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STUDY ON THE SYNTHESIS OF g-C_3N_4/$LaFeCuO_3$ HYBRID MATERIALS FOR THE DEGRADATION OF CIPROFLOXACIN

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Abstract. In this study, the photocatalysts g-C_3N_4, $LaFeCuO_3$, and the composite 10%g-C_3N_4/$LaFeCuO_3$ were prepared using a combination of sol-gel, pyrolysis, and ultrasonic-assisted synthesis methods.Their surface characteristics, including the crystal structure, elemental composition, morphology, optical absorption, and electron-hole recombination, were investigated through XRD, EDX, FE-SEM, UV-vis DRS, and photoluminescence (PL) spectroscopy. The role of ammonium persulfate agent (APS) in the degradation of ciprofloxacin (CIP) under light irradiation in the absence of photocatalysts was also examined. Results indicated that APS generated highly oxidative radicals ($SO_4^{\bullet-}$ and •OH), contributing significantly to the degradation of CIP. The optimal APS volume was 0.8 mL of 0.25M APS, achieving 82.35% CIP degradation after 180 minutes. In the presence of both APS and photocatalysts, $LaFeCuO_3$, g-C_3N_4, and 10%g-C_3N_4/$LaFeCuO_3$ materials achieved degradation efficiencies of 81.78%, 78.61%, and 96.3%, respectively, under the same experimental conditions, specifically, a CIP concentration of 20 ppm, photocatalyst loading of 1.5 g/L, and solution pH of 4.6. This improvement is attributed to the enhanced formation of free radicals facilitated by the presence of the photocatalysts.

Keywords: g-C_3N_4/$LaFeCuO_3$ composite, persulfate activation, ciprofloxacin degradation.

1. Introduction

Ciprofloxacin (CIP) is a broad-spectrum fluoroquinolone antibiotic widely used in human and veterinary medicines, as well as in animal husbandry and aquaculture. A significant portion of CIP is excreted unmetabolized and enters wastewater systems, where conventional treatment processes are often ineffective in completely removing it. Consequently, CIP persists in the environment, contaminating water bodies, soil, and agricultural fields through hospital and livestock wastewater, pharmaceutical effluents, sludge application, and surface runoff. The presence of CIP in the environment poses significant ecological and public health concerns. It can disrupt microbial communities, interfere with nutrient cycling, inhibit plant growth, reduce crop yields, and promote the emergence of antibiotic-resistant bacteria. Humans can be exposed to CIP through contaminated food and water, leading to potential liver and kidney damage, gut microbiota disruption, and increased antibiotic resistance [1], [2].

Due to the limitations of traditional treatment methods, the development of more advanced and eco-friendly technologies is necessary. One promising approach is persulfate oxidation using ammonium persulfate (APS), which produces strong oxidizing sulfate radicals ($SO_4^{\bullet -}$, E = 2.6 - 3.1 V $vs.$ NHE-Normal Hydrogen Electrode) and hydroxyl ($^{\bullet}OH$, E = 2.0 - 2.8 V $vs.$ NHE) upon activation by heat, UV, transition metals, or alkaline conditions. These radicals degrade persistent organic pollutants effectively [3]-[5]. To enhance APS activation, photocatalysts like $LaFeCuO_3$ and g-C_3N_4 have shown great potential. $LaFeCuO_3$, a perovskite oxide with a band gap energy in the range 1.8 - 2.0 eV, generates multiple reactive oxygen species (ROS) through electron transfer facilitated by Fe^{2+} and Cu^{+}, with oxygen vacancies boosting catalytic performance under visible light irradiation [6]-[8]. Similarly, g-C_3N_4, a carbon-based photocatalyst having a band gap energy of 2.7 eV, utilizes UV-visible light to produce ROS, offering stability and easy recovery [5], [9]-[11]. Individually, these materials show limited efficiency. However, combining APS with $LaFeCuO_3$ and g-C_3N_4 in optimal ratios enhances ROS production, photocatalyst stability, and pollutant degradation, providing an effective and sustainable solution for removing ciprofloxacin from water [12], [13].

