 rpm to remove suspended catalyst particles. The phenol concentration in the supernatant was analyzed using a UV-Vis Spectrophotometer (Shimadzu UV–1900i model) by measuring the absorbance at the characteristic wavelength of phenol (270 nm). This approach ensured precise monitoring of phenol degradation and provided reliable data to assess the photocatalytic performance of the catalysts.

A Simple Batch Photoreactor System

A simple batch photoreactor system was designed and utilized for testing the photocatalytic activity of the synthesized catalysts. The system comprised a fluorescent lamp (Philip TLD 36W/865, lifespan 15,000 hours, color temperature 6500 K, emission range 420–520 nm) mounted at a fixed height of 15 cm above the reaction solutions. A magnetic stirrer was employed to ensure uniform dispersion of the catalyst within the solution throughout the reaction. Photocatalytic experiments were conducted in batches using 200 mL Pyrex conical flasks placed on the magnetic stirrer. To optimize light radiation, the entire setup was enclosed within a specially designed box. The interior walls of the box were lined with aluminum foil, which served to reflect and concentrate light radiation back into the solution, thereby maximizing the exposure of the catalyst to the emitted light. The reaction temperature was monitored continuously using a thermometer inserted into the Pyrex conical flasks. This setup ensured consistent and reproducible experimental conditions, facilitating reliable evaluation of the photocatalytic performance of the catalysts.

Determination of Phenol Concentration

To determine the concentration of phenol during the photocatalytic degradation process, a UV-Vis spectrophotometer (Shimadzu UV–1900i) was employed, targeting the characteristic absorption peak of phenol at 270 nm. At predetermined time intervals, 2.5 mL of the reaction mixture was carefully withdrawn and centrifuged using a Hettich Zentrifugen Micro 120 centrifuge at 75,000 rpm. This step ensured the removal of catalyst particles from the solution, allowing for accurate measurement of the phenol concentration in the supernatant. The percentage degradation of phenol was calculated using the following formula:

$$\text{Degradation (\%)} = (Co\text{-}Ci)/Co \times 100\% \qquad \text{(Equation 1)}$$

where Co represents the initial concentration (mg L^{-1}) and Ct denotes a variable concentration (mg L^{-1}).

This approach provided a quantitative evaluation of the catalyst's photocatalytic efficiency in degrading phenol under the experimental conditions.

III. RESULTS AND DISCUSSION

The degradation of phenol under optimal conditions was analyzed using three different catalysts: ZnO-commercial, TiO_2-commercial, and the synthesized catalyst MsEgZn. Photolysis, which involves the direct exposure of phenol solution to light without a catalyst, was also included as a control to determine the baseline degradation efficiency. According to Figure 1 and Table 1, the results indicate that the presence of a photocatalyst significantly enhances the degradation of phenol compared to photolysis alone, underscoring the importance of catalysts in promoting the breakdown of organic pollutants under light irradiation.

Among the commercial catalysts, ZnO and TiO_2 exhibited substantial photocatalytic activity, with ZnO achieving approximately 66% degradation of phenol and TiO_2 slightly lower at 54%. The enhanced performance of ZnO can be attributed to its superior light absorption properties and its ability to generate reactive oxygen species more efficiently under light exposure. However, both catalysts showed minimal activity in the dark, emphasizing their dependence on light energy to drive the photocatalytic reaction. This highlights the importance of optimizing light conditions when utilizing such catalysts for pollutant removal [24-26].

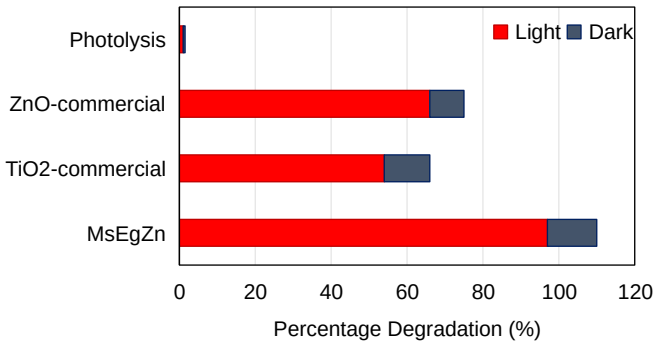

FIGURE I. Performance studies on phenol removal (pH = 3, catalyst dosage = 1.25 g/L, concentration = 10 ppm, time = 60 mins, temperature = 303 K)

