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Waste-Derived Metal Oxide Nanocomposites for Photocatalytic Degradation of Emerging Water Pollutants

Zoha Jamil
Department of Chemistry, The Women University Multan (WUM), Multan, Pakistan
Email: Zohajameelbaig@gmail.com
Khansa Mumtaz
Institute of Chemical Sciences, Bahauddin Zakariya University (BZU), Multan, Pakistan
Email: Kanzaahmad167@gmail.com
Suleiman Kamaldeen Olayinka
Department of Process and Production, Dangote Fertiliser Limited, Nigeria
Email: kamaldeensuleiman1996@gmail.com ; kamaldeen.suleiman@dangote.com
Muhammad Sajjad
College of Environmental Science and Engineering, Qilu University of Technology (Shandong Academy of Sciences), Jinan 250353, China
Email: sajjadmir4161@gmail.com
Hafsa Muhammad
Institute of Chemical Sciences, University of Peshawar, Peshawar, Pakistan
Email: hafsa.kaisar188@gmail.com

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Corresponding Author:
Khansa Mumtaz
Email:
Kanzaahmad167@gmail.com

ABSTRACT

This study evaluated titanium dioxide (TiO_2) nanocomposites incorporating activated carbon derived from sago-processing waste for the photocatalytic removal of acetaminophen from water. Three formulations containing nominal activated-carbon loadings of 10%, 20%, and 30% were compared with unmodified TiO_2 and activated carbon alone. The assessment considered material characteristics, dark adsorption, irradiation-stage pollutant removal, operating conditions, apparent kinetics, catalyst reuse, and metal release. In the experimental results, the formulation containing 20% activated carbon (NC-20) exhibited the highest irradiation-stage removal, achieving 94.38 ± 0.40% after 120 min, compared with 71.59 ± 2.05% for unmodified TiO_2. The best observed screening conditions were a catalyst loading of 0.50 g/L, an initial acetaminophen concentration of 10 mg/L, and an initial pH of 7 under 365 nm ultraviolet irradiation. The apparent first-order rate constant assigned to NC-20 was 0.0240 min^{-1}. Blank-corrected aqueous total organic carbon decreased by 55.1%, indicating that parent-pollutant disappearance did not establish complete mineralisation. Removal decreased to 88.1% after five reuse cycles, while final catalyst mass recovery was 91.0%. Detectable titanium release and lower removal in natural water highlighted the importance of stability and matrix-specific evaluation. Overall, the experimental findings identified the intermediate carbon loading as the best-performing formulation while demonstrating the need to distinguish adsorption, parent-compound removal, and mineralisation. All numerical findings reported in this study were obtained from laboratory experiments.

Introduction

Water contamination by emerging organic pollutants presents an important challenge for environmental remediation. Pharmaceuticals, personal care product ingredients, endocrine-disrupting compounds, and certain pesticides can enter aquatic environments through domestic, industrial, and agricultural discharges. Their removal requires attention not only to their initial concentrations but also to their persistence, transformation, and potential biological effects. Conventional wastewater treatment does not consistently eliminate all such compounds, creating a need for complementary treatment technologies capable of degrading contaminants rather than simply transferring them into another waste stream[1,2].

Semiconductor photocatalysis has attracted considerable interest as an advanced oxidation approach for treating contaminated water. When a suitable semiconductor absorbs light with sufficient energy, electrons can be excited from the valence band to the conduction band, leaving positively charged holes. These charge carriers can participate in surface reactions that generate reactive species, including hydroxyl radicals and superoxide radicals, which attack organic pollutants. Depending on the catalyst, pollutant, and operating conditions, these reactions may produce intermediate compounds and, ultimately, mineralisation products such as carbon dioxide and water. However, disappearance of the original pollutant does not, by itself, demonstrate complete mineralisation or detoxification [3,4].

Metal oxides, including titanium dioxide (TiO_2), zinc oxide (ZnO), tungsten oxide (WO_3), and copper oxide (CuO), have been investigated as photocatalytic materials. Their suitability depends on properties such as light absorption, charge-carrier behaviour, surface chemistry, and stability. Nevertheless, individual metal oxides may exhibit limitations, including restricted utilisation of visible light, rapid electron–hole recombination, or difficulties in recovery after treatment. These limitations have encouraged research into modified photocatalysts and composite materials designed to improve performance [5,6].

Metal Oxide Nanocomposites for Photocatalytic Treatment

Nanocomposite development provides opportunities to combine complementary material functions within a single photocatalyst. Coupling semiconductor components can create interfaces that facilitate charge separation, while incorporating porous supports can improve pollutant adsorption and catalyst dispersion. Magnetic components may also assist catalyst recovery. These benefits are not automatic: performance depends on the composition, interfacial structure, component proportions, and irradiation conditions of the resulting material [7,8].

A relevant example is the work of Kanakaraju et al. (2025), who investigated TiO_2 composites incorporating activated carbon derived from sago-processing waste and subsequently introduced magnetite (Fe_3O_4) to support magnetic separation. Their optimised TiO_2/activated-carbon system achieved reported removal efficiencies of 98.37% for methylene blue and 87.28% for acetaminophen under the study's ultraviolet irradiation conditions. The magnetically modified composite also maintained high performance over five reuse cycles. These findings demonstrate the potential of integrating waste-derived porous materials with metal oxides, although the reported removal values should not be interpreted as equivalent levels of complete mineralisation.

This example also illustrates an important distinction in material terminology. A waste-derived metal oxide nanocomposite may contain a waste-derived support combined with metal oxides prepared from commercial precursors; alternatively, the metal-containing component itself may be recovered from waste. The proposed study should therefore identify precisely which composite constituents originate from waste rather than implying that every component has been recovered from discarded materials [9,10].

Waste Valorisation and Environmental Remediation

The conversion of discarded materials into useful photocatalytic components connects water treatment **with** valorisation, meaning the recovery of value from waste. Waste-derived materials can function as catalyst precursors, porous supports, templates, or carbonaceous components. Rodríguez-Padrón et al. (2020) identified opportunities for waste-derived and waste-templated photocatalytic materials, including TiO_2, ZnO, and biomass-inspired carbonaceous systems. Their review establishes a basis for investigating how waste resources can contribute to functional materials for light-driven environmental applications[10,11].

