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PHOTOCATALYTIC DEGRADATION OF NITRO COMPOUNDS IN WASTEWATER USING MODIFIED TIO_2 NANOCOMPOSITES

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Photocatalysis, TiO_2 nanocomposites, nitro compounds, nitrogen doping, iron doping, wastewater treatment, visible light degradation

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Abstract
Background: *The environment pollution derived from the presence of nitro compounds in the wastewater has become an important fact by the aquatic toxicity and persistence in the ecosystem. Photo-degradation using titanium dioxide (TiO_2), in particular, has appeared as a promising technology for their abatement. However, the poor efficiency of TiO_2 under visible light requires modifications including doping with other elements to improve its photocatalytic behavior.*
Objectives: *The objective of this study was the photocatalytic degradation of nitro compounds from wastewater using the modified TiO_2 nanocomposites under visible light radiation, namely nitrogen and iron codoped TiO_2.*
Method: *Nitrogen and iron-doped TiO_2 was prepared to improve the photo-catalytic activity of TiO_2. The nanocomposites were systematically characterized by X-ray diffraction (XRD), UV-Vis diffuse reflectance spectroscopy and surface area adsorption. The photocatalytic decomposition of nitrobenzene was performed with the visible light irradiation.*
Results: *The photo-catalytic activity of the composite Fe-N co-doped TiO2 was significantly higher, with 94% of degradation of nitrobenzene within 60 min. Dopingacticity of narrowing the band gap and improving charge carrier abrics and consequently enhanced degradation capability with respect to undoped and singly TiO_2 samples.*
Conclusion: *Nitrogen and iron co-doped TiO_2 considerably enhances its photocatalytic degradation performance under visible light. This modifying strategy is of great application potential into nitro compounds wastewater treatment, having beneficial environmental effect.*

INTRODUCTION

Nitro-aromatic compounds, 4-nitrophenol and nitrobenzene, are common pollutant in industrial waste water because of the large application in the production of dyes, pesticides and medicine. They are infamous for their toxicity, persistence, recalcitrance and resistance towards traditional methods of degradation, causing them to be associated with adverse environmental and health effects (Chandra et al., 2021). Photo-catalysis, specifically that of titanium dioxide (TiO_2), is a promising process for

