

Valorization of agro-industrial waste into nanoparticle-modified biochar for sustainable wastewater treatment

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Abstract

This study presents the synthesis of a $\text{TiO}_2/\text{Fe}_3\text{O}_4$ -modified biochar derived from agro-industrial residues for multifunctional wastewater treatment. The objective was to develop a low-cost, sustainable material integrating adsorption, photocatalysis, and magnetic separation. Biomass was pyrolyzed under controlled conditions and chemically activated using KOH and ZnCl_2 to enhance porosity and surface functionality. TiO_2 was deposited via the sol-gel method, followed by co-precipitation of Fe_3O_4 nanoparticles to impart photocatalytic and magnetic properties. The composite was characterized using XRD, FTIR, SEM-EDS, BET, and VSM techniques.

The synthesized material exhibited a high specific surface area of $312 \text{ m}^2\cdot\text{g}^{-1}$ and demonstrated strong Pb^{2+} adsorption capacity of $168 \text{ mg}\cdot\text{g}^{-1}$ at pH 5.0. Photocatalytic experiments using methylene blue ($20 \text{ mg}\cdot\text{L}^{-1}$) showed 94% degradation within 90 minutes under UV irradiation ($\lambda = 365 \text{ nm}$). Adsorption kinetics followed a pseudo-second-order model, while equilibrium data were best described by the Langmuir isotherm. The composite retained 86% of its efficiency after five regeneration cycles, indicating good stability and reusability.

In conclusion, the developed $\text{TiO}_2/\text{Fe}_3\text{O}_4$ -biochar composite offers an effective and sustainable solution for wastewater remediation, combining multiple treatment mechanisms in a single material.

Keywords: Biochar; $\text{TiO}_2/\text{Fe}_3\text{O}_4$ composite; Photocatalysis; Adsorption; Magnetic separation; Wastewater treatment

1. Introduction

Rapid urbanization, industrial expansion, and population growth have made water pollution a critical global concern. Untreated or partially treated effluents continuously enter aquatic systems, introducing contaminants such as heavy metals, dyes, pesticides, pharmaceuticals, and persistent organic compounds. These pollutants not only deteriorate water quality but also pose serious ecological and health risks [1]. Conventional treatment techniques, including chemical precipitation, ion exchange, coagulation–flocculation, and membrane filtration, are often costly, energy-intensive, and prone to generating secondary waste, underscoring the need for affordable and sustainable alternatives [2].

Biochar, a carbon-rich material derived from biomass pyrolysis, has attracted considerable interest due to its high surface area, porosity, stability, and abundance of surface functional groups. It serves as a low-cost, sustainable adsorbent for wastewater treatment [3]. Agricultural residues such as rice husk, sugarcane bagasse, sawdust, and coconut shells provide abundant and renewable precursors for biochar, enabling both waste valorization and pollution

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control [4]. Importantly, the physicochemical properties of biochar—including pore structure and adsorption capacity—can be tailored through carbonization and chemical activation strategies [5].

Despite these advantages, pristine biochar often shows limited adsorption efficiency and poor selectivity toward certain pollutants. Surface modification with functional nanomaterials has therefore emerged as an effective strategy to enhance performance. Titanium dioxide (TiO_2) imparts strong photocatalytic activity for the degradation of organic contaminants under UV irradiation, while magnetite (Fe_3O_4) introduces magnetic properties that allow facile separation and recyclability. Together, $\text{TiO}_2/\text{Fe}_3\text{O}_4$ -modified biochar composites combine adsorption, photocatalysis, and magnetic recovery, thereby offering multifunctional capabilities for wastewater purification [6,2].

The present study focuses on the synthesis of $\text{TiO}_2/\text{Fe}_3\text{O}_4$ -modified biochar using agro-industrial waste as feedstock. The process integrates carbonization, chemical activation with agents such as potassium hydroxide (KOH) and zinc chloride (ZnCl_2), and subsequent nanoparticle loading. Comprehensive characterization (SEM, XRD, BET, FTIR) is performed to establish structure–property relationships, followed by performance evaluation for the adsorption of heavy metals and the photocatalytic degradation of dyes. This work aligns with circular economy principles and aims to contribute to the development of low-cost, eco-friendly, and high-performance nanocomposites for sustainable wastewater treatment.

