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Reduction of Phenol Levels in Textile Wastewater Using ZnO-$NiFe_2O4$ Photocatalysts with Variations in Mass and Contact Time

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Abstract: The textile industry in Indonesia has an important role in the economy, but the presence of phenol is also one of the main causes of environmental pollution. Therefore, innovative solutions are needed to reduce this problem. By using Zinc Acetate Dihydrate you can obtain ZnO compounds, which are included in the category of photocatalyst compounds. ZnO has the potential as a catalyst in chemical reactions to break down organic and inorganic compounds that are harmful to the environment. Apart from that, this research also uses the $NiFe_2O_4$ compound synthesized from $NiCl_2$ and $FeCl_3$ as a doping agent which can reduce the ZnO band gap, thereby increasing the efficiency of photodegradation of dangerous compounds such as phenol. The research methods used consisted of ZnO synthesis and ZnO-$NiFe_2O_4$ synthesis and photocatalyst activation testing with variations in contact time of 0, 60, 120, 180 minutes and variations in the weight of the ZnO-NiFe2O4 photocatalyst of 0.5; 0.75; 1 gram. The characteristics of the resulting ZnO-Zeolite show a crystal size of 24.98 nm and absorption bands of Zn-O/Ni-O and Fe-O at wave numbers of 455 cm-1 and 580 cm-1. To determine the effectiveness of the ZnO-zeolite photocatalyst on textile liquid waste, pH, Chemical Oxygen Demand (COD), Total Suspended Solid (TSS) analysis and reduction in the concentration of phenolic compounds were carried out. Results Reduction of TSS value from 235 mg/L to 25.3 mg/L with a reduction percentage reaching 89.3% and degradation of phenol content from 3.625 mg/L to 0.067 mg/L which occurred at a mass variation of 0.75 grams and a contact time of 180 minutes.

Keywords: Phenol, Photocatalyst, Textile wastewater

1. Introduction

The textile industry is a major manufacturing sector contributing significantly to the national economy, particularly within the fashion-based creative industry. Statistics from the Ministry of Tourism and Creative Economy indicate that the fashion sub-sector accounts for approximately 55% of the total Gross Domestic Product (GDP) associated with Indonesia's creative industries. High levels of production activity simultaneously increase the volume of waste generated annually. It is estimated that Indonesia produces around 33 million tons of textiles each year, with approximately 1 million tons classified as textile waste, posing a potential risk of environmental pollution if not managed properly [1].

A common form of pollution associated with textile industrial activities is wastewater, which arises from washing, dyeing, and finishing processes involving various chemicals and synthetic dyes. This wastewater typically contains key pollutants such as synthetic dyes and organic compounds, the latter measured in terms of Chemical Oxygen Demand (COD). High COD levels result from the extensive use of dyes and chemicals during dyeing; since some dyes do not bond completely with the fibers, they are discharged into the wastewater. The complex and stable structures of these dyes make them resistant to biological degradation, thereby increasing both COD levels and color intensity in textile wastewater [2]. The presence of dyes in water bodies can inhibit light penetration and disrupt the photosynthesis of aquatic organisms, while also potentially exerting toxic effects on the aquatic environment [3].

Textile wastewater contains various hazardous organic compounds, including phenols. Phenols are toxic, relatively stable, and resistant to natural degradation. High phenol concentrations can disrupt aquatic ecosystem balance and degrade the quality of water used by the community. Furthermore, prolonged exposure to phenols can lead to health issues in humans, such as skin irritation, nervous system disorders, and organ damage [4]. Consequently, many countries have established maximum permissible limits for phenol content in textile wastewater. In Indonesia, the quality standard for textile wastewater stipulates a permissible phenol concentration of 0.5 mg/L [5]. This highlights the need for an effective treatment method to reduce its concentration in the wastewater before discharge into the environment.

Various conventional methods have been employed to address textile wastewater pollution, such as coagulation, flocculation, adsorption, and biological processes [6]. However, these methods have limitations, including low efficiency for recalcitrant dyes, high operational costs, and the generation of secondary sludge requiring further treatment. Furthermore, some textile dyes are resistant to conventional biodegradation, necessitating more effective alternative technologies [7].