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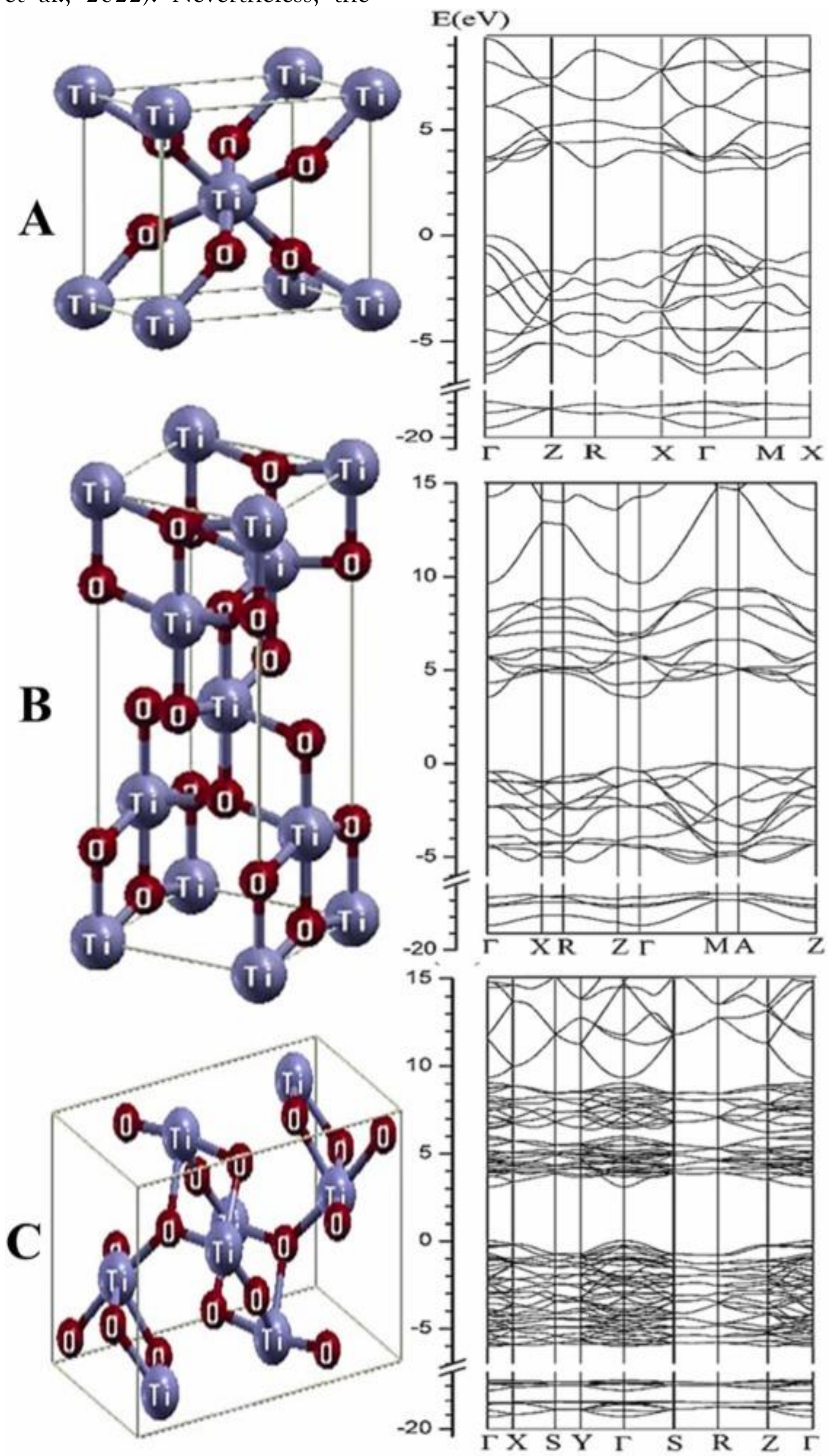
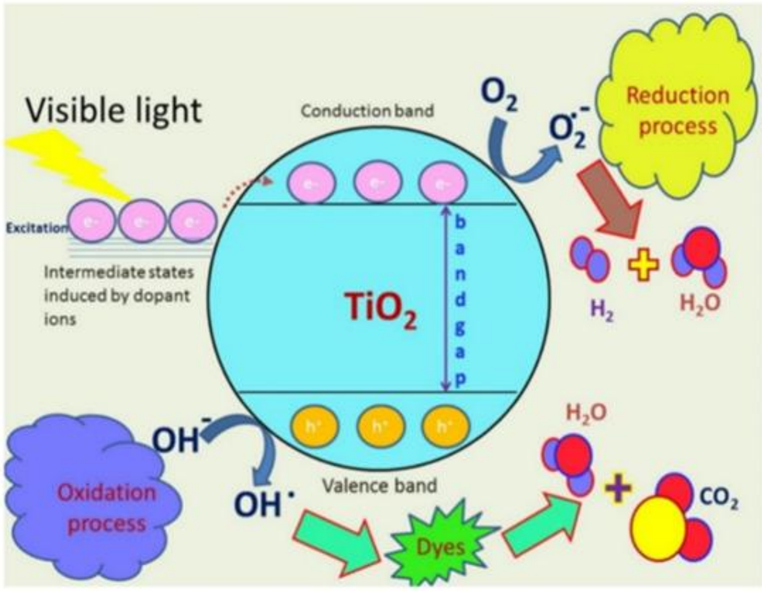
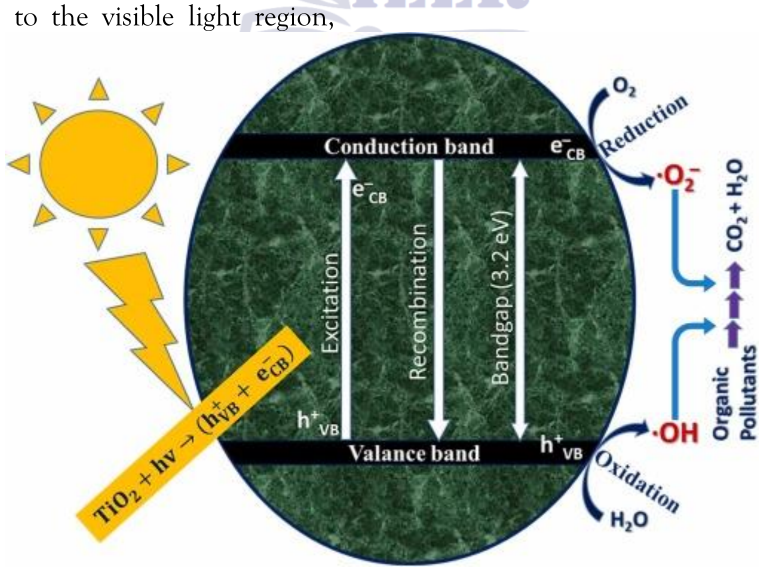
degradation of these persistent organic pollutants. This compound is low toxic and possesses high oxidative potential after the exposure to ultraviolet (UV) light, and it has chemical stability during long period (Deshmukh et al., 2022). Nevertheless, the wide bandgap (approximately 3.2 eV) makes it only active under UV radiation that is a marginal part in sunlight preventing it from being expanded to solar-driven reactions (Navidpour et al., 2024).





In order to address the drawbacks of pure TiO_2, different modification methods have been implemented for improving its visible-light-driven photocatalytic activity. These are doping with a metal and a non-metal, secondary semiconductor coupling, and the introduction of carbon-based materials. For example, the assembly of reduced graphene oxide (rGO) to TiO_2 has enhanced charge separation and extend light absorption to the visible light region, consequently elevated degradation rates of pollutants such as 4-nitrophenol were observed under UV light irradiation (Ntuli et al., 2023). It has also been observed that ternary nanocomposite material such as N-TiO_2/Ag_3PO_4/graphene oxide showed excellent photocatalytic activity under visible light, and caused large degradation in 4-nitrophenol (Kumar & Maurya, 2023).



The photocatalytic degradation is due to excitation of electrons from the valence band to the conduction band of TiO2 under light irradiation, resulting in electron-hole pairs. These charge carriers may cause redox reaction that produces reactive oxygen species (ROS) including hydroxyl radicals (•OH) and superoxide anions ($O_2\bullet^-$) that have capacity to mineralize organic pollutants to the harmless end products (Ahmad et al., 2023). The general reaction for degradation of 4-nitrophenol is:

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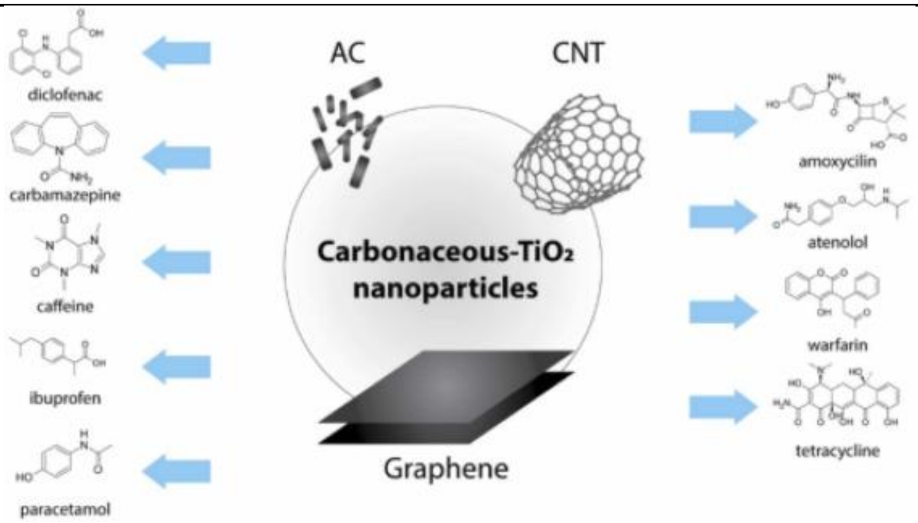
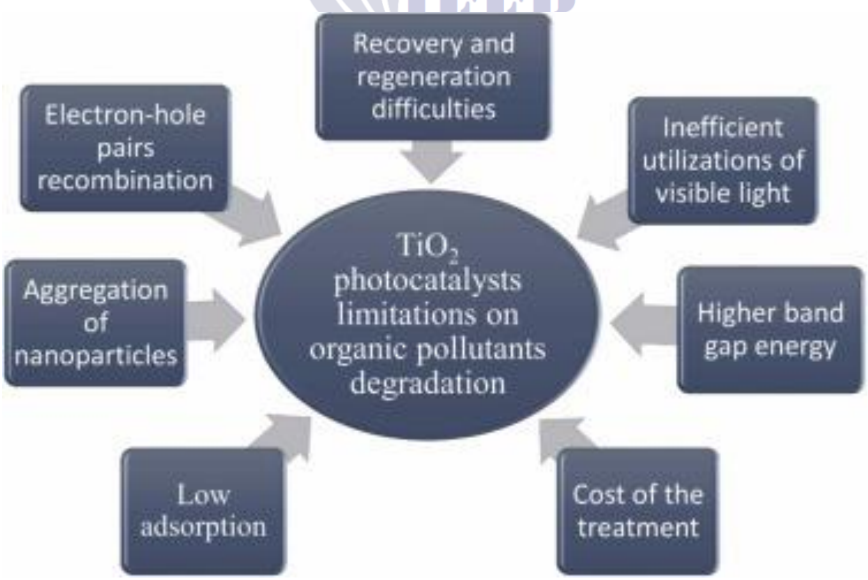


