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Effect of Gd Doping on Ni Bead Nanoparticles for Enhanced Pharmaceutical Degradation

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***Abstract*— The presence of pharmaceutical pollutants, including theophylline, in the environment presents considerable risks owing to their persistence and toxicity. This research examines the improvement of photocatalytic degradation of theophylline using gadolinium (Gd) doped nickel (Ni) bead nanoparticles. The objectives encompass the synthesis of Gd-Ni nanoparticles and the assessment of their photocatalytic efficiency under different conditions. Gd-Ni nanoparticle beads were synthesized using precipitation and sol-gel techniques. The evaluation of photocatalytic performance involved measuring the degradation rates of theophylline when exposed to UV light. The results demonstrated that the best conditions for photocatalytic activity were achieved at a neutral pH of 7, with a catalyst dosage of 20 g and an initial concentration of 10 ppm of theophylline. The 5 % Gd-Ni bead nanoparticles exhibited a degradation efficiency of 88.571 % within four hours of irradiation, surpassing the 83.758 % removal achieved by the 3 % Gd-Ni bead nanoparticles. The findings demonstrate the efficacy of Gd-doped Ni bead nanoparticles as photocatalysts for the sustainable elimination of pharmaceutical pollutants, thereby aiding environmental remediation initiatives.**

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Keywords— ***Photocatalytic; Gadolinium doped nickel bead; Theophylline; removal***

I. INTRODUCTION

The occurrence of pharmaceutical pollutants in the environment is a significant global issue, attributed to their potential toxicity, persistence, and bioaccumulation characteristics. Traditional wastewater treatment facilities often fail to adequately eliminate antibiotics, analgesics, and hormones, leading to their common presence in aquatic environments. These pollutants disrupt aquatic ecosystems and present risks to human health.

Theophylline is commonly utilized as a bronchodilator for the management of respiratory conditions, including asthma and chronic obstructive pulmonary disease (COPD). Theophylline, while beneficial therapeutically, is a persistent compound that can infiltrate aquatic environments via industrial discharge, improper disposal, and inadequate removal by conventional wastewater treatment systems. The presence of theophylline and analogous pharmaceuticals in

aquatic environments necessitates the development of effective and sustainable degradation methods [1].

Photocatalysis represents a significant advancement in environmental remediation, utilizing semiconductor materials to convert light energy into the degradation of pollutants into non-toxic byproducts[2-3]. The process entails the generation of reactive species, including hydroxyl radicals (OH•) and superoxide anions (O_2^-), following the absorption of light energy by a photocatalyst. The photocatalyst, generally a semiconductor material, experiences photoexcitation upon exposure to light of appropriate energy, resulting in the excitation of electrons from the valence band to the conduction band, thereby generating electron-hole pairs. Charge carriers initiate redox reactions, wherein holes (h^+) oxidize water or hydroxide ions to produce hydroxyl radicals, while electrons (e^-) reduce oxygen to generate superoxide anions. Reactive species facilitate the degradation of complex organic pollutants into smaller, non-toxic molecules, including CO_2 and H_2O [4-5]

Nickel (Ni) nanoparticles exhibit significant potential as photocatalysts, attributed to their advantageous electronic properties, elevated surface area, and catalytic activity. The efficiency of Ni nanoparticles in photocatalytic reactions is frequently constrained by rapid charge carrier recombination, which diminishes the availability of reactive species, alongside their limited light absorption and low surface reactivity. Researchers have investigated several strategies to address these limitations, including doping, which entails the introduction of foreign atoms into the material's crystal lattice to improve its physicochemical properties [6].

Gadolinium (Gd), a rare earth element, has garnered significant interest as a dopant for its capacity to alter the structural, electrical, and surface characteristics of host materials. Gd doping provides advantageous defects, such as oxygen vacancies, and establishes intermediate energy levels inside the bandgap, facilitating enhanced charge carrier separation and improved light absorption in the visible spectrum [7]. Furthermore, Gd ions can augment surface active sites, promoting the interaction between the catalyst and reactants. The attributes of Gd-doped Ni nanoparticles render them a viable option for sophisticated photocatalytic applications, particularly in the breakdown of pharmaceutical contaminants like theophylline.