Heterogeneous photocatalysis has emerged as a promising alternative technology for the degradation of organic pollutants in wastewater. This technology is capable of degrading organic pollutants into harmless products such as CO2 and H2O [8]. The photocatalysis process involves the activation of a semiconductor material by photon energy to generate electron-hole pairs, which subsequently form reactive radical species—primarily hydroxyl radicals (•OH)—that play a crucial role in the degradation process [9].

Zinc oxide (ZnO) has been widely used as a photocatalytic material due to its suitable band gap (3.35 eV), good chemical stability, non-toxicity, and relatively low cost [8]. ZnO materials exhibit high photocatalytic activity, achieving degradation rates of up to 99.4% for organic dyes [10]. However, ZnO has certain limitations: it is active only under UV irradiation due to its wide band gap, it suffers from a high electron-hole recombination rate, and it is difficult to separate from the medium after the photocatalytic process.

To overcome the limitations of ZnO, the development of composite materials by combining ZnO with other substances has become a focus of intensive research. Nickel ferrite (NiFe2O4) is an attractive magnetic material for combination with ZnO due to several advantages, including magnetic properties that facilitate catalyst separation and recovery, high chemical stability, and the ability to modify the composite's electronic properties [11]. Nickel ferrite-based composites have demonstrated high effectiveness in degrading various textile dyes, such as methylene blue, rhodamine B, methyl orange, and Congo red [11].

2. Materials and Methods

2.1 Tools and Materials

The tools and materials used in this study included a magnetic stirrer, a hot plate, thermometer, furnace, LED lamps, filter paper, crucible and glassware, while the required materials consisted of textile wastewater, zinc acetate dihydrate, Nickel (II) chloride, Ferric chloride and distilled water.