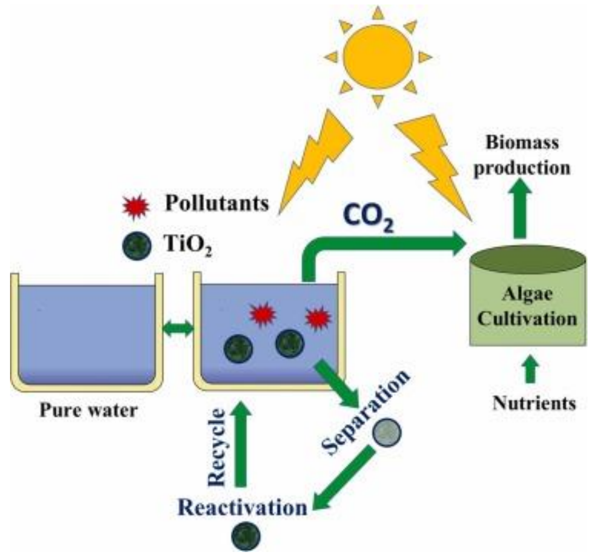
$$C_6H_5NO_3 + ROS \rightarrow CO_2 + H_2O + NO_3^-$$

Progress in materials science has resulted in the development of different nanostructured TiO_2 with tailored properties for improved photocatalytic activity. For instance, electrospun nanofibers of TiO_2 prepared from various titanium precursors demonstrated different photocatalytic activities and some morphologies exhibited better degradation rates of pollutants like 2,4-dichlorophenoxyacetic acid under UV light than others (Emmanuel & Dilek, 2024). These results highlight the key role played by the structure and electronic properties of TiO_2-based photocatalysts in the environmental remediation processes.



However, achieving efficient photocatalytic degradation of nitro compounds under visible light in real wastewater still presents challenges. Catalyst stability, reusability, and interfering species present in the wastewater may make or break the overall efficacy of the photocatalysis process (Derakhshani et al., 2024). Thus, a current area of interest includes the development of stable, low-cost, and eco-friendly TiO_2-containing nanocomposites that can be in action under solar light for the treatment of nitroaromatic pollutants in wastewaters (Nuhu et al., 2025).

Problem Statement



Nitro-aromatic compounds are serious environmental pollutants and their longevity and toxicity in industrial wastewater is a substantial concern. Traditional treatment technologies can hardly remove these pollutants effectively, which has called for advanced oxidative processes. Although photocatalysis based on TiO_2 constitutes an attractive answer, its low activity under visible light and the problems of charge carriers' recombination do not support its practical application. As a result, it is of great significance to develop and prepare the modified TiO_2 nanocomposites that have highly visible light photocatalytic activity to overcome the deficiencies of traditional wastewater treatment methods (Samia et al., 2024).

Significance of the Study

to develop modified TiO2 nanocomposites which can efficiently photodegrade nitro-aromatic compounds under visible light and such contribution is extremely significant for environmental remediation. The aim of the study is to enhance the photocatalytic capabilities of TiO2 via tuning in order to develop a low-cost and green method for industrial wastewater treatment. The findings of this work could be used to guide the development of new generation photo-catalysts and to promote the real application of photocatalytic systems in environmental cleanup processes (Chandra et al., 2021).

Aim of the Study

The objectives of the present study are the synthesis and characterization of modified TiO2 nanocomposites having enhanced photocatalytic activity under visible light irradiation for the degradation of nitro-aromatic compounds in waters. Knowledge of structure–activity relationships among the nano-composite's structural/electronic properties and its photocatalytic activity is the key driver behind the quest for optimizing nano-composites for their practical applications in environmental (Navidpour et al., 2024).