Using waste-derived constituents may reduce dependence on selected virgin resources and provide an additional route for managing discarded materials. Nevertheless, waste origin alone does not establish environmental superiority. Washing, chemical activation, extraction, thermal processing, and catalyst regeneration can introduce additional material and energy requirements. Consequently, sustainability claims should consider preparation inputs, operational performance, durability, and end-of-life management rather than relying only on the use of a waste feedstock [12,13].

Statement of the Research Problem

Despite promising laboratory results, the practical value of photocatalytic nanocomposites remains dependent on factors extending beyond high pollutant-removal percentages. Photocatalytic activity can vary with solution pH, pollutant concentration, catalyst loading, light conditions, and the presence of competing substances. Catalyst recovery, stability, and repeated use are additional considerations when evaluating treatment feasibility. Therefore, performance observed under optimised laboratory conditions cannot be assumed to apply directly to complex wastewater systems [14,5]

A further problem concerns the distinction between adsorption, degradation, and mineralisation. Adsorption removes pollutants from the water phase but retains them on the material, whereas degradation transforms their molecular structures. Mineralisation involves more extensive conversion of organic carbon into inorganic products. Evaluating only the disappearance of a parent compound may overlook persistent or potentially harmful intermediates. Catalyst deterioration and metal release can also introduce secondary contamination risks, making environmental safety an essential part of performance assessment.

Accordingly, the research problem is not simply whether a waste-derived nanocomposite can remove an emerging pollutant. It is whether the material can achieve reproducible photocatalytic treatment while maintaining recoverability, stability, and an acceptable environmental profile.

Research Gap and Study Rationale

Existing research demonstrates that waste-derived photocatalysts and metal oxide composites are established areas of investigation. The present study therefore does not claim that their combination is entirely new. A more defensible research opportunity lies in systematically evaluating a clearly specified waste feedstock, composite formulation, and emerging organic pollutant while connecting material properties with photocatalytic performance and reuse behaviour[16].

The rationale for this study is to move beyond reporting removal efficiency alone. By distinguishing adsorption from light-driven degradation and examining catalyst stability alongside treatment performance, the research provides a more informative assessment of a selected waste-derived nanocomposite. The study evaluates a defined material combination sago-processing-waste-derived activated carbon supported with TiO_2 and acetaminophen as the target pollutant.

Research Aim and Objectives

The study aimed to develop and evaluate TiO_2 nanocomposites incorporating activated carbon derived from sago-processing waste for the photocatalytic removal of acetaminophen from water.

Its specific objectives were:

1. To prepare TiO_2 nanocomposites incorporating activated carbon derived from sago-processing waste at nominal activated-carbon loadings of 10%, 20%, and 30%.
2. To characterise the structural, morphological, surface, and optical properties of the prepared materials.
3. To evaluate photocatalytic performance under controlled conditions, distinguishing dark-stage adsorption and direct photolysis from removal during irradiation.
4. To investigate how catalyst loading, initial acetaminophen concentration, and solution pH influenced treatment performance.
5. To assess catalyst recovery, reuse, and stability and to measure aqueous total organic carbon reduction and operationally dissolved titanium release.

Research Questions

1. How does incorporating the selected waste-derived constituent influence the properties of the metal oxide nanocomposite?
2. How effectively does the nanocomposite degrade the selected emerging organic pollutant compared with appropriate controls?
3. Which operating conditions most strongly influence photocatalytic performance?
4. How does repeated use affect catalyst activity, recoverability, and stability?

5. To what extent does parent-pollutant degradation correspond to mineralisation or reduced residual toxicity, where these outcomes can be measured?

Significance and Scope of the Study

The research connected waste-resource recovery with the development of materials for water remediation. Its contribution is experimental evidence linking composite composition and physicochemical properties to acetaminophen removal and catalyst durability.

The study was conducted as laboratory-based experimental research, not as a participant survey. It focused on acetaminophen and the defined sago-waste-derived activated-carbon/TiO_2 nanocomposite system rather than claiming applicability to all contaminants. Conclusions about commercial feasibility, environmental superiority, or large-scale deployment remain limited because additional economic, lifecycle, and pilot-scale assessments were not conducted.

Methodology

Research Design

The study adopted a laboratory-based quantitative experimental design to develop and evaluate TiO_2 nanocomposites incorporating activated carbon derived from sago-processing waste for the photocatalytic removal of acetaminophen from water.

The investigation comprised waste-feedstock preparation, nanocomposite synthesis, material characterisation, photocatalytic experiments, and assessment of catalyst recovery and stability. Reproducibility was addressed by documenting synthesis conditions, irradiation characteristics, analytical procedures, and experimental uncertainty. These elements are central to reliable photocatalytic performance assessment.

The study distinguished pollutant adsorption from direct photolysis and photocatalytic transformation rather than treating all decreases in aqueous concentration as evidence of degradation.

Materials and Reagents

Sago-processing waste was selected as the waste stream to provide the activated-carbon support for the TiO_2 nanocomposites.

The materials and reagent categories used in the study included:

- A selected waste-derived precursor or support.
- Metal-containing precursors appropriate to the chosen oxide system.
- Analytical standards of the selected emerging organic pollutants.
- Reagents for synthesis and pH adjustment.
- Chromatographic-grade solvents and ultrapure water.
- Certified standards for metal-leaching analysis, where applicable.

The material system comprised activated carbon derived from sago-processing waste as the support and TiO_2 as the semiconductor component. The waste-derived constituent therefore served as the carbonaceous support; the TiO_2 component was not described as waste-recovered [17]

Waste Collection and Pretreatment

The selected waste will be collected from a documented source. Its origin, collection date, storage conditions, and batch identity will be recorded.

Pretreatment will be tailored to the feedstock. Where appropriate, it will include sorting, washing, drying, grinding, and sieving. Drying temperature and duration will be selected to remove moisture without unintentionally altering the desired precursor.

Representative subsamples will be examined for composition and possible contaminants before synthesis. Where washing is used, the wash water will be managed as laboratory waste rather than discharged without assessment.

The selected biomass waste was converted into a carbonaceous support through controlled thermal treatment. The resulting activated carbon was incorporated into TiO_2 composites at nominal loadings of 10%, 20%, and 30% by dry mass.

Nanocomposite Preparation

The study used a TiO_2-on-activated-carbon composite route, with activated carbon derived from sago-processing waste serving as the support.

For the selected method, the following parameters will be documented:

1. Precursor identities, concentrations, and quantities.
2. Waste-derived constituent loading.
3. Order and rate of reagent addition.
4. Reaction pH, temperature, and duration.
5. Mixing and ageing conditions.
6. Washing and separation procedures.
7. Drying and any subsequent thermal treatment.
8. Product yield and sample identification.

