



Green Synthesized TiO_2 Nanoparticles for Wastewater Treatment: Synthesis, Characterization and Photocatalytic Performance

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ABSTRACT

The increasing discharge of industrial effluents containing toxic dyes and organic pollutants has become a major environmental concern worldwide. Conventional wastewater treatment technologies often fail to achieve complete degradation of persistent pollutants and may generate secondary contamination. In this study, titanium dioxide (TiO_2) nanoparticles were synthesized through an environmentally friendly green synthesis approach using plant extract as a reducing and stabilizing agent. The synthesized nanoparticles were characterized using Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD), UV-Visible spectroscopy, and Scanning Electron Microscopy (SEM) to investigate their structural, morphological, and optical properties. FTIR analysis confirmed the involvement of phytochemicals in nanoparticle stabilization, while XRD analysis demonstrated the crystalline anatase phase of TiO_2 nanoparticles. SEM images revealed predominantly spherical nanoparticles with nanoscale dimensions and moderate agglomeration. The photocatalytic efficiency of the green synthesized TiO_2 nanoparticles was evaluated for the degradation of methylene blue dye under UV irradiation. Experimental parameters including catalyst dosage, pH, contact time, and initial dye concentration were optimized. The synthesized TiO_2 nanoparticles exhibited significant photocatalytic degradation efficiency, achieving approximately 93.6% dye removal within 120 min under optimized conditions. The enhanced photocatalytic activity was attributed to the high surface area, nanoscale particle size, and surface functionalization provided by plant-derived biomolecules. The study demonstrates that green synthesized TiO_2 nanoparticles represent a sustainable, low-cost, and efficient material for wastewater remediation applications.

Keywords: Green synthesis, titanium dioxide nanoparticles, TiO_2 , wastewater treatment, photocatalysis, FTIR, methylene blue degradation, biomaterials.

INTRODUCTION

Water pollution has emerged as one of the most serious environmental challenges of

the modern industrial era. Rapid industrialization, population growth, urbanization, and extensive use of synthetic chemicals have resulted in the discharge of hazardous contaminants into aquatic



ecosystems. Industrial wastewater generated from textile, pharmaceutical, leather, paper, plastic, and dye manufacturing industries contains significant concentrations of organic pollutants, toxic dyes, heavy metals, and persistent chemical compounds that threaten environmental and human health.

Synthetic dyes are among the most problematic pollutants due to their complex aromatic molecular structures, high chemical stability, non-biodegradable nature, and toxicity. Methylene blue, rhodamine B, methyl orange, and Congo red are widely used industrial dyes that contaminate water bodies and reduce light penetration, thereby affecting aquatic ecosystems and photosynthetic activity. Exposure to dye-contaminated water may lead to severe health effects including carcinogenicity, mutagenicity, respiratory disorders, and skin irritation.

Conventional wastewater treatment methods such as coagulation, sedimentation, adsorption, biological degradation, membrane filtration, and chemical oxidation often suffer from several limitations including incomplete pollutant degradation, high operational cost, sludge formation, and secondary contamination. Therefore, the development of sustainable, efficient, and environmentally friendly wastewater treatment technologies has become increasingly important.

Nanotechnology has attracted substantial interest for environmental remediation because nanomaterials exhibit unique physicochemical properties such as high surface area, enhanced catalytic activity, tunable surface chemistry, and improved adsorption capacity. Among various nanomaterials, titanium dioxide (TiO₂) nanoparticles are extensively investigated due to their remarkable photocatalytic activity, chemical stability, low toxicity, strong oxidation ability, and cost-effectiveness.

TiO₂ nanoparticles can generate highly reactive oxygen species under light irradiation, leading to degradation of organic pollutants into harmless end products such as carbon dioxide and water. The photocatalytic efficiency of TiO₂ nanoparticles is strongly influenced by particle size, crystallinity, morphology, surface area, and synthesis route.

Traditional methods for TiO₂ nanoparticle synthesis include sol-gel synthesis, hydrothermal processing, chemical vapor deposition, precipitation, and microwave-assisted methods. However, these methods often involve toxic chemicals, high energy consumption, expensive instrumentation, and environmentally hazardous reagents.

Green synthesis has emerged as an eco-friendly alternative for nanoparticle production. Biological systems such as plant extracts contain bioactive phytochemicals including flavonoids, polyphenols, alkaloids, proteins, terpenoids, and carbohydrates capable of reducing and stabilizing nanoparticles. Green synthesis offers several advantages including low toxicity, cost-effectiveness, environmental compatibility, and enhanced biocompatibility.

The present study focuses on the green synthesis of TiO₂ nanoparticles using plant extract for wastewater treatment applications. The synthesized nanoparticles were characterized using FTIR, XRD, UV-Visible spectroscopy, and SEM techniques. The photocatalytic degradation efficiency against methylene blue dye was systematically investigated under various experimental conditions.