The aim of this study is to evaluate the efficiency of CIP degradation using APS in the presence of $LaFeCuO_3$ and g-C_3N_4-based photocatalysts. Additionally, the study investigates key influencing factors such as solution pH, photocatalyst dosage, and the presence of radical scavengers to determine the optimal conditions for effective CIP degradation under light irradiation. The study proposes a degradation mechanism of CIP on the $LaFeCuO_3$/g-C_3N_4 heterojunction material under light irradiation conditions.

2. Content

2.1. Experiments

2.1.1. Materials and synthesis

**** Synthesis of $LaFeCuO_3$***

$LaFeCuO_3$ was synthesized via a sol-gel method. Precisely weighed amounts of $La(NO_3)_3 \cdot 6H_2O$, $FeCl_3 \cdot 6H_2O$, $Cu(NO_3)_2 \cdot 3H_2O$, citric acid, acrylamide, and glucose were dissolved in 2 mL of dilute HNO_3 and 8 mL of distilled water to form a homogeneous solution. The mixture was continuously stirred and heated at 80°C for 2 h to form a gel, then dried at 120 °C for 24 h. The resulting xerogel was ground into a fine powder and calcined at 500 °C for 3 h to form powder $LaFeCuO_3$. After natural cooling, the product was reground into a uniform fine powder for further application.

***** Synthesis of g-C_3N_4**

g-C_3N_4 was synthesized by thermal polymerization of urea. Ten grams of urea were placed in a semi-closed crucible to allow controlled gas release and heated in a muffle furnace at a rate of 5 °C/min up to 550 °C. The temperature was maintained for 4 h to promote polymerization.

Once cooled, the resulting yellow powder is removed from the crucible, which is the synthesized bulk g-C_3N_4.

***** Synthesis of 10%g-C_3N_4/ $LaFeCuO_3$**

The g-C_3N_4/$LaFeCuO_3$ composite was synthesized by mixing $LaFeCuO_3$ with 10 wt% g-C_3N_4. The mixture was ground into a fine powder, then dispersed in 2 mL of ethylene glycol and 15 mL of absolute ethanol. After stirring, the solution was sonicated for 30 min and transferred to a Teflon-lined autoclave for hydrothermal treatment at 80 °C for 6 h. The product was cooled to room temperature, filtered, and dried at 120 °C for 24 h. The final dried solid was ground into a fine powder, yielding the g-C_3N_4/$LaFeCuO_3$ composite.

2.1.2. Research method

***** Characteristics**

The crystalline phases of materials were evaluated through XRD pattern (X-ray diffraction) carried out on a Bruker D8-Advance diffractometer with Cu Kα radiation (λ = 0.015405 nm). The surface morphology of the phosphor samples was observed via scanning electron microscopy (FESEM) using a HITACHI S-4800 instrument. Elemental composition analysis was performed using energy-dispersive X-ray spectroscopy (EDX) equipped with a HORIBA 7593-H detector attached to the FESEM system. UV-Visible Diffuse Reflectance Spectroscopy (UV-Vis DRS) was performed on a Shimadzu UV-2600 device. The FLS1000 Photoluminescence Spectrometer was used to measure the photoluminescence (PL) spectra of the photocatalysts.

***** Persulfate oxidation of Ciprofloxacin over g-C_3N_4/$LaFeCuO_3$ photocatalyst**

The photocatalytic degradation of ciprofloxacin was carried out in a photoreactor, where a quartz beaker containing the solution was placed in a temperature-controlled water bath maintained at 25 °C. A 250 W xenon lamp was positioned 20 cm above the beaker to serve as the light source. A known volume (200 mL) of 20 ppm ciprofloxacin solution, with 0.8 mL 0.25 mM APS (ammonium persulfate) were mixed with the “set mass” of catalyst in units (e.g., 0.3 g of photocatalyst, which corresponds to 1.5 g/L, based on later text) and irradiated for 210 minutes under stirring after 60 minutes of dark adsorption to reach adsorption equilibrium. At 30-minute intervals, samples were collected and analyzed using UV-vis spectroscopy at 273 nm. CIP concentration was calculated using the standard curve: $A = 0.0949 * C + 0.0593$, $R^2 = 0.998$, to evaluate photocatalytic performance (A-absorption, C-concentration).

