Reaktor

p-ISSN 0852 – 0798
e-ISSN 2407 – 5973

Website: http://ejournal.undip.ac.id/index.php/reaktor/

Reaktor, Vol. 26 No. 1, April Year 2026, pp. 1-15

Assessment of TiO_2–C-dots Nanocomposites Derived from Tofu Liquid Waste for Post-Mining Soil Remediation: Its Application in Bengkulu Province

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(Received: 3 December 2025; Accepted: 27 March 2026; Published: 24 April 2026)

Abstract

This paper critically evaluates the technological feasibility of producing TiO_2–carbon dots (C-dots) nanocomposites from tofu liquid waste as a biomass-derived carbon precursor for soil remediation in a post-mining environment. Rather than assuming conversion efficiency or economic viability, the analysis is structured around the synthesis routes, composite fabrication strategies, and process–structure–performance relationships curated from the relevant literature. Biomass-to-C-dots conversion pathways, including hydrothermal, microwave-assisted, and pyrolytic methods, are assessed with respect to feedstock tolerance, operational conditions, and product characteristics. Integration strategies between C-dots and TiO_2, namely in situ growth, impregnation, and sol–gel hybridization, are assayed in terms of their interfacial coupling, stability, and photocatalytic relevance for heavy-metal immobilization. A regional case context from Bengkulu Province is used solely to illustrate feedstock availability and chemical relevance, without extrapolating to production yield or economic feasibility. The results demonstrate that the functional performance of TiO_2–C-dots systems is governed primarily by synthesis parameters and composite architecture rather than by precursor volume. Current evidence situates this technology at an early development stage, where reproducible fabrication and interfacial engineering remain the principal determinants of applicability. These findings provide a process-centered framework for evaluating biomass-derived photocatalytic composites while avoiding premature feasibility claims unsupported by mature conversion technologies.

Keywords: Photocatalysis, post-mining, soil remediation, TiO_2–C-dots Nanocomposites, Tofu Liquid Waste Valorization.

How to Cite This Article: Silalahi, J.L., and Khatami, M., (2026) Assessment of TiO_2–C-dots Nanocomposites Derived from Tofu Liquid Waste for Post-Mining Soil Remediation: Its Application in Bengkulu Province, Reaktor, 26 (1), 1–15, https://doi.org/10.14710/reaktor.79958

INTRODUCTION

Bengkulu Province is one of Indonesia's gold-producing centers, yet mining activities simultaneously pose a serious threat to the sustainability of the agricultural sector. Weak regulatory enforcement has facilitated the proliferation of illegal mining operations that are increasingly difficult to control. Gold extraction not only alters land use, but also contributes to the release of hazardous heavy metals, as Pb^{2+}, Cd^{2+}, and Hg^{2+} into the surrounding environment. For instance, gold mining activities in Bukit Sanggul, Seluma Regency, have endangered approximately 2,378 hectares of rice fields (Wicaksono, 2024). According to (Chu *et al*., 2019), elevated concentrations of Pb and Cd can immobilize phosphorus (P) in soil, thereby reducing its availability to needy plants. Similarly, Afrisa *et al*. (2023) reported that soils in South Bengkulu, particularly in Seluma, exhibit highly acidic conditions (pH 4.89–5.52), low phosphorus availability, and variable potassium level. A comparable condition is found in coastal areas of Bengkulu City, where entisol soils contain low levels of phosphorus and potassium due to their sandy texture and poor organic matter content, resulting in a lower fertility (Herman *et al*., 2024). These conditions highlight the urgent need for effective interventions to restore soil quality in Bengkulu Province.

One of the strategic approaches widely explored for environmental remediation is the use of titanium dioxide (TiO2)-based photocatalytic technology. This technology employs electron excitation on the TiO_2 surface under UV irradiation, generating electron–hole pairs that subsequently produce hydroxyl radicals (•OH) and superoxide radicals (O_2•⁻) that are capable of reducing heavy-metal contaminants (Pavel *et al*., 2023). However, TiO_2 possesses some drawbacks, including the reliance on pure chemical precursors such as citric acid or urea for the synthesis of carbon nanodots (C-dots), which is neither cost-effective nor sustainable, and its predominant applicability in aqueous media. Moreover, TiO_2 absorbs light only within the UV region, representing approximately 5% of the solar spectrum, due to its wide energy band gap (3.2 eV), resulting in relatively low photocatalytic efficiency (Peslinof, 2018).

Tofu industry in Bengkulu Province represents a rapidly expanding MSME sector, yet it generates large large quantities of untreated liquid waste that remains underutilized and is often discharged directly into the environment, possessing risks of soil and river pollution. A study carried out by Sarina & Hasibuan (2021) reported that in Sukaraja District, Seluma Regency, there are nine tofu production units, which each process 200–400 kg of soybeans per day, producing approximately 96–210 blocks of tofu. Each kilogram of soybean generates roughly 8–10 liters of liquid waste (Cahyani *et al*., 2021), resulting in a total liquid waste output of about 1,600–4,000 liters per day. In fact, this effluent is rich in soluble organic compounds, including carbohydrates, proteins, and residual lipids, which constitutes a readily available carbon source for hydrothermal or microwave-assisted conversion into carbon nanomaterials. In addition, a study by (Chu *et al*., 2019) highlighted that C-dots derived from tofu liquid waste can function as effective sensitizers capable of extending light absorption to enhance photocatalytic reactions. Previous studies have demonstrated that TiO_2-based photocatalysts are effective agents for the removal and transformation of a wide range of environmental contaminants, including several heavy-metal species, by means of photo-induced electron–hole generation and subsequent redox reactions that promote adsorption, reduction and immobilization processes at the catalyst surface. Comprehensive reviews and experimental reports document TiO_2's capacity to completely remove or reduce dissolved metal ions under irradiation and to serve as a scaffold for further modification to extend light absorption and selectivity (Gopinath, *et al*., 2020; Gao & Meng, 2021). Concurrently, carbon quantum dots (CQDs) have emerged as multifunctional nanomaterials that (i) act as visible-light sensitizers when coupled with wide energy band gap semiconductors, (ii) improve charge separation and transfer in heterojunction photocatalysts, and (iii) provide abundant surface functional groups (–COOH, –OH, $–NH_2$) that chelate metal ions and promote adsorption. Several recent reports (Falara *et al*., 2024; Ghamarpoor *et al*., 2024) show that C-dots, when composited with TiO_2, shift optical response into the visible region, reduce electron–hole recombination, and increase photocatalytic removal efficiencies for a variety of aqueous contaminants relative to pristine TiO_2. Beyond contaminant degradation, an expanding body of literature indicates that C-dots can positively modulate nutrient dynamics and plant responses. Experimental studies and reviews (Gogoi *et al*., 2023; Li *et al*., 2023) report that appropriately engineered C-dots (including N- and P-doped variants) can increase solubilization or bioavailability of otherwise immobile phosphate fractions, enhance nutrient uptake, and stimulate plant growth and photosynthetic performance, mechanisms that include chelation/ complexation of ions, interaction with rhizosphere microbiota, and up-regulation of nutrient-transport pathways in plants. These agronomic effects suggest a dual function for C-dots in remediation systems. Simultaneously reducing bioavailable heavy-metal concentrations while promoting nutrient availability.