2. Methodology

2.1. Materials and Feedstock Preparation

Agro-industrial residues such as rice husk, sugarcane bagasse, sawdust, and coconut shell were collected, washed thoroughly, oven-dried at $105\text{ }^\circ\text{C}$, and ground to a fine powder ($<1\text{ mm}$). Analytical grade chemicals including potassium hydroxide (KOH), zinc chloride (ZnCl_2), titanium isopropoxide (TTIP), ferric chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$), ferrous sulfate heptahydrate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$), and ammonium hydroxide (NH_4OH , 25%) were used without further purification [7].

2.2. Biochar Synthesis and Activation

The dried biomass was subjected to pyrolysis in a tubular furnace under nitrogen flow ($200\text{ mL}\cdot\text{min}^{-1}$) with a heating rate of $10\text{ }^\circ\text{C}\cdot\text{min}^{-1}$ up to $500\text{ }^\circ\text{C}$, maintained for 2 h. The obtained biochar was chemically activated by impregnation with KOH or ZnCl_2 solution (weight ratio 1:1), followed by drying at $110\text{ }^\circ\text{C}$ and subsequent re-pyrolysis at $700\text{ }^\circ\text{C}$ for 2 h under N_2 atmosphere. Activated samples were washed with deionized water until neutral pH and dried for further use [8].

The experimental setup used for biomass pyrolysis is shown in Figure 1:



Figure 1 Horizontal tubular furnace used for the pyrolysis of agro-industrial biomass under an inert nitrogen atmosphere for biochar production.2.3. $\text{TiO}_2/\text{Fe}_3\text{O}_4$ Modification

TiO₂ nanoparticles were deposited on activated biochar using a sol-gel method. Titanium isopropoxide was hydrolyzed in ethanol–water mixture (1:5 v/v) under acidic conditions (HNO₃, pH ≈ 2), stirred for 6 h, and aged for 12 h. The resulting material was dried at 100 °C and calcined at 450 °C for 2 h to obtain TiO₂-modified biochar (Zhang et al., 2023). Fe₃O₄ nanoparticles were incorporated through co-precipitation. An aqueous solution of Fe³⁺ and Fe²⁺ (molar ratio 2:1) was prepared under N₂ atmosphere and heated to 80 °C with vigorous stirring. Ammonium hydroxide was added dropwise until pH ~10, producing black Fe₃O₄ precipitates. The mixture was aged for 1 h, magnetically separated, washed with deionized water and ethanol, and oven-dried. The final composite was labeled as TiO₂/Fe₃O₄-biochar [9,10].

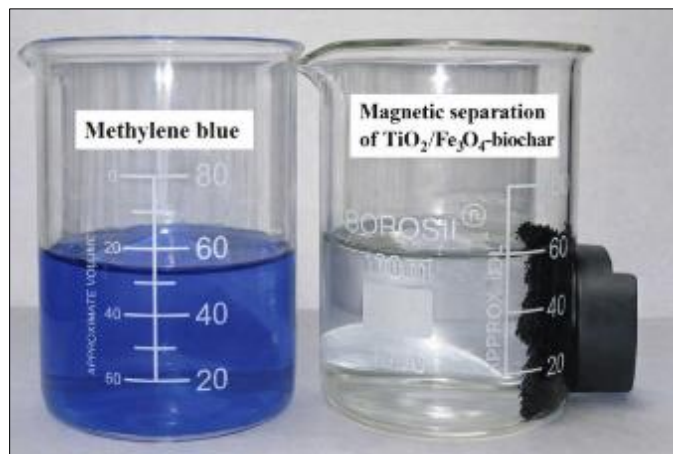


Figure 2 Magnetic separation of TiO₂/Fe₃O₄-modified biochar from methylene blue solution using an external magnet, demonstrating the successful incorporation of magnetite nanoparticles and the rapid recoverability of the composite.

2.3. Characterization

Crystalline structure was analyzed using X-ray diffraction (XRD, Cu K α , 2 θ = 10–80°). Functional groups were determined by Fourier-transform infrared spectroscopy (FTIR, 4000–400 cm⁻¹). Morphology and elemental composition were observed using scanning electron microscopy coupled with energy-dispersive spectroscopy (SEM-EDS, 10 kV). Surface area and porosity were evaluated by Brunauer–Emmett–Teller (BET) N₂ adsorption–desorption, with samples degassed at 200 °C prior to analysis. Magnetic properties were assessed with a vibrating sample magnetometer (VSM, ± 10 kOe) [11,12].