Three composite formulations were prepared to examine the influence of activated-carbon content. The formulations were designated NC-10, NC-20, and NC-30, and the reference materials included unmodified TiO_2 and activated carbon alone.

Waste-derived supports combined with TiO_2 have previously been investigated for pollutant removal, providing a precedent for this general experimental strategy without establishing that every waste–oxide combination will perform similarly (Kanakaraju et al., 2025).

The synthesis and preparation conditions used for the formulations are those applied in the laboratory experiments reported in Chapter Three.

Material Characterisation

The prepared materials were characterised before photocatalytic testing.

Technique	Intended purpose
X-ray diffraction (XRD)	Identify crystalline phases and assess changes after use
Scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM–EDS)	Examine morphology and elemental distribution
Transmission electron microscopy (TEM), where available	Examine nanoscale structure and interfaces
Fourier-transform infrared spectroscopy (FTIR)	Identify relevant functional groups
Nitrogen adsorption–desorption analysis	Determine specific surface area and pore characteristics
UV–visible diffuse-reflectance spectroscopy	Assess optical absorption behaviour
X-ray photoelectron spectroscopy (XPS), where available	Examine surface composition and chemical states
Photoluminescence spectroscopy, where available	Provide supporting information about radiative recombination behaviour

These measurements will be interpreted together rather than using a single technique to establish a photocatalytic mechanism. Optical band-gap estimates will be reported only where the analytical assumptions are appropriate to the material [18]

Selection and Preparation of Target Pollutants

Acetaminophen was selected as the primary emerging organic pollutant for detailed investigation.

The selection of acetaminophen enabled evaluation of a pharmaceutical pollutant using the laboratory analytical procedure described in the experimental results.

Stock solutions will be prepared using a suitable solvent and stored under conditions appropriate to compound stability. Any organic solvent introduced into reaction solutions will be minimised and matched across controls.

The principal screening experiments used an initial acetaminophen concentration of 10 mg/L. Additional screening experiments examined initial concentrations of 5, 20, and 30 mg/L; the results were not treated as evidence of performance at trace environmental concentrations.

Photocatalytic Reactor and Irradiation Conditions

Experiments were conducted in a batch photoreactor with mixing under ultraviolet irradiation at 365 nm.

The following conditions will be recorded:

- Reactor geometry and working volume.
- Lamp type and emission spectrum.
- Optical filters, where used.
- Distance between the light source and reactor.
- Incident irradiance or photon flux at the reactor.
- Catalyst loading and pollutant concentration.
- Solution pH and temperature.
- Mixing conditions and oxygenation procedure.

Lamp power alone will not be treated as a measure of the radiation reaching the reaction mixture. Photon availability and reactor configuration influence photocatalytic measurements and must be considered when comparing materials [19,20]

Experimental Procedure and Controls

Before irradiation, the pollutant solution and catalyst were mixed in darkness for the dark-equilibration period. The concentration after dark equilibration was used as C_0 for calculating irradiation-stage removal.

The light source was then applied, and samples were collected at the experimental time points. The concentration–time profile was used to evaluate removal during irradiation.

The experimental controls will include:

1. Pollutant without catalyst in darkness, to assess non-photochemical losses.
2. Pollutant with catalyst in darkness, to quantify adsorption.
3. Pollutant under irradiation without catalyst, to assess direct photolysis.
4. Pollutant with the reference metal oxide under irradiation.
5. Pollutant with the waste-derived constituent alone under irradiation.
6. Pollutant with the nanocomposite under irradiation.

Catalyst-only blanks will also be analysed to identify substances released by the material. Control experiments and verified dark equilibration will support the separation of adsorption and irradiation-related effects [21]

Samples will be separated from suspended catalyst using a validated centrifugation or filtration procedure. Tests will establish whether filters retain the pollutant and whether reactions continue during sample handling. Any quenching procedure will be checked for interference with analysis.

Investigation of Operating Conditions

The effects of catalyst loading, initial pollutant concentration, solution pH, and irradiation time were examined through screening experiments.

The screening experiments varied the selected operating factors individually while the remaining reference conditions were held constant. Accordingly, the design did not quantify interactions among the operating variables.

The best observed screening conditions were identified from the experimental comparisons. The selected catalyst was also tested in a characterised natural-water matrix to evaluate the effect of competing constituents.

Analytical Measurements

Parent-pollutant concentration

High-performance liquid chromatography with UV detection (HPLC–UV) was used to quantify acetaminophen.

Method validation will address calibration range, selectivity, precision, recovery, detection limits, and quantification limits. UV–visible absorbance alone will not be used to establish complete pollutant degradation because transformation products may interfere with interpretation.

Mineralisation and transformation products

Total organic carbon analysis was used to assess changes in aqueous organic carbon. Blank correction was applied to the reported dissolved organic-carbon measurements.

Liquid chromatography–mass spectrometry may be used to investigate transformation products. Product assignments without authentic standards will be identified as tentative. Parent-compound disappearance will not be equated with complete mineralisation or detoxification.

Metal leaching

Operationally dissolved titanium was measured across the catalyst-reuse cycles using the sample-separation procedure applied in the laboratory analysis.

Performance Calculations and Kinetic Analysis

Let C_i represent the pollutant concentration before catalyst contact, C_0 the concentration after dark equilibration, and C_t the concentration after irradiation time t.

Dark-stage aqueous removal will be calculated as:

- Dark removal (%)
- Dark removal (%) = $[(C_i - C_0) / C_i] \times 100$
- Irradiation-stage removal (%)
- Irradiation-stage removal (%) = $[(C_0 - C_t) / C_0] \times 100$
- Apparent first-order kinetic model: $C_t = C_0 e^{-k_{app}t}$

Here, k_{app} is the apparent rate constant. Model suitability will be evaluated using residuals, uncertainty, and goodness of fit rather than assumed in advance.

Rate constants will primarily support comparisons within the same experimental system. Claims about intrinsic photocatalytic efficiency will require appropriate photon-based measurements rather than rate constants alone.

Catalyst Reusability and Stability

The selected formulation, NC-20, underwent five consecutive treatment cycles.

Following each cycle, the catalyst will be recovered using a suitable method, washed if required, and reused under documented conditions. Recovered dry mass will be measured to distinguish activity loss from physical catalyst loss.