Taken together, these prior findings justify the rational design of green-synthesized TiO_2–C-dots nanocomposites as a recommended material for integrated soil remediation and fertility restoration. A green synthesis route (biomass or food-waste precursors, hydrothermal or facile solvothermal methods without harsh reagents) can produce C-dots with beneficial surface functionalities while minimizing environmental footprint; when anchored onto TiO_2, such composites combine photocatalytic redox activity (for heavy-metal transformation/ immobilization) with adsorption/ chelation and nutrient-mobilizing functions of C-dots. Hence, a carefully optimized TiO_2– C-dot nanocomposites, characterized by charge-transfer properties, metal-binding capacity, photostability, and ecotoxicity, represents a promising, scalable candidate for simultaneous degradation/ immobilization of Pb^{2+}, Cd^{2+} and Hg^{2+} and for enhancement of soil P and K availability in contaminated agricultural lands.

SYNTHESIS AND PROCESSING REVIEW METHODOLOGY

Literature Selection and Scope Definition

The literature surveyed in this review was systematically retrieved from well-established scientific databases, including Scopus, Science Direct, and Web of Science (WoS), covering publications from 2014 to 2025 to ensure technological relevance and methodological consistency. The selection criteria prioritized peer-reviewed studies that explicitly reported synthesis routes and processing parameters for biomass-derived C-dots and TiO_2–carbon nanocomposite systems, including information on precursor type, reaction temperature, residence time, pH conditions, and reported material yields. Particular emphasis was placed on studies describing composite fabrication pathways (in situ growth, impregnation, or sol–gel integration) and their influence on band-gap modulation and interfacial charge-transfer behavior. Investigations addressing photocatalytic applications for heavy-metal removal (Pb^{2+}, Cd^{2+}, and Hg^{2+}) were included only when accompanied by sufficient methodological detail on material preparation. Studies limited to performance evaluation without clear description of synthesis and processing steps were excluded from the analysis to maintain a process-oriented scope.

Biomass to C-Dots Conversion Routes

Tofu liquid waste is primarily composed of soluble organic compounds originating from soybean processing, including carbohydrates (oligosaccharides and soluble starch fractions), proteins and amino acids, as well as minor lipid residues. These components provide abundant carbon frameworks and heteroatoms (N and O), which are favorable for the formation of functionalized carbon nanostructures under hydrothermal or thermochemical conditions. The presence of nitrogen-containing species enables the in-situ generation of N-doped C-dots without additional dopants, enhancing surface polarity and electron-donating capability. From a process perspective, the high water content of tofu liquid waste allows direct utilization in aqueous-phase synthesis routes, minimizing the need for drying or solvent exchange. However, its variable composition depending on soybean quality and coagulation method introduces feedstock heterogeneity, which necessitates controlled processing conditions to achieve reproducible C-dots yield and size distribution. These characteristics position tofu liquid waste as a chemically suitable yet compositionally variable carbon precursor for hydrothermal and microwave-assisted carbonization pathways.

Hydrothermal carbonization is the most widely preferred route for converting biomass-derived precursors into C-dots, owing to its compatibility with aqueous feedstocks and moderate operating conditions. In this process, dissolved organic constituents undergo dehydration, polymerization, and aromatization reactions at temperatures typically ranging from 150 to 250°C under autogenous pressure. Reaction times commonly vary between 2 and 12 h, depending on precursor complexity and desired particle size. Biomass-derived hydrothermal C-dots generally exhibit particle diameters between 2 and 10 nm with numerous surface hydroxyl, carboxyl, and amine groups inherited from the precursor. Reported yields vary substantially due to differences in carbon content, reaction severity, and purification strategy, typically spanning from sub-gram to several grams per liter of precursor solution. For tofu liquid waste specifically, hydrothermal synthesis offers a direct aqueous conversion pathway in which soluble carbohydrates and proteins act as carbon sources and passivating agents simultaneously. Nevertheless, the process remains constrained by batch operation, limited volume of autoclaves, and the need for post-treatment steps such as dialysis or centrifugation to remove the unreacted organics.

Microwave-assisted synthesis provides an alternative conversion pathway characterized by rapid volumetric heating and significantly reduced reaction time compared with conventional hydrothermal treatment. Under microwave irradiation, polar organic molecules in biomass precursors absorb electromagnetic energy, promoting rapid carbonization and nucleation of C-dots within minutes. Typical processing temperatures correspond to internal heating in the range of 120–200°C, with reaction times between 5 and 30 min. This route offers advantages in energy efficiency and throughput, yet suffers from challenges in controlling nucleation homogeneity and particle size distribution when complex feedstocks such as tofu liquid waste are employed.

Pyrolytic routes involve high-temperature thermal decomposition of dried biomass or concentrated residues, generally above 300°C under inert or limited-oxygen conditions. C-dots obtained from pyrolysis tend to exhibit higher graphitization degrees, but lower surface functionalization than those produced by hydrothermal or microwave routes. As tofu liquid waste is predominantly aqueous, pyrolytic processing requires energy-intensive drying or concentration prior to treatment, reducing its process attractiveness. Consequently, microwave-assisted and pyrolytic routes are considered technically viable but less directly compatible with wet effluents than hydrothermal synthesis, and their applicability to tofu liquid waste remains largely at laboratory proof-of-concept level.