The integration of Gd into Ni bead nanoparticles aims to resolve significant issues related to traditional photocatalysts. The modification of the bandgap via Gd doping enables the photocatalyst to harness a wider spectrum of solar radiation, thereby improving light absorption and overall photocatalytic efficiency. The formation of oxygen vacancies and other defect sites enhances charge carrier separation, reduces recombination losses, and facilitates more efficient redox reactions [7]. The enhanced surface properties from Gd doping facilitate improved adsorption of theophylline molecules, leading to better interaction and subsequent degradation. The combined effects lead to increased degradation rates and enhanced performance in environmental remediation applications.

Prior research has shown that rare earth doping enhances the photocatalytic efficiency of different metal-based nanoparticles. Gd-doped TiO_2 and ZnO demonstrate notable improvements in the degradation of organic pollutants when subjected to visible light irradiation. Doping transition metals such as Ni with rare earth elements enhances their catalytic efficiency, stability, and reusability in environmental applications [8-9]. Nevertheless, there is a paucity of research specifically examining the impact of Gd doping on Ni nanoparticles in the context of degrading pharmaceutical pollutants like theophylline. The existing knowledge gap provides an opportunity to investigate the potential of Gd-doped Ni bead nanoparticles as advanced photocatalysts.

This research intends to produce Gd-doped Ni bead nanoparticles and assess their photocatalytic efficacy in the breakdown of theophylline under regulated conditions. The photocatalytic activity will be evaluated by quantifying the breakdown rate of theophylline under simulated UV light exposure for four hours, emphasizing the impact of Gd doping on the material's catalytic efficiency.

This study investigates the impact of Gd doping on Ni bead nanoparticles to elucidate the principles of photocatalytic amplification and advance the development of effective and sustainable remedies for pharmaceutical pollution. The results are anticipated to illustrate the efficacy of Gd-doped Ni nanoparticles as a formidable photocatalyst for environmental remediation, facilitating their implementation in actual wastewater treatment systems. Furthermore, the research will enhance the field of nanomaterial design by emphasizing the significance of rare earth doping in the progression of photocatalytic technologies aimed at mitigating pressing environmental issues.

II. METHOD

Materials

The Nickel (Ni) powder and gadolinium (Gd) were sourced from Macklin, China, with a purity surpassing 99.5 %. The powder was utilized in its unaltered state. The utilized materials comprise alginate powder from Sigma-Aldrich Laborchemikalien GmbH, calcium chloride dihydrate of A.R. grade from Uchem, epichlorohydrin solution, and theophylline anhydrous with a minimum purity of 99 % from Sigma-Aldrich Laborchemikalien GmbH. Hydrochloric acid (HCl) and sodium hydroxide (NaOH) were obtained from QReCTM. All reagents were of analytical grade and were employed without additional processing. Deionized water was employed to make solutions, comprising synthetic pharmaceutical substances, alginate solution, calcium chloride solution, and pH solution. pH adjustments were conducted utilizing 0.1 M HCl and 0.1 M NaOH solutions.

Gd doped Ni Bead Catalyst Preparation

The bead catalyst preparation procedure was refined and enhanced based on previous study [10]. Three weight percent of powdered alginate will be dissolved in deionized water to make the precursor. The solution will be agitated for 2 hours to

get a 3 %wt. alginate solution (viscous solution). Subsequently, Gd-Ni nanoparticles in a 1:1 ratio are incorporated into the produced alginate solution. The mixture is agitated for two hours and allowed to rest overnight. Subsequently, inject it with a syringe into an 800 mL precipitation bath (non-solvent) containing 0.5 M aqueous $CaCl_2$ to produce wet magnetic catalyst gel beads. Thereafter, the gel beads are cross-linked to make them insoluble in an acidic (low pH) aqueous solution. The wet Gd-Ni bead catalyst was then cross-linked with epichlorohydrin via a modified method [11]. The final phase of the Gd-Ni bead involved meticulously rinsing the cross-linked alginate beads with deionized water, thereby preparing them for photocatalytic evaluation.