2.2. Results and discussion

2.2.1. Characteristics of the as-synthesized materials

X-ray diffraction (XRD) patterns were used to analyze the phase composition and structural characteristics of the materials. The patterns for g-C_3N_4, $LaFeCuO_3$, and the 10%g-C_3N_4/$LaFeCuO_3$ composite are presented in Figure 1, revealing their distinct crystalline structures. From Figure 1, the characteristic diffraction peaks of the $LaFeCuO_3$ perovskite material were observed at 2θ angles are 24.6°, 27.9º, 32.2°, 35.4°, 39.6°, 46.2°, 57.4°, 67.4° and 76.7°, corresponding to specific crystal planes: (101), (111), (121), (210), (220), (202), (240), (242) and (204) (JCPDS No. 37-1493) [3], [6]-[8].

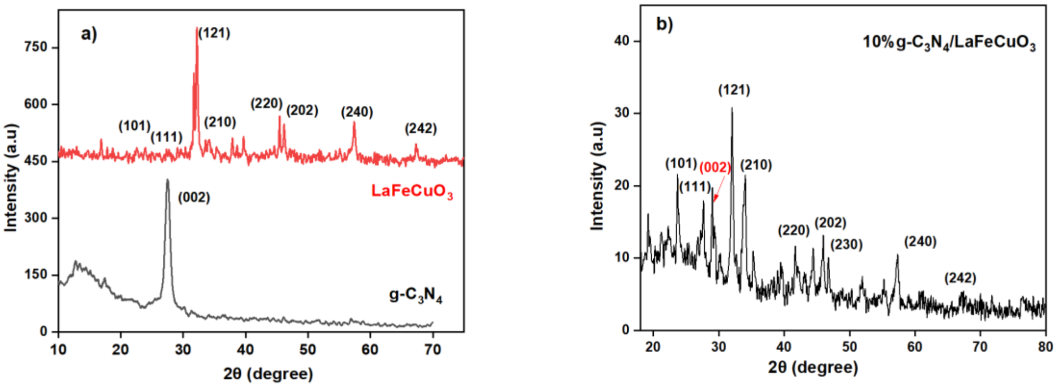


Figure 1. XRD pattern of (a) g-C_3N_4, $LaFeCuO_3$ and (b) 10%g-C_3N_4/$LaFeCuO_3$

For g-C_3N_4, the characteristic diffraction peak is observed at 2θ = 27.2°, corresponding to the (002) crystal plane. (JCPDS 87-1526) [12], [13]. When combining 10%g-C_3N_4 with $LaFeCuO_3$ to form the 10%g-C_3N_4/$LaFeCuO_3$, the characteristic diffraction peaks of both g-C_3N_4 and $LaFeCuO_3$ in the composite exhibit reduced intensities, indicating a decrease in crystallinity. This likely results from the intimate mixing of the two phases. The absence of any new diffraction peaks suggests that no new crystalline phase is formed, implying that g-C_3N_4 and $LaFeCuO_3$ coexist and form a heterojunction structure.

The morphology of the materials was evaluated using FE-SEM images. Figure 2 shows the FE-SEM images of the investigated materials.