Integration of C-Dots with TiO_2

In situ growth refers to the formation of C-dots directly on the TiO_2 surface during hydrothermal or solvothermal treatment of a carbon precursor in the presence of TiO_2 particles.

Table 1. Comparison of Biomass-Derived C-dots Synthesis Routes

Method	Typical Temperature	Reaction Time	Typical Yield Range	Particle Size (nm)	Advantages	Limitations	References
Hydrothermal carbonization	150–250°C	2–12 h	0.1–5 g L^{-1} (depending on precursor and purification)	2–10	Compatible with wet biomass; rich surface functional groups; simple equipment	Batch process; long residence time; variable yield	Cai *et al.*, 2025; Kang, *et al.*, 2020
Microwave-assisted synthesis	120–200°C (internal heating)	5–30 min	0.2–3 g L^{-1}	2–8	Rapid heating; reduced energy consumption; short processing time	Limited control of size distribution; hot-spot formation	De Medeiros *et al.*, 2019; Selvaraj *et al.*, 2025; Jorns *et al.*, 2022
Pyrolytic carbonization	>300°C	0.5–3 h	0.05–2 g g^{-1} dry biomass	3–15	Higher graphitic content; solvent-free	Requires drying; low surface functionality; higher energy demand	Yang *et al.*, 2024; Cui *et al.*, 2021; He *et al.*, 2021

Table 2. TiO_2–C-dots Nanocomposites Fabrication Methods

Integration Route	Typical Temperature	Bonding Type	Composite Morphology	Advantages	Limitations	References
In situ growth (hydrothermal)	150–200°C	Ti–O–C (partial covalent)	C-dots anchored on TiO_2 surface	One-step synthesis; strong interfacial contact; reduced recombination	Limited control of C-dots loading; batch process	Yu *et al.*, 2014; Ge *et al.*, 2012; Shen et al., 2021
Impregnation/ physical mixing	60–120°C (drying)	Electrostatic/ H-bonding	C-dots adsorbed on TiO_2 particles	Simple procedure; independent control of components	Weak bonding; possible leaching; lower stability	Zhang *et al.*, 2021; Yuan *et al.*, 2025
Sol–gel/ hybrid integration	≤300°C (post-gel treatment)	Ti–O–C (network integration)	C-dots embedded in TiO_2 matrix	Uniform dispersion; tunable porosity; high stability	Thermal degradation risk of C-dots; multistep process	Hua *et al*, 2020; Falara *et al.*, 2024

In this route, organic molecules undergo carbonization while simultaneously anchoring onto surface hydroxyl groups of TiO_2, promoting the formation of Ti–O–C interfacial bonds. Typical processing conditions involve temperatures of 150–200°C and reaction times of 4–12 h, allowing nucleation of nanoscale carbon domains at the semiconductor interface. This approach enhances interfacial charge transfer by minimizing physical separation between TiO_2 and C-dots, thereby reducing electron–hole recombination. However, the method offers limited control over C-dots loading and size distribution, as nucleation occurs concurrently with carbonization. From a process standpoint, in situ growth is attractive due to its single-step operation, yet reproducibility remains sensitive to precursor concentration and surface chemistry of TiO_2.

Impregnation and physical mixing constitute post-synthesis integration strategies in which pre-formed C-dots are deposited onto TiO_2 via adsorption, stirring, or sonication. In this method, TiO_2 powders are dispersed in aqueous or alcoholic C-dots solutions and subjected to mild thermal drying (60–120°C) to immobilize the nanocarbon species. The resulting interaction is dominated by electrostatic attraction and hydrogen bonding between surface hydroxyl groups of TiO_2 and functional moieties (–COOH, $–NH_2$) on C-dots. This route allows independent optimization of C-dots synthesis and composite formulation, enabling more precise control of C-dots loading. Nevertheless, weak interfacial bonding increases the risk of leaching during aqueous or soil-contact applications. Consequently, impregnation-based composites are more suitable for mechanistic evaluation and screening studies than for long-term environmental deployment.

Sol–gel and hybrid fabrication routes integrate C-dots into the TiO_2 matrix during the hydrolysis and condensation of titanium precursors (titanium isopropoxide or titanium butoxide). In this process, C-dots are introduced into the sol prior to gelation, allowing uniform dispersion within the growing TiO_2 network. Subsequent drying and mild thermal treatment (≤300°C) yield nanocomposites with embedded carbon domains and enhanced interfacial contact. This route provides superior control over composite morphology and porosity while promoting stable Ti–O–C bonding. However, high-temperature calcination commonly employed to crystallize TiO_2 must be limited to avoid thermal degradation of C-dots. Thus, sol–gel integration is technically advantageous for tailoring nanostructure but requires careful optimization of thermal treatment to preserve carbon functionality.

Key Process Parameters Governing Yield and Performance

The yield of biomass-derived C-dots and the functional performance of TiO_2–C-dots nanocomposites are governed by a set of interrelated processing parameters that control carbonization kinetics, surface chemistry, and interfacial charge-transfer behavior. Among these, reaction temperature is the dominant factor influencing dehydration and aromatization of organic precursors; temperatures below 150°C generally lead to incomplete carbonization, whereas excessive severity (>250°C) promotes aggregation and loss of surface functional groups. Reaction time further modulates nucleation and growth, with prolonged treatment increasing carbon core size but potentially reducing photoluminescence efficiency due to excessive graphitization.

Solution pH critically affects precursor polymerization and heteroatom incorporation. Acidic environments favor dehydration and nucleation of carbonaceous clusters, while alkaline conditions promote surface oxidation and formation of carboxyl-rich C-dots, which enhance hydrophilicity and interfacial interaction with TiO_2. Precursor concentration determines nucleation density and particle size distribution: dilute systems favor small, well-dispersed C-dots, whereas concentrated feeds increase yield at the expense of size uniformity.

For composite fabrication, the mass ratio of C-dots to TiO_2 dictates light-harvesting efficiency and charge-separation behavior. Insufficient C-dots loading yields negligible sensitization, while excessive loading can shield TiO_2 active sites and act as recombination centers. The choice of integration route (in situ growth, impregnation, or sol–gel) further governs bonding strength and electron-transfer pathways, with Ti–O–C interfacial linkages generally associated with improved charge mobility and reduced recombination rates.

Post-synthesis treatment, including drying temperature and calcination, also exerts a critical influence. Mild thermal treatment (<300°C) enhances crystallinity of TiO_2 while preserving C-dots functionality, whereas higher temperatures induce carbon degradation and loss of visible-light response. Collectively, these parameters determine not only the attainable C-dots yield but also the electronic structure and surface reactivity of the composite, underscoring the need for process-level optimization rather than purely performance-driven evaluation.

Table 3. Critical Parameters Affecting C-dots Yield and Composite Performance

Parameter	Typical Range	Effect on C-dots Yield	Effect on Composite Performance	References
Reaction temperature (hydrothermal/ microwave)	150–250°C	Higher temperature increases carbonization degree and yields up to an optimum; excessive temperature causes aggregation	Controls band-gap narrowing and surface functionalization influencing visible-light absorption	Ozyurt *et al.*, 2023; Xu *et al.*, 2023; Ng, et al., 2021
Reaction time	2–12 h (hydrothermal); 5–30 min (microwave)	Longer time increases particle growth and apparent yield	Excessive time reduces photoluminescence and charge-transfer efficiency	Quaid *et al.*, 2022; Ng *et al.*, 2021
Solution pH	2–10	Acidic pH promotes nucleation; alkaline pH increases surface oxidation	Determines surface charge and TiO_2–C-dots interaction strength	Chu *et al.*, 2019; Zhang *et al.*, 2021
Precursor concentration	0.1–10 wt% carbon equivalent	Higher concentration increases yield but broadens size distribution	Influences light absorption and recombination pathways	Ozyurt *et al.*, 2023
C-dots/TiO_2 mass ratio	0.5–10 wt%	Does not directly affect C-dots yield but affects utilization efficiency	Low ratio: weak sensitization; high ratio: active-site shielding and recombination	Mahmood *et al.*, 2021; Yuan *et al.*, 2025
Integration route	In situ/ impregnation/ sol–gel	In situ yields lower recoverable C-dots but higher utilization	Sol–gel and in situ routes enhance interfacial bonding and stability	Yuan *et al.*, 2025; Rawat, et al., 2024
Post-treatment temperature	80–300°C	Mild heating preserves functional groups; high temperature reduces carbon yield	Affects crystallinity and visible-light activity	Dong *et al.*, 2020; Cong *et al* 2025

Characterization Techniques Relevant to Process Evaluation

Characterization techniques play a central role in linking synthesis conditions to the structural and electronic properties of biomass-derived C-dots and TiO_2–C-dots nanocomposites. Transmission electron microscopy (TEM) and high-resolution TEM provide direct information on particle size, morphology, and dispersion, enabling assessment of nucleation and growth behavior as a function of reaction temperature and precursor concentration. X-ray diffraction (XRD) is used to identify the crystalline phase of TiO_2 (anatase, rutile, or mixed phases) and to evaluate the impact of post-treatment temperature on crystallinity, which governs charge transport and recombination kinetics.

Surface chemistry is primarily evaluated through Fourier-transform infrared spectroscopy (FTIR) and X-ray photoelectron spectroscopy (XPS). These techniques reveal the presence of oxygen- and nitrogen-containing functional groups inherited from biomass precursors and allow identification of Ti–O–C bonding at the composite interface. Such information is critical for interpreting interfacial electron transfer and stability of the integrated system. Ultraviolet–visible diffuse reflectance spectroscopy (UV–Vis DRS) and photoluminescence (PL) spectroscopy are employed to assess band-gap modification and recombination behavior, respectively, providing indirect evidence of the effectiveness of C-dots integration routes in extending light absorption into the visible region.

Textural properties, including specific surface area and pore structure, are determined using Brunauer–Emmett–Teller (BET) analysis, which reflects the influence of sol–gel processing and thermal treatment on porosity and adsorption capacity. Together, these characterization tools enable process-level evaluation by correlating synthesis parameters with particle size distribution, interfacial bonding, electronic structure, and surface reactivity. Consequently, material characterization is not merely descriptive but constitutes a diagnostic framework for optimizing synthesis routes and integration strategies of TiO_2–C-dots nanocomposites derived from biomass precursors.

TECHNOLOGY REVIEW: TiO_2–C-DOTS NANOCOMPOSITES

C-Dots as Biomass-Derived Functional Additives

C-dots derived from biomass precursors have emerged as multifunctional additives for semiconductor photocatalysts due to their tunable surface chemistry, optical activity, and electron-transport capability. Biomass-derived C-dots typically contain abundant oxygen- and nitrogen-bearing functional groups (–OH, –COOH, $–NH_2$), originating from carbohydrates and proteins in the precursor, which impart high dispersibility in aqueous media and facilitate interfacial coupling with metal oxides. These surface functionalities also act as anchoring sites for TiO_2, enabling intimate contact and promoting interfacial charge transfer. Compared with C-dots synthesized from pure organic reagents, biomass-derived C-dots offer comparable

Table 4. Characterization-Process Linkage

Technique	Information Obtained	Relevant Process Parameter	Interpretation for Process Evaluation
TEM/ HRTEM	Particle size, morphology, dispersion of C-dots on TiO_2	Reaction temperature; precursor concentration; integration route	Smaller and uniformly distributed particles indicate controlled nucleation and effective in situ or sol–gel integration
XRD	Crystalline phase and crystallite size of TiO_2	Calcination temperature; sol–gel conditions	Higher anatase crystallinity at mild temperatures indicates preserved photocatalytic framework without C-dots degradation
FTIR	Functional groups (–OH, –COOH, $–NH_2$) on C-dots and TiO_2 surface	Precursor composition; pH; synthesis route	Presence of oxygen/ nitrogen functionalities confirms successful biomass-derived carbonization and surface passivation
XPS	Elemental composition and bonding states (Ti–O–C, C–N)	Integration method; post-treatment temperature	Detection of Ti–O–C bonds indicate strong interfacial coupling and efficient charge-transfer pathways
UV–Vis DRS	Optical absorption edge; band-gap shift	C-dots loading ratio; synthesis severity	Red-shifted absorption reflects effective sensitization and visible-light activation
Photoluminescence (PL)	Recombination behavior of charge carriers	Composite morphology; interface quality	Lower PL intensity indicates suppressed electron–hole recombination due to improved interfacial contact
BET surface area analysis	Specific surface area and pore distribution	Sol–gel parameters; drying and calcination temperature	Higher surface area and suitable porosity suggest improved adsorption capacity and reactant accessibility

optoelectronic behavior while introducing intrinsic heteroatom doping without the need for additional chemical modifiers, making them particularly suitable as low-complexity functional additives in composite systems (Cai *et al*., 2025; Dubey, 2023).

From a functional standpoint, C-dots contribute to TiO_2-based photocatalysts through three principal mechanisms. First, their π-conjugated carbon cores enable absorption of visible light and subsequent electron donation to the conduction band of TiO_2, extending photoresponse beyond the ultraviolet region. Second, C-dots can act as transient electron reservoirs, capturing photogenerated electrons and thereby suppressing electron–hole recombination within the TiO_2 lattice. Third, the polar surface groups of C-dots enhance surface wettability and adsorption affinity for metal ions, indirectly improving photocatalytic and immobilization performance. These roles depend strongly on C-dots size, degree of graphitization, and functional group density, which are governed by the severity of the carbonization process and precursor composition (Shen *et al*., 2022; Chen *et al*., 2021; Wang *et al* 2014).

When derived from biomass such as agricultural residues or food-processing effluents, C-dots additionally exhibit precursor-dependent heterogeneity that influences their additive behavior. Protein-rich feedstocks tend to yield nitrogen-doped C-dots with enhanced electron-donating capacity, whereas carbohydrate-dominant precursors favor oxygenated surfaces that improve hydrophilicity and interfacial adhesion. This variability underscores the importance of process control in converting biomass into reproducible functional additives rather than merely carbonaceous by-products. In TiO_2–C-dots nanocomposites, the functional contribution of C-dots is therefore inseparable from their synthesis route: hydrothermal and microwave-assisted processes typically generate highly functionalized surfaces suitable for sensitization and interfacial bonding, whereas high-temperature pyrolytic routes produce more graphitic domains with reduced surface polarity and weaker coupling to TiO_2 (Chen *et al*., 2021; Zikalala *et al*., 2021).

Overall, biomass-derived C-dots should be viewed not as passive carbon fillers but as active functional components whose optical and electronic roles are dictated by their precursor chemistry and conversion pathway. Their effectiveness as additives in TiO_2-based systems arises from a combination of light-harvesting capability, interfacial electron mediation, and surface chemical compatibility. Consequently, their utilization in composite photocatalysts must be evaluated in relation to synthesis-induced properties rather than solely on the basis of precursor availability or nominal carbon content.

Fabrication Pathways of TiO_2–C-Dots Nanocomposites

The fabrication of TiO_2–C-dots nanocomposites can be broadly classified into three technological routes: in situ growth, impregnation/physical mixing, and sol–gel or hybrid integration. Each pathway imposes distinct constraints on interfacial bonding, spatial distribution of carbon domains, and structural stability, which in turn determine the electronic interaction between C-dots and the TiO_2 matrix. Consequently, the selection of fabrication route is a primary determinant of composite architecture rather than a secondary formulation step.

In situ growth involves the concurrent carbonization of an organic precursor in the presence of pre-formed TiO_2 under hydrothermal or solvothermal conditions. During this process, nucleation of carbon nanodomains occurs directly on the TiO_2 surface through interaction with surface hydroxyl groups, leading to partial formation of Ti–O–C linkages. This one-pot route minimizes interfacial distance and promotes intimate electronic coupling, which is advantageous for interfacial charge transfer. Typical conditions range from 150–200°C with reaction times of 4–12 h. However, control over C-dots size and loading is limited because nucleation and growth are governed by the precursor concentration and TiO_2 surface chemistry rather than by independent tuning parameters. From a processing standpoint, in situ growth simplifies synthesis but sacrifices compositional precision and reproducibility when complex biomass-derived precursors are used (He *et al*., 2019; Wang *et al*., 2014).

Impregnation and physical mixing represent post-synthesis integration strategies in which pre-formed C-dots are deposited onto TiO_2 particles via adsorption, sonication, or mechanical stirring, followed by low-temperature drying. The interfacial interaction is primarily electrostatic or hydrogen bonding between oxygen- or nitrogen-containing groups on C-dots and surface –OH groups on TiO_2. This route decouples C-dots synthesis from composite formation, allowing independent optimization of each step and accurate control of C-dots loading. Nevertheless, the weak bonding nature renders the composite susceptible to C-dots leaching under aqueous or soil-contact conditions, and the electronic coupling is generally inferior to that obtained by covalent or semi-covalent interfacial bonding. As a result, impregnation-based composites are well suited for mechanistic studies but less robust for long-term environmental deployment (Phiri *et al*. 2023; Yu *et al*., 2014; Zhou *et al*., 2023).

Sol–gel and hybrid integration routes embed C-dots within the TiO_2 matrix during the hydrolysis and condensation of titanium alkoxides.

Table 5. Fabrication Routes and Structural Outcome

Fabrication Route	Key Process Conditions	Interfacial Bonding Type	Structural Outcome	Advantages	Limitations	References
In situ growth (hydrothermal/ solvothermal)	150–200 °C; 4–12 h; TiO_2 dispersed in carbon precursor solution	Partial covalent (Ti–O–C) and strong interfacial contact	C-dots nucleated and anchored on TiO_2 surface	One-step process; intimate electronic coupling; reduced interfacial resistance	Limited control of C-dots loading; batch operation; sensitivity to precursor composition	He *et al.*, 2019; Wang *et al.*, 2014
Impregnation/ physical mixing	Pre-formed C-dots mixed with TiO_2; drying at 60–120 °C	Electrostatic interaction and hydrogen bonding	C-dots adsorbed on TiO_2 particle surface	Simple procedure; independent optimization of C-dots and TiO_2	Weak bonding; possible leaching; lower structural stability	Phiri *et al.* 2023; Yu *et al.*, 2014; Zhou *et al*, 2023
Sol–gel/ hybrid integration	Hydrolysis of titanium alkoxide with dispersed C-dots; calcination ≤300 °C	Ti–O–C network bonding within oxide matrix	C-dots embedded in TiO_2 framework with controlled porosity	Uniform dispersion; tunable morphology; high stability	Multi-step process; risk of C-dots degradation at high temperature	Cerro-Prada *et al.*, 2018; Miditana *et al.*, 2022

In this approach, C-dots are introduced into the sol prior to gelation, resulting in homogeneous dispersion within the growing oxide network. Subsequent drying and mild thermal treatment (≤300°C) produce composites with improved interfacial contact and controlled porosity. The sol–gel route enables precise tailoring of morphology and facilitates formation of stable Ti–O–C linkages while preserving surface functional groups when low calcination temperatures are employed.However, excessive thermal treatment leads to degradation of C-dots and loss of surface functionality. Process-wise, sol–gel methods offer superior control over composite structure but involve multiple steps and stricter control of thermal history (Cerro-Prada *et al.,* 2018; Miditana *et al.*, 2022).

Across these fabrication pathways, the structural outcome of TiO_2–C-dots nanocomposites are governed by the balance between interfacial bonding strength and spatial distribution of carbon domains. Routes that promote chemical bonding and uniform dispersion generally yield enhanced interfacial charge-transfer efficiency, whereas routes dominated by physical adsorption tend to produce heterogeneous architectures with lower stability. Thus, fabrication strategy should be selected based on the desired balance between simplicity, structural precision, and long-term integrity rather than on performance metrics alone.

Interfacial Charge-Transfer and Photocatalytic Mechanism

The primary limitations of pristine TiO_2 photocatalysts lie in their wide band gap energy, which restricts light absorption predominantly to the UV region, and the rapid recombination of photo-generated electron–hole (e^-/h^+) pairs. Consequently, the development of TiO_2–C-dots nanocomposites has emerged as a key strategy to overcome these challenges. Mechanistically, C-dots exhibit dual functionality: they act as sensitizers that extend optical absorption into the visible-light region, and as efficient electron donors or acceptors that facilitate charge transfer, thereby forming a heterojunction that enhances charge separation. This mechanism involves several critical steps, including the excitation of C-dots under visible light, electron transfer to the conduction band (CB) of TiO_2, and subsequent redox reactions that generate ROS such as $O_2{\bullet}^-$ and •OH, which are fundamental to photocatalytic degradation processes.

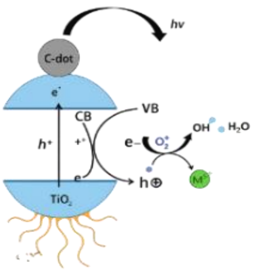

Figure 2. Photocatalytic Mechanism of TiO_2-C-Dots Nanocomposites

The TiO_2–C-dots nanocomposites operate through a visible-light-driven photocatalytic mechanism. Upon solar irradiation, the TiO_2 component absorbs photon energy and undergoes electronic excitation from the valence band to the conduction band, forming e^-/h^+ pairs. In conventional TiO_2 systems, these charge carriers recombine rapidly, limiting photocatalytic efficiency. The incorporation of C-dots effectively mitigates this limitation: C-dots function as electron mediators that absorb visible light and transfer electrons to TiO_2, thereby prolonging the lifespan of charge carriers. This extended charge separation significantly enhances the production of •OH and $O_2{\bullet}^-$, which are highly reactive toward the oxidation of organic pollutants and the reduction of heavy-metal ions to more stable species. Moreover, the active functional groups present on C-dots (–OH, –COOH, and $–NH_2$) can interact with metal ions such as Pb^{2+}, Hg^{2+}, and Cd^{2+} through complexation and chemisorption, further contributing to the nanocomposite's remediation performance.

C-dots Excitation (Sensitization):

$$Cdots + hv \rightarrow Cdots^{*}(e^{-} + h^{+}) \quad (1)$$

Electron Injection into TiO_2 (Charge Separation):

$$Cdots^{*}(e^{-}) + TiO_2 \rightarrow Cdots(h^{+}) + TiO_2(e^{-}_{CB}) \quad (2)$$

Reduction Reaction in the CB of TiO_2 (Formation of Superoxide Radicals):

$$TiO_2(e^{-}_{CB}) + O_2 \rightarrow TiO_2 + O_2^{\cdot -} \quad (3)$$

Oxidation Reaction on the TiO_2 Surface (Formation of Hydroxyl Radicals):

$$TiO_2(h^{+}_{VB}) + H_2O \rightarrow TiO_2 + OH + H^{+} \quad (4)$$

$$TiO_2(h^{+}_{VB}) + OH^{-} \rightarrow TiO_2 + OH \quad (5)$$

Degradation of Organic Pollutants (P):

$$P + \frac{OH}{O_2^{\cdot -}} \rightarrow degradation\ products \rightarrow CO_2 + H_2 \quad (6)$$

Reported Process and Performance Relationships in Literature

Literature on TiO_2–C-dots nanocomposites demonstrates that photocatalytic and immobilization performance for heavy metals is strongly contingent on fabrication route, interfacial bonding, and composite morphology rather than on the mere presence of carbonaceous additives. In situ growth and sol–gel routes consistently yield stronger interfacial coupling (often evidenced by Ti–O–C signatures) and more homogeneous dispersion of carbon domains, which correlate with enhanced visible-light absorption and suppressed electron–hole recombination. By contrast, impregnation-based composites typically show modest performance gains attributable to physical adsorption of C-dots, with improvements limited by weak bonding and partial surface shielding. Process severity during C-dots synthesis further modulates composite performance. Hydrothermal or microwave-derived C-dots with abundant oxygen- and nitrogen-containing groups promote better wettability and interfacial contact with TiO_2, facilitating electron transfer and improving adsorption of metal ions. Excessive carbonization (high-temperature pyrolytic routes) increases graphitization but diminishes surface functionality, leading to poorer coupling with TiO_2 and lower photocatalytic gains. Consequently, performance enhancements reported for Pb^{2+}, Cd^{2+}, and Hg^{2+} removal is typically associated with composites prepared under mild-to-moderate thermal conditions that preserve functional groups while ensuring nanoscale carbon domains.