2.2 Research Flow Diagram

Synthesis of ZnO Nanoparticles

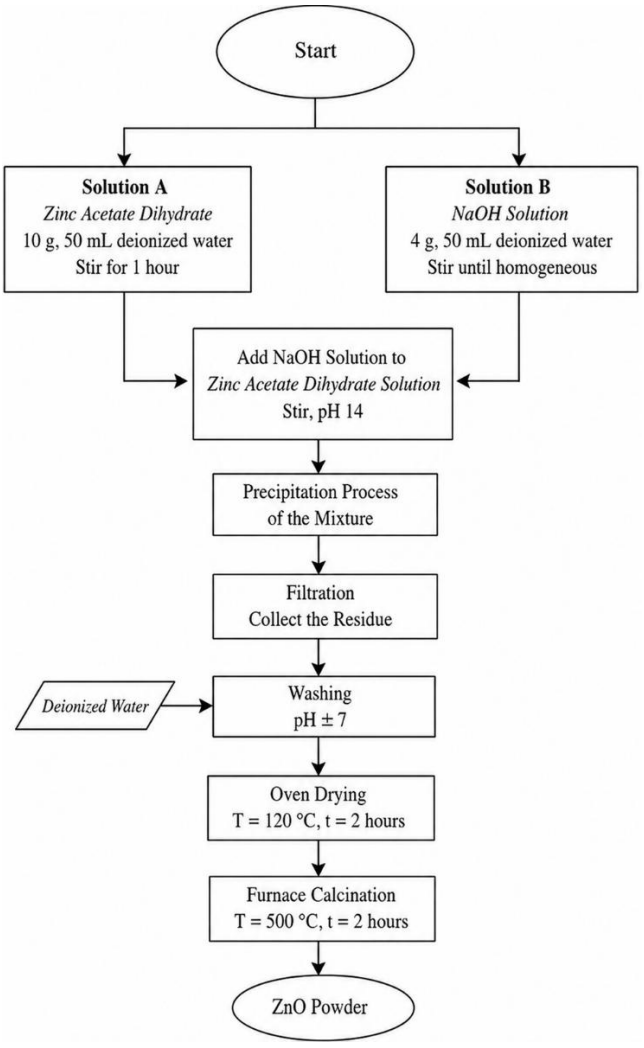

Figure 1. Synthesis of ZnO Nanoparticles

Synthesis of ZnO-$NiFe_2O_4$ Nanocomposite

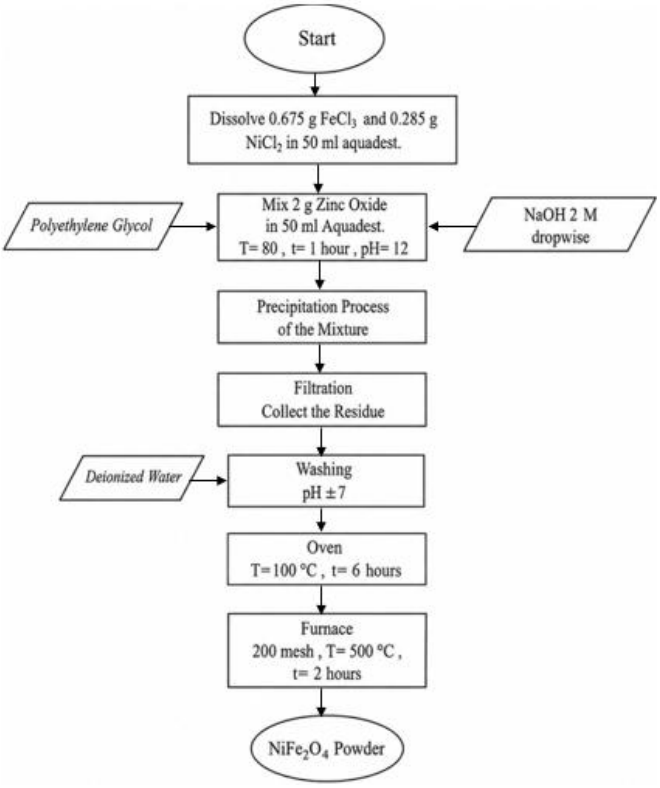

Figure 2. Synthesis of ZnO-$NiFe_2O_4$ Nanocomposite

Photocatalytic Activity Test

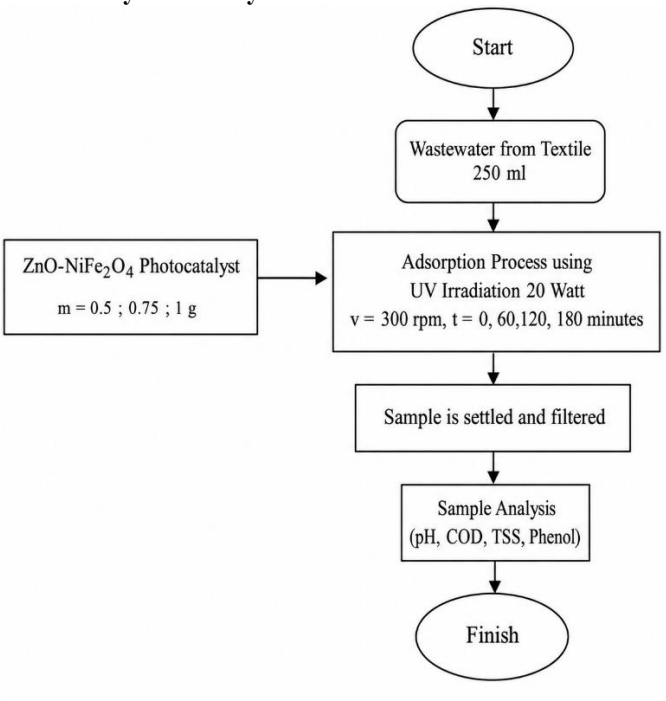

Figure 3. Photocatalytic Activity Test

2.3 Research Procedure

Preparation of Raw Materials

Prepare the raw materials to be used in the research—such as zinc acetate dihydrate, NaOH, $NiCl_2 \cdot 6H_2O$, $FeCl_3 \cdot 6H_2O$, and distilled water—for the synthesis of the ZnO-$NiFe_2O_4$ nanoparticle photocatalyst, as well as textile wastewater as the sample for phenol degradation.