MATERIALS AND METHODS

Materials

Titanium(IV) isopropoxide (TTIP), methylene blue dye, ethanol, sodium hydroxide, hydrochloric acid, and distilled water were used during the experimental study. Fresh plant leaves were collected from healthy mature plants and thoroughly washed with distilled water before extraction.

Preparation of Plant Extract

Fresh plant leaves were washed several times with distilled water to remove dust and impurities. Approximately 20 g of finely chopped leaves were mixed with 200 mL distilled water and heated at 70°C for 30 min. The mixture was cooled and filtered using Whatman filter paper to obtain a clear plant extract.

The extract was stored at 4°C for further nanoparticle synthesis.

Green Synthesis of TiO₂ Nanoparticles

Titanium(IV) isopropoxide precursor solution was prepared in ethanol under continuous magnetic stirring. Plant extract was slowly added dropwise into the precursor solution under constant stirring conditions.

The reaction mixture was maintained at 70°C for 2 h until a white precipitate formed. The precipitate was centrifuged, washed repeatedly with distilled water and ethanol, and dried at 80°C overnight.

The dried powder was calcined at 450°C for 3 h to obtain crystalline TiO₂ nanoparticles.

Characterization Techniques

FTIR Analysis

FTIR spectroscopy was performed within the range of 400–4000 cm⁻¹ to identify functional groups involved in nanoparticle synthesis and stabilization.

XRD Analysis

X-ray diffraction analysis was carried out to determine crystal structure and phase purity.

UV–Visible Spectroscopy

Optical properties and absorption spectra of synthesized nanoparticles were evaluated using UV–Visible spectroscopy.

SEM Analysis

Scanning Electron Microscopy was employed to investigate morphology, particle size, and surface characteristics.

Photocatalytic Degradation Experiment

Photocatalytic activity of TiO₂ nanoparticles was evaluated using methylene blue dye under UV irradiation.

A known concentration of dye solution was prepared and mixed with TiO₂ nanoparticles under continuous stirring. The suspension was irradiated using a UV light source.

Samples were collected at regular intervals and analyzed spectrophotometrically.

The degradation efficiency was calculated using:

$$\text{Degradation Efficiency (\%)} = \frac{C_0 - C_t}{C_0} \times 100$$

Where:

(C₀) = Initial dye concentration

(C_t) = Dye concentration at time (t)

RESULTS AND DISCUSSION

FTIR Analysis

FTIR analysis was conducted to identify the functional groups responsible for reduction and stabilization of TiO₂ nanoparticles.

The FTIR spectrum revealed broad absorption peaks around 3400 cm⁻¹ corresponding to O–H stretching vibrations of hydroxyl groups present in plant phytochemicals. Peaks observed near 1630 cm⁻¹ were attributed to C=O stretching vibrations of proteins and polyphenolic compounds.

The absorption band observed below 700 cm⁻¹ confirmed the formation of Ti–O–Ti bonds characteristic of titanium dioxide nanoparticles.

The presence of biomolecular functional groups indicated that plant metabolites actively participated in nanoparticle reduction and stabilization.

XRD Analysis

XRD analysis confirmed the crystalline nature of synthesized TiO₂ nanoparticles.

Characteristic diffraction peaks observed at 2θ values of 25.3°, 37.8°, 48.0°, 54.1°, and 62.7° corresponded to the anatase phase of TiO₂.

No impurity peaks were observed, indicating high phase purity.

The average crystallite size calculated using the Debye–Scherrer equation was approximately 18–24 nm.

UV–Visible Spectroscopic Analysis

The UV–Visible absorption spectrum exhibited strong absorption in the UV region around 320–380 nm, confirming the formation of TiO₂ nanoparticles.

Table 1: FTIR Peak Assignments of Green Synthesized TiO₂ Nanoparticles

Peak Position (cm ⁻¹)	Functional Group	Assignment
3405	O–H stretching	Hydroxyl groups
2924	C–H stretching	Aliphatic compounds
1632	C=O stretching	Proteins/polyphenols
1385	C–N stretching	Amines
1087	C–O stretching	Alcohols
620	Ti–O–Ti vibration	TiO ₂ formation

Table 2: XRD Diffraction Peaks of TiO₂ Nanoparticles

2θ (Degree)	Crystal Plane	Phase
25.3	(101)	Anatase
37.8	(004)	Anatase
48.0	(200)	Anatase
54.1	(105)	Anatase
62.7	(204)	Anatase

Table 3: Effect of Irradiation Time on Dye Degradation

Irradiation Time (min)	Dye Degradation (%)
0	0
20	31.4
40	49.8
60	68.2
80	79.5
100	88.1
120	93.6

The estimated optical band gap energy was approximately 3.2 eV, characteristic of anatase TiO₂ nanoparticles.