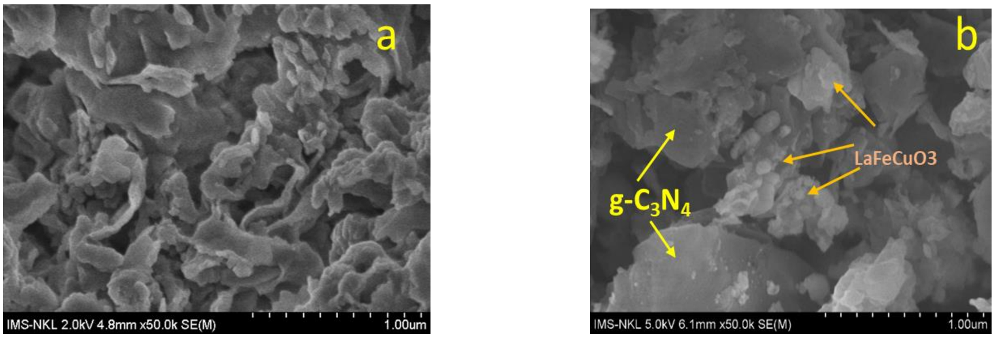


Figure 2. FE-SEM images of (a) g-C_3N_4 and (b) 10%g-C_3N_4/$LaFeCuO_3$

The g-C_3N_4 material exhibits a two-dimensional planar structure with thin layers and a certain degree of porosity. In the 10 % g-C_3N_4/$LaFeCuO_3$ composite, $LaFeCuO_3$ particles are distributed on the surface of the g-C_3N_4. Although the $LaFeCuO_3$ particles are not perfectly uniformly distributed on g-C_3N_4, the FE-SEM image confirms that the two materials are combined, indicating the successful formation of the g-C_3N_4/$LaFeCuO_3$ composite heterojunction

The EDX spectra of $LaFeCuO_3$ (Figure 3a) indicate that the elements like La (20.89 wt%), Fe (6.09 wt%), Cu (3.24 wt%), and O (30.77 wt%) were presented in $LaFeCuO_3$ component. Additionally, trace amounts of Na and Cl were detected on the surface of the $LaFeCuO_3$, likely originating from residual chloride salts and other precursors that were not completely removed during synthesis. For the modified material 10%g-C_3N_4/$LaFeCuO_3$ (Figure 3b), the elemental composition was also analyzed to confirm the presence of C and N from the g-C_3N_4 structure. The detected elements included La (12.17 wt%), Fe (3.72 wt%), Cu (1.51 wt%), O (25.58 wt%), C (46.66 wt%), and N (10.36 wt%).

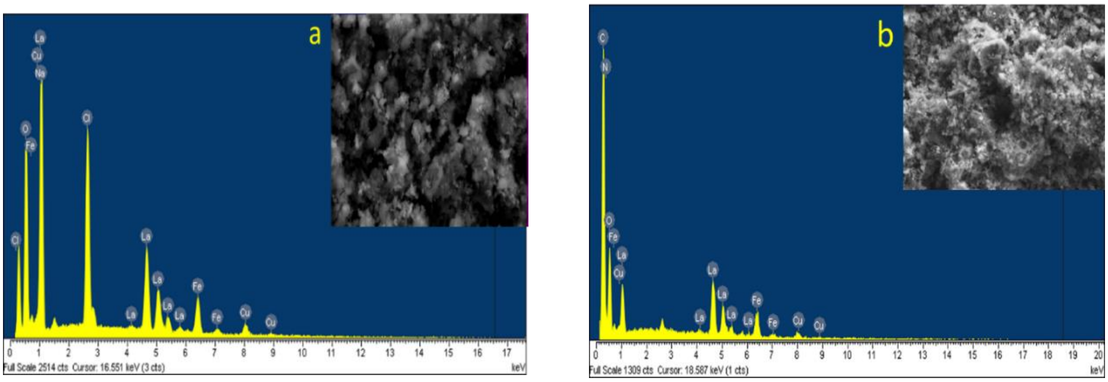


Figure 3. EDX spectra of (a) $LaFeCuO_3$ and (b) 10 % g-C_3N_4/$LaFeCuO_3$

The optical absorption properties of g-C_3N_4, $LaFeCuO_3$, and 10 % g-C_3N_4/$LaFeCuO_3$ were investigated using UV-vis DRS spectra, as shown in Figure 4.

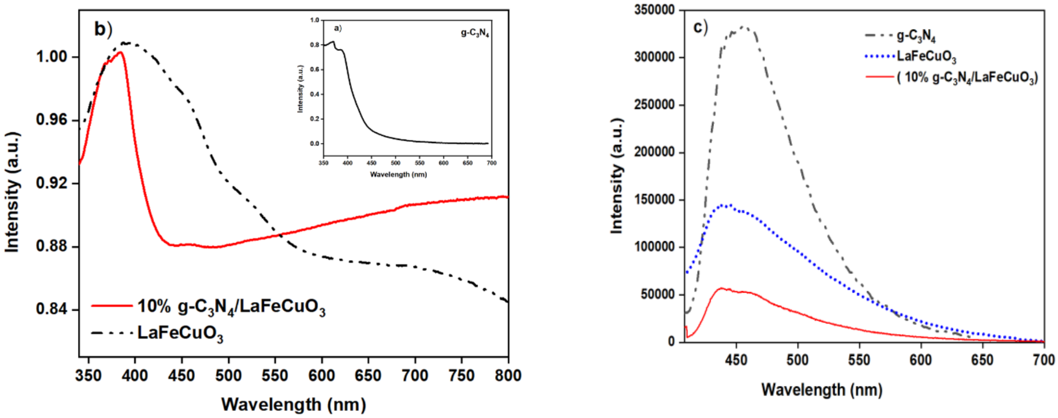


Figure 4. UV vis DRS (a, b) and PL spectra of the as-synthesized materials (c)