Photocatalytic Testing

The photocatalytic effectiveness of the synthesized Gd-doped Ni bead catalyst was evaluated for the degradation of theophylline concentrations in an aqueous solution. The solution was subjected to radiation from an 8 W UV lamp as the light source, while ensuring a constant temperature and agitation for four hours. The light emanates from a distance of 15 cm between the lamp and the beaker carrying 100 mL of theophylline solution with differing pH levels. The beaker possesses a capacity of 250 mL. The influence of catalyst dosage and the initial concentration of theophylline was evaluated in photocatalytic experiments. A degradation sample was collected, and the concentration of analytes in the sample was quantified using a UV-Vis spectrophotometer (UV 1900). Theophylline was measured by measuring its distinctive absorption bands at 273 nm. A 5 mL sample was obtained and thereafter underwent centrifugation at specified time intervals. The rate of photodegradation for different percentages of Gd-doped Ni bead catalyst at each time interval (degradation quantity) was calculated using Equation (1).

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

where Co represents the initial concentration and Ci denotes a variable concentration.

III. RESULTS AND DISCUSSION

Effect of pH

Figure I depicts the influence of pH on the breakdown of theophylline in an aqueous solution utilizing two distinct concentrations of doping: 3 % Gd-Ni bead and 5 % Gd-Ni bead as photocatalysts. The pH values of the analysed solution were 3, 5, 7, 9, and 11. The graph in Figure I illustrates that the observed value rises from pH 3 to pH 7 across all three doping amounts, indicating optimal performance or photocatalytic activity at neutral pH. Nonetheless, the performance trend for both doping agents diminishes from pH 7 to pH 11 owing to diminished efficacy in alkaline environments. 5 % Gd-Ni exhibits superior performance compared to 3 % Gd-Ni at pH 3. Nonetheless, at pH 5 and pH 7, the disparities in concentrations decrease, signifying comparable performance. At elevated pH levels (pH 9 and pH 11), the discrepancies become less uniform; however, 5 % Gd-Ni continues to exhibit marginally superior performance or remains equivalent.

Table I presents the pH performance analysis, indicating that the optimal pH for the removal of theophylline is 7, achieving a maximum removal rate of 87.895 % with 5 % Gd-Ni beads, followed by 79.041 % with 3 % Gd-Ni beads. The lowest percentage of theophylline removed at a pH of 3 during the photocatalytic reaction is 11.770 % for the 3 % Gd-Ni bead and 13.349 % for the 5 % Gd-Ni bead. The research established that the optimal pH for the efficient removal of theophylline is pH 7.

The pH of a solution is essential for generating hydroxyl radicals during photocatalytic degradation. OH^- ions elevate hydroxyl radical concentrations, which are extremely reactive and expedite the degradation of medicinal molecules [12]. Photocatalysis transpires when •OH radicals convey their charge to the target molecule. The effectiveness of degradation enhances with a rise in pH from 3 to 7, during which negatively charged photocatalysts generate a greater quantity of hydroxyl radicals [12]. Photocatalysts exhibit optimal performance at pH 7, where they effectively harness energy. At pH values beyond 7, energy absorption diminishes, and chemicals such as theophylline, which acquire a negative charge in alkaline environments, resist the photocatalyst, hence diminishing efficiency [12].

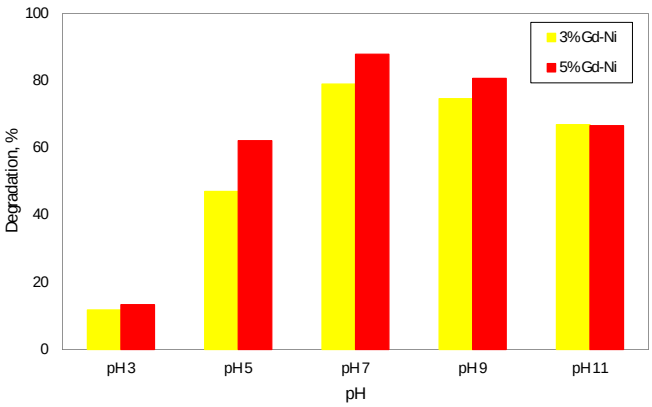

FIGURE I. Effect of pH on theophylline removal (catalyst dosage: 30g, initial concentration: 10 ppm)