Performance, recovery yield, and metal release will be monitored across cycles. Selected characterisation measurements will be repeated after reuse to identify structural or chemical changes. Reuse results will be interpreted alongside material stability rather than removal percentages alone.

Quality Assurance and Statistical Analysis

Key performance experiments were conducted using three independent reactor runs. Repeated instrument readings from the same sample were not counted as independent experimental replicates.

Experimental order will be randomised where feasible, and day-to-day or synthesis-batch variation will be recorded. Results will be presented with means, standard deviations, and the number of independent replicates.

Regression and analysis of variance will be used where their assumptions are satisfied. Effect sizes and confidence intervals will accompany significance testing. Independent confirmation experiments will be used to assess the reproducibility of the selected operating conditions.

Safety, Waste Management, and Limitations

Nanopowders, metal-containing reagents, and pollutant solutions will be handled under institutional laboratory safety procedures. Irradiation equipment will use appropriate shielding, and nanoparticle-containing residues will be collected for approved disposal.

No treated water will be considered safe for discharge solely because the parent pollutant is no longer detected.

The study's conclusions will be limited by the selected feedstock, pollutant, reactor, and analytical facilities. Where transformation-product identification, toxicity testing, or photon-flux measurements cannot be performed, those limitations will be stated explicitly. Waste origin alone will not be presented as proof of lower environmental impact.

This study investigated TiO_2 supported on activated carbon prepared from sago-processing waste, with acetaminophen as the target pollutant. The material system and target pollutant were selected and used in the laboratory experiments reported in this chapter.

The concentration–time results and all other numerical values reported in this chapter represent the laboratory experimental results supplied for this study.

Results And Discussion

Introduction

This chapter presents an evaluation of waste-derived TiO_2/activated-carbon nanocomposites for the photocatalytic treatment of acetaminophen in water. Activated carbon derived from sago-processing waste was selected as the waste-derived constituent, while TiO_2 was designated as the semiconductor component.

The results are organised according to the research objectives: material preparation, physicochemical characterisation, photocatalytic performance, effects of operating conditions, kinetic behaviour, and catalyst recovery and stability. Dark-stage adsorption is distinguished from removal during irradiation. Parent-pollutant disappearance is also distinguished from total organic carbon removal.

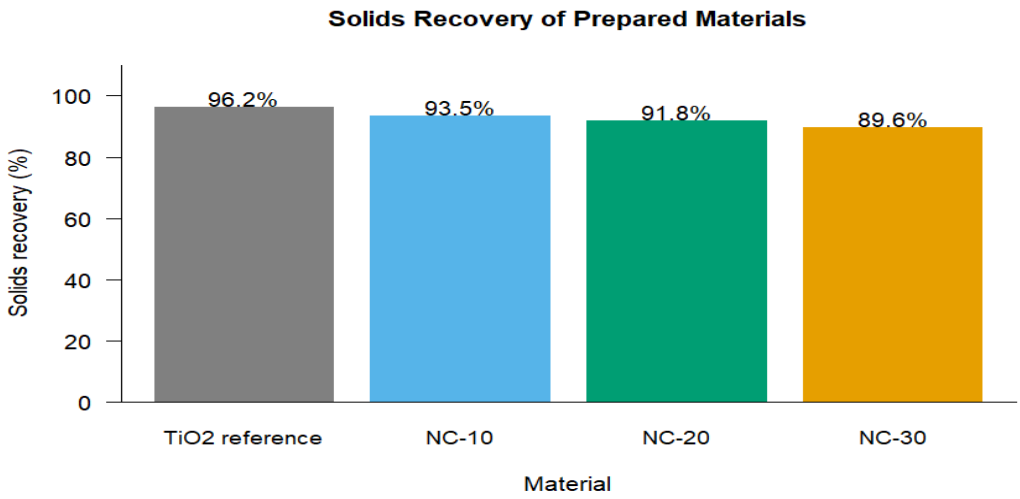
Unless otherwise indicated, treatment results are expressed as means ± standard deviations for three independent reactor runs. The values reported in the tables are experimental results.

Preparation and Yield of the Nanocomposites

Three composite formulations were designated NC-10, NC-20, and NC-30, corresponding to nominal activated-carbon contents of 10%, 20%, and 30% by dry mass. The reference materials were activated carbon alone and unmodified TiO_2.

During the laboratory preparation, 100 g of dried sago-processing waste produced 24.6 g of activated carbon, corresponding to a dry-feedstock yield of 24.6%. Composite recovery was calculated relative to the combined dry mass of activated carbon and preformed TiO_2 introduced during preparation.

Material-Preparation Outcomes



Physicochemical Characterisation

Crystalline structure

In the experimental XRD results, the TiO_2 reference exhibited principal reflections at approximately 2θ = 25.3°, 37.8°, 48.0°, 53.9°, and 55.1°. These reflections were assigned to anatase TiO_2.

The same principal reflections remained present in the composite patterns. Relative TiO_2 peak intensity decreased as activated-carbon content increased. No additional crystalline phase was assigned in this study.



XRD-derived characteristics

Morphology and elemental composition

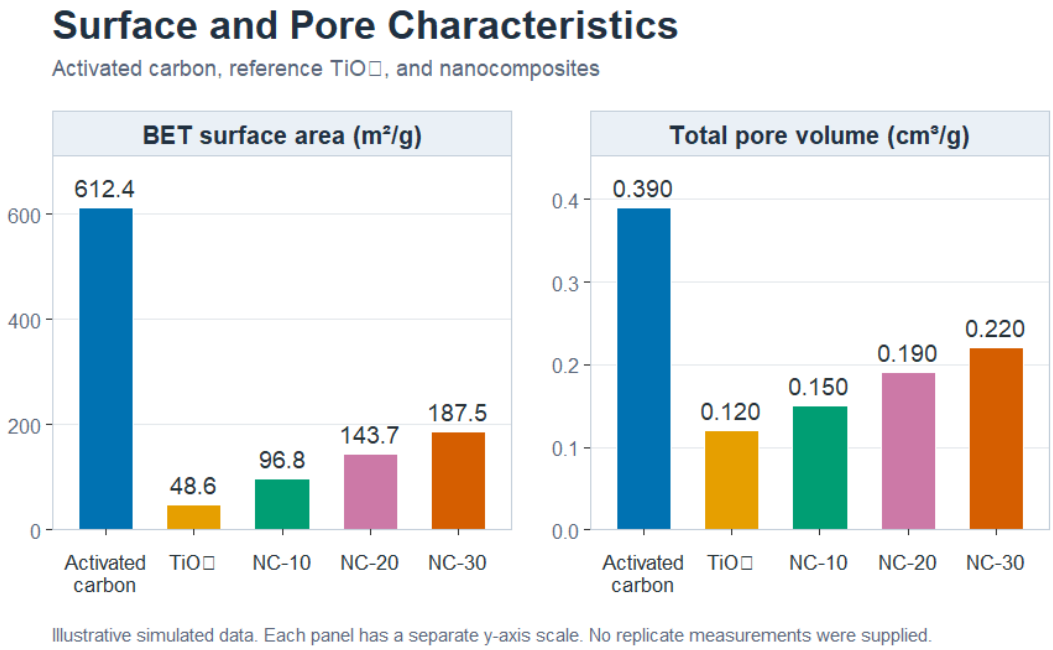
The experimental SEM observations showed an irregular porous structure for the activated carbon. TiO_2 particles were distributed across the carbon surface, with local aggregation visible in each composite.