The g-C_3N_4 showed strong UV absorption below 400 nm, with an absorption edge around 460 nm, but weak visible light absorption. $LaFeCuO_3$ exhibited broader absorption extending into the visible region, with an edge near 550 nm. Based on the Tauc plot (plotting $(Ahv)^{0.5}$versus hv), the band gaps of g-C_3N_4 and $LaFeCuO_3$ are determined to be 2.51 eV and 2.25 eV, respectively.

For the 10 % g-C_3N_4/$LaFeCuO_3$ composite, the absorption peak was slightly blue-shifted (~380 nm), with a sharper band edge at 430 nm. Notably, absorption above 550 nm increased, perhaps due to interaction between phases. The finding likely indicates some change in optical properties with coupling. It implies that the composite has better visible-light harvesting than pure g-C_3N_4 or $LaFeCuO_3$ alone, which supports the synergistic effect and improves charge separation for enhanced photocatalytic activity.

Figure 4c shows that g-C_3N_4 exhibits higher PL intensity than $LaFeCuO_3$, indicating a faster electron-hole recombination rate. In the 10 % g-C_3N_4/$LaFeCuO_3$ composite, the PL intensity significantly decreases, which improves charge separation and reduces recombination. Additionally, the reduced recombination is likely due to type-II heterogeneous scheme charge transfer between $LaFeCuO_3$ and g-C_3N_4.

2.2.2. Study on the persulfate activation of ciprofloxacin under light irradiation

In this study, the simultaneous presence of persulfate oxidizing agent ($S_2O_8^{2-}$) and as-synthesized photocatalysts was investigated to evaluate the degradation efficiency of CIP at an initial concentration of 20 ppm, pH 4.6. The results are shown in Figure 5.

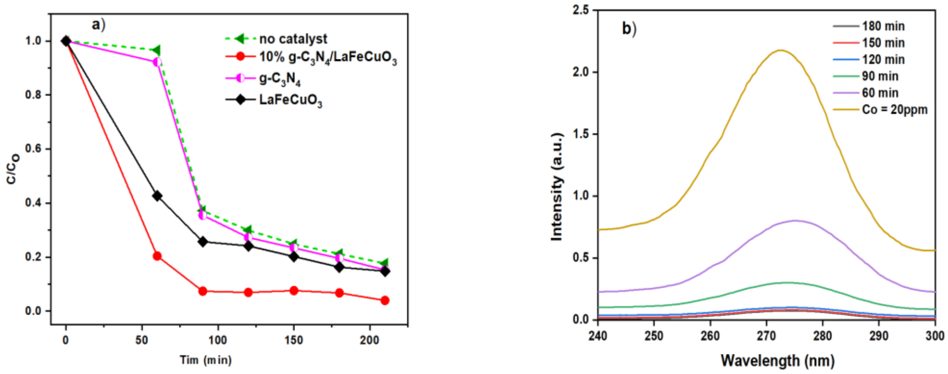


Figure 5. (a) The CIP degradation efficiency of as-synthesized photocatalysts, (b) UV vis absorption spectra of CIP before and after degradation time of 180 min

As illustrated in Figure 5a, three photocatalysts like g-C_3N_4, $LaFeCuO_3$, and 10 % g-C_3N_4/$LaFeCuO_3$ were tested, with a constant addition of 0.8 mL of 0.25 M APS solution. The degradation efficiency of CIP in the presence of APS alone (no photocatalyst) was also included for comparison. The results indicate that CIP degradation efficiency, represented as C/C_o, varied significantly even within the first 60 minutes of irradiation. For g-C_3N_4, the degradation efficiency was only 7.82%, while $LaFeCuO_3$ degraded 57.30% of CIP under the same conditions. This efficiency further increased to 79.58% with the 10 % g-C_3N_4/$LaFeCuO_3$ composite. The trend became more pronounced at 180 minutes of illumination. g-C_3N_4 and $LaFeCuO_3$ degraded 78.61% and 81.78% of CIP, respectively, while the 10 %g-C_3N_4/$LaFeCuO_3$ composite achieved a significantly higher degradation efficiency of 96.3%. For reference, APS alone achieved

~ 82% CIP removal in 180 min under these conditions. This provides a quantitative basis for evaluating the photocatalyst's performance.