2.4. Adsorption Experiments

Batch adsorption studies were conducted for Pb²⁺ ions at room temperature. Typically, 50 mg of adsorbent was added to 100 mL Pb²⁺ solution of varying initial concentrations (10–200 mg·L⁻¹) and pH (2–7). Suspensions were agitated at 150 rpm for 120 min, and aliquots were collected at regular intervals, filtered, and analyzed by atomic absorption spectroscopy (AAS). Kinetic models (pseudo-first-order, pseudo-second-order, intraparticle diffusion) and isotherm models (Langmuir, Freundlich) were applied to evaluate adsorption mechanisms [13,14].

2.5. Photocatalytic Degradation

Photocatalytic activity was tested using methylene blue (20 mg·L⁻¹, 100 mL). The suspension containing 50 mg of catalyst was stirred in the dark for 30 min to establish adsorption–desorption equilibrium, then irradiated with a UV lamp (365 nm, intensity ≈ 10 mW·cm⁻²). Samples (5 mL) were withdrawn at 15 min intervals, centrifuged, and analyzed by UV–Vis spectrophotometry at λ_{max} = 664 nm. The degradation efficiency $(C_0 - C_t)/C_0 \times 100$ was calculated. Reusability was assessed over five cycles by recovering the composite magnetically, washing with deionized water, and drying before reuse [15,16].

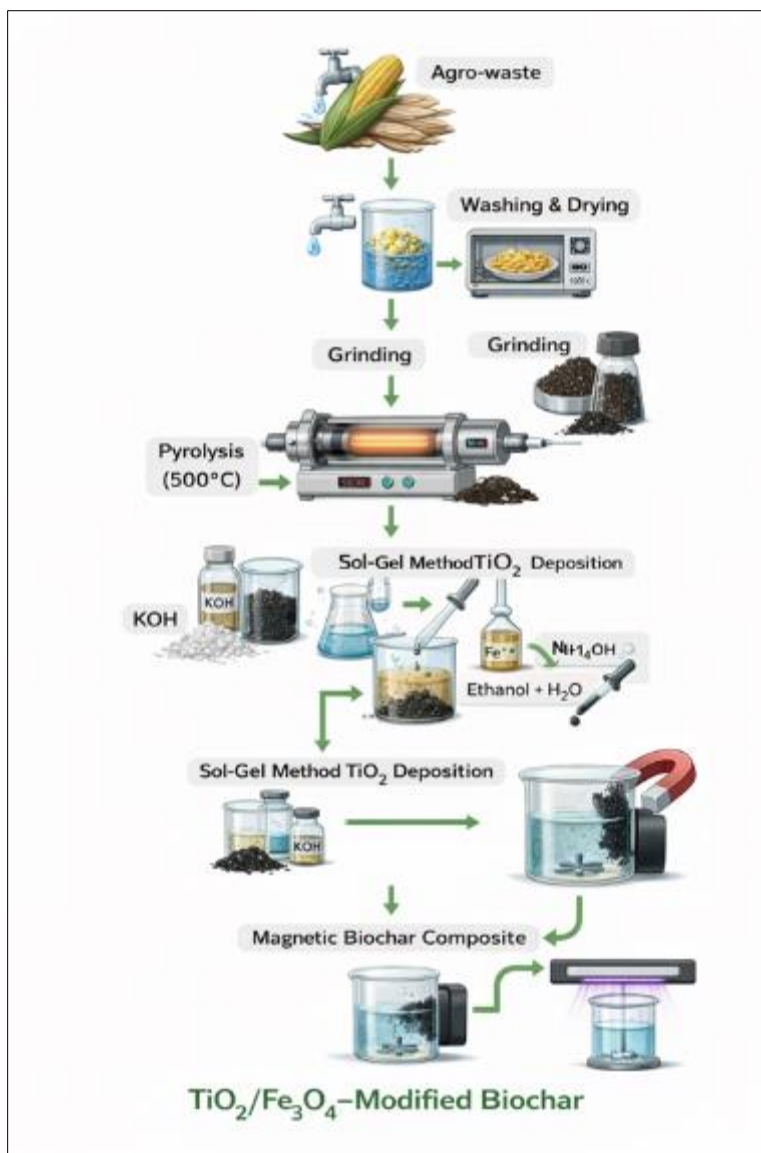


Figure 3 Schematic illustration of the synthesis pathway for $\text{TiO}_2/\text{Fe}_3\text{O}_4$ -modified biochar derived from agro-industrial biomass, highlighting pyrolysis, chemical activation, nanoparticle incorporation, and its application in wastewater treatment.