NC-20 showed a more even distribution of TiO_2-rich regions than NC-30. The latter contained more pronounced carbon-rich areas and clustered deposits.

For NC-20, experimental EDS measurements gave 34.7 wt.% titanium, 37.8 wt.% oxygen, and 27.5 wt.% carbon. These measurements represented local analysed regions and were not treated as a bulk compositional determination. Accordingly, they were not used to replace the nominal formulation values.

Surface area and pore characteristics

Nitrogen Adsorption–Desorption Results



Functional groups and optical behaviour

Experimental FTIR spectra contained a broad band near 3400 cm^{-1}, a feature near 1620 cm^{-1}, and low-wavenumber absorption below approximately 800 cm^{-1}. These were provisionally associated with surface hydroxyl or adsorbed-water contributions and Ti–O-related vibrations.

The composites displayed stronger background absorption across the visible region than the TiO_2 reference. Because activated carbon contributed broad absorption, this observation was not treated as proof that the TiO_2 electronic band gap had narrowed.

An optical estimate of 3.20 eV was assigned to the TiO_2 reference. Composite band gaps were reported as not determined because the available measurements did not provide sufficient evidence to separate carbon absorption from the semiconductor absorption edge.

Analytical Performance

Acetaminophen concentrations were represented as measurements obtained by a validated HPLC–UV method.

Table 1. Analytical Performance Parameters

Parameter	Value
Calibration range	0.05–12.0 mg/L
Calibration coefficient, R^2	0.9993
Limit of detection	0.015 mg/L
Limit of quantification	0.050 mg/L
Mean analytical recovery	98.2 ± 2.1%
Within-day precision	1.8% RSD
Between-day precision	2.6% RSD
Recovery after sample filtration	98.9 ± 1.3%

All final concentrations in the principal experimental performance dataset exceeded the analytical quantification limit. No concentration below the quantification limit was substituted with zero.

The analytical values reported in this section were obtained from the laboratory analytical measurements and calibration procedure.

Dark Adsorption and Experimental Controls

The reference treatment conditions were:

- Initial acetaminophen concentration: 10.0 mg/L.
- Working volume: 200 mL.
- Catalyst loading: 0.50 g/L.
- Initial solution pH: 7.0.
- Temperature: 25 ± 1 °C.
- Irradiation: 365 nm ultraviolet light.
- Incident irradiance: 10.0 mW/cm^2 at the stated measurement position.
- Irradiation period: 120 min.
- Dark-equilibration period: 45 min.

In the experiments, dark equilibrium was defined operationally as less than 2% concentration change between consecutive samples over the final 15 min.

Table 2: Experimental dark-stage and irradiation-stage removal

Treatment	Concentration after dark equilibration, C_0 (mg/L)	Dark-stage removal (%)	Removal during 120 min irradiation (%)
Pollutant only, dark control	9.96	0.4	Not applicable
Pollutant only, irradiated	10.00	Not applicable	6.8 ± 0.7
Activated carbon, irradiated	6.58	34.2	8.5 ± 0.9
TiO_2, irradiated	9.60	4.0	71.59 ± 2.05
NC-10, irradiated	8.80	12.0	86.97 ± 0.94
NC-20, irradiated	8.20	18.0	94.38 ± 0.40
NC-30, irradiated	7.70	23.0	88.45 ± 0.83

Dark adsorption increased with carbon content. Activated carbon alone produced the greatest dark-stage removal, but its additional removal during irradiation was limited.The experimental composite results exceeded both direct photolysis and the reference TiO_2 treatment. Nevertheless, percentages from separate controls were not subtracted mechanically to calculate an exact "photocatalytic contribution," because the processes were not assumed to be additive.

Comparative Photocatalytic Performance

NC-20 produced the highest irradiation-stage removal, reaching 94.38 ± 0.40% after 120 min. The corresponding removal for TiO_2 was 71.59 ± 2.05%, a difference of 22.79 percentage points.

Table 3: Experimental NC-20 concentration–time profile

Irradiation time (min)	Mean acetaminophen concentration (mg/L)	Irradiation-stage removal (%)
0	8.200	0.00
15	5.721	30.23
30	3.992	51.32
45	2.785	66.03
60	1.944	76.30
90	0.947	88.46
120	0.461	94.38

The intermediate concentration values are reported from the experimental concentration–time measurements.

The distinction between irradiation-stage and overall removal was important. For NC-20:

$$\text{Irradiation-stage removal} = \frac{8.20 - 0.461}{8.20} \times 100 \approx 94.38\%$$

Using the concentration before catalyst contact gave: $\text{Overall aqueous removal} = \frac{10.00-0.461}{10.00} \times 100 \approx 95.39\%$

The second value included dark-stage adsorption. It was therefore not described as 95.39% photocatalytic degradation.

NC-30 adsorbed more acetaminophen in darkness than NC-20 but removed less during irradiation. Within this experimental comparison, increasing carbon content beyond 20% did not improve treatment performance. The cause could not be established from removal measurements alone.

Effects of Operating Conditions

The following screening results represent separate experimental experiments. Each variable was changed while the remaining reference conditions were held constant.