Figure 5b shows the UV-Vis absorption spectra of the CIP solution before and after degradation under the experimental conditions. As illustrated, the intensity of the characteristic absorption peaks in the range of 240 - 300 nm gradually decreases as the irradiation time increases from 0 to 180 minutes. In particular, CIP exhibits a prominent absorption peak at approximately 273 nm, whose intensity steadily declines over time. This suggests that the CIP molecules were effectively degraded by APS oxidative agents in the presence of the 10%g-C_3N_4/$LaFeCuO_3$ photocatalyst under light irradiation. Notably, the 10% g-C_3N_4/$LaFeCuO_3$ composite exhibits higher degradation efficiency compared to its individual components (e.g. g-C_3N_4 and $LaFeCuO_3$) as well as the control condition without a catalyst.

The degradation of CIP using APS agent and a photocatalyst can be explained as follows: Upon light irradiation, g-C_3N_4 and $LaFeCuO_3$ absorb photons and generate electron-hole pairs. The excited electrons (e^-) in the conduction band and holes (h^+) in the valence band react with oxidants like dissolved O_2 and peroxydisulfate ($S_2O_8^{2-}$, E° = 1.8 - 2.0 V), producing reactive radicals such as $^{\bullet}O_2^-$, $^{\bullet}OH$ (E° = 2.4 - 2.8 V), and $SO_4^{\bullet-}$ (E° = 2.6 - 3.1 V). These species oxidize CIP into CO_2, H_2O, and other non-toxic byproducts.

To investigate the roles of reactive species in the photocatalytic degradation of ciprofloxacin (CIP), the contributions of hydroxyl radicals ($^{\bullet}OH$), sulfate radicals ($SO_4^{\bullet-}$), and photogenerated holes (h^+) were examined using specific radical scavengers. Isopropyl alcohol was used to quench $^{\bullet}OH$, and methanol for $SO_4^{\bullet-}$ and Na-EDTA for h^+. In each experiment, 300 mg of the 10%g-C_3N_4/$LaFeCuO_3$ photocatalyst was dispersed in 200 mL of a 20 ppm CIP solution at pH 4.6. Subsequently, 10 mL of the appropriate scavenger-pure isopropyl alcohol, concentrated methanol, or 0.1 M Na-EDTA-was added to the solution. The suspension was then irradiated with a xenon lamp, and aliquots were collected at 60, 90, 120, 150, and 180 minutes to measure the residual CIP concentration. This approach allowed for the assessment of the relative contribution of each reactive species to the overall degradation process. The results were presented in Figure 6.

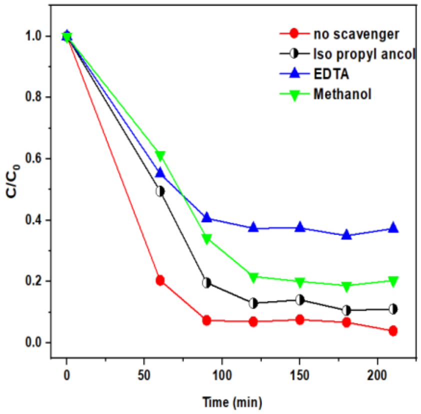
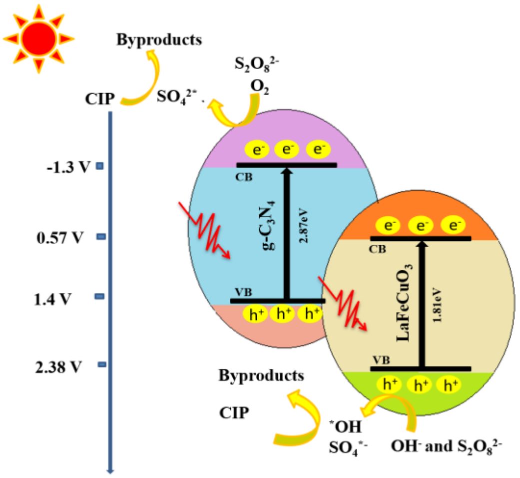


Figure 6. Effect of radical scavenger on CIP degradation efficiency over 10% C_3N_4/$LaFeCuO_3$ photocatalyst during 210 min of irradiation time

As shown in Figure 6, the degradation efficiency of CIP using 10% g-C_3N_4/$LaFeCuO_3$ significantly decreased upon the addition of radical scavengers such as methanol, isopropyl alcohol, and Na-EDTA to the reaction system. In the absence of scavengers, the removal efficiency of ciprofloxacin (CIP) reached 96.3%, which is approximately 1.2 to 1.5 times higher than the efficiencies observed in the presence of scavengers. For comparison, about 89.0% of CIP was degraded in the presence of isopropanol, around 62.7% with Na-EDTA, and approximately 79.7% in the presence of methanol. This indicates that hydroxyl radicals ($^{\bullet}OH$), sulfate radicals ($SO_4^{\bullet-}$), and photogenerated holes (h^+) all play essential roles in the degradation process. Among the scavengers, Na-EDTA exhibited the strongest inhibitory effect during the range from 60 to 180-minute irradiation period, suggesting that h^+ plays a more dominant role in CIP degradation compared to $^{\bullet}OH$ and $SO_4^{\bullet-}$radicals. Although photogenerated holes had the most pronounced effect, sulfate radicals ($SO_4^{\bullet-}$) are still considered more potent oxidizing agents than hydroxyl radicals ($^{\bullet}OH$) due to their higher redox potential. Therefore, the degradation mechanism involves a synergistic action of $^{\bullet}OH$, $SO_4^{\bullet-}$radicals, and photogenerated h^+ holes. The persulfate process can be activated by photons, either directly through UV irradiation or indirectly via photocatalysts that generate electron–hole pairs (e^-/h^+), leading to the subsequent formation of reactive radicals.

From the obtained results, the proposed mechanism for the formation of $SO_4^{\bullet-}$ and $^{\bullet}OH$ radicals by the 10%g-C_3N_4/$LaFeCuO_3$ material is illustrated in Figure 7.



$S_2O_8^{2-}$ + UV/Heat → $2SO_4^{\bullet-}$

$S_2O_8^{2-} + 2H_2O \rightarrow HO_2^- + 2SO_4^{2-} + 3H^+$

$S_2O_8^{2-} + HO_2^- \rightarrow SO_4^{\bullet-} + O_2^{\bullet-} + SO_4^{2-} + H^+$

$SO_4^{\bullet-} + OH^- \rightarrow SO_4^{2-} + {}^{\bullet}OH$

${}^{\bullet}OH + H^+ + e^- \rightarrow H_2O$

$SO_4^{\bullet-} + H^+ + e^- \rightarrow HSO_4^-$

$S_2O_8^{2-} + h^+ \rightarrow 2SO_4^{\bullet-}$

$SO_4^{\bullet-} + H_2O \rightarrow H^+ + SO_4^{2-} + {}^{\bullet}OH$

$SO_4^{\bullet-} + {}^{\bullet}OH$ + CIP → Byproducts + CO_2 + $H_2O + SO_4^{2-}$

Figure 7. The proposed mechanism for the formation of $SO_4^{\bullet-}$and $^{\bullet}OH$ radicals by the 10%g-C_3N_4/$LaFeCuO_3$ material

In this study, the effect of pH and dosage of photocatalyst on CIP degradation efficiency was evaluated using the 10%C_3N_4/$LaFeCuO_3$ photocatalyst. The results were shown in Figure 8.