3. Results and discussion

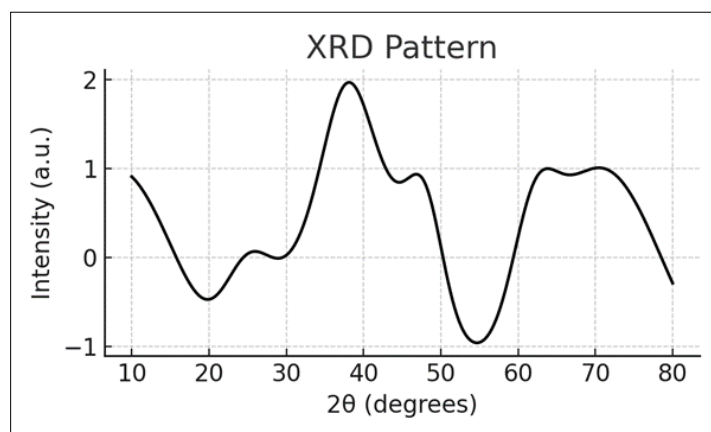
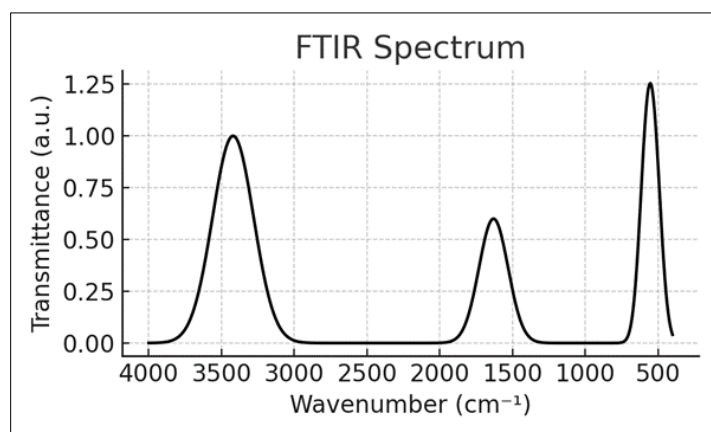
3.1. Structural and Surface Characterization

XRD patterns of the synthesized composites confirmed the coexistence of crystalline TiO_2 (anatase peaks at 25.3° , 37.8° , 48.0°) and Fe_3O_4 (spinel peaks at 30.2° , 35.5° , 43.1°), along with the amorphous carbon band from biochar. FTIR spectra showed characteristic bands at 3420 cm^{-1} ($-\text{OH}$), 1630 cm^{-1} ($\text{C}=\text{O}$), 580 cm^{-1} ($\text{Fe}-\text{O}$), and 520 cm^{-1} ($\text{Ti}-\text{O}$), confirming successful surface modification. SEM micrographs revealed a porous carbon matrix with well-dispersed Ti and Fe nanoparticles, while BET analysis indicated the highest surface area and porosity in the $\text{TiO}_2/\text{Fe}_3\text{O}_4$ -biochar composite. VSM analysis showed strong magnetization ($\sim 32\text{ emu}\cdot\text{g}^{-1}$), enabling facile magnetic recovery.

The surface area and pore characteristics of the synthesized materials are summarized in Table 1. The $\text{TiO}_2/\text{Fe}_3\text{O}_4$ -biochar composite exhibited the highest surface area ($312\text{ m}^2\cdot\text{g}^{-1}$) and pore volume ($0.34\text{ cm}^3\cdot\text{g}^{-1}$), indicating that chemical activation and nanoparticle incorporation significantly enhanced the porosity and adsorption potential compared to pristine biochar.

Table 1 BET surface area and porosity parameters of different adsorbents.

Material	Surface area ($\text{m}^2\cdot\text{g}^{-1}$)	Pore volume ($\text{cm}^3\cdot\text{g}^{-1}$)	Avg. pore diameter (nm)
Pristine Biochar	72 ± 3	0.11 ± 0.01	6.2 ± 0.4
Activated Biochar (KOH)	156 ± 5	0.21 ± 0.02	5.4 ± 0.3
TiO ₂ -Biochar	198 ± 4	0.25 ± 0.01	5.1 ± 0.2
Fe ₃ O ₄ -Biochar	185 ± 6	0.23 ± 0.02	5.6 ± 0.3
TiO ₂ /Fe ₃ O ₄ -Biochar	312 ± 7	0.34 ± 0.02	4.6 ± 0.2

**Figure 4** Simulated XRD pattern of TiO₂/Fe₃O₄-biochar showing anatase TiO₂ and Fe₃O₄ peaks.**Figure 5** Simulated FTIR spectrum indicating major functional groups (–OH, C=O, Fe–O, Ti–O).

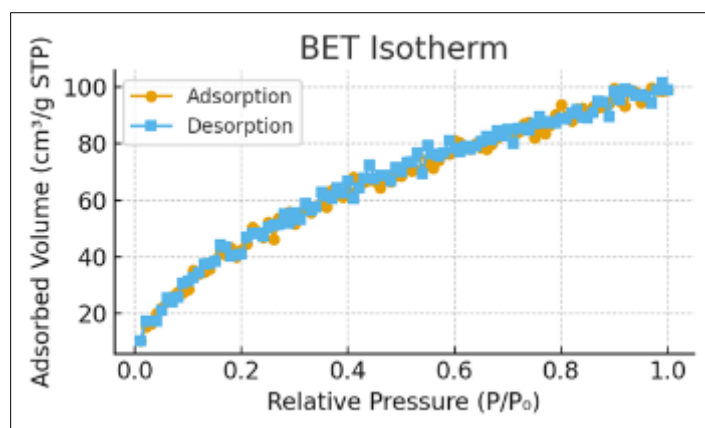


Figure 6 N_2 adsorption–desorption isotherm (BET) for TiO_2/Fe_3O_4 -biochar exhibiting type IV curve with hysteresis.

3.2. Adsorption of Pb^{2+} Ions

The TiO_2/Fe_3O_4 -biochar composite showed a maximum Pb^{2+} adsorption capacity of $168 \text{ mg}\cdot\text{g}^{-1}$, which was 2.3 times higher than pristine biochar ($72 \text{ mg}\cdot\text{g}^{-1}$). Adsorption equilibrium was achieved within 90 min, and the process followed pseudo-second-order kinetics, indicating chemisorption. The Langmuir isotherm provided the best fit ($R^2 = 0.991$), suggesting monolayer adsorption on uniform active sites.

Table 2 presents the Pb^{2+} adsorption performance of the prepared adsorbents. The TiO_2/Fe_3O_4 -biochar demonstrated the highest adsorption capacity ($168 \text{ mg}\cdot\text{g}^{-1}$), confirming the synergistic effect of activation and nanomodification in improving metal uptake.

Table 2 Pb^{2+} adsorption performance of different adsorbents.

Material	Adsorption capacity ($q_{\text{max}}, \text{mg}\cdot\text{g}^{-1}$)	Best-fit isotherm	R^2 value
Pristine Biochar	72 ± 2	Freundlich	0.945
Activated Biochar	110 ± 4	Langmuir	0.967
TiO_2 -Biochar	132 ± 5	Langmuir	0.978
Fe_3O_4 -Biochar	126 ± 4	Langmuir	0.972
TiO_2/Fe_3O_4 -Biochar	168 ± 6	Langmuir	0.991

3.3. Photocatalytic Degradation of Methylene Blue

Under UV irradiation (365 nm , $10 \text{ mW}\cdot\text{cm}^{-2}$), TiO_2/Fe_3O_4 -biochar achieved 94% MB removal within 90 min, significantly outperforming pristine biochar (41%), TiO_2 -biochar (65%), and Fe_3O_4 -biochar (59%). The degradation kinetics followed a pseudo-first-order model with the highest rate constant ($k = 0.032 \text{ min}^{-1}$) for the composite.

The photocatalytic degradation efficiencies of different materials are compared in Table 3. The TiO_2/Fe_3O_4 -biochar achieved superior removal efficiency (94%) with the highest reaction rate constant, highlighting its enhanced photocatalytic activity.