TABLE I Percentage removal of theophylline in varying pH

Type of catalyst	Degradation rate, %				
	pH 3	***pH 5***	***pH 7***	***pH 9***	***pH 11***
3 % Gd-Ni bead	11.770	47.054	79.041	74.700	66.944
5 % Gd-Ni bead	13.349	62.154	87.895	80.719	66.631

Effect of Catalyst dosage

Figure II illustrates the effect of Gd-Ni nanoparticles bead catalyst dosage on the efficacy of the photocatalytic degradation process across two distinct doping concentrations. The studied catalyst dosage varied from 10 g to 50 g, in

increments of 10 g. At a catalyst dosage of 20 g, both doping concentrations (3 % and 5 %) demonstrate high performance, with 5 % Gd-Ni achieving the highest removal efficiency, closely followed by 3 % Gd-Ni beads.

Table II presents the catalyst dosage performance analysis, indicating that the optimal catalyst dosage for the removal of theophylline is 20 g, achieving a maximum removal rate of 88.571 % with 5 % Gd-Ni bead, followed by 83.758 % with 3 % Gd-Ni bead. The minimum percentage of theophylline eliminated during the photocatalytic reaction with a catalyst dosage of 50 g was 68.643 % for the 3 % Gd-Ni bead and 66.607 % for the 5 % Gd-Ni bead. The research identified that the optimal catalyst dosage for the effective removal of theophylline is 20 g.

As the dosage rises from 10 g to 20 g, the removal efficiency improves; however, from 20 g to 50 g, the measured values for all concentrations progressively decline, suggesting a potential saturation point or diminishing returns in performance with an excess of catalyst.

The degradation rate rose with the increase in catalyst mass until equilibrium was attained. This was likely attributable to the increased availability of active sites, facilitating enhanced absorption of photons and pharmaceutical molecules [13,14]. Enhanced photon absorption elevates the generation of hydroxyl radicals and positive holes. When the catalyst dose surpasses a specific threshold, the suspension becomes excessively turbid, obstructing light and diminishing photodegradation efficiency. Excessive amounts of catalyst can occasionally reduce photocatalytic activity [14–16].

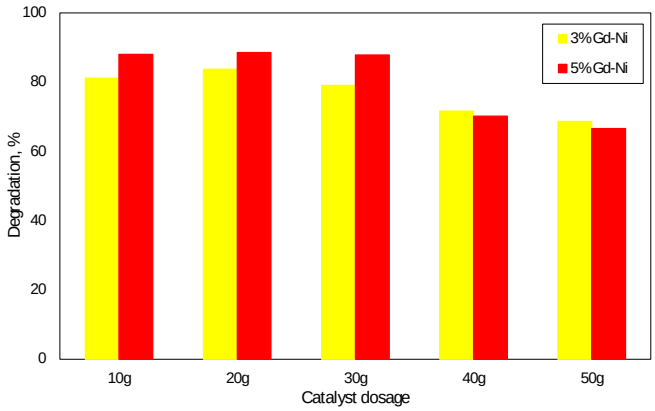

Figure II Effect of catalyst dosage on theophylline removal (pH: 7, initial concentration: 10 ppm)

Table II Percentage removal of theophylline in varying of catalyst dosage

Type of catalyst	Degradation rate, %				
	10 g	***20 g***	***30 g***	***40 g***	***50 g***
3 % Gd-Ni bead	81.183	83.758	79.041	71.660	68.643
5 % Gd-Ni bead	88.036	88.571	87.895	70.179	66.607

Effect of initial concentration

Figure III illustrates the effect of the initial concentration of theophylline on photocatalytic degradation. The pharmaceutical ingredient under investigation exhibited initial concentrations of 10, 20, 30, 40, and 50 ppm. At 10 ppm, all doping concentrations (3 % and 5 %) exhibit high performance, with 5 % Gd-Ni attaining the highest value, closely succeeded by 3 % Gd-Ni.