Synthesis of ZnO Nanoparticles

Weigh 10 g of zinc acetate dihydrate ($Zn(CH_3COO)_2 \cdot 2H_2O$). Dissolve the zinc acetate dihydrate in 50 mL of deionized water in a 100 mL beaker. Stir the solution using a magnetic stirrer for 1 hour at room temperature (±25°C) until homogeneous. Weigh 4 g of sodium hydroxide (NaOH). Dissolve the NaOH in 50 mL of deionized water in a separate beaker. Stir until homogeneous. Add the NaOH solution dropwise to the zinc acetate solution while continuously stirring with the magnetic stirrer. Monitor the solution's pH, maintaining it at 14 throughout the stirring process. Continue stirring for 2 hours until a white, gel-like precipitate forms. Allow the mixture to stand overnight at room temperature for the precipitation process. Separate the precipitate from the solution via filtration or decantation. Wash the precipitate several times with deionized water until the pH of the wash water approaches neutral (±7) to remove residual ions or unreacted chemicals. Place the precipitate in a hot air oven at 120°C for 2 hours until dry. Grind the dry powder using an agate mortar until uniform. Perform calcination in a muffle furnace at 500°C for 2 hours to produce crystalline ZnO nanoparticles. Store the resulting ZnO in a sealed container for further characterization.

Synthesis of ZnO-NiFe2O4 Nanoparticles

A mixture was prepared by combining 2 g of ZnO and 1 g of PEG-4000 in 50 mL of distilled water. This mixture was stirred using a magnetic stirrer at 300 rpm and 80°C for 1 hour until a homogeneous suspension was formed. Subsequently, a solution containing 0.6758 g of FeCl3 and 0.2852 g of NiCl2 dissolved in 50 mL of distilled water was slowly added to the ZnO suspension while stirring until the mixture became homogeneous. A 2 M NaOH solution was added gradually to the mixture until the pH reached 12, resulting in the formation of a brown precipitate. The precipitate was separated and washed with deionized water until neutral pH was achieved, followed by washing with absolute alcohol to remove residual impurities. The resulting precipitate was dried in an oven at 100°C for 6 hours. The dried sample was then pulverized and sieved using a 200-mesh sieve. Finally, the sieved powder was calcined at 500°C for 2 hours to obtain ZnO-NiFe2O4 nanoparticles.

Photocatalytic Activity Test

Textile wastewater (250 mL) was placed into each of three beakers. The ZnO-NiFe2O4 photocatalyst was added to the textile wastewater at varying masses (0.5, 0.75, and 1.0 g). The samples were irradiated using a 20-watt UV lamp for varying durations (0, 60, 120, and 180 minutes) while being stirred at 600 rpm. Subsequently, the samples were allowed to settle, and the solid phase was separated from the filtrate. Water

quality parameters (pH, phenol, TSS, and COD) were then measured.

3. Result and Discussion

3.1 Analysis of the Characteristics of ZnO- NiFe2O4 Synthesis Results.

a. XRD analysis

Characteristics using XRD are carried out to determine the size of the crystallity of the ZnO-NiFe2O4 that has been synthesized so that the particle size and crystal structure of ZnO-NiFe2O4 can be known. This analysis was carried out at the FMIPA Laboratory at Sriwijaya University. The following are the results of the ZnO-NiFe2O4 test using XRD which can be seen in Figure 4.