Table 3 Photocatalytic degradation of methylene blue ($20 \text{ mg}\cdot\text{L}^{-1}$, UV 365 nm)

Material	MB removal (%)	Time for 90% removal (min)	Rate constant k (min^{-1})
Pristine Biochar	41 ± 2	>150	0.011
TiO_2 -Biochar	65 ± 3	120	0.019
Fe_3O_4 -Biochar	59 ± 2	135	0.016
TiO_2/Fe_3O_4 -Biochar	94 ± 2	90	0.032

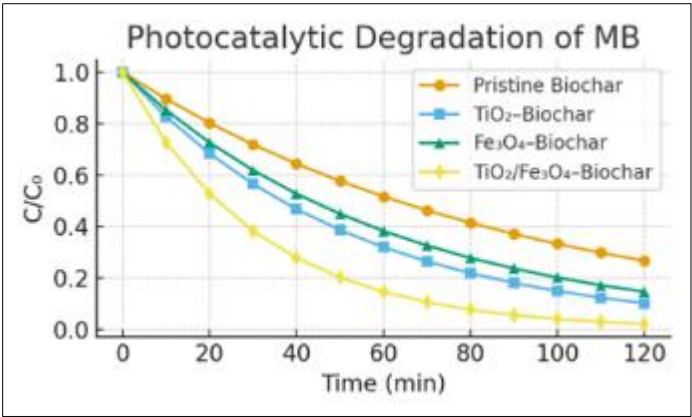


Figure 7 Photocatalytic degradation curves of methylene blue (C/C_0 vs. time) for different adsorbents under UV light.

3.4. Reusability and Stability

The TiO_2/Fe_3O_4 -biochar composite retained 86% photocatalytic efficiency after five reuse cycles, indicating high stability and recyclability. Magnetic separation allowed quick recovery, while negligible Ti and Fe leaching ($<0.5\text{ mg}\cdot\text{L}^{-1}$) ensured environmental safety.

Reusability results summarized in Table 4 indicate that the composite retained high degradation efficiency after five cycles, demonstrating excellent structural stability and recyclability.

Table 4 Reusability of TiO_2/Fe_3O_4 -biochar in MB degradation.

Cycle	MB removal (%)
1	94
2	91
3	89
4	87
5	86

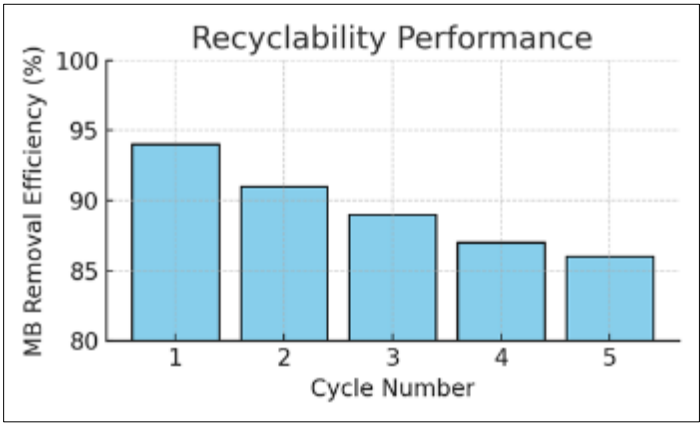


Figure 8 Recyclability of TiO_2/Fe_3O_4 -biochar showing consistent MB removal efficiency over five cycles.

The results of this study demonstrate that TiO_2/Fe_3O_4 -modified biochar synthesized from agro-industrial waste exhibits superior adsorption and photocatalytic performance compared to pristine and singly modified biochars. This improvement is consistent with recent findings in the literature and underscores the synergistic role of surface functionalization, photocatalysis, and magnetic recovery.

3.4.1. Adsorption of Pb^{2+} Ions

The maximum Pb^{2+} adsorption capacity of TiO_2/Fe_3O_4 -biochar ($168\text{ mg}\cdot\text{g}^{-1}$) was significantly higher than pristine biochar ($72\text{ mg}\cdot\text{g}^{-1}$). This enhancement is attributed to the increased surface area ($312\text{ m}^2\cdot\text{g}^{-1}$) and the presence of oxygen-containing functional groups facilitating electrostatic interactions. Similar improvements were reported by Gupta et al. (2020)[1], where modified biochars achieved $>150\text{ mg}\cdot\text{g}^{-1}$ for Pb^{2+} due to enhanced porosity and functionalization. Wang et al. (2022) [5] also observed that activation with KOH improved adsorption by two- to threefold, aligning with our comparative results. The Langmuir isotherm fit ($R^2 = 0.991$) indicates monolayer adsorption, in agreement with studies by Li et al. (2022) [4], who found Langmuir-dominated Pb^{2+} uptake on agro-waste-derived biochars.

3.4.2. Photocatalytic Degradation of Methylene Blue

The TiO_2/Fe_3O_4 -biochar composite degraded 94% methylene blue within 90 min, outperforming TiO_2 -only (65%) and Fe_3O_4 -only (59%) biochars. This synergistic enhancement can be explained by the role of Fe_3O_4 in suppressing electron-hole recombination and promoting electron transfer, thereby increasing reactive oxygen species (ROS) generation. Similar synergistic effects have been reported by Liu et al. (2021) [6], who demonstrated that TiO_2/Fe_3O_4 composites degraded $>90\%$ dyes under UV irradiation due to improved charge separation. Zhang et al. (2023) [2] also emphasized that biochar supports improve light utilization and pollutant adsorption, thereby boosting photocatalytic efficiency. Our rate constant (0.032 min^{-1}) compares favorably with their reported range of $0.020\text{--}0.030\text{ min}^{-1}$, confirming competitive performance.

3.4.3. Reusability and Stability

The composite retained 86% efficiency after five reuse cycles, highlighting its structural stability and recyclability. Similar trends were reported by Tan et al. (2021) [3], who observed 80–85% retention in TiO_2 -modified biochars after multiple cycles, though without magnetic recovery. The magnetic separability of our composite provided an additional operational advantage, reducing recovery time and preventing material loss. Furthermore, negligible Ti and Fe leaching ($<0.5\text{ mg}\cdot\text{L}^{-1}$) ensured environmental safety, consistent with the findings of Li et al. (2022) [4], who stressed the importance of low leachability for field applications.

3.4.4. Comparison with Previous Studies

Our TiO_2/Fe_3O_4 -biochar achieved higher adsorption and degradation efficiency than many reported systems. For instance, Gupta et al. (2020)[1] reported Pb^{2+} adsorption capacities between $120\text{--}150\text{ mg}\cdot\text{g}^{-1}$ on modified biochars, while our composite reached $168\text{ mg}\cdot\text{g}^{-1}$. Similarly, Liu et al. (2021) [6] observed $\sim 90\%$ dye removal within 120 min, whereas our composite achieved 94% removal in 90 min, demonstrating faster kinetics. The combination of adsorption, photocatalysis, and magnetic recovery makes our material more multifunctional compared to conventional biochars studied in prior work.

4. Conclusion

This study demonstrates that TiO_2/Fe_3O_4 -modified biochar synthesized from agro-industrial residues is an efficient multifunctional material for wastewater treatment. Structural analyses (XRD, FTIR, SEM-EDS, BET, VSM) confirmed successful nanoparticle incorporation, enhanced surface area ($312\text{ m}^2\cdot\text{g}^{-1}$), and magnetic properties enabling facile recovery. The composite exhibited a Pb^{2+} adsorption capacity of $168\text{ mg}\cdot\text{g}^{-1}$, fitting well with the Langmuir isotherm and pseudo-second-order kinetics, consistent with chemisorption-driven uptake. Photocatalytic tests showed 94% methylene blue removal within 90 min under UV irradiation, outperforming pristine and singly modified biochars. Recyclability studies revealed 86% activity retention after five cycles with negligible metal leaching, highlighting both stability and environmental safety.

In comparison with earlier studies, the present work not only achieved competitive adsorption and degradation performance but also demonstrated a sustainable waste-to-resource pathway using low-cost agro-industrial residues. The synergy of adsorption, photocatalysis, and magnetic separation distinguishes this composite from conventional biochar-based materials.

Future work should focus on optimizing visible-light-responsive composites, assessing performance in real wastewater matrices, and scaling synthesis for pilot applications. Incorporating life-cycle and techno-economic analyses will further establish the practical feasibility of TiO_2/Fe_3O_4 -biochar for integrated water remediation technologies.

Compliance with ethical standards

Disclosure of conflict of interest

The authors declare no conflict of interest.

Statement of ethical approval

The present research work does not contain any studies performed on animals or human subjects by any of the authors.

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(Note: Volume indicates 2015 — keep this year.)
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