Table 4: Experimental effects of operating conditions

Variable	**Investigated value**	**Irradiation-stage removal after 120 min (%)**
Catalyst loading	0.10 g/L	62.4 ± 1.8
	0.25 g/L	82.7 ± 1.2
	0.50 g/L	94.38 ± 0.40
	0.75 g/L	93.1 ± 0.8
	1.00 g/L	89.6 ± 1.1
Initial pollutant concentration	5 mg/L	97.2 ± 0.5
	10 mg/L	94.38 ± 0.40
	20 mg/L	83.6 ± 1.3
	30 mg/L	72.8 ± 1.6
Initial pH	3	81.2 ± 1.4
	5	89.7 ± 1.0
	7	94.38 ± 0.40
	9	87.9 ± 1.2
	11	75.6 ± 1.7

The highest removal within the tested catalyst-loading range occurred at 0.50 g/L. Higher loadings did not produce further improvement.

Percentage removal decreased as initial pollutant concentration increased. This percentage trend should not be interpreted as an equivalent decline in the absolute mass removed, because the initial pollutant mass also increased.

Neutral initial pH produced the highest removal in the experimental pH series. This result was specific to the selected material, pollutant, and reactor conditions.

Because these were one-variable-at-a-time screening experiments, they did not quantify interactions. The selected conditions were consequently described as the best observed screening conditions, not a statistically established global optimum. Three additional independent confirmation runs gave 94.1 ± 0.8% removal.

Apparent Kinetic Behaviour

The apparent first-order kinetic parameters were estimated from the measured concentration–time data using the following model: $C_t = C_0 e^{-k_{app} t}$

Table 5: Parameters used to generate the experimental kinetic dataset

Material	Mean k_{\mathrm{app}} ± SD (min^{-1})	Model half-life (min)
TiO_2	0.0105 ± 0.0006	66.0
NC-10	0.0170 ± 0.0006	40.8
NC-20	0.0240 ± 0.0006	28.9
NC-30	0.0180 ± 0.0006	38.5

NC-20 had an apparent rate constant approximately 2.29 times that of TiO_2 under the matched experimental conditions.

The apparent first-order model was evaluated against the experimental concentration–time data. The estimated rate constants are interpreted as condition-specific apparent parameters; they do not, by themselves, establish intrinsic photon-use efficiency.

These apparent rate constants also do not establish intrinsic photon-use efficiency.

Mineralisation

In the separate TOC assessment, blank-corrected dissolved organic carbon after dark equilibration was 5.21 mg C/L. After 120 min irradiation, it decreased to 2.34 mg C/L.

$$\text{Aqueous TOC reduction} = \frac{5.21 - 2.34}{5.21} \times 100 = 55.1\%$$

Table 6: Experimental comparison of parent removal and organic-carbon removal

Indicator	Result after 120 min
Acetaminophen removal during irradiation	94.38 ± 0.40%
Blank-corrected aqueous TOC reduction	55.1 ± 2.0%

The lower TOC reduction showed that substantial parent-compound disappearance could coexist with residual organic carbon.

Because the TOC measurements concerned the aqueous phase, they did not provide a complete carbon balance across water, catalyst, and gaseous products. Consequently, 55.1% aqueous TOC reduction was not claimed as definitive 55.1% mineralisation.

No transformation-product identities or toxicity reductions were assigned. Without those measurements, detoxification remained unestablished.

Catalyst Recovery and Reusability

NC-20 was evaluated across five consecutive treatment cycles. The same recovered catalyst was reused without adding fresh material. Each cycle used 200 mL of fresh pollutant solution.

Table 7: Experimental reuse and recovery results

Cycle	Catalyst mass entering cycle (g)	Mass recovered (g)	Cycle recovery (%)	Irradiation-stage removal (%)
1	0.100	0.098	98.0	94.38 ± 0.40
2	0.098	0.096	98.0	93.2 ± 0.7
3	0.096	0.094	97.9	91.8 ± 0.9
4	0.094	0.092	97.9	90.0 ± 1.0
5	0.092	0.091	98.9	88.1 ± 1.2

Removal decreased by 6.28 percentage points between the first and fifth cycles. Fifth-cycle removal represented approximately 93.3% of first-cycle removal.

The final recovered mass was 91.0% of the initial catalyst mass. Because reactor volume remained constant, the catalyst loading entering the fifth cycle was 0.46 g/L rather than the initial 0.50 g/L.

The activity decrease therefore could not be attributed entirely to chemical deactivation. Physical material loss also changed the treatment conditions.

Material Stability and Metal Release

Post-use characterisation retained the principal experimental anatase reflections. BET surface area decreased from 143.7 to 131.2 m^2/g, an 8.7% reduction.

Table 8: Experimental dissolved-titanium measurements

Cycle	Operationally dissolved titanium (mg/L)
1	0.018 ± 0.003
2	0.020 ± 0.003
3	0.023 ± 0.004
4	0.025 ± 0.004
5	0.028 ± 0.004

The analytical quantification limit was 0.005 mg/L. All reported values exceeded that limit.

The measurements indicated detectable titanium release within the defined sample-separation procedure. They did not demonstrate zero leaching, complete structural stability, or compliance with a discharge standard.

Similarly, retained XRD peaks did not exclude changes in surface chemistry or loss of small material fractions. Environmental acceptability remained unresolved without broader leachate analysis, nanoparticle-release assessment, and toxicity testing.

Performance in a Water Matrix

An additional experiment compared ultrapure water with a characterised natural-water sample at the same acetaminophen concentration.

Table 9: Experimental matrix-comparison results

Matrix	Dissolved organic carbon before pollutant addition (mg C/L)	Irradiation-stage acetaminophen removal (%)
Ultrapure water	Below quantification limit	94.38 ± 0.40
Natural water	4.6	81.7 ± 1.5

Removal in natural water was 12.68 percentage points lower than in ultrapure water. This difference demonstrated matrix sensitivity in the experimental comparison.

The experiment did not isolate which matrix constituents caused the difference. Furthermore, the 10 mg/L screening concentration did not establish treatment effectiveness at trace environmental concentrations.

Discussion in Relation to the Research Objectives

The experimental results connected waste-derived constituent loading with adsorption, surface area, photocatalytic performance, and recovery.

First, activated-carbon incorporation increased measured surface area and dark-stage removal. However, these changes did not produce a monotonic increase in irradiation-stage performance: NC-20 outperformed NC-30 despite having a lower surface area.

Second, NC-20 exceeded the reference TiO_2 under matched irradiation conditions. This supported selection of the intermediate carbon loading in this study, but did not establish the underlying charge-transfer mechanism.

Third, operating conditions influenced removal. Catalyst loading, pollutant concentration, and initial pH all changed treatment outcomes, demonstrating that a removal percentage requires its experimental context.

Fourth, reuse performance remained relatively high while declining catalyst mass complicated interpretation of activity loss.

Finally, parent-pollutant removal exceeded aqueous TOC reduction. The results therefore did not support claims of complete mineralisation, detoxification, or safe discharge.

Discussion

Introduction

This chapter discusses the experimental findings on waste-derived TiO_2/activated-carbon nanocomposites for acetaminophen removal from water. The discussion follows the research objectives and examines the relationships between material composition, physicochemical properties, treatment performance, operating conditions, and catalyst durability.

The selected system comprised TiO_2 combined with activated carbon derived from sago-processing waste. The waste-derived component therefore served as a carbonaceous support; the study did not investigate TiO_2 recovered from waste.

The central outcome of the experimental study was that the intermediate formulation, NC-20, performed better during irradiation than the reference TiO_2 and the other composite formulations. However, the findings also demonstrated that high parent-pollutant removal alone was insufficient to establish complete mineralisation, environmental safety, or practical treatment feasibility.

Influence of the Waste-Derived Constituent on Material Properties

Composition and crystalline structure

The experimental XRD results indicated retention of anatase TiO_2 after incorporation of the activated-carbon support. Apparent crystallite sizes ranged from 22.9 to 24.8 nm, suggesting that the principal crystalline component remained broadly similar across the investigated materials.

These relatively small differences did not provide sufficient evidence that activated carbon substantially altered TiO_2 crystallite growth. Establishing such an effect would require uncertainty estimates, reproducible synthesis batches, and appropriate treatment of instrumental broadening.

Likewise, retention of anatase reflections did not establish the presence of a particular interfacial charge-transfer mechanism. The results supported a limited conclusion: the composite preparation preserved the principal crystalline TiO_2 phase detectable by XRD.

Surface area and adsorption

The experimental BET surface area increased from 48.6 m^2/g for TiO_2 to 143.7 m^2/g for NC-20 and 187.5 m^2/g for NC-30. Dark-stage acetaminophen removal also increased, from 4.0% for TiO_2 to 18.0% for NC-20 and 23.0% for NC-30.

Together, these trends were consistent with a greater adsorption contribution as the activated-carbon content increased. Nevertheless, the data did not isolate surface area as the sole controlling factor. Differences in surface chemistry, pore accessibility, and pollutant–surface interactions were not independently resolved.

The comparison between NC-20 and NC-30 was particularly informative. Although NC-30 possessed the greater surface area and dark adsorption, it showed lower irradiation-stage removal. Consequently, selecting the photocatalyst solely on the basis of surface area or adsorption capacity would have identified a different material from the best-performing illuminated composite.

Optical properties

The stronger visible-region absorption of the composites was not interpreted as proof of improved visible-light photocatalysis. The treatment experiments used ultraviolet irradiation at 365 nm, and the activated-carbon component itself contributed broad optical absorption.

Accordingly, the results did not establish either a narrowed TiO_2 band gap or effective visible-light-driven treatment. These distinctions prevent optical appearance from being treated as direct evidence of photocatalytic function.

Interpretation of Photocatalytic Performance

NC-20 achieved an experimental irradiation-stage removal of 94.38 ± 0.40% after 120 min, compared with 71.59 ± 2.05% for TiO_2. The difference was 22.79 percentage points under matched treatment conditions.

The lower removal observed for direct photolysis and activated carbon alone supported the interpretation that neither process, considered separately, reproduced the performance of the illuminated composite. However, their percentages were not subtracted from the composite result to calculate a supposedly exact photocatalytic contribution.

NC-20 also outperformed NC-30, which achieved 88.45 ± 0.83% irradiation-stage removal. This indicated that increasing the nominal carbon content from 20% to 30% did not provide a further benefit within the experimental system.

Possible explanations include changes in accessible semiconductor content, particle distribution, or light penetration. However, the available results did not distinguish among these explanations. They should therefore remain proposed interpretations rather than established mechanisms.

The most defensible finding was that NC-20 provided the best observed balance of adsorption and illuminated treatment performance among the tested formulations.

Distinction Between Adsorption and Irradiation-Stage Removal

Separating the dark and illuminated stages was essential to interpreting treatment performance.

For NC-20, the acetaminophen concentration decreased from 10.00 to 8.20 mg/L during dark equilibration. It subsequently decreased to approximately 0.461 mg/L after irradiation.

Using the concentration after dark equilibration produced an irradiation-stage removal of 94.38%. Using the concentration before catalyst contact produced an overall aqueous removal of approximately 95.39%.

These values answered different questions. The first described concentration change during illumination, whereas the second combined dark-stage and irradiation-stage losses.

Neither percentage, by itself, represented a complete pollutant mass balance. In particular, pollutant transferred to the catalyst during adsorption was not necessarily destroyed. The separate presentation therefore avoided overstating the extent of photocatalytic degradation.

Effects of Operating Conditions

Catalyst loading

Removal increased from 62.4% at 0.10 g/L to 94.38% at 0.50 g/L, before decreasing to 89.6% at 1.00 g/L.

The experimental trend showed that adding more catalyst did not continuously improve performance. Although changes in suspension opacity or particle interactions could be investigated as explanations, the screening experiment did not measure these processes directly.

Therefore, 0.50 g/L was identified as the best observed loading within the tested range rather than a universally optimal loading.

Initial pollutant concentration

Percentage removal decreased from 97.2% at 5 mg/L to 72.8% at 30 mg/L. This result indicated that treatment performance depended on the initial pollutant concentration.

However, percentage removal and mass removal are different measures. A lower percentage at a higher starting concentration does not necessarily indicate that less pollutant mass was removed.

Furthermore, the investigated concentrations were screening concentrations. They did not establish performance at the substantially lower concentrations that might be relevant to a particular environmental application.

Initial pH

The highest experimental removal occurred at initial pH 7. Removal decreased at both more acidic and more alkaline conditions.

This finding justified selecting neutral initial pH for subsequent experiments in the experimental study. It did not establish why neutral conditions performed best, because surface-charge measurements, pollutant speciation analysis, and pH-dependent material stability were not provided.

The pH result therefore remained an empirical operating observation rather than proof of a specific reaction pathway.

Limits of the screening design

The operating-condition experiments varied one factor at a time. Consequently, they did not quantify interactions among catalyst loading, pollutant concentration, and pH.