The synthesized catalyst MsEgZn outperformed both ZnO and TiO_2, achieving over 97% degradation of phenol under light conditions. This exceptional efficiency can be attributed to its advanced physicochemical properties, such as an increased surface area, enhanced light absorption, and a higher density of active catalytic sites. Interestingly, MsEgZn also demonstrated notable degradation activity in dark conditions, indicating its strong adsorption capacity and potential intrinsic catalytic activity. This dual functionality makes MsEgZn particularly promising for hybrid systems that combine photocatalysis and adsorption for enhanced pollutant removal [24-26].

TABLE I Percentage removal of phenol in varying type of catalysts

Type of Catalyst	Degradation (%)	
	Light	Dark
MsEgZn	97	13
TiO_2-commercial	54	12
ZnO-commercial	66	9
Photolysis	1	0.5

In contrast, photolysis alone resulted in negligible phenol degradation, confirming that light exposure without a catalyst is insufficient for efficient pollutant breakdown. This reinforces the critical role of photocatalysts in water treatment, as they facilitate the generation of reactive species necessary for the decomposition of organic contaminants. The negligible activity

observed in the photolysis control further underscores the potential of advanced catalysts to harness light energy and significantly accelerate reaction kinetics [24-26].

The findings of this study highlight the superior performance of the synthesized MsEgZn catalyst compared to commercial alternatives, making it a promising candidate for industrial-scale water treatment applications. Its high photocatalytic efficiency under light conditions and significant activity in the dark offer considerable flexibility for various operational environments. Future research should focus on evaluating the long-term stability, reusability, and cost-effectiveness of MsEgZn to confirm its feasibility for real-world applications and to optimize its deployment in large-scale pollutant removal systems.

IV. CONCLUSIONS

In this study, ZnO nanoparticle catalysts were successfully synthesized using a straightforward electrogeneration method in combination with microwave-assisted technology for the photodegradation of phenol in aqueous solutions. The catalyst preparation process was designed to optimize the reaction efficiency, leveraging the unique properties of ZnO nanoparticles for environmental applications. The microwave-assisted synthesis was particularly effective, leading to an improved catalyst structure with enhanced surface area and reactivity. Importantly, the synthesized MsEgZn nanoparticles demonstrated a high degree of efficacy in the degradation of phenol, a toxic organic pollutant, without producing any harmful or secondary pollutants, which is a critical factor for maintaining environmental sustainability in water treatment applications.

The experimental results showed that the prepared electrogenerated microwave-assited ZnO catalyst exhibited the highest catalytic performance, achieving a remarkable 97% phenol degradation within just 60 minutes of reaction time. This high degradation rate underscores the advantages of using microwave-assisted synthesis, which significantly enhances the catalyst's photodegradation efficiency. The method not only improves the degradation rate but also promotes key structural modifications in the ZnO nanoparticles. These modifications included an increase in pore size, better mechanical strength, and superior chemical stability, all of which contribute to the overall efficiency and longevity of the catalyst. The enhanced stability of the catalyst ensures its effective performance over multiple cycles, making it a reliable option for long-term applications in water purification.

Overall, the findings of this study highlight the potential of microwave-assisted synthesis for optimizing ZnO nanoparticle catalysts, offering a promising solution for environmental pollution control. The enhanced properties of the MsEgZn nanoparticles, including their high surface area, stability, and improved catalytic efficiency, position them as a highly effective tool for photodegradation processes. This study not only demonstrates the successful removal of phenol but also suggests that the developed catalysts could be adapted for the removal of other organic pollutants from water. By offering an efficient, cost-effective, and environmentally friendly solution for water treatment, the use of MsEgZn nanoparticles opens up new possibilities in addressing global water quality challenges.

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