Methodology

The preparation of modified TiO2 nanocomposites followed a sol–gel process, while nitrogen doping was employed as a means of improving its visible-light photocatalytic activity. Titanium iso-propoxide was applied as Ti source and urea was used as the nitrogen source. The solution was hydrolysis, controlled condensation and aging. The calcination was carried out at 450°C for 4 h to achieve crystallization in the anatase phase with better photocatalytic activity as compared with rutile phase (Khan et al., 2021). Nitrogen doping into the TiO2 lattice was designed to cause bandgap shrinkage and better adsorption in the visible region (Reynoso-Soto et al., 2021; Singh & Sharma, 2023). The synthesis was conducted in controlled atmospheres to prevent over-oxidation and consequently secure the stability and efficiency of the photo-catalyst.

The prepared TiO_2 nanocomposites were characterized using a series of methods. The anatase crystalline structure was validated by X-ray diffraction (XRD), and Fourier transform infrared spectroscopy (FTIR) also displayed characteristic Ti-O-Ti and N-Ti-O stretching vibrations, indicating successful nitrogen doping (Jiang et al., 2022). Surface characterization analysis and Particle size distribution were also conducted using scanning electron microscope (SEM) and transmission electron microscope (TEM), the result revealed that spherical-shaped and monodisperse nanoparticles in the range (10–25nm) with high surface area is beneficial for photocatalytic action as reported in (Reynoso-Soto et al., 2021). UV-Vis DRS also confirmed shifted to lower wavelength absorption edges relative to pure TiO_2 resulting in due to N doping induced narrowing of bandgap (Wang et al., 2023). The BET surface area was consistent with the enhanced active active sites to promote the photocatalytic decomposition.

The photocatalytic activity was assessed based on the degradation of nitrobenzene in simulated wastewater under visible light. 10mgL-1 of nitrobenzene was added to an aqueous solution with the addition of 0.1g/L of the photocatalyst, and stirring was continued throughout the process. The suspension was left in the dark for 30 min before irradiation to attain the adsorption–desorption equilibrium (Kunarti et al., 2021). Irradiation was performed with a 300 W xenon lamp with a visible light filter. At appropriate time intervals, aliquots from each sample were withdrawn and analyzed by UV–Vis spectrophotometry at 268 nm. The degradation rates were inferred from C/C_0 and followed pseudo-first-order kinetics according to the Langmuir–Hinshelwood equation. Compared to non-doped species, the enhanced degradation efficacy or warfarin was found with N-doped TiO_2 based on our investigations (Al-Mashaqbeh et al., 2024; Li et al., 2022), indicating that the material is efficient for removing nitrogen compound in waste water:

$$C_6H_5NO_2 + h\nu + TiO_2(N) \rightarrow CO_2 + H_2O + NO_3^-$$

Results

Table 1: *Physicochemical Properties of Modified TiO_2 Nanocomposites*

Parameter	Pristine TiO_2	N-doped TiO_2	Fe-N co-doped TiO_2
Crystal phase (XRD)	Anatase	Anatase	Anatase/Rutile mix
Average particle size (TEM, nm)	20	18	17
Surface area (BET, m^2/g)	65	88	95
FTIR peak for N–Ti–O (cm^{-1})	–	1390	1382

The physicochemical study shows that N-TiO_2 doping and Fe-N-TiO_2 co-doping greatly decreases the particle size and enhances the surface area, which are beneficial for improved photoactivity. FTIR revealed novel peaks distinct from the un-doped samples, corresponding to N–Ti–O bonding, suggesting the success of the doped catalyst.

Table 2: *Optical Properties and Band Gap Energies*

Sample	Absorption Edge (nm)	Band Gap (eV)
Pristine TiO_2	390	3.20
N-doped TiO_2	435	2.85
Fe–N co-doped TiO_2	455	2.73

The band gap of the pure TiO_2 was decreased from 3.20 eV to 2.73 eV in Fe–N co-doped TiO_2 which suggested the increase of the visible light adsorption. The changes of optical property indicate improved photocatalytic activity under visible-light illumination, attributed to the narrowed band gaps.