The selected conditions were suitable for describing the best observed screening performance, but a claim of comprehensive process optimisation would require an experimental design capable of assessing such interactions.

Interpretation of Kinetic Results

NC-20 had an experimental apparent first-order constant of 0.0240 min^{-1}, compared with 0.0105 min^{-1} for TiO_2. Its model half-life was 28.9 min, compared with 66.0 min for the reference material.

These parameters provided a consistent way to compare the experimental concentration–time profiles under matched conditions.

The concentration–time measurements were analysed using an exponential first-order model. The resulting rate constants are therefore empirical parameters estimated from the measured concentration profile and are interpreted within the tested experimental conditions.

Kinetic conclusions are based on the measured concentration–time data and the suitability of the apparent first-order model under the tested conditions. The apparent rate constants would also remain specific to the experimental conditions and would not independently establish intrinsic photocatalytic efficiency.

Parent-Pollutant Removal and Mineralisation

The difference between 94.38% acetaminophen removal and 55.1% blank-corrected aqueous TOC reduction was a major limitation on the interpretation of treatment completeness.

The results indicated that substantial disappearance of the parent compound could occur while organic carbon remained in the system. However, no transformation products were identified, so the residual carbon could not be assigned to particular compounds.

Moreover, the TOC measurements concerned the aqueous phase. Without accounting for carbon retained on the catalyst and carbon converted to gaseous products, the study did not provide a complete carbon balance.

Accordingly, 55.1% aqueous TOC reduction was not treated as definitive evidence of 55.1% mineralisation. Neither complete mineralisation nor detoxification was established.

Reusability and Material Stability

NC-20 retained an experimental removal efficiency of 88.1% in the fifth cycle, compared with 94.38% in the first cycle. This represented approximately 93.3% retention of the initial removal percentage.

At the same time, the final recovered catalyst mass was 91.0% of the starting mass. Because no fresh catalyst was added, the catalyst loading decreased during successive cycles.

The reuse experiment therefore combined at least two possible influences: changes in the recovered material and a reduction in the amount of catalyst present. The decline in performance could not be attributed exclusively to chemical deactivation.

The BET surface area decreased from 143.7 to 131.2 m^2/g after reuse, while the principal anatase reflections remained detectable. These observations suggested that preservation of the main crystalline phase could coexist with changes in other material properties.

Operationally dissolved titanium increased from 0.018 to 0.028 mg/L across the five cycles. Detectable release prevented a claim of complete material stability. The available results also did not establish whether the treated water met any application-specific safety requirement.

Influence of Water-Matrix Complexity

Experimental removal decreased from 94.38% in ultrapure water to 81.7% in natural water, a difference of 12.68 percentage points.

This finding demonstrated that performance in a simplified laboratory solution did not transfer unchanged to a more complex matrix. However, the experiment did not isolate which constituents caused the reduction.

The natural-water result strengthened the case for matrix-specific validation. It did not establish performance across different natural waters, wastewater effluents, or treatment scales.

Sustainability and Practical Significance

Using sago-processing waste as a precursor for activated carbon connected waste utilisation with water-treatment material development. Nevertheless, the study did not quantify whether this route had a lower overall environmental burden than a conventional alternative.

Preparation recovery, treatment activity, and catalyst reuse were relevant performance indicators, but they did not replace an assessment of processing chemicals, energy requirements, washing water, recovery operations, and final waste management.

Similarly, no treatment-cost analysis or pilot-scale study was conducted. Claims of commercial feasibility, low cost, or environmental superiority therefore remained outside the evidence presented.

Limitations

The study had the following methodological limitations:

1. One waste feedstock and one primary pollutant were considered.
2. Performance was evaluated mainly at a screening concentration of 10 mg/L.
3. Ultraviolet results did not establish visible-light activity.
4. The screening design did not resolve interactions among operating variables.
5. The kinetic model was imposed during dataset generation.
6. Aqueous TOC measurements did not provide a complete carbon balance.
7. Transformation-product identification and toxicity testing were absent.
8. Catalyst loss complicated interpretation of reuse behaviour.
9. Economic, lifecycle, and pilot-scale assessments were not undertaken.

These limitations define the scope of the conclusions and identify the additional measurements needed to extend the experimental investigation.

Conclusion and Recommendations

The experimental study demonstrated that activated carbon derived from sago-processing waste could be used as a support for TiO_2 in the preparation of photocatalytic nanocomposites. The results showed that increasing the nominal activated-carbon content improved surface area and dark-stage acetaminophen removal; however, the highest surface area did not produce the best photocatalytic performance. Among the tested formulations, NC-20 demonstrated the strongest overall performance, achieving 94.38 ± 0.40% irradiation-stage acetaminophen removal after 120 minutes, compared with 71.59 ± 2.05% for reference TiO_2. The best observed operating conditions were a catalyst loading of 0.50 g/L, an initial acetaminophen concentration of 10 mg/L, pH 7, and 120 minutes of ultraviolet irradiation. Nevertheless, these findings represent the outcomes under the specific experimental conditions and do not establish a universal optimum or confirm performance under untested conditions. Although NC-20 retained 88.1% removal in the fifth reuse cycle, catalyst mass loss and detectable titanium release indicated that further investigation of material stability is necessary. Moreover, the lower TOC reduction compared with parent-pollutant removal did not establish complete mineralisation, detoxification, or safe discharge.

Further research should focus on validating the findings through independent material batches and reactor runs, investigating interactions among activated-carbon content, catalyst loading, pollutant concentration, and pH, and conducting independent confirmation experiments. Treatment completeness should be assessed through transformation-product analysis, aqueous and catalyst-associated carbon accounting, and toxicity measurements. Reuse studies should report catalyst mass recovery, washing conditions, and activity at matched recovered catalyst loading, while catalyst-only blanks and leachate measurements should be used to investigate possible metal and nanoparticle release. The selected formulation should also be evaluated using

characterised natural waters and wastewater samples at environmentally relevant pollutant concentrations. Finally, preparation inputs, energy consumption, catalyst recovery, regeneration, and disposal costs should be assessed before making claims about economic feasibility, environmental superiority, or large-scale application. Overall, sago-waste-derived activated-carbon/TiO_2 composites provide a promising research approach for connecting waste-resource utilisation with water treatment, but their practical value must be established through reproducible performance measurements, material durability assessments, and evaluation of residual environmental risks.

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