Reported removal efficiencies vary widely (from moderate to high) because they are inseparable from process parameters such as C-dots loading, integration route, and post-treatment temperature. Studies employing in situ growth or sol–gel integration generally report higher stability over multiple cycles than impregnation-based systems, reflecting the role of chemical or semi-covalent interfacial bonding in preventing leaching. Importantly, most reported performances are derived from aqueous-phase

Table 6. Technology and Performance Mapping

Composite Type	**Synthesis Route**	**Target Metal**	**Reported Removal Efficiency***	**Key Process Parameter**	**Structural Outcome**	**Reference**
TiO_2/C-dots (in situ)	Hydrothermal in situ growth	Pb^{2+}	85–95% (aqueous)	180°C; 8 h; low C-dots loading	C-dots anchored on TiO_2; Ti–O–C linkage	Shen *et al*, 2021; Chen *et al*., 2021
TiO_2/C-dots (hybrid)	Sol–gel with embedded C-dots	Cd^{2+}	70–90% (aqueous)	Calcination ≤ 300°C; homogeneous dispersion	C-dots embedded in TiO_2 matrix	Hua *et al*., 2020; Song *et al*., 2022
TiO_2/C-dots (impregnated)	Physical mixing with drying	Pb^{2+}	60–75% (aqueous)	C-dots/TiO_2 = 2–5 wt%	Surface-adsorbed C-dots; weak bonding	Alves *et al*., 2023
N-doped C-dots/TiO_2	Hydrothermal with post-impregnation	Cd^{2+}	65–85% (aqueous)	pH-controlled synthesis; N-rich precursor	Functionalized C-dots; enhanced adsorption	Martins *et al*., 2016; Thanh Thuy *et al*., 2022
TiO_2/C-dots composite	Solvothermal in situ	Hg^{2+}	70–88% (aqueous)	160°C; 6 h	Strong interfacial contact; reduced recombination	Chen *et al*., 2021
TiO_2/C-dots from biomass	Hydrothermal C-dots with sol–gel TiO_2	Pb^{2+}, Cd^{2+}	60–90% (aqueous)	Biomass-derived C-dots; mild calcination	High surface polarity; uniform dispersion	Akhtar *et al*., 2025

*Reported efficiencies are drawn from aqueous-phase experiments under visible or simulated solar irradiation; values vary with initial concentration, pH, and irradiation intensity.

experiments; soil-based demonstrations remain limited, indicating that current relationships between process and performance are primarily validated at laboratory scale and in controlled media.

Overall, the literature indicates a consistent trend: fabrication strategies that maximize interfacial contact and preserve surface functionality translate into improved charge separation and higher metal-removal efficiency, whereas routes prioritizing simplicity at the expense of structural control result in lower and less stable performance. These findings reinforce the necessity of selecting synthesis and integration pathways based on targeted structural outcomes rather than on nominal additive content.

Stability and Reusability of TiO_2–C-Dots Nanocomposites

The operational stability and reusability of TiO_2–C-dots nanocomposites are governed primarily by the nature of interfacial bonding and the structural integrity of the composite during repeated photocatalytic cycles (Huang *et al*., 2023). Composites prepared via in situ growth and sol–gel integration generally exhibit higher stability than those obtained by impregnation or physical mixing, owing to the formation of Ti–O–C linkages or embedded carbon domains that reduce the likelihood of C-dots detachment. In contrast, physically adsorbed C-dots are prone to gradual leaching, particularly under aqueous or mildly acidic conditions, leading to progressive loss of photocatalytic activity.

Experimental studies commonly report that TiO_2–C-dots nanocomposites retain a substantial fraction of their initial activity over three to five reuse cycles, provided that the integration route ensures sufficient interfacial coupling (Syafei *et al*., 2017). Activity decay observed after multiple cycles is typically associated with surface fouling by reaction intermediates, partial oxidation of carbon domains, or aggregation of TiO_2 particles rather than with intrinsic degradation of the TiO_2 lattice. Mild regeneration procedures, such as low-temperature drying or washing with deionized water, have been shown to partially restore activity, indicating that deactivation is largely physical rather than chemical in nature (Nawaz & Saravanan, 2020).

From a process perspective, stability is closely linked to synthesis conditions. Excessive thermal treatment during composite fabrication can degrade C-dots and weaken their functional contribution, whereas insufficient bonding during low-temperature impregnation leads to poor retention under repeated use. These findings underscore that reusability is not an intrinsic property of TiO_2–C-dots systems but a direct outcome of fabrication strategy and post-treatment protocol. Consequently, stability assessments reported in the literature primarily reflect laboratory-scale durability and do not yet constitute evidence of long-term operational robustness in soil-based or field-scale applications.

Technology Readiness and Scalability Considerations

Current TiO_2–C-dots nanocomposite technologies remain predominantly at the laboratory proof-of-concept stage, corresponding to low technology readiness levels. Most reported studies employ batch hydrothermal or solvothermal reactors with small working volumes and controlled precursor compositions, conditions that are difficult to replicate at scale without substantial process modification (Mamaghani *et al*., 2019). The scalability of hydrothermal in situ growth is constrained by autoclave volume, heat-transfer efficiency, and reproducibility of carbonization from heterogeneous biomass-derived precursors. Microwave-assisted routes offer reduced reaction times but introduce challenges in achieving uniform heating and consistent particle nucleation when processing larger volumes (Albert *et al*., 2025; Miditana *et al*., 2022).

Sol–gel and hybrid routes provide better control over morphology and dispersion but require multiple processing steps, including solvent handling, gel ageing, and thermal treatment, which complicate scale-up and increase sensitivity to process deviations. In addition, the variability of biomass-derived C-dots, arising from differences in precursor composition and carbonization severity, introduces feedstock-related uncertainty that must be managed through standardization of synthesis parameters rather than through downstream performance correction.

Importantly, the majority of reported performances for TiO_2–C-dots nanocomposites are derived from aqueous-phase experiments under controlled irradiation, whereas soil-based demonstrations remain scarce. This gap indicates that technology readiness is currently limited not only by reactor scale but also by the absence of validated process–performance relationships in realistic environmental matrices. Therefore, scalability considerations should focus on reactor design, feedstock preconditioning, and reproducibility of composite structures, rather than on economic metrics or deployment scenarios.