Table III indicates that the maximum degradation achieved was 88.571 % and 83.758 % when utilizing a concentration of 10 ppm of theophylline for the 5 % Gd-Ni bead and the 3 % Gd-Ni bead, respectively. The lowest degradation rates observed were 52.103 % for the 3 % Gd-Ni bead and 48.086 % for the 5 % Gd-Ni bead at a concentration of 50 ppm. Elevating the initial concentration of a pharmaceutical ingredient leads to a reduction in its degradation through photocatalytic reaction, as evidenced by the observed trend. The optimal initial concentration for the photocatalytic reaction of theophylline is 10 ppm.

Pollutant concentrations substantially influence the rate and effectiveness of degradation processes. At low contamination levels, degradation occurs more rapidly due to the presence of numerous reactive sites and the interaction of particles with impurities. As pollutant concentration exceeds the optimal level, efficiency declines due to a deficiency of hydroxyl radicals [16]. Increased impurity concentrations absorb photons prior to their interaction with the catalyst, thereby diminishing photocatalytic efficiency [17–18].

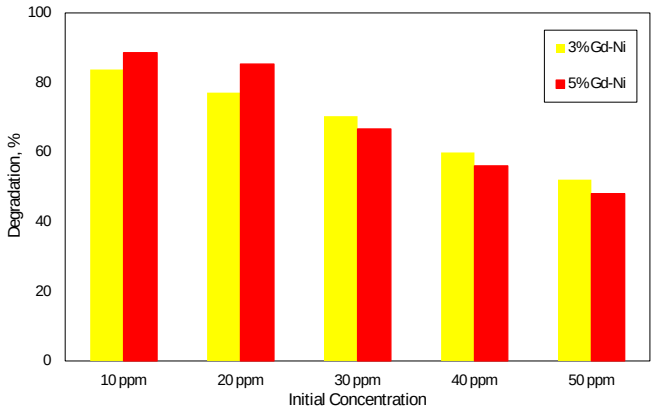

Figure III Effect of initial concentration on theophylline removal (pH: 7, catalyst dosage: 20g)

Table III Percentage removal of theophylline in varying of initial concentration

Type of catalyst	Degradation rate, %				
	10 ppm	***20 ppm***	***30 ppm***	***40 ppm***	***50 ppm***
3 % Gd-Ni bead	83.758	77.115	70.313	59.934	52.103
5 % Gd-Ni bead	88.571	85.345	66.644	56.064	48.086

The optimal conditions for the three different doping scenarios were identified as pH 7, a catalyst dosage of 20 g, and an initial concentration of 10 ppm. In comparison of doping concentration, a higher doping level of Gd-Ni (5 %)

demonstrates superior performance, suggesting that the proportion of Gd-Ni is crucial in enhancing the measured effect.

IV. CONCLUSIONS

This study investigates the efficiency of Gd doping on Ni nanoparticle beads for the photocatalytic degradation of theophylline in aqueous solutions under various conditions. 5 % Gd-Ni nanoparticle beads demonstrate superior performance in the removal of theophylline compared to 3 % Gd-Ni nanoparticle beads. The pH significantly affects the photo-degradation percentage of theophylline, with an optimum pH identified at 7. The degradation escalates with an increase in catalyst mass, resulting in greater absorption of photons and pharmaceutical molecules. Excessive use of photocatalysts may lead to a reduction in activity. The optimal catalyst dosage was determined to be 20 g. Pollution levels affect efficiency and degradation rates. Higher concentrations lead to accelerated breakdown; however, efficiency diminishes due to insufficient hydroxyl radical availability. The ideal starting concentration is 10 ppm. The highest degradation rates obtained at four hours of irradiation for theophylline were 88.571 % and 83.758 %, respectively, when 5 % Gd-Ni nanoparticle bead and 3 % Gd-Ni nanoparticle bead were utilized as photocatalysts. In comparison of doping concentration, a higher doping level of Gd-Ni (5 %) demonstrates superior performance, indicating that the proportion of Gd-Ni is critical in enhancing the measured effect. It can be concluded that the photocatalytic process utilizing Gd doped Ni nanoparticle beads is effective. has a relatively high potential of pharmaceutical compound removal from aqueous solution and can be used as a convenient method for easy operation and has a low cost for the operational scale.

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