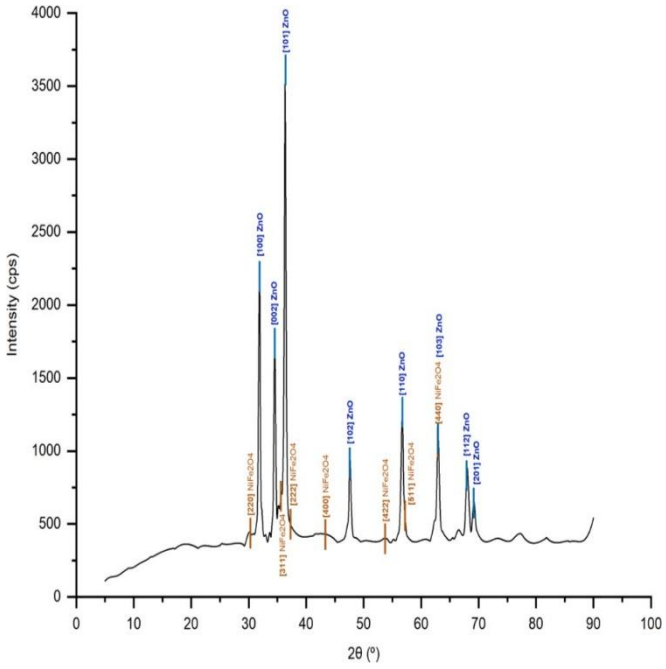

Figure 4. XRD test results

Based on the XRD diffraction pattern in Figure 4, several diffraction peaks are observed, indicating the presence of two main crystalline phases: ZnO and $NiFe_2O_4$. The primary diffraction peaks appear at 2θ angles of approximately 31.89°, 34.52°, 36.34°, 47.56°, 56.72°, 62.92°, 67.92°, and 69.16°. These peaks correspond to the characteristic crystal planes of hexagonal wurtzite-structured ZnO—specifically the (100), (002), (101), (102), (110), (103), (112), and (201) planes—in accordance with the Joint Committee on Powder Diffraction Standards (JCPDS) reference No. 36-1451 [12].

The presence of these peaks indicates that ZnO was successfully formed during the synthesis process. The highest intensity is observed at the 2θ peak of 36.34°, corresponding to the (101) plane, which suggests that the ZnO crystal growth orientation is predominantly along this plane. The high intensity of the diffraction peaks indicates that the resulting ZnO possesses a high degree of crystallinity. The wurtzite structure is commonly found in ZnO materials and is known for its high photocatalytic activity, attributed to semiconductor properties that enable the generation of electron-hole pairs upon exposure to ultraviolet radiation [12].

In addition to the ZnO phase, phase identification also reveals peaks corresponding to the cubic spinel-structured $NiFe_2O_4$ phase at 2θ diffraction angles of approximately 30.3°, 35.6°, 37.3°, 43.3°, and 53.7°, 57.2° and 62.9° correspond to the NiFe2O4 crystal planes (220), (311), (222), (400), (422), (511), and (440), referencing the JCPDS standard No. 10-0325 [13]. These peaks tend to exhibit lower intensity compared to those of ZnO, as the material is a composite. The presence of identified NiFe2O4 peaks alongside the ZnO peaks indicates that both phases are well-distributed and interact effectively within the composite. Furthermore, the absence of other sharp extraneous peaks (impurity phases) in the XRD diffractogram confirms the successful synthesis of the ZnO-NiFe2O4 nanocomposite with high purity.

b. FTIR Analysis

Characterization via Fourier Transform Infrared Spectroscopy (FTIR) was performed to identify the functional groups and chemical bonds present in the ZnO-$NiFe_2O_4$ composite material. FTIR analysis was used to identify bond vibrations appearing at specific wavenumbers, thereby providing information on the chemical structure of the formed material. The presence of characteristic Zn-O and Fe-O bonds serves as an indicator of the successful synthesis of the ZnO-$NiFe_2O_4$ composite. The results of the FTIR characterization, conducted at the Integrated UPA Laboratory of the University of Lampung, are shown in Figure 5.

The FTIR characterization analysis results shown in Figure 5 reveal several absorption bands indicating the presence of metal-oxygen bonds within the composite. An absorption band at a wavenumber of approximately 580 cm^{-1} corresponds to the stretching vibration of Fe-O bonds—a characteristic feature of ferrite materials—indicating the formation of the $NiFe_2O_4$ spinel structure. These results align with reports stating that Fe-O bonds in ferrites typically appear in the 550–600 cm^{-1} range; the Fe-O band signifies that iron ions have bonded with oxygen to form the ferrite crystal lattice [14].