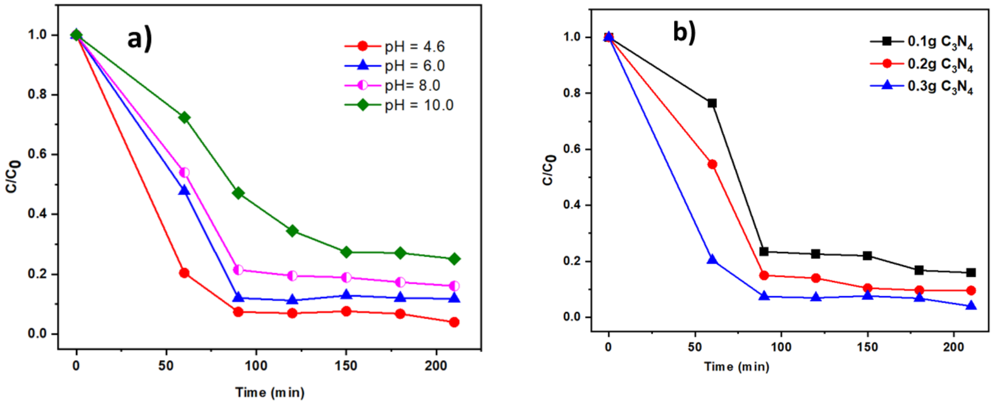


Figure 8. (a) The effect of pH and (b) dosage of photocatalyst on CIP degradation efficiency over 10%$C_3N_4/LaFeCuO_3$ photocatalyst

The obtained results showed that, at all tested pH levels, the degradation efficiency of CIP by 10% $C_3N_4/LaFeCuO_3$ material increased with illumination time, reaching its maximum after 180 minutes.

As the pH increased from 4.6 to 10, the CIP degradation efficiency exhibited a decreasing trend. Specifically, at 90 minutes of illumination, the efficiency decreased from 93.01% to 65.48% as the pH increased from 4.6 to 10. However, with continued illumination up to 180 minutes, the degradation efficiency improved across all pH levels and eventually stabilized. The final efficiencies recorded at 180 minutes were 96.3% at pH 4.6, 88.26% at pH 6.0, 83.85% at pH 8.0, and 77.68% at pH 10.0, respectively. These results indicate that the CIP degradation process of 10% $C_3N_4/LaFeCuO_3$ material is more favorable under acidic conditions compared to alkaline conditions. In acidic solution, more H^+ are available to react with $SO_4^{\bullet-}$ to form OH (as seen in the mechanism equations), and also CIP (a zwitterionic compound) may be more susceptible to radical attack in its protonated form [6], [14], [15].

As the dosage of 10%g-$C_3N_4/LaFeCuO_3$ was varied from 0.1g to 0.2g, the CIP removal efficiency increased from 84.3% to 90.5%. While the photocatalyst dosage increased from 0.2 g to 0.3g, the removal efficiency reached 96.3%. The observed improvement in CIP removal efficiency can be attributed to the increased availability of active sites resulting from the higher photocatalyst dosage, which enhanced the activation of ammonium persulfate (APS) and subsequently generated more reactive free radicals. Beyond 0.3 g, the presence of excessive catalyst can lead to scavenging of radicals (by the catalyst itself) and increased opacity of the suspension, resulting in a reduced reaction rate. Based on these findings, 0.3 g of g-$C_3N_4/LaFeCuO_3$ was selected as the optimal photocatalyst dosage for this study.

3. Conclusions

In summary, g-C_3N_4, $LaFeCuO_3$, and 10%g-$C_3N_4/LaFeCuO_3$ photocatalysts were successfully synthesized through a combined approach involving thermal decomposition, the sol-gel technique, and ultrasonic-assisted synthesis. The 10%g-$C_3N_4/LaFeCuO_3$

photocatalysts enhanced catalytic activity for the removal of ciprofloxacin. Under optimal conditions: 0.8 mL APS 0.25 M and 1.5 g/L of photocatalyst-the CIP removal efficiency reached approximately 96.3% within 180 minutes of irradiation using a 250 W xenon lamp. The primary reactive species responsible for CIP degradation were identified as hydroxyl radicals (${}^{\bullet}OH$), sulfate radicals ($SO_4^{\bullet-}$), and holes. Overall, this study presents an effective strategy for evaluating the catalytic potential of g-C_3N_4/$LaFeCuO_3$ heterojunction materials for APS activation, offering a promising approach for the treatment of organic pollutants in wastewater.

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