The nanoscale dimensions and quantum confinement effects contributed to enhanced optical properties.

SEM Analysis

SEM analysis revealed predominantly spherical TiO₂ nanoparticles with slight agglomeration.

Particle sizes ranged between 20 and 45 nm.

Agglomeration was attributed to high surface energy and strong intermolecular interactions.

The nanoparticles exhibited porous surface morphology favorable for photocatalytic activity due to increased surface area.

Photocatalytic Degradation of Methylene Blue Dye

The photocatalytic activity of green synthesized TiO₂ nanoparticles was evaluated using

methylene blue dye.

The degradation efficiency increased significantly with irradiation time.

Under optimized conditions, approximately 93.6% degradation was achieved within 120 min.

The enhanced photocatalytic activity was attributed to:

1. High surface area
2. Small particle size
3. Efficient electron-hole separation
4. Surface functionalization by biomolecules

Effect of Catalyst Dosage

Catalyst dosage strongly influenced photocatalytic degradation efficiency.

Increasing catalyst concentration increased degradation efficiency due to availability of more active surface sites.

However, excessive catalyst dosage reduced efficiency because of light scattering and reduced light penetration.

Effect of pH

The solution pH significantly affected photocatalytic degradation.

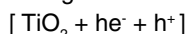
Maximum degradation efficiency was observed under slightly alkaline conditions.

Alkaline pH enhanced hydroxyl radical formation and pollutant adsorption.

Proposed Photocatalytic Mechanism

Upon UV irradiation, TiO_2 nanoparticles absorb photons with energy equal to or greater than their band gap.

This generates electron-hole pairs:



The generated electrons and holes react with oxygen and water molecules to produce reactive oxygen species such as hydroxyl radicals and superoxide radicals.

These radicals oxidize dye molecules into harmless products.

Table 4: Effect of Catalyst Dosage on Photocatalytic Degradation

Catalyst Dosage (g/L)	Degradation Efficiency (%)
0.2	54.7
0.4	68.5
0.6	81.2
0.8	93.6
1.0	90.4

Table 5: Effect of pH on Dye Degradation

pH	Degradation Efficiency (%)
3	48.6
5	66.8
7	82.1
9	93.6
11	88.2

DISCUSSION

The present study demonstrates that plant-mediated green synthesis represents an efficient and environmentally sustainable approach for TiO_2 nanoparticle production.

FTIR analysis confirmed the participation of biomolecules in reduction and stabilization processes. The presence of hydroxyl, carbonyl, and amine functional groups suggested strong interactions between plant metabolites and nanoparticle surfaces.

XRD analysis verified the anatase crystalline phase, which is highly favorable for photocatalytic applications because anatase TiO_2 exhibits superior photocatalytic efficiency compared with rutile and brookite phases.

SEM analysis revealed nanoscale particle dimensions and porous morphology that enhanced active surface area and photocatalytic performance.

The photocatalytic degradation results demonstrated remarkable dye degradation efficiency under optimized conditions.

The observed photocatalytic behavior may be attributed to efficient charge separation, increased reactive oxygen species generation, and enhanced pollutant adsorption on nanoparticle surfaces.

Compared with conventional chemical synthesis methods, green synthesis offers several advantages including:

1. Reduced environmental toxicity
2. Lower synthesis cost
3. Biocompatibility
4. Simple experimental conditions
5. Renewable biological resources

The study confirms the potential of green synthesized TiO_2 nanoparticles for wastewater remediation applications.

CONCLUSION

In the present investigation, titanium dioxide nanoparticles were successfully synthesized through

an eco-friendly green synthesis approach using plant extract as a biological reducing and stabilizing agent. FTIR analysis confirmed the involvement of phytochemicals in nanoparticle synthesis and stabilization. XRD analysis demonstrated the formation of crystalline anatase TiO₂ nanoparticles, while SEM analysis revealed nanoscale spherical morphology.

The synthesized TiO₂ nanoparticles exhibited excellent photocatalytic degradation efficiency against methylene blue dye under UV irradiation, achieving approximately 93.6% degradation under optimized conditions.

The enhanced photocatalytic performance was attributed to nanoscale particle dimensions, high surface area, and biomolecule-assisted surface functionalization.

The results indicate that green synthesized

TiO₂ nanoparticles represent a sustainable, efficient, and environmentally friendly material for wastewater treatment applications.

Future investigations should focus on:

1. Visible-light-responsive TiO₂ nanocomposites
2. Pilot-scale wastewater treatment systems
3. Reusability studies
4. Toxicity assessment
5. Industrial-scale implementation

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Conflict of Interest

The authors declare no conflict of interest.

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