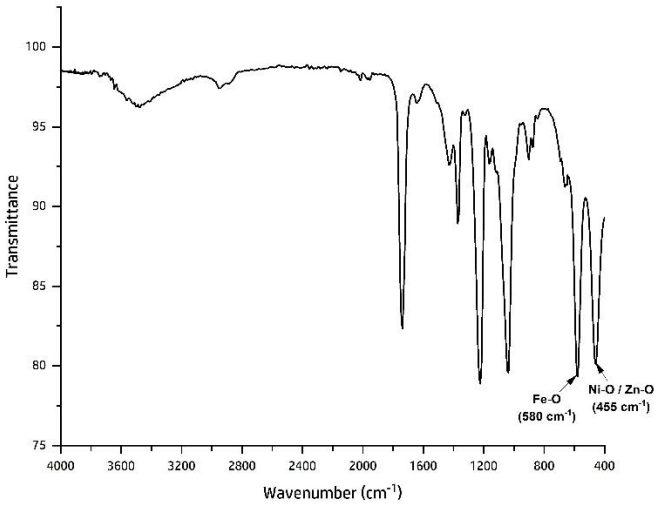

Figure 5. FT-IR test results

Furthermore, the absorption band at a wavenumber of approximately 455 cm^{-1} is attributed to the vibration of the Zn-O bond. Zn-O vibrations are typically observed in the 400–500 cm^{-1} range. This band is characteristic of ZnO and $NiFe_2O_4$ materials. The presence of this absorption band indicates that the synthesis process successfully established an interaction

between the ZnO and $NiFe_2O_4$ phases within the composite. Vibrations in this region generally arise from the stretching of metal-oxygen bonds within the metal oxide crystal structure.

Overall, the FTIR analysis results confirm the presence of Fe–O, Ni–O, and Zn–O bonds, which are key characteristics of the ZnO–$NiFe_2O_4$ nanocomposite. These results support the XRD characterization data, indicating the successful formation of ZnO and $NiFe_2O_4$ phases in the synthesized material.

3.2 Effect of Catalyst Mass and Contact Time on Wastewater pH

pH is a measure of the acidity or alkalinity of a solution, determined by the concentration of hydrogen ions (H^+) within it. Based on the pH analysis results shown in Figure 6—which examines variations in ZnO-$NiFe_2O_4$ photocatalyst mass and contact time during the degradation process—the initial pH of the textile wastewater prior to treatment was 8.3.

Figure 6 shows that, starting from an initial pH of 8.3, a decrease in pH occurred across all tested photocatalyst masses following treatment. For a mass of 0.5 g, the pH dropped from 7.2 at 0 minutes to 6.9 at 180 minutes; and remained relatively stable after 60 minutes; whereas for 1 g, the pH decreased from 7.0 to 6.8 and then remained constant up to the 180-minute mark. This indicates that increasing the photocatalyst mass accelerates the system's initial response; however, at longer contact times, the change in pH diminishes as the process approaches a more stable state [15].

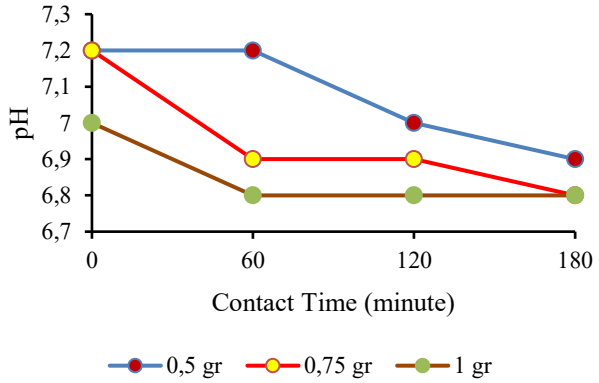

Figure 6. Effect of Mass and Contact Time on Wastewater pH

The decrease in pH can be attributed to the fact that, during the photocatalysis process, electron-hole pairs formed on the ZnO-NiFe2O4 surface trigger the generation of hydroxyl radicals and other oxidative species that facilitate the degradation of organic compounds in textile wastewater [16]. This indicates that the degradation process proceeds actively—particularly during the initial contact phase—but tends to slow down as the quantity of easily oxidizable pollutants diminishes and the system approaches equilibrium.