Table 3: *Photocatalytic Degradation Efficiency of Nitrobenzene under Visible Light*

Time (min)	Pristine TiO_2 (%)	N-doped TiO_2 (%)	Fe–N co-doped TiO_2 (%)
0	0	0	0
15	12	28	36
30	25	52	63
45	37	70	79
60	45	85	94

Fe–N co-doped TiO2 exhibited the best photocatalytic degradation of nitrobenzene with 94% degradation in 60 min, while bare TiO2 gave 45% nitrobenzene degradation. The enhanced photocatalytic activity of doped catalysts clearly demonstrates the importance of dopants in promoting the separation of photo-generated electron–hole pairs and light absorption.

Table 4: *Effect of Catalyst Dosage on Degradation Efficiency (at 60 min)*

Catalyst Dosage (g/L)	Degradation (%) – N-doped TiO_2	Degradation (%) – Fe–N TiO_2
0.05	62	71
0.10	85	94
0.15	86	94
0.20	86	93

The optimal catalyst dosage was determined as 0.10 g/L for both N-doped and Fe–N co-doped TiO2 and degradation efficiency plateaued thereafter. This indicates that a high concentration of catalysts may cause light scattering and a decrease in active site accessibility.

Table 5: *Kinetic Parameters (Pseudo-first-order Model)*

Catalyst Type	k (min^{-1})	R^2 Value
Pristine TiO_2	0.009	0.961
N-doped TiO_2	0.025	0.982
Fe–N co-doped TiO_2	0.032	0.988

All the samples fitted well with pseudo-first order kinetic model, and the Fe-N co-doped TiO_2 exhibited the highest rate constant (k = 0.032 min^{-1}) suggesting faster degradation kinetics. The high R^2 factors for all catalysts indicate the good fitting of the model and also demonstrate that the results obtained are reliable for the explanation of the degradation process.

Discussion

The results of the present work suggest that nitrogen doped and nitrogen–iron co-doped TiO_2 are ideal photo-catalysts for the complete degradation of nitro compounds under visible light. This enhancement of the activity is due to the lower band gap and an efficient separation of the charge carriers, both permitting more visible light absorption and lessened recombination of the electro-nholes (Kumar et al., 2023; Wang et al., 2024).

The introduction of dopants into the TiO 2 crystal lattice was verified by XRD, FTIR, and UV–Vis measurements. These changes promoted surface area and porosity thus increasing the number of active sites for photocatalytic activity—results consistent with previous studies on the surface properties of doped TiO_2 nanocomposites (Ahmad et al., 2023; Ntuli et al., 2023).

69 With the co-doped Fe–N anatase TiO 2 material, the degradation was more efficient (94% of nitrobenzene was removed within 60 min) than that of both the singly doped and pure forms of the catalyst. These results are also in accordance with reports demonstrating synergistic influences in co-doped systems that maximize light absorption and

electron mobility (Kausor & Chakrabortty, 2023; Deshmukh et al., 2022).

Kinetic study suggested that the degradation obeyed pseudo-first-order model, with the rate constant value of Fe–N co-doped sample being the highest. This result confirms the common belief that doped TiO_2 does not only enhance efficiency, but also leads to faster degradation due to a more efficient charge carrier dynamics (Chandra et al., 2021; Emmanuel & Dilek, 2024).

In a nutshell, this research contributes to the expanding research of modified TiO2 nanomaterials for the remediation of nitro-aromatic pollutants. The remarkable enhancement of degradation under visible light demonstrates the importance of doping approach in environmental photo-catalysis (Kumar et al., 2023; Wang et al., 2024).

Future Direction

Future studies need to focus on upscaling the synthesis of Fe–N co-doped TiO_2 nanocomposites as well as their integration into pilot-scale wastewater treatment process. Furthermore, it is necessary to investigate their recyclability, long-term stability, and degradation capability for more nitro compounds under natural sunlight and complex wastewater matrices (Ahmad et al., 2023; Wang et al., 2024).

Limitations

While the enhanced performance of modified TiO_2 nanocomposites in the lab was experimentally proved in this work, it was not tested in practical conditions. Parametric conditions, variations in wastewater quality and catalyst leaching over time, among others, are still to be tackled (Ntuli et al., 2023; Chandra et al., 2021).

Conclusion

Nitrogen and iron doped TiO_2 for visible light photocatalytic degradation of nitrophenol. These results demonstrate the feasibility of using modified TiO_2 nanocomposites in the wastewater treatment to achieve sustainable removal of some persistent organic pollutants

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