Overall, the literature suggests that TiO_2–C-dots nanocomposites occupy an early developmental phase in which synthesis strategies and interfacial engineering dominate technological feasibility. Advancement toward higher readiness levels will require a systematic transition from batch laboratory synthesis to continuous or semi-continuous processing and from model aqueous systems to soil-relevant testing environments.

CASE CONTEXT: TOFU WASTE AVAILABILITY IN BENGKULU

Tofu production in Bengkulu Province is dominated by small and medium-scale enterprises that operate with simple processing lines and stable daily throughput of soybeans. According to (Sarina & Hasibuan, 2021), nine tofu production facilities operate in Sukaraja District, Seluma Regency, each processing approximately 200–400 kg of soybeans per day. Given that every kilogram of soybeans generates an estimated 8–10 liters of liquid waste (Cahyani *et al*., 2021), the total volume of tofu liquid waste produced in this region reaches approximately 1,600–4,000 liters per day. Similar production patterns have been reported in other Indonesian tofu clusters, indicating that liquid waste generation is a structurally inherent outcome of traditional tofu manufacturing rather than an incidental by-product. From a material's perspective, this regional production profile defines the upper bound of feedstock availability and constrains process design to low-cost, decentralized handling strategies rather than centralized industrial-scale treatment systems.

Liquid tofu waste is generated primarily during soybean soaking, grinding, and pressing stages, resulting in an aqueous effluent with high organic content and suspended colloidal matter. Reported characteristics typically include elevated chemical oxygen demand (COD), biological oxygen demand (BOD), and turbidity, reflecting the presence of soluble carbohydrates, peptides, and residual lipids. The waste stream is weakly acidic to near-neutral, depending on coagulant use and washing intensity. Importantly, the volumetric output scales directly with soybean input rather than with tofu block yield, making waste generation predictable at the facility level but variable across clusters. Such variability implies that any downstream material conversion process must accommodate fluctuations in solid content and organic loading without relying on tightly controlled feedstock purity.

The chemical composition of tofu liquid waste confers intrinsic suitability as a carbon precursor for nanomaterial synthesis. It contains dissolved polysaccharides, oligosaccharides, and protein fragments that serve as carbon skeletons under hydrothermal or thermal treatment, while its nitrogen-containing amino acids enable in-situ heteroatom doping. Previous studies have demonstrated that protein-rich and carbohydrate-rich biomass sources yield C-dots with surface functionalities such as –OH, –COOH, and $–NH_2$, which are critical for photogenerated charge transfer when integrated with semiconductors (Liu *et al*., 2025). The presence of endogenous nitrogen in tofu waste is particularly advantageous, as N-doped C-dots exhibit narrower band gaps and enhanced visible-light absorption relative to undoped analogues. Therefore, the relevance of tofu waste lies not in its volume alone but in its molecular composition, which aligns with known precursor requirements for carbon dot formation.

In Bengkulu and comparable regions, tofu liquid waste is commonly discharged directly into drainage systems or nearby water bodies with minimal pretreatment. The absence of on-site treatment infrastructure reflects both economic constraints and the decentralized nature of production units. Conventional wastewater treatment technologies, such as aerobic digestion or chemical coagulation, are rarely adopted due to space limitations and operational complexity. Consequently, liquid tofu waste remains largely unmanaged despite its high pollutant load. From a process development perspective, this context implies that any valorization pathway must be compatible with small-scale collection, tolerate compositional variability, and operate without extensive preconditioning. These constraints also explain why laboratory-based conversion routes must be critically examined before extrapolating to field-relevant implementation.

The heterogeneous nature of tofu liquid waste imposes fundamental constraints on synthesis route selection for C-dots. Variations in organic concentration and nitrogen content necessitate process conditions that are robust against feedstock fluctuations, favoring hydrothermal or microwave-assisted carbonization over highly stoichiometric methods. Mild pre-treatment steps, such as filtration or sedimentation, are sufficient to remove coarse particulates without altering chemical composition, aligning with low-complexity process design. Importantly, feedstock inconsistency shifts the feasibility criterion from maximizing yield to ensuring reproducibility of carbonization behavior and surface functionalization. Thus, in the Bengkulu context, tofu liquid waste should be regarded as a chemically relevant but operationally variable precursor, whose utilization depends primarily on adaptable synthesis strategies rather than on volumetric abundance.

CONCLUSION

This review has reoriented the assessment of tofu liquid waste utilization toward a technology-driven framework centered on synthesis routes and composite fabrication mechanisms. The analysis shows that tofu liquid waste is chemically compatible with carbon dot formation due to its carbohydrate- and protein-rich composition, which supports carbonization and heteroatom doping under hydrothermal or thermal conditions. However, the conversion of such heterogeneous biomass into reproducible C-dots is controlled by operational parameters rather than by precursor availability.

Among reported synthesis pathways, hydrothermal and microwave-assisted methods exhibit the highest tolerance to feedstock variability, whereas pyrolytic routes require stricter control of precursor concentration and thermal history. The performance of TiO_2–C-dots nanocomposites is dictated by the nature of interfacial coupling achieved during fabrication. In situ and sol–gel hybrid routes promote stronger Ti–O–C interactions and improved charge-transfer behavior compared with physical mixing, which remains vulnerable to leaching and structural instability.

Stability and reusability studies indicate that activity retention is mainly influenced by fabrication strategy and post-treatment conditions, highlighting that durability is an emergent property of processing rather than an intrinsic characteristic of the material system.

Technology readiness remains limited to laboratory-scale demonstrations, with scale-up constrained by batch reactor dependence, feedstock heterogeneity, and the absence of validated soil-based performance data.

Overall, the feasibility of TiO_2–C-dots nanocomposites derived from tofu liquid waste should be interpreted as a function of process maturity and structural control rather than as a function of waste volume or theoretical yield. Future research should prioritize reproducible synthesis protocols, interfacial engineering, and performance validation in realistic soil matrices before any techno-economic or deployment-oriented assessment is attempted. This process-centered perspective provides a more defensible basis for evaluating biomass-derived nanocomposites for environmental remediation.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

ACKNOWLEDGEMENTS

The authors are also grateful to institutional partners and scholars whose contributions to discussions on photocatalytic materials, soil remediation strategies, and green nanotechnology significantly informed the direction of this research.

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