3.3 Effect of Catalyst Mass and Contact Time on Phenol Concentration

Phenol is a hazardous and carcinogenic organic compound. Its presence at certain concentrations can have negative impacts on the environment and human health. Phenol-containing waste can pollute water bodies, thereby reducing dissolved oxygen levels [17].

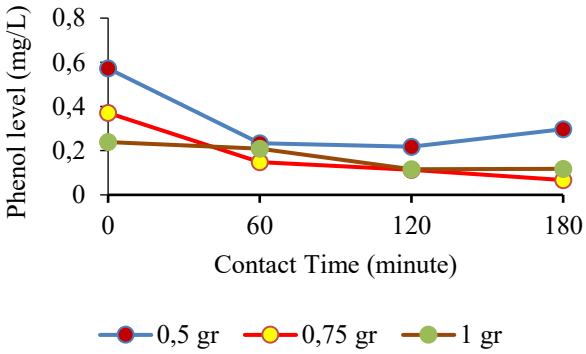

Figure 7. Effect of mass and contact time on phenol concentration.

Figure 7 illustrates the effect of photodegradation on phenol, based on data obtained using ZnO-$NiFe_2O_4$ photocatalyst masses of 0.5, 0.75, and 1 gram, with irradiation contact times of 0, 60, 120, and 180 minutes. The initial phenol concentration in the textile wastewater prior to treatment was 3.256 mg/L. With a photocatalyst mass of 0.5 grams, the phenol concentration was 0.572 mg/L at 0 minutes; it dropped significantly to 0.233 mg/L at 60 minutes and continued to decrease, reaching a minimum concentration of 0.218 mg/L at 120 minutes. However, when irradiation was extended to 180 minutes, the phenol concentration rose again to 0.297 mg/L.

At a photocatalyst mass of 0.75 grams, the phenol concentration exhibited the most stable and progressive downward trend, with no subsequent fluctuation. The measured phenol concentration was 0.371 mg/L at 0 minutes, decreasing to 0.148 mg/L at 60 minutes and 0.113 mg/L at 120 minutes, before reaching the most optimal degradation point at 180 minutes with a residual concentration of 0.067 mg/L. This indicates that a photocatalyst mass of 0.75 grams represents the most ideal and stable dosage for the gradual generation of free hydroxyl radicals to degrade phenol pollutants.

Meanwhile, with a catalyst mass of 1 gram, the phenol concentration was 0.239 mg/L at minute 0, decreased to 0.209 mg/L at minute 60, and reached a minimum of 0.116 mg/L at minute 120. However, similar to the 0.5-gram mass, an increase was observed at minute 180, where the concentration rose slightly to 0.118 mg/L. This increase or fluctuation in phenol levels during the final stages (as seen with both the 0.5-gram and 1-gram masses at minute 180) indicates the desorption of phenol compounds or intermediate products—previously adsorbed onto the catalyst's active surface—back into the solution phase; this occurs due to a limited supply of active radicals required to sustain the complete mineralization process. Furthermore, an excessive amount of photocatalyst (such as the 1-gram dose) can lead to a screening effect or particle agglomeration within the wastewater suspension, thereby obstructing light penetration and reducing the efficiency converting photon energy into active radicals [18].

The reduction in phenol levels is attributed to the photocatalytic oxidation process utilizing the ZnO-$NiFe_2O_4$ photocatalyst. This process begins with the activation of the photocatalyst by UV-Vis light. Upon irradiation, electrons in

the valence band are excited to the conduction band, generating electron-hole (e^--h^+) pairs. These e^--h^+ pairs subsequently react with water and dissolved oxygen to form active radicals—such as hydroxyl radicals (•OH) and superoxide radicals ($•O_2^-$)—which serve as the primary oxidizing agents in the degradation of phenol compounds. The optimal conditions for the photocatalysis of phenolic compounds in textile wastewater were observed with a ZnO-$NiFe_2O_4$ mass of 0.75 g and an irradiation contact time of 180 minutes, achieving a degradation percentage of 98.2%.

3.4 Effect of Catalyst Mass and Contact Time on Total Suspended Solids (TSS) Values

Total Suspended Solids (TSS) refers to the residue of total solids retained by a filter with a maximum particle size of 2 µm—or particles larger than the colloidal size range. TSS consists of various components such as silt, clay, metal oxides, sulfides, and bacteria. Typically, TSS is removed through flocculation and filtration processes. TSS contributes to increased water turbidity by limiting light penetration, thereby affecting—and potentially inhibiting—photosynthesis within aquatic ecosystems [19]. The results of the TSS analysis for textile wastewater are shown in Figure 8.

Figure 8 illustrates the decrease in TSS levels observed during the test. The initial TSS concentration prior to treatment was 235 mg/L. However, once the photocatalysis process commenced, the suspended solids concentration dropped significantly across all test variations: to 72.2 mg/L for the 0.5 g mass, 63.3 mg/L for the 0.75 g mass, and 55.1 mg/L for the 1 g mass. This strongly indicates that the reduction in pollutants was not merely driven by dark adsorption (the physical uptake of solids onto the catalyst's pore surfaces in the absence of light) but was triggered by the onset of photocatalytic activity. Exposure of the solution to UV light was sufficient to excite electrons in the conduction band of the ZnO-NiFe2O4 nanocomposite. This process immediately stimulated the formation of hydroxyl radicals (•OH) during the initial seconds; these radicals simultaneously attacked and broke down the macromolecular chains of the suspended organic solids into simpler intermediate compounds [18].

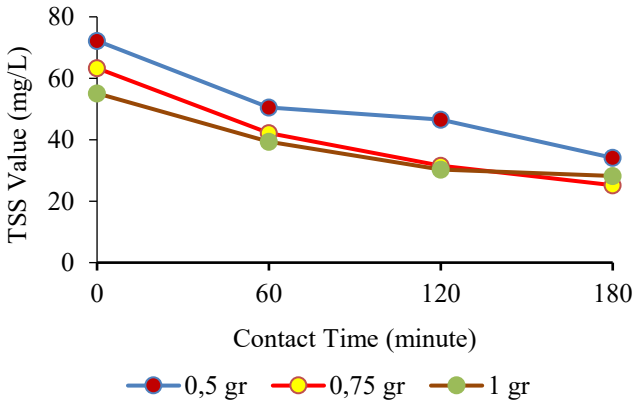

Figure 8 Effect of Mass and Contact Time on TSS Value

Following the initial stage, the oxidative degradation process driven by these active radicals proceeded continuously throughout the irradiation period, extending up to 180 minutes. Although a downward trend in TSS levels was observed across all variations, the 0.75-gram mass variation consistently demonstrated the most stable and optimal reduction efficiency, successfully lowering solids content to 25.2 mg/L by the end of the contact time. This dosage is considered the ideal spatial density, as it allows for uniform UV light distribution across the catalyst's active surface without causing optical interference [20].

In contrast, at the highest mass variation of 1 gram, the rate of reduction began to slow and plateau between the 120-minute mark (30.3 mg/L) and the 180-minute mark (28.2 mg/L). This deceleration in degradation kinetics at such a high dosage is attributed to the high concentration of powder within the reactor, which triggered agglomeration—or the clumping together—of semiconductor particles [20]. This mass agglomeration caused light scattering, thereby obstructing UV photon penetration to the catalyst's active sites. Consequently, the productivity of •OH radicals in breaking down the remaining suspended solid molecules was diminished by the end of the reaction period [20].

4. Conclusion

Based on XRD results, the crystallite size of the ZnO-NiFe2O4 composite was determined to be 11.86 nm, while FTIR analysis identified functional groups at 455 cm^{-1} (Zn-O/Ni-O) and 580 cm^{-1} (Fe-O). These findings confirm the formation of nanoparticles and the successful synthesis of a ZnO-NiFe2O4 composite optimized for photocatalytic activity.

The best results were achieved with a contact time of 180 minutes and a mass of 0.75 grams. The process yielded an 87.1% reduction in COD, 98.2% degradation of phenol content, and an 89.3% reduction in TSS, while the pH reached 6.8 all meeting the required quality standards.

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