

Review

TiO₂–Biochar-Based Photocatalysts for Organic Pollutants Removal: From Synthesis Parameters to Degradation Performance

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Abstract

TiO₂–biochar-based photocatalysts are one of the materials exhibiting adsorption-photocatalytic synergy. They have been widely used in the remediation of water systems. Current reviews in this field predominantly focus on the following aspects. These include the preparation methods for composite materials, the pollutant removal performance, the adsorption–photocatalytic synergy, and environmental applications. However, there are still gaps in understanding the intrinsic relationships among photocatalyst morphology, surface functional groups, reactive oxygen species (ROS) generation, pollutant removal, and interfacial charge-transfer mechanisms. This restricts the potential for further enhancement of photocatalytic performance. To fill this gap, this review provides a comprehensive summary of the impact of various parameters on the morphology of TiO₂–biochar-based photocatalysts during in situ synthesis. These factors include titanium sources, carbon sources, preparation methods, solvents, pyrolysis conditions, and doping modifications. Further analysis is conducted to investigate the effects of morphological structure on the distribution characteristics of surface functional groups (e.g., oxygen- and nitrogen-containing groups), the generation of ROS, and the removal behavior of organic pollutants. Furthermore, this review focuses on the effects of three typical morphologies. The three typical morphologies include surface-adhered, pore-embedded, and interlayer-distributed. The role of morphology in charge transport behavior at interfaces is also examined. We systematically elucidate the mechanisms of coupled interactions among material morphology, surface functional groups, ROS, interfacial charge transport, and photocatalytic performance. An analytical framework is established to explore the relationships among morphology control, structural characteristics, and photocatalytic performance. Lastly, the limitations of TiO₂–biochar-based photocatalysts in environmental remediation processes are summarized. It also points the way forward for future development. Overall, this review provides a new theoretical perspective on the rational design and environmental applications of high-performance TiO₂–biochar-based photocatalysts.

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Keywords: TiO₂-biochar-based photocatalysts; morphology; adsorption–photocatalysis; pollutants

1. Introduction

With the rapid development of industrialization and the continuous improvement in living standards, large quantities of organic pollutants are generated [1–4]. They pose a serious threat to ecosystems and human health. The organic pollutants include dyes, antibiotics, per- and polyfluoroalkyl substances (PFASs), and microplastics (MPs) [5–7]. They are characterized by their resistance to degradation, environmental persistence, and high toxicity [8,9]. However, the concentration of organic pollutants in wastewater treatment plants often exceeds standard limits. Consequently, it is difficult to ensure that the effluent meets discharge requirements. This is because traditional techniques such as filtration, flocculation, and sedimentation are insufficient [10]. Therefore, exploring efficient wastewater treatment technologies has become an important topic in wastewater management.

Advanced oxidation processes (AOPs) can mineralize pollutants, producing CO₂ and H₂O. These emerging water treatment technologies are environmentally friendly and sustainable [11]. AOPs primarily involve Fenton/Fenton-like, electrocatalytic, and photocatalytic processes [12]. Specifically, solar energy (light energy) can be converted into chemical energy via photocatalytic technology, thereby enabling efficient energy utilization [13]. Currently, the technique is widely used in areas such as water splitting, carbon dioxide reduction, hydrogen peroxide production, and pollutant degradation [13–16]. In photocatalytic degradation, photocatalysts break down pollutants. The pollutants are further converted into non-toxic, clean products. Photocatalysts can also reduce the chemical oxygen demand of wastewater [17,18]. During photocatalytic degradation experiments, common semiconductor materials include titanium dioxide (TiO₂), cadmium sulfide (CdS), and graphitic carbon nitride (g-C₃N₄) [19,20]. They show great potential for application in water pollution treatment. Furthermore, metal–organic frameworks (MOFs), covalent organic frameworks (COFs), and the novel material MXene also play an important role in pollutant removal [21–23]. Among the many photocatalytic materials available, researchers strongly favor TiO₂ for its strong oxidizing power, high chemical stability, excellent cost-effectiveness, and favorable safety profile [24]. The three common crystal forms of TiO₂ are anatase, rutile, and brookite [25]. The crystal structure of anatase is more loosely packed than that of rutile and exhibits greater distortion. These characteristics improve the electron transport properties of anatase TiO₂ relative to rutile TiO₂ [26]. The bandgap of anatase TiO₂ is approximately 3.2 eV. This means it can only absorb the ultraviolet component of sunlight, which accounts for approximately 4–6% of solar radiation. The resulting low sunlight utilization severely limits its photocatalytic performance [27]. In addition, when the TiO₂ catalyst is exposed to light, the photogenerated charge carriers recombine quickly. This results in a low quantum yield, which also reduces its performance [28]. Therefore, to improve the catalytic activity of TiO₂ and its ability to use visible light, modification is necessary. Common strategies for modification include controlling morphology and structure, adjusting the bandgap, and constructing composite structures. Typical techniques include surface modification, metal/non-metal doping, forming heterojunctions, and using composite carriers [29]. However, achieving the desired efficiency when purifying wastewater using photocatalysis alone is difficult. There are two main reasons. Firstly, there is the rapid recombination of photo-generated charge carriers. Secondly, the kinetics of the photocatalytic reaction are slow [30]. TiO₂ performance can be improved to some extent by conventional modification. However, there are still some issues to be resolved, such as rapid recombination of photogenerated electron–hole pairs, a

narrow visible-light response range, and poor pollutant adsorption. Meanwhile, TiO₂-based photocatalysts still face challenges in practical applications. These include poor pollutant removal efficiency, high costs, and unstable performance.

Biochar (BC) is a carbon-based material that is produced when biomass waste (e.g., wood, agricultural residues, and livestock manure) is heated at a moderate temperature. A synergistic adsorption-photocatalytic effect can be achieved by combining BC with TiO₂ [31]. A search was conducted in Web of Science using the keywords “photocatalyst”, “adsorption”, and “photocatalysis + adsorption”. The relevant literature from 2016 to 2025 was retrieved. The initial retrieval yielded 69,413, 469,063, and 13,361 relevant articles, respectively. In the “Refine results” filter, enter each target pollutant individually. A total of 3951 articles were retrieved on dyes (e.g., methylene blue, methyl orange, and Congo red). Antibiotics (e.g., ciprofloxacin, tetracycline, and sulfamethoxazole) were the focus of 1011 articles in total. Aromatic compounds (e.g., benzene, phenol, and aniline) accounted for a total of 77 articles. This category included 133 articles on pesticides, such as herbicides and insecticides. There was a total of 24 papers on MPs. The number of papers on PFASs was 41. Then, the selection was further refined based on the titles, keywords, and abstracts of the publications. Literature relating solely to adsorption, photocatalysis, or systems not involving both photocatalysis and adsorption was excluded. As shown in Figure 1a, the core-associated nodes are TiO₂, carbon materials, photocatalytic degradation, adsorption, and visible light. This indicates that current research is primarily focused on TiO₂–carbon materials. The research focuses on expanding the range of light-responsive properties and enhancing pollutant degradation performance. Furthermore, there are also connections between nodes for keywords related to nanosheets, nanocomposites, properties, and mechanisms. However, they are less connected. This implies that their correlation is relatively weak. This is because research into the links between microstructure, photocatalytic performance, and mechanisms is still insufficient. There has been a limited exploration of the mechanisms governing microstructural manipulation. This makes it difficult to achieve the targeted design of high-performance photocatalysts. As shown in Figure 1b, the number of publications on single adsorption techniques far exceeds those on photocatalysis and coupled adsorption–photocatalysis systems. This trend reflects the simplicity of the adsorbent’s preparation and the single mechanism. Research into adsorption-photocatalytic synergistic systems is even more challenging. This is because it involves multiple aspects, including interface coupling, control of microstructure, and electron transport. However, relevant research remains insufficient. Based on current publications, relatively little attention has been given to adsorption-photocatalytic synergistic systems. This has held back innovation in high-performance composites to some extent. Over the past decade, research papers focusing on adsorption–photocatalysis have shown an overall steady upward trend. It peaked at around 175 publications in 2024 (Figure 1c). This suggests that research into the material has reached a plateau. There is limited scope for innovation in fundamental areas. The focus of the research shifted to the level of core mechanisms. The number of review articles has also shown a similar upward trend. Yet, there are no systematic reviews that examine regulatory mechanisms. This highlights the significance of the research in the present review. In 2025, the number of publications in this field declined. This indicates that the development of high-performance synergistic composites is becoming more challenging. Figure 1d displays a statistical chart for the publication output of different pollutants. The figure shows that dyes have long been a subject of considerable research interest. This is because dyes are widely studied organic pollutants. As emerging pollutants, antibiotics have attracted the attention of scholars. The proportion of articles published rose from 2% to 26%. Although there is relatively little literature on PFASs and MPs as emerging pollutants, they have garnered some attention. Based on their degradation characteristics, ROS readily attack the conjugated structure of

dye molecules. By breaking the chain, mineralization can be achieved gradually. Moreover, antibiotics are highly chemically stable, strongly resistant to degradation, and persistently ecotoxic. The degradation of antibiotics is currently one of the key subjects of research in the field of aquatic environments. Most studies have focused merely on the degradation behavior of a single pollutant. The summary fails to highlight the differences in the removal behavior of these two types of pollutants. It is also the focus of this review.

Biochar alone has limited capacity for removing pollutants as a photocatalyst. However, biochar has become an important carbon-based support and co-catalyst [32]. Its abundant pore structure and considerable specific surface area provide favorable conditions for pollutant adsorption [33]. Combining biochar with TiO_2 increases the contact area between the pollutants and the photocatalyst. This shortens the migration path of the photogenerated electron–hole pairs. This enhances photocatalytic performance [32]. Furthermore, introducing biochar encourages the formation of oxygen and carbon vacancies, as well as lattice defects. These defect sites capture photogenerated electrons and prevent the recombination of photogenerated carriers. Simultaneously, the thermal stability of the calcined TiO_2 -biochar-based photocatalyst is enhanced. The bandgap can be narrowed, and the spectral response range broadened [34]. Consequently, the use of biochar as a carrier for TiO_2 has been extensively studied over the past decade. Many review articles have summarized the latest developments in this field (Table 1). Existing reviews have primarily focused on the sources of biochar, methods for preparing composite materials, synthesis parameters, pollutant removal performance, and synergistic mechanisms. However, the crucial role of morphological features in the removal of pollutants has not been systematically discussed. Previous reviews have not systematized the principles of regulating morphology by synthetic parameters. They also overlook the fact that the three typical forms (surface-adhered, pore-embedded, and interlayer-distributed) differ in terms of charge transport pathways. Furthermore, relevant reviews lack a framework that organizes the material around morphology as the central concept. Consequently, this review takes morphology as its central theme to address the research gaps. The mechanism by which preparation parameters influence morphology is analyzed in depth. The correlation is also discussed among surface morphology, surface functional groups, ROS, and interfacial charge transport. This will further highlight the important role of microstructure in photocatalytic systems. The specific objectives of this review are as follows:

- (1) To systematically investigate the influence of key preparation parameters for photocatalysts (e.g., in situ synthesis methods, carbon precursors, TiO_2 precursors, solvent types, preparation temperature, and time) on morphological characteristics and optical properties. This lays the groundwork for improving the performance of photocatalytic reactions.
- (2) To discuss the influence of morphological characteristics, surface functional groups, and the generation of reactive oxygen species on pollutant removal performance and to reveal the relationships among these factors.
- (3) To investigate the charge transfer mechanisms in the three typical morphologies of TiO_2 -BC-based photocatalysts (surface-adhered, pore-embedded, and interlayer-distributed).

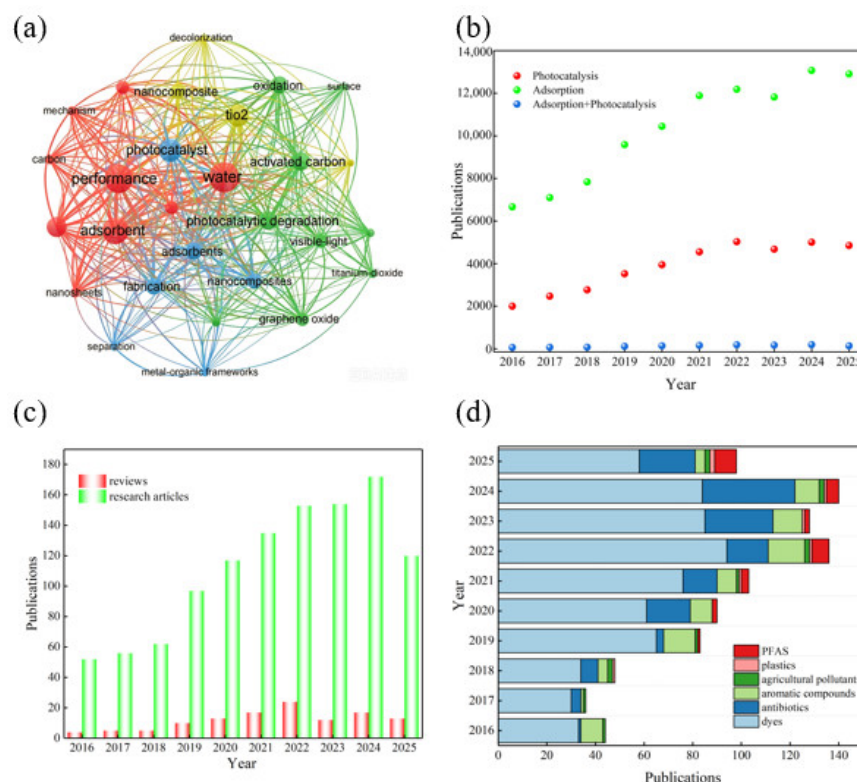


Figure 1. (a) Keyword co-occurrence map, (b) number of publications retrieved from the Web of Science database between 2016 and 2025 using the keywords “photocatalyst”, “adsorbent”, and “photocatalysis-adsorption”, (c) number of research and review articles published on adsorption-photocatalysis systems, and (d) distribution of publications by pollutant type.

Table 1. Comparative overview of pollutant degradation by TiO₂–biochar-based photocatalysts.

Contents of the Review	Limitations	Contributions of the Present Review	Ref.
Summary of TiO ₂ preparation methods, sources of biochar, and applications of TiO ₂ –biochar-based photocatalysts.	(1) The effects of different preparation methods on the morphological evolution of photocatalysts were not systematically examined in this review. (2) The relationship between morphological characteristics and photocatalytic performance was not addressed either.	The review focuses on morphological evolution, and it systematically outlines the function of synthesis parameters in regulating the morphology of TiO ₂ –biochar. Existing reviews have largely ignored the morphological evolution of TiO ₂ –biochar composites.	[31]
Overview of the preparation methods of biochar and TiO ₂ –biochar composites and advantages.	(1) This is simply a preliminary summary of the methods used to prepare composite materials. (2) The regulatory role of morphological evolution has not been systematically explored.	A systematic analysis is provided to examine the effects of preparation methods and conditions on the dispersion and pore structure.	[32]
Review of the synthesis methods, physico-chemical properties, and degradation performance of biochar/TiO ₂ .	The reviews did not investigate how various preparation parameters influence morphological characteristics, nor how morphological characteristics affect the degradation of pollutants.	A framework is established that correlates synthetic parameters with microstructural characteristics (e.g., specific surface area, pore structure and particle size), surface functional groups, ROS, and application.	[35]
Investigation of the properties, applications, and degradation mechanisms of TiO ₂ , ZnO, and Fe ₃ O ₄ /biochar systems.	A brief analysis of the effects of pre- and post-treatment on the morphology of TiO ₂ –biochar. However, the regulatory	A comparative analysis was carried out of the electronic transport pathways at the interface for three typical morphol-	[36]

	role of morphology in the mechanism of charge transfer at the interface has not been demonstrated.	ogies: surface-adhered, pore-embedded, and interlayer-distributed.	
Application of biochar-based catalysts and activity mechanism supported by DFT calculations.	The analysis of the microstructural characteristics of composite materials and their role is still insufficient.	A sufficient explanation of the regulatory effects of three typical morphologies on surface functional groups and the generation of ROS is presented.	[37]
Reviews of bottom-up and top-down approaches to the preparation methods, structural characteristics, applications, and reaction mechanisms.	Only the two factors of temperature and precursor are considered. The key parameters (e.g., solvent, pH, doping, and the Ti/C ratio) and their impact on morphological evolution were not discussed.	A full analysis is provided to explore the effect of different factors on the patterns of morphological control. These factors include the titanium source, carbon source, solvent, Ti:C ratio, pyrolysis process, and doping.	[38]
Review of sources of pollutants, TiO ₂ and biochar, as well as the applications of composite materials.	The influence of the microstructure of composite materials on the mechanism of action has not been clarified.	The pollutant removal behavior under the influence of the microstructure of composite materials was discussed by category.	[39]
(1) Summary of sources, physicochemical properties, preparation methods, and applications of biochar in composite materials. (2) Analysis of its advantages in terms of pollutant removal.	Ignoring the intrinsic link between microstructure and electronic transport at interfaces.	The relationship between morphology control, structural characteristics, and photocatalytic performance is systematically discussed.	[40]

2. Synthesis of TiO₂–Biochar-Based Catalysts

TiO₂–biochar-based photocatalysts are emerging dual-function materials that combine the synergistic effects of adsorption and photocatalytic degradation [41]. This photocatalyst can be prepared using non-in situ synthesis strategies, such as the hydrothermal method, impregnation–calcination, or microwave-assisted synthesis. In situ synthesis strategies include the sol–gel method, coprecipitation, and pyrolysis [32] (Figure 2). These methods differ in the carbon sources, titanium sources, solvents, and doping modifications used. Photocatalysts exhibit different morphologies and photoresponse characteristics. These factors are key to the photocatalyst’s performance in degrading dyes and antibiotics (Table 2). This review focuses on the effects of in situ synthesis on the microstructure of photocatalysts.

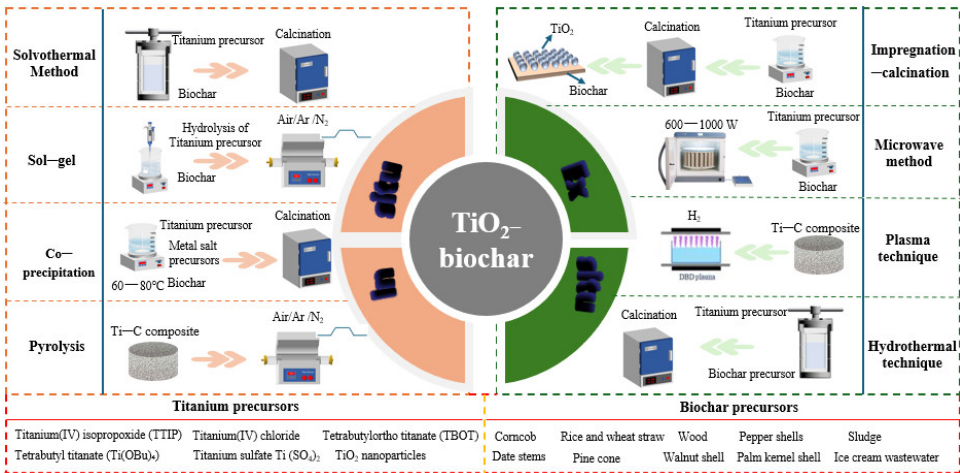


Figure 2. Preparation methods of TiO₂–biochar-based photocatalysts.

Table 2. In situ preparation of TiO₂-biochar photocatalysts: Parameters, morphology, and bandgap.

Titanium Source	Carbon Source	Method	Ti:C	Type of Solvent	Heating Conditions	Modification	Morphology	Band Gap (eV)	Ref.
Titanium isopropoxide	Coffee husk	Sol-gel	23% mTiO ₂ /77% mAC	Ethyl alcohol, acetic acid, deionized water	350 °C	CaCl ₂	TiO ₂ nanoparticles supported on amorphous AC; 700 m ² /g; Pore size = 4.80 nm	2.8	[42]
Tetrabutyl titanate	-	Sol-gel	TiO ₂ :PMBC = 3:1	Ethanol, HNO ₃ , deionized water	-	H ₃ PO ₄	Ordered vessel structure and irregular particles; 62.29 m ² /g	-	[43]
Butyl titanate	Coconut shell	Sol-gel	-	Ethanol, deionized water, acetic acid, polyethylene glycol	450 °C	-	Porous surface	-	[44]
Titanium (IV) isopropoxide	Macadamia nut shells	Sol-gel	2.87 mLTTIP:250 mg AC	Anhydrous isopropanol	500 °C	NaOH	Clusters of particles; 129 m ² /g; Pore size = 9.26 nm	3.04	[45]
Titanium isopropoxide	Peanut shells	Sol-gel	3.4 mL Ti(OiPr) ₄ :1 g PSB	Isopropanol, nitric acid	400 °C	PMS activation	Compressed and wrinkled morphology; 57.69 m ² /g; Pore size = 4.35 nm	2.59	[46]
Titanium isopropoxide	Corn cob	Sol-gel	2.5 g BC:20 mL TTIP	Ethanol, HCl	325 °C	HNO ₃	Tiny TiO ₂ granules were well dispersed on the biochar with little agglomeration	-	[47]
Tetrabutyl titanate	Corn cobs	Sol-gel	-	-	-	g-C ₃ N ₄	Layered and granular structures; 163.66 m ² /g	2.39	[48]
Tetrabutyl titanate	Corn cobs and straw	Sol-gel	2 g BC:100 mL tetrabutyl titanate	Ethanol, NH ₃ ·H ₂ O	550 °C	Al ₂ O ₃	Core-shell; 200.62 m ² /g; Pore size = 3.43 nm	-	[49]
Tetrabutyl titanate	Corn cobs	Sol-gel	1 g BC:10 mL tetrabutyl titanate	-	500 °C	g-C ₃ N ₄	Layered and granular structures	2.39	[50]
Titanium butoxide	Ramie bars	Sol-gel	2 g biomass	Ethanol, glacial acetic acid, and water	500 °C	-	TiO ₂ nanoparticles attached to BC; 10.55 m ² /g	-	[51]
Tetrabutyl titanate	Reed straw	Sol-gel	-	Absolute ethanol, acetic acid, ultra-pure water	500 °C	HCl	Distributions of TiO ₂ on the exterior and within the internal channels of pBC; 102.16 m ² /g; Pore size = 2.58 nm	-	[52]
Tetrabutyl titanate	Reed straw	Sol-gel	0.1 g pBC:20 mL tetrabutyl titanate	Absolute ethanol, acetic acid, ultra-pure water	300 °C	ZnO	Original vessels structure and cylindrical morphology	-	[53]

Titanium (IV) Iso-propoxide	Mangrove plants (twigs and roots)	Sol-gel	5 g BC: 20 mL TTIP	Ethanol, deionized water and acetic acid	500 °C	-	Aggregation of TiO ₂ nanoparticles	-	[54]
Titanium (IV) tetra-chloride	Giant reed	Sol-gel	10 wt% BC	Distilled water, ethanol, ammonia solution	600 °C	-	Rough and fractured texture	2.69	[55]
Titanium Isopropoxide	Prosopis juliflora	Sol-gel	TiO ₂ :BC = 3:7	Isopropanol and absolute ethanol	600 °C	N	Porous morphology with a consistent distribution of TiO ₂ nanoparticles within the BC surface.	2.23	[56]
Titanium (IV) isopropoxide	Wood	Sol-gel	-	Absolute ethanol, HCl, deionized water	500 °C	-	-	-	[57]
Titanium butoxide	Wood	Sol-gel	10% AC	Anhydrous alcohol, distilled water, glacial acetic acid	500 °C	Ni	Mesoporous structure; 222.89 m ² /g; Pore size = 5.41 nm	-	[58]
Titanium tetra-isopropoxide, Titanium oxysulfate	Salvinia molesta	Sol-gel and mechanical mixing	1:1	Isopropyl alcohol	350 °C	-	Bar; 8.55 m ² /g; Pore size = 32.55 nm	3.22 ± 0.002	[59]
Titanium butoxide	Furfural residue	Ultrasonic-assisted sol-gel treatment, solvothermal treatment, microwave-assisted heating	1 g AC: 52.5 mL titanium butoxide	Ethanol, deionized water	-	KOH	Porous structure; 241.70 m ² /g; Pore size = 4.43 nm	2.3	[60]
Titanium tetrakis (2-ethylhexoxide)	Wheat straw	Wet chemical precipitation-sol-gel	5 g C:17.8 g Titanium tetrakis (2-ethylhexoxide)	Ethanol, deionized water	-	La ³⁺ , Fe ³⁺	grape-cluster	2.9	[61]
Butyl titanate	Wheat straw	Sol-gel-calcination synthesis	-	Ethanol, PTFE solution	500 °C	N	Irregular, clustered morphology	-	[62]
Titanium isopropoxide	Textile sludge	Sol-gel and wet precipitation	0.1 g BC:0.376 mL titanium isopropoxide	-	450 °C	-	Denser TiO ₂ distributions with reduced particle sizes	2.1	[63]
Titanium (IV) Iso-propoxide	Macroalgae	Wet precipitation	-	Isopropanol	400 ± 1 °C	-	Spherical TiO ₂ nanoparticles uniformly distributed on the BC layer surface	2.60–2.70	[64]
Titanium tetra iso-propoxide	Manilkara zapota leaf	co-precipitation	2 g AC:1 M TTIP	Plant extract	800 °C	-	Equal spherical shape	2.73	[65]

Titanium (IV) Iso-propoxide	Corn cob	Wet precipitation	800 mg: 4 mL TTIP	Isopropyl alcohol	400 ± 5 °C	KOH	TiO ₂ distributed across rock-like AC	3.05	[66]
Tetrabutyl titanate	Walnut shell	Photodeposition	BC:Ti = 0.2:1	Ultrapure water, AgNO ₃ solution	500 °C	Ag	Small spherical particles	3.28	[67]
Titanium isopropoxide	Pepper shells	Deposition-precipitation	0.3 mL titanium isopropoxide:0.135 g BCP	-	400 °C	KOH, HAuCl ₄ , urea	Nanorod-like structure; 1287.6 m ² /g; Pore size = 2.08/2.60 nm	1.79	[68]
TiO ₂	Chitosan	Co-precipitation/gelation	5 g CS:2.5 g TiO ₂	Acetic acid	600 °C	ZnCl ₂	Loose mesoporous structure	3.03	[69]
Tetrabutyl titanate	Bagasse	hydrothermal, calcination and photo-deposition	-	Acetic acid	650 °C	Ag urea	Floating flower-like	2.73	[70]
Titanium (IV) iso-propoxide	Ice cream wastewater	Solvothermal	1 g HC: 1.853 mL TTIP	Absolute ethanol, distilled water	- 500 °C	-	Irregularly shaped and exhibit a wide size distribution; 216.49 m ² /g; Pore size = 7.83–10.73 nm	2.05	[71]
Titanium dioxide	Furniture	Pyrolysis	10% <i>w/w</i> TiO ₂	Isopropyl alcohol	500 °C	-	TiO ₂ anchored on the cellulose-like surface of BC	3.66	[72]
Anatase TiO ₂	Cornstalk	Pyrolysis	0.75 Ti/1 AC	-	500 °C	-	Particles distributed on carbon lump; 4.37 m ² /g; Pore size = 35.96 nm	2.77	[73]
Tetrabutyl titanate	Sludge	Co-pyrolysis	9 g (BC): 1 g g-C ₃ N ₄ /La-TiO ₂	Deionized water	500 °C	g-C ₃ N ₄ La	Lamellar BC with uniformly dispersed g-C ₃ N ₄ /La-TiO ₂ (A); 172.99 m ² /g; Pore size = 15.53 nm	-	[74]
Titanium isopropoxide	Lignocellulosic biomass	Sol-gel	1:2–1:5	Ethanol/Isopropanol	450–550 °C	N, g-C ₃ N ₄	Uniformly dispersed nanoparticles with porous structure	2.2–3.0	General trend

2.1. Titanium Precursors

As shown in Table 2, the microstructure of TiO_2 is closely linked to the physicochemical properties of the titanium source. The molecular structure, ligand type, and electronic properties of the titanium source play a critical role in the hydrolysis process of the titanium source. These properties further regulate the crystal growth, surface morphology, electronic structure, and photocatalytic activity of TiO_2 . This section primarily discusses the influence of the physicochemical properties of the titanium source. These properties influence the crystal structure, microstructure, and photocatalytic performance. Typical titanium sources include titanium isopropoxide, tetrabutyl titanate, and titanium butyrate. In these titanates, the alkyl groups act as electron-donating groups. This reduces the positive charge on the titanium atom, thereby inhibiting hydrolysis. The electron-withdrawing groups increase the positive charge on the Ti atom. This makes the Ti atom more prone to forming bonds with H_2O . As a result, the hydrolysis reaction is promoted [75].

The hydrolysis rate determines the crystal growth of TiO_2 [76]. In the hydrolysis stage, the number of Ti-OH groups gradually increases. First, titanium-oxygen dimers are formed via ololation. Subsequently, the Ti-O-Ti network is established through oxolation. Different crystal structures are formed by the gel after calcination. The rapid rate of hydrolysis tends to result in the formation of fine, agglomerated crystals. On the other hand, slow hydrolysis leads to the formation of larger crystals, better dispersion, and higher crystallinity [77,78]. Controlling the hydrolysis rate of the titanium source enables uniform dispersion of TiO_2 particles on the surface of biochar. Crystal growth directly affects the microstructure of TiO_2 . Anisotropic growth exposes crystal faces with high surface energy. TiO_2 nanorods or nanowires can more easily form. Isotropic growth tends to result in spherical TiO_2 particles, whereas rapid growth leads to particle agglomeration [79]. The transport pathways of photo-generated charge carriers, bandgap width, and photocatalytic performance are influenced by TiO_2 microstructures. As Table 2 shows, the TiO_2 prepared from titanium alcoholates exists mainly as near-spherical nanoparticles or attached to the surface of biochar. However, particle agglomeration is still a problem. Further confirmation that the prepared TiO_2 exhibits a bimodal particle size distribution, with a mean of 190–220 nm, is obtained by DLS. This indicates moderate agglomeration (Figure 3a). Taking titanium isopropoxide (TTIP) as an example, reference [63] reported that the crystal structure of pure TiO_2 is of the rutile type, with a bandgap of 3.1 eV. TiO_2 particles prepared using titanium tetrachloride (TiCl_4) as a precursor exhibit uniform particle sizes and smooth surfaces with no discernible texture (Figure 3b). The XRD pattern exhibited distinct rutile-type TiO_2 features, with a bandgap of 3.03 eV [55]. When titanium sulfate (TiOSO_4) is used as the titanium source, its specific surface area ($166.25 \text{ m}^2/\text{g}$) is higher than that of TiO_2 produced using TTIP as the precursor ($56.37 \text{ m}^2/\text{g}$). However, it is difficult to clearly observe the morphological features of TiO_2 particles in SEM analysis. No significant characteristic peaks were observed in the XRD pattern, making it difficult to determine the crystal type [59] (Figure 3c). This may indicate that the specific surface area is inversely proportional to crystallinity. The crystal structure of commercial TiO_2 consists of 80% anatase and 20% rutile. Its bandgap is approximately 3.2 eV, and its specific surface area is approximately $52 \text{ m}^2/\text{g}$. The commercial TiO_2 often appears as irregular aggregates [69] (Figure 3d). Studies have shown that TiO_2 prepared using titanate as the titanium source exhibits more distinct morphology, a greater specific surface area, and a narrower bandgap than commercial TiO_2 . This significantly improves the material's ability to absorb sunlight, thereby boosting its photocatalytic activity [80]. The choice of titanium source affects the crystalline phase, crystallinity, microstructure, and active sites of TiO_2 , among other factors. These microstructures are mutually coupled and influenced. Following careful modulation, the photocatalytic performance of the material is enhanced in a synergistic approach. Different preparation methods and reagents are employed in exist-

ing studies. There is a lack of uniform, quantitative evaluation criteria for the microstructural characteristics of catalysts. In the future, a quantitative relationship between preparation conditions and microstructural characteristics should be established. The aim is to provide mechanistic guidance for creating high-performance photocatalysts.

In addition to these traditional commercial titanium sources, developing green titanium sources is also a key priority at present. Green titanium sources have potential applications in the remediation of aquatic environments. The industrial waste product ferrotitanium (FeTiO_3) is chemically stable and environmentally safe and exhibits oxidative activity [81,82]. FeTiO_3 consists of Fe^{2+} and Ti^{4+} ions and has been used for the photocatalytic degradation of pollutants [83]. Mostafa [82] synthesized flower-like nanostructured $\text{FeTiO}_3/\text{SiO}_2$ using natural ilmenite and recycled glass as raw materials. The removal of heavy metal Cr(VI) and 4-nitrophenol (4-NP) was achieved via synergistic adsorption-photocatalysis. This photocatalyst exhibited adsorption capacities of 277.0 mg/g and 173.4 mg/g for Cr(VI) and 4-NP, respectively, and achieved 100% photocatalytic degradation within 2 h. Li [84] prepared silicon-based TiO_2 photocatalysts containing rutile and anatase phases derived from titanium blast furnace slag. After 2 h of ultraviolet irradiation, the degradation rates of MB and MO reached 97.37% and 92.15%, respectively. Perovskiy [85] utilized leucoxene as a titanium source to prepare TiO_2 mixed phases (rutile and anatase) with a purity of approximately 80%. Natural minerals and industrial solid waste can be used as sources of titanium, which reduces the cost of production and enables the waste to be recycled. However, these materials usually contain impurities that reduce the purity of the TiO_2 . These impurities are incorporated into the TiO_2 lattice. They hasten the recombination of photogenerated charge carriers, thus degrading photocatalytic performance.

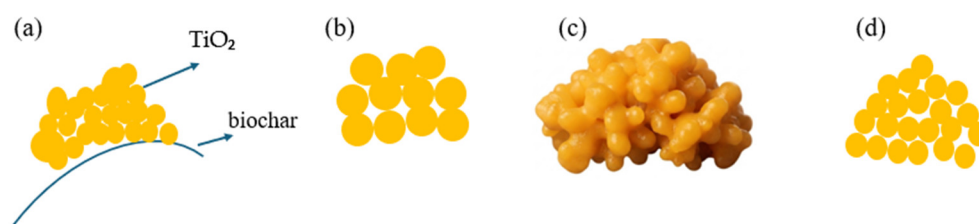


Figure 3. Morphology and XRD patterns of TiO_2 : (a) TTIP, (b) TiCl_4 , (c) TiOSO_4 , (d) commercial TiO_2 .

2.2. Biochar Precursors

The performance of biochar-based photocatalytic systems is critically influenced by the elemental and chemical composition of biomass. The three components of plant biomass are lignin, hemicellulose, and cellulose [86]. The pyrolysis of these materials can produce biochar with various carbon structures [87]. Compared with hemicellulose and cellulose, lignin typically decomposes over a wider temperature range. The decomposition promotes the formation of porous or hierarchical carbon frameworks [88]. The oxygen-containing functional groups on the surface of biochar predominantly depend on the chemical composition of the biomass. These functional groups not only boost the ability to adsorb pollutants but also provide anchoring sites for TiO_2 . Thus, an interfacial contact is formed, which promotes charge transport [32,64]. In summary, variations in the biomass feedstock result in differences in pore structure, surface chemical characteristics, and the nature of the interface. These factors collectively regulate the adsorption–photocatalytic synergy. Ultimately, this synergy determines the photocatalytic degradation performance of the composite material.

Table 2 lists the sources of biochar. These sources can be broadly categorized into six types: wood, crop straw, agricultural and forestry husks, fruit and vegetable waste, aquatic algae, and industrial sludge. Among these, straw and fruit husks are the most

widely used. The biomass source has substantial implications for the microstructure, specific surface area, active sites, and material bandgap of biochar [89]. The transformation of biomass waste into biochar allows for the effective reuse of waste while reducing environmental pressures [90]. According to relevant reports, biochar produced from cellulose-rich crops such as wheat straw and maize cobs has a more developed porous structure than biochar derived from sewage sludge. Biochar also demonstrates superior performance when used as a photocatalyst support [91–93]. This suggests that the composition of the carbon source determines the biochar's pore structure and loading capacity. Yang [61] used wheat straw as a carbon source to prepare a dual photocatalyst (biochar/La-FeO₃/TiO₂) with particle diameters ranging from 188 to 290 nm. The presence of biochar provides effective support sites for the photocatalytically active components (Figure 4a). Kim [47] prepared maize cob-derived biochar with a microporous layered framework. The unique layered pore structure effectively suppressed the aggregation of TiO₂ nanoparticles (Figure 4b). In addition, waste sludge is also used to produce layered biochar. Masud and Jiang [63,74] developed highly porous biochar with a layered morphology using sludge as the carbon source. The layered porous structure provided a stable support framework for the photocatalyst particles, facilitating the adsorption and degradation of pollutants (Figure 4c). The shells of agricultural and forestry products are rich in lignin. Nevertheless, the resulting biochar typically exhibits a granular or honeycomb-like porous structure due to its inherent hardness and irregular morphology. The pore structures of biochar produced from fruit peel waste are less developed compared to those of biochar produced from crop straw. However, the biochar exhibits outstanding mechanical stability and offers superior load-bearing capacity. Zhang [44] prepared a honeycomb-structured, mesoporous activated carbon from coconut shells. TiO₂ nanoparticles were successfully immobilized on the surface of the carbon and embedded them within the honeycomb pores (Figure 4d). Martins [45] found that biomass-derived activated carbon prepared from macadamia nut shells not only serves as support for TiO₂ nanoparticles but also acts as a dopant in TiO₂ composites. This modification resulted in granulated porous structures with an average pore diameter of 9.26 nm (Figure 4e). Zouggari [46] prepared biochar with a network-like porous structure from peanut shells. This uniform distribution of TiO₂ within the micropores and mesopores of the carbon matrix is shown in Figure 4f. This embedded structure serves three main purposes: (1) suppressing TiO₂ agglomeration; (2) minimizing the loss of photocatalytically active components; and (3) inhibiting the recombination of photogenerated charge carriers. Shan [67] produced lump biochar with a surface area of 66.06 m²/g and pore sizes of 10.01 nm, using walnut shells as the carbon source (Figure 4g). The resulting biochar framework provided support for silver and TiO₂. Liu [68] produced biochar from pepper shells with a specific surface area of 3.16 m²/g, exhibiting a layered, wrinkled morphology (Figure 4h). Following KOH activation, the specific surface area increased to 2665.63 m²/g. The microstructure changed to a ridge-like structure with a hierarchical pore system comprising crack-like and mesopores (Figure 4i). Biochar generated from medium-density fiberboards also exhibited a layered, crinkled structure. It had an overall rod-like morphology [44]. A similar morphology was also observed in biochar prepared by *Salvinia molesta* [59]. The large specific surface area of this layered, wrinkled biochar makes it suitable for adsorption of pollutants. TiO₂ can also be loaded due to the abundance of sites and space that is offered by biochar. Porous and layered structures promote uniform dispersion of TiO₂ nanoparticles and electron transport. However, these microstructures depend on the preparation conditions. The relationship between microstructure and photocatalytic performance is difficult to describe in isolation. Therefore, it is important to reveal the intrinsic relationship between the evolution of microstructure, charge transport, and photocatalytic performance during the pyrolysis process.

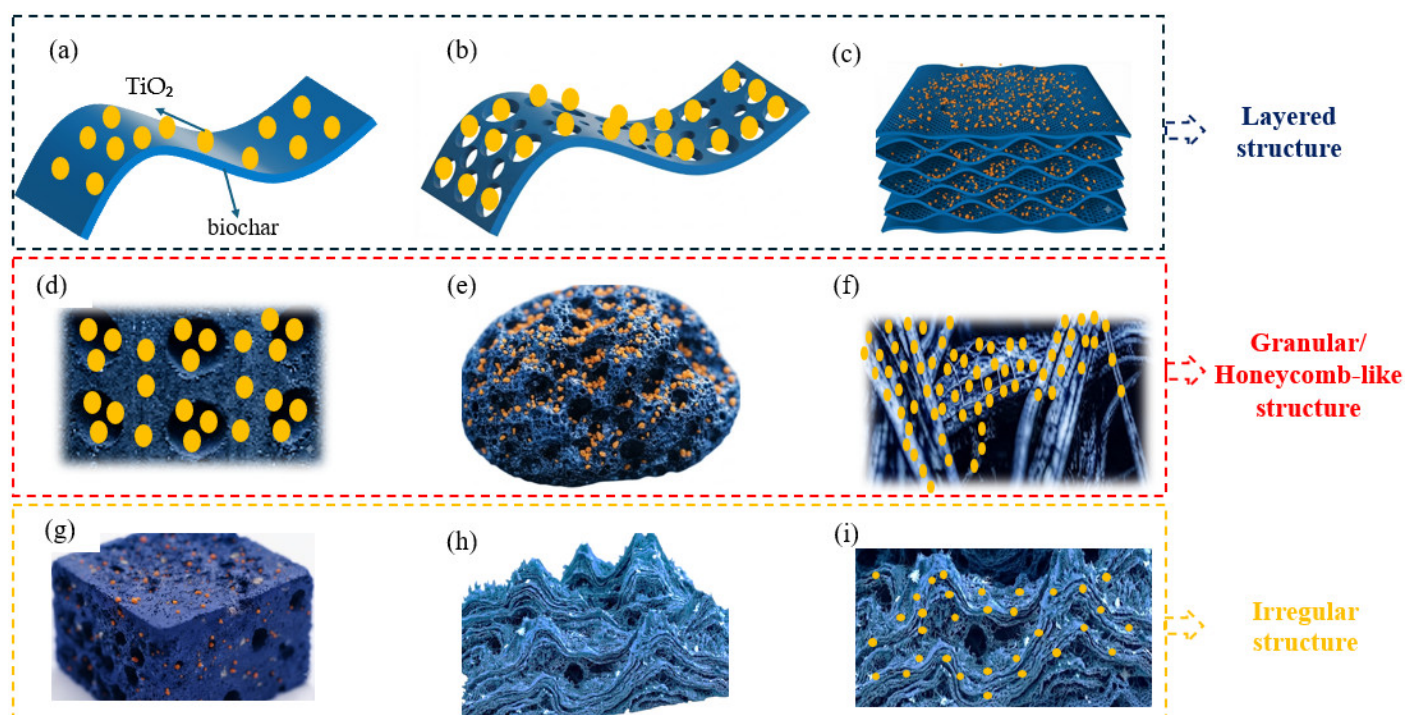


Figure 4. Morphology of biochar: (a) wheat straw, (b) corn cobs, (c) textile sludge, (d) coconut shell, (e) macadamia nut shells, (f) peanut shells, (g) walnut shell, (h,i) pepper shells.

In summary, biochar produced from cellulose-based materials typically exhibits a layered, porous structure. Biochar produced from lignin-based materials tends to form a high-strength, honeycomb-like framework. This indicates that biochar precursors play a significant role in the microstructure of biochar. Therefore, to regulate the microstructure and optimize photocatalytic performance, the carbon source should be selected carefully.

2.3. Titanium–Carbon Mass Ratio

The removal of pollutants is more efficient when the titanium-to-carbon ratio is optimized. Optimizing the ratio is crucial for enhancing photocatalyst performance. If the mass ratio is too low, the biochar may shield TiO_2 , preventing light from reaching it and covering some active sites [94]. Xie [95] prepared TiO_2/BC photocatalysts with titanium-to-carbon mass ratios of 1:4, 1:2, and 3:4. The results demonstrated that the efficacy of wastewater purification diminished as the titanium-to-carbon mass ratio decreased. Figures 5a and 5b show the results for titanium-to-carbon mass ratios of 1:4 and 1:2, respectively. As illustrated in Figure 5a,b, when the titanium-to-carbon mass ratio is 1:4 rather than 1:2, TiO_2 becomes embedded in the biochar pores and is more effectively shielded. Ashebir [56] synthesized the N- TiO_2/BC photocatalyst with a titanium-to-carbon mass ratio of 3:7. Figure 5c indicates that TiO_2 was uniformly dispersed on the BC surface. These conditions favored the light-induced excitation of TiO_2 and the suppression of the recombination of photo-generated carriers. The UV-vis DRS results revealed that the porous structure of biochar provided more abundant active sites for adsorbing pollutants and enhanced light penetration. When the mass ratio is too high, it prevents the full utilization of biochar's adsorption properties and exacerbates TiO_2 agglomeration. Lu [96] developed photocatalysts with titanium-to-carbon mass ratios of 1:0.1, 1:0.2, 1:0.5, 1:0.8, and 1:1. The highest degradation rate for the anionic dye methyl orange was observed at a ratio of 1:0.2. Figures 5d and 5e show photocatalysts with titanium-to-carbon mass ratios of 1:0.2 and 1:0.5, respectively. Compared with a Ti/C mass ratio of 1:0.2, TiO_2 particle agglomeration is more pronounced at a Ti/C mass ratio of 1:0.5. The interface contact area between the two phases is reduced,

and the charge transport pathways are restricted. The rate of recombination of photo-generated charge carriers rises, which in turn reduces photocatalytic activity.

The above analysis shows that the efficiency of photocatalytic degradation is influenced by two factors. The first is the dispersion state of TiO_2 . The second is the efficiency of photogenerated carrier separation at the TiO_2 /biochar interface. At an ideal Ti/C ratio, TiO_2 nanoparticles are evenly distributed on the surface of biochar, leading to beneficial interfacial interactions. Conductive biochar can act as both an electron capture site and an electron transport channel. This promotes the separation and migration of photogenerated charge carriers within TiO_2 [32]. The separated electrons and holes subsequently participate in redox reactions, generating $\bullet\text{O}_2^-$ and $\bullet\text{OH}$. As a result, the photocatalytic degradation performance is enhanced [97].

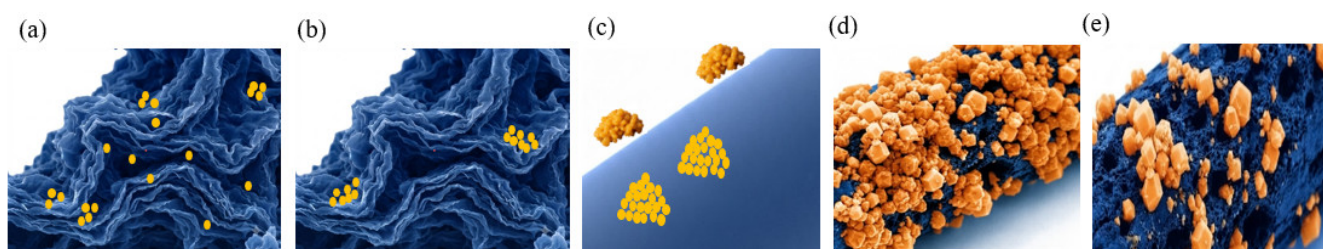


Figure 5. Morphology at different titanium-to-carbon ratios: (a,b) 1:4 and 1:2, (c) 3:7, (d,e) 1:0.2 and 1:0.5.

2.4. Preparation Methods and Solvents

Table 2 summarizes the in situ synthesis strategies for TiO_2 -biochar-based photocatalysts. These strategies have been used in the photodegradation of dyes and antibiotics over the past decade. As the table shows, the preparation methods include sol-gel, coprecipitation/precipitation, pyrolysis, solvothermal synthesis, and photodeposition. The TiO_2 prepared using various methods exhibits differences. These differences are reflected in crystallinity, particle size, microstructure, interfacial contact between the two phases, and photocatalytic performance [98].

The sol-gel method uses titanium precursors to prepare TiO_2 via a controlled hydrolysis reaction. During the synthesis process, the pH is typically adjusted with an acid solution, such as HNO_3 or HCl . Different particle sizes and crystal morphologies of TiO_2 can be obtained by varying the synthesis conditions [98]. On the one hand, acid regulation inhibits the hydrolysis rate of the titanium precursor, enabling the uniform growth of TiO_2 . On the other hand, mild acidification is used to increase the oxygen-containing functional groups on the surface of the biochar. TiO_2 prepared using the sol-gel method exhibits favorable characteristics such as low impurity content, small particle size, and high crystallinity. Therefore, the sol-gel method is the most widely used method. A long preparation cycle and the need for subsequent calcination to improve crystallinity are also limitations of this method [99]. Biochar, or biomass, is added to the titanium precursor solution when preparing TiO_2 -biochar photocatalysts using the sol-gel method. The pH is adjusted to an appropriate level using acid, allowing it to gradually form a gel. The composite material was obtained after aging, washing, drying, and calcination [100]. The morphology of photocatalysts can be modified through this method to yield various structures, such as particle-supported, porous, and core-shell forms. Portela [42] applied ethanol, acetic acid, and deionized water as solvents for tetrabutyl titanate. The process slows down the rapid hydrolysis of the titanium precursor while also preventing the agglomeration of TiO_2 . Activated carbon made from coffee grounds was then added and heated. The results showed that TiO_2 particles had been loaded onto the surface of the activated carbon (Figure 6a). This photocatalyst had a surface area of around $700 \text{ m}^2/\text{g}$ and a bandgap of 2.8 eV. The selection of solvents can facilitate the dispersion of TiO_2 particles.

Solvent selection can also regulate the specific surface area and bandgap structure. In reference [45], isopropanol and water were used as solvents, and activated carbon was added following stirring. After gelation, drying, and calcination, the resulting composite material had a specific surface area of 129 m²/g. TiO₂ exhibited a clustered morphology (Figure 4e). The comparisons demonstrated that the type of solvent and the synthesis procedure influenced the microstructure of TiO₂. Wang [49] constructed a TiO₂/Al₂O₃@BC photocatalyst with a core-shell structure using ethanol as the solvent for tetrabutyl titanate. The addition of ammonia water during mixing accelerated hydrolysis and condensation reactions. The addition of ammonia water also enhances interfacial bonding by adjusting the pH (Figure 6b). The introduction of ammonia solution enabled the formation of a hierarchical structure. This laid a solid foundation for the separation of electron-hole pairs. Ashebir [56] adopted isopropanol and anhydrous ethanol as solvents for TTIP. A mesoporous composite material was prepared by grinding and calcination (Figure 5c).

The sol-gel method can be combined with techniques such as ultrasonication, hydrothermal treatment, microwave irradiation, and mechanical agitation. These combined approaches can significantly enhance photocatalytic performance. The multi-method approach combines physical interactions to closely link the dispersion of TiO₂ nanoparticles with controlling their crystalline phase. This strategy is an effective method of addressing the tendency of particles to agglomerate during the conventional sol-gel process. Ao [60] successfully synthesized TiO₂@AC using a combination of the sol-gel and solvothermal methods with microwave-assisted technology. The sol-gel method used ethanol, acetic acid, and water. The solvothermal method used ethanol and water. TiO₂ was distributed on the surface of the porous biochar structure. Nevertheless, when composite materials were prepared using a sol-gel method combined with microwave assistance, the loading of TiO₂ nanoparticles onto the biochar surface was relatively low (Figure 6c). In contrast, the photocatalyst prepared using the solvothermal method combined with microwave-assisted synthesis exhibited a uniform distribution of TiO₂. TiO₂ was uniformly distributed both on the surface and within the pores of the biochar (Figure 6d). The structure provided a basis for synergistic removal of antibiotics via adsorption and photocatalysis. It has been indicated that the physical interactions and the type of solvent are directly linked to the form of TiO₂ loading and the degree of dispersion on the biochar.

The coprecipitation/precipitation methodologies are low-cost and efficient in producing spherical or granular TiO₂ nanomaterials, with a more uniform particle size distribution. However, this method can be affected by issues such as uneven reactant precipitation rates, product contamination, and the production of large volumes of waste liquid [101]. Moreover, the fact that certain materials cannot be obtained using this method means that the scope of application is more limited than other methods. Reference [61] reported the precipitation method to produce metal-ion-modified biochar. This involved stirring a mixture of biomass precursors and La³⁺ and Fe³⁺ solution under alkaline conditions, followed by calcination. The photocatalyst was formed by mixing the modified biochar with the titanium precursor. Figure 4a shows the 'grape-like' morphology and uniform particle size of TiO₂. Its bandgap width was 2.9 eV. Parvathiraja [65] prepared hemispherical and spherical TiO₂ nanoparticles by mixing titanium with the plant extract (Figure 6e). The presence of plant molecules may induce TiO₂ to form hemispherical nanoparticles. When combined with activated carbon, the composite formed a uniform spherical structure with regularly spaced particles 22 nm in diameter (Figure 6f). Its bandgap was reduced from 3.11 eV to 2.73 eV. Thus, biochar not only serves as a support for TiO₂ but also modulates its microstructure and bandgap through interfacial interactions, thereby broadening the light response range.

Pyrolysis involves mixing TiO₂ directly with biomass and subjecting the mixture to high-temperature treatment. It offers convenient operability and scalability. The resulting

photocatalysts usually have a granular, carbon-based structure consisting of layered blocks. Moreover, variations in microstructural morphology are closely linked to the pyrolysis atmosphere and temperature. Furthermore, under high-temperature conditions, the crystalline phase of TiO_2 undergoes a significant transformation. This makes particle agglomeration likely. The microstructure of composite materials can be controlled by the pyrolysis method. However, the strength of the interfacial bond and the efficiency of electron transport are inferior to those of the sol-gel and solvothermal methods. Jiang [73] applied a pyrolysis method to prepare a photocatalyst consisting of TiO_2 nanoparticles attached to the surface of bulk biochar (Figure 6g). With increasing titanium-to-carbon mass ratio, the particle adhesion effect became more pronounced. Following pyrolysis treatment, the bandgap of TiO_2/BC (2.77 eV) was narrower than that of pure TiO_2 (3.4 eV). The report [72] described preparing TiO_2 nanoparticles on the surface of cellulose biochar using a pyrolysis method (Figure 6h). These particles had an optical bandgap of 3.66 eV. This suggests that the source and microstructure of the biochar affect the bandgap of the composite material.

The solvothermal method is a process that involves dissolving and reacting sparingly soluble substances. It takes place within a closed system [102]. The system is subject to specific conditions of temperature, pressure, and time [103]. The composite materials prepared using this method typically exhibit superior crystallinity. Nevertheless, this method is expensive, hard to control precisely, and not suitable for materials sensitive to high temperatures and pressures [101]. Naribi [71] successfully synthesized TiO_2 with non-uniform particle sizes and irregular shapes using a solvothermal method. The TiO_2 particles uniformly covered the solvothermally treated carbon surface (Figure 6i,j). This structure enhanced both the separation of photogenerated carriers and the efficiency of charge transport. Liu [104] employed a solvothermal method to prepare C, N-doped TiO_2/C photocatalysts. The carbon source and titanium source were reacted under the solvothermal method at 180 °C for 12 h. Flower-like TiO_2 was obtained following calcination, supported on the biochar surface (Figure 6k,l). These results demonstrate that the solvothermal method is more effective for synthesizing photocatalysts with specific morphologies. This approach increases the number of active sites and provides a convenient method for preparing multi-level morphological composites.

The photodeposition method is better suited to synthesizing TiO_2/BC photocatalysts with noble metal particles. The photodeposition method involves loading noble metal nanoparticles (e.g., Ag, Au, and Pt) onto the surface of TiO_2/BC photocatalysts. A Schottky junction is formed at the interface between the noble metal nanoparticles and TiO_2 . The Schottky junction effectively captures photogenerated electrons [105,106]. This method is more suitable for optimizing the electronic structure at interfaces than controlling microstructure.

In general, the sol-gel method and the solvothermal method are better for promoting TiO_2 crystals, controlling the microstructure, and improving interfacial contact. In contrast, the pyrolysis and precipitation methods are limited in terms of their applications and structural control. Therefore, when selecting a method for preparing composite materials, a range of factors must be considered. These include interfacial bonding, the influence of microstructure on electron transport pathways, and cost.

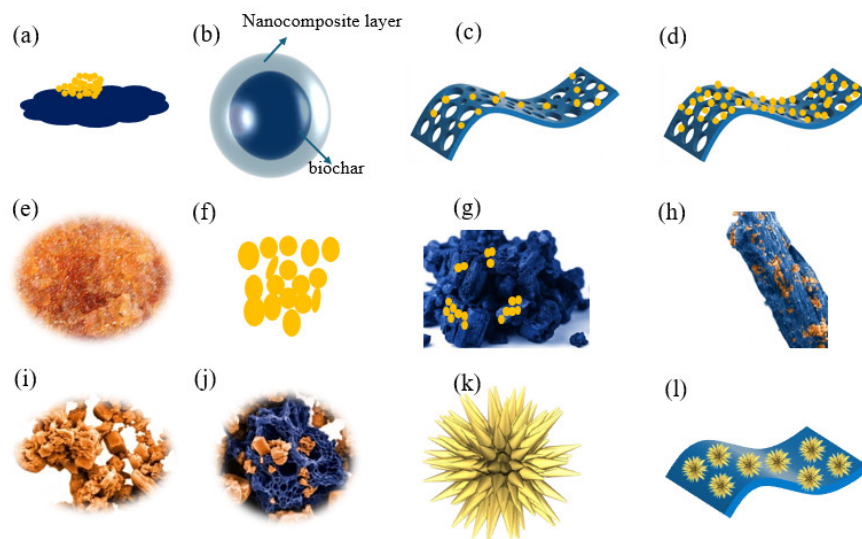


Figure 6. The effect of synthesis methods and solvents on morphology: (a) Sol-gel, ethyl alcohol, acetic acid, deionized water, (b) Sol-gel, ethanol, $\text{NH}_3 \cdot \text{H}_2\text{O}$, (c,d) Ultrasonic-assisted sol-gel treatment, solvothermal treatment, microwave-assisted heating, ethanol, and deionized water, (e,f) Coprecipitation, plant extract. (g) Pyrolysis. (h) Pyrolysis, isopropyl alcohol. (i,j) Solvothermal, absolute ethanol, distilled water. (k,l) Hydrothermal, calcination and photo-deposition, acetic acid.

2.5. Heat Treatment Conditions

For TiO_2 -biochar-based photocatalysts, the pyrolysis temperature is one of the key factors that influence their crystal structure and microstructure [107]. Typically, the crystallinity of TiO_2 increases, the grain size grows, and the number of photogenerated charge carriers rises when the pyrolysis temperature rises ($<450^\circ\text{C}$). These changes improve the efficiency of electron transport [108]. Research has demonstrated that after 4 h of pyrolysis of walnut shell biochar/ TiO_2 at 400°C , TiO_2 is uniformly distributed across the biochar's reticulated porous structure [46] (Figure 4g). Pyrolysis was carried out with TiO_2 precursors using macroalgae as a carbon source at 350°C . The composite material was prepared by dispersing TiO_2 on the surface of a rod-shaped carbon matrix [59]. It can therefore be concluded that, at temperatures below 450°C , TiO_2 exhibits better dispersion and crystallinity. Biochar can retain a more intact microstructure. The photocatalytic properties of TiO_2 depend on its crystalline phase. However, it is subject to a combination of factors, including calcination temperature, time, and preparation methods. Anatase TiO_2 exhibits superior performance in photocatalytic degradation. The reason for this is its higher conduction band potential and lower photogenerated carrier recombination rate. At temperatures ranging from 400 to 600°C , the biochar's surface area and pore structure show enhancement compared to those at 300 to 400°C . At this stage, the anatase phase of TiO_2 gradually transforms into the rutile phase. The phase transformation of TiO_2 is suppressed within the composite material by biochar. The TiO_2 still retains anatase characteristics. Corn cob biochar can be taken as an example. When calcined at 325°C , anatase TiO_2 particles are uniformly dispersed across the biochar surface. Fewer particles were embedded within its pores [47] (Figure 7a). Under conditions of 500°C during the process of pyrolysis, the surface of the microporous biochar was covered with granular TiO_2 . In this case, the TiO_2 retained the anatase crystal structure [48] (Figure 7b). As the temperature increased to 550°C , the composite displayed a morphology in which anatase TiO_2 nanoparticles occupied the pores of the biochar. Its specific surface area was $200.62\text{ m}^2/\text{g}$, approximately 20 times greater than that of pure biochar prepared under the same pyrolysis conditions (Figure 7c). The typical particle size of composite materials was 5 nm [49]. When giant reed stems were pyrolyzed at 600°C , their specific surface area was $147.1\text{ m}^2/\text{g}$. It

contained both the anatase and rutile crystal phases. With increasing temperature, slight agglomeration of the TiO_2 particles in the composite material was observed [55] (Figure 7d). Although rutile TiO_2 has a wider band gap, higher crystallinity, and better thermal stability than anatase-type titanium dioxide, it has a faster electron–hole pair recombination rate, resulting in poorer photocatalytic performance. The composite system of rutile and anatase TiO_2 has the capacity to generate a heterojunction structure. Their interface speeds up the separation of photo-generated charge carriers, boosting the performance of photocatalytic degradation. In TiO_2 -biochar-based composites, the biochar serves not only as a dispersing medium for the TiO_2 but also contributes significantly to regulating the crystalline phase. At temperatures of 800 °C, TiO_2 still exhibited the characteristics of the anatase phase. TiO_2 particles appear as dispersed spherical particles attached to the surface of the biochar. This further demonstrates that biochar can simultaneously moderate the evolution of TiO_2 crystal phases and the electronic structure at the interface.

In summary, the pyrolysis temperature affects both the degree of biochar carbonization and the phase transformation, grain growth, and crystallinity of TiO_2 . The synergistic effect at the interface between TiO_2 and biochar is enhanced within the appropriate temperature range.

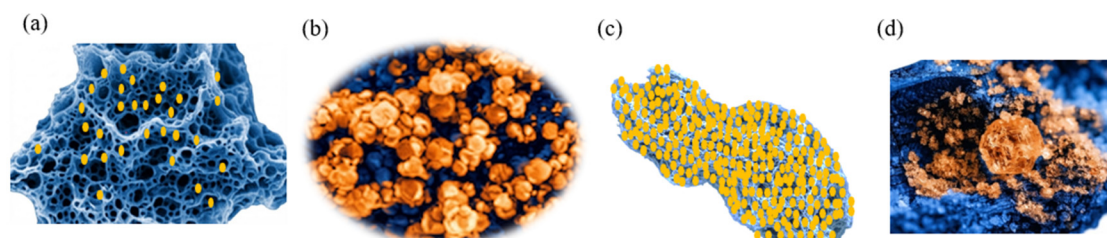


Figure 7. The effect of pyrolysis temperature on morphology: (a) 325 °C, (b) 500 °C, (c) 550 °C, (d) 600 °C.

2.6. Doping Modification

Several issues have been identified with TiO_2 -biochar-based photocatalysts. These include TiO_2 's tendency to sinter and agglomerate, a low specific surface area, poor utilization of visible light, and a lack of active sites. These factors collectively affect the capacity to treat pollutants. Thus, doping and modifying photocatalysts have become an effective means of enhancing their performance. Common strategies for modifying materials include doping with metals and non-metals, as well as acid and base modification. Element doping inhibits the agglomeration of TiO_2 . These elements include metals and non-metals. This results in the formation of relatively uniformly dispersed nanoparticles, thereby enriching the biochar's pore structure. According to Ref. [56], the use of urea as a nitrogen source for doping TiO_2 -biochar photocatalysts resulted in an excellent porous structure (Figure 5c). The introduction of C and N into the TiO_2 lattice has effectively reduced the material's bandgap width. This has further broadened the material's response range to visible light, thereby creating favorable conditions for enhancing its photocatalytic performance. The addition of metal ions helps to create heterojunctions and controls the structure at the interface between the active components and biochar (e.g., grape-like structures). This process promotes the separation and transport of photo-generated electron–hole pairs [58,61]. Incorporating noble metal (Au, Ag) enhances the local electromagnetic field on the composite material's surface and broadens the range of light absorption. These are typically loaded onto the surface of TiO_2 -biochar in the form of metal nanoparticles [29,67,68,109]. The bandgap of TiO_2 -biochar-based photocatalysts can be engineered to range from 1.79 to 3.28 eV by doping with metallic or non-metallic elements. The composite's morphology, interfacial bonding, and electronic energy levels are effectively modulated by doping. This strategy offers a vital method of improving visible-light responsivity

and encouraging the separation of photo-generated electron–hole pairs. Incorporating semiconductors (e.g., g-C₃N₄, ZnO, and Al₂O₃) into TiO₂-biocarbon systems facilitates the formation of type II, Z-type, and S-type heterojunctions. Such heterojunctions not only facilitate the separation of photogenerated carriers, but also enable the active components to couple synergistically [110]. Thanks to its morphology and robust photoresponsive properties, g-C₃N₄ promotes the formation of layered particulate composite structures, thereby improving interfacial interactions [48]. ZnO functions as both a carrier and a catalyst, promoting the dispersion of TiO₂ particles. Al₂O₃ acts as an interfacial bridge, further enhancing the interfacial bonding between TiO₂ and biochar. The presence of semiconductor components regulates the bandgap to a narrower range.

Additionally, the pore structure of biochar that has undergone acid-base modification is more developed. Through chemical activation (e.g., KOH, NaOH, NaCl, and H₃PO₄), the specific surface area and porosity of the material can be further enhanced [111,112]. Hydrothermally synthesized TiO₂ can be modulated into rod-, tubular-, flake-, and flower-like morphologies in the presence of acids and alkalis [113,114]. Meanwhile, biochar is transformed into a porous carbon material following activation. This enables TiO₂ with various nanostructures to be embedded within its pores. Consequently, this extends the migration path of photo-generated carriers to some extent and inhibits their recombination. In terms of band structure modulation, acid-base modification is generally used as an auxiliary method. It has a limited effect on the bandgap of photocatalysts.

The doping modification strategy has been shown to effectively address issues such as particle agglomeration, a low specific surface area, poor utilization of visible light, and insufficient active sites. Metal and non-metal doping promotes the uniform dispersion of TiO₂ nanoparticles and significantly enhances the visible light response range. It also promotes the separation of photogenerated carriers. Semiconductor doping forms heterojunctions by strong interfacial coupling between components, thereby achieving complementary advantages. Acid and alkali modification serves as a supplementary method for regulating interfacial bonding in composites, but it cannot directly regulate the bandgap.

Based on the above analysis, the in situ synthesis strategies are crucial in the preparation of TiO₂-biochar-based photocatalysts. This strategy improves the adsorption-photocatalytic degradation of pollutants through the regulation of morphology, specific surface area, and bandgap. The in situ synthesis process is influenced by multiple parameters. These include titanium sources, biochar precursors, the titanium-to-carbon mass ratio, preparation methods, solvent types, and doping modifications. These parameters interact in multiple ways to influence the microstructure and optical properties of composite materials. This section provides a systematic overview of how various key parameters affect the material's morphology, specific surface area, and bandgap. The review provides insights and perspectives based on relevant research developments.

The photocatalytic activity, microstructure, and physicochemical properties of TiO₂ prepared by in situ synthesis are directly influenced by the type of titanium precursor [115]. Currently, TTIP is the most widely used titanium source in in situ synthesis strategies. Compared with TiCl₄, TTIP is more suitable for preparing TiO₂ in the anatase phase with a higher degree of crystallinity. TiOSO₄ tends to produce TiO₂ with a larger specific surface area and relatively lower crystallinity. Previous reports have examined the effect of titanium precursors on the morphology of TiO₂, revealing that the size of TiO₂ particles at 25 °C is related to the length of the alkyl chain. Among these, the TiO₂ particles prepared using ethanol-based titanium sources exhibited the largest particle size. Those prepared using butanol-based titanium sources showed the smallest particle size [116]. Furthermore, the morphology, specific surface area, and bandgap of TiO₂ are influenced by a variety of factors, including the solvent type, preparation method, temperature, and doping modifications [117]. Optimizing various parameters is essential for the preparation of high-per-

formance TiO₂ nanomaterials. However, the use of these chemical titanium sources increases the cost of the photocatalytic process. Therefore, it is necessary to seek alternative titanium sources (e.g., ilmenite and titanium blast-furnace slag) that can reduce costs while addressing real-world environmental issues. These “green” sources of titanium enable the recycling of solid waste. These strategies also point the way forward for future environmental management.

Different biochar sources influence the pore structure and loading behaviors of the matrix [118]. A highly porous matrix for TiO₂ loading can be achieved using straw-based charcoal that has undergone appropriate pyrolysis treatment and is rich in lignocellulose [119]. Fruit shell-derived biochar enables a more stable environment for the immobilization of active components. Biochar with layered, folded structures is commonly obtained from sludge or algal precursors. The selection of the most suitable carbon source is paramount in determining the interfacial binding strength and dispersion of active components. Optimizing the titanium–carbon mass ratio is a critical parameter for achieving synergy between adsorption and photocatalysis. Insufficient titanium-to-carbon ratios lead to a shortage of active components and a limited distribution range, which decreases photocatalytic performance. Excessively high proportions cause TiO₂ particles to agglomerate, thereby diminishing their adsorption capacity. Furthermore, the titanium-to-carbon mass ratio affects the photocatalyst’s photoresponse characteristics and electron transport pathways to some extent. An optimized titanium-to-carbon ratio enhances light responsiveness and suppresses the rapid recombination of electron–hole pairs.

Among in situ synthesis strategies, the sol–gel method is the most widely used. However, the sol–gel method alone is insufficient. It fails to disperse the particles effectively. By combining sol–gel methods with additional methods such as ultrasonication and microwave treatment, the agglomeration phenomenon can be mitigated to some extent. Photocatalysts prepared by the coprecipitation method tend to form uniform spherical or near-spherical particles. Materials with flower-like morphology and a hierarchical structure are easier to prepare using the solvothermal method. During the preparation of composite materials, the type of solvent also influences their morphological characteristics. Relevant studies indicate that particle size increases with a high concentration of alcoholic solvents [120–122].

During low-temperature pyrolysis, biochar initially develops micropores and is more likely to retain anatase TiO₂. Biochar has a confinement effect during medium- and high-temperature pyrolysis. This confinement effect provides favorable conditions for enhancing the thermal stability of the TiO₂ crystalline phase.

Modifying doping strategies can effectively solve the problems associated with TiO₂-biochar-based photocatalysts. These problems include photocatalysts’ narrow visible-light response range and the tendency of the particles to agglomerate. Specifically, element doping extends the range of light responsiveness by introducing dopant elements and modulating the bandgap. Heterojunctions formed by combining semiconductor materials can enhance synergistic effects. The bandgap of the composite material remains unaffected by acids and alkalis. However, its morphology is more likely to be porous.

Existing studies often employ a variety of parameters to assess the impact of doping modifications on photocatalytic performance. The chief evaluation indicators consist of specific surface area, bandgap width, heterojunction structure, and the recombination rate of photogenerated carriers. Nevertheless, there is an intense interaction between the structural features and the optical properties. It is difficult to determine which characteristic parameter holds the predominant role.

The preparation conditions in Table 2 are compared in detail. The results show that high-performance photocatalysts based on TiO₂ and biochar can be produced. The sol–gel method is the sol–gel method, which uses TTIP as the titanium source and lignocellulosic

biomass as the carbon source. The composite materials are calcined at temperatures of 450–550 °C. The high-performance photocatalysts are produced when the titanium-to-carbon mass ratio is kept between 1:2 and 1:5. This results in high particle dispersion, moderate crystallinity, and a regular microstructure. However, the performance of the TiO₂-biochar binary composite in degrading pollutants may be limited. Consequently, urea and thiourea can be utilized as precursors for g-C₃N₄ to modify the material. Composites that have been doped or modified are more likely to exhibit a layered microstructure. The bandgap can be narrowed to 2.2–3.0 eV, which significantly enhances the composite material's visible-light response.

3. In Situ Synthesis of TiO₂-Biochar Photocatalysts and Their Performance in Removing Organic Pollutants

Due to its excellent adsorption-photocatalytic synergy, the TiO₂-biochar-based photocatalyst is widely used to remove organic pollutants.

3.1. Dyes

Cationic and anionic dyes are among the typical organic pollutants found in aquatic environments. Typical cationic dyes include methylene blue (MB), Rhodamine B (Rh-B), crystal violet (CV), and malachite green (MG). Anionic dyes mainly include methyl orange (MO), Congo red (CR), and naphthol blue black (NBB) [123]. Table 3 lists the functional groups, dye types, degradation conditions, major reactive oxygen species (ROS), and degradation performance of the TiO₂-biochar-based photocatalysts. As shown in Table 3, the overall removal efficiency of the TiO₂-biochar-based photocatalysts toward dyes ranges from 57% to 100%. This can be attributed to the synergistic effects of adsorption and photocatalysis. The biochar component adsorbs and concentrates dye molecules using biochar with various morphologies, bringing the pollutants into proximity with the catalytically active sites. The separation of photogenerated carriers is accelerated by the Ti-O-C bonds in the composite material. When exposed to light, photogenerated electrons/holes react with water and oxygen to produce ROS. Notably, there are hydroxyl radicals ($\bullet\text{OH}$) and superoxide radicals ($\bullet\text{O}_2^-$).

The morphological characteristics of TiO₂-biochar-based photocatalysts can affect the formation and distribution of functional groups. Meanwhile, functional groups play a regulatory role in the catalyst's redox properties and surface charge distribution [124]. Yang [124] reported that the distribution of functional groups is selectively influenced by microporous, mesoporous/microporous, and hierarchical porous structures. Nitrogen-based basic functional groups are distributed in microporous structures. Oxygen-containing acidic functional groups are distributed in mesoporous/microporous structures. Non-polar oxygen-containing functional groups are distributed in multi-scale pore structures. As particle size decreases and porosity increases, the characteristic peaks of oxygen-containing functional groups detected by FTIR become more pronounced [125]. Table 3 lists photocatalysts that primarily contain oxygen-containing functional groups, such as -OH, C=O, and -COOH. These oxygen-containing functional groups include the hydrophilic -OH, the strongly acidic -COOH, and the redox-active C=O. They perform a crucial function in the catalytic degradation of pollutants [126,127]. The -OH and -COOH groups provide active adsorption and surface reactivity sites [128]. During this process, the -OH group acts as an electron donor and is oxidized. The carbonyl group (in ketones/quinones) acts as an electron acceptor, accepting electrons and participating in reduction reactions. The surface charge properties of biochar can be modulated by nitrogen-containing functional groups (-NH₂, -NH-) that appear following modification. These groups increase its hydrophilicity. This modification will enhance pollutant removal performance [129]. The

Ti-O-C bond indicates the formation of a tightly bound interface between TiO₂ and biochar. The Ti-O-C bond can shorten the electron migration path and suppress the recombination of photogenerated carriers.

From the perspective of ROS, biochar itself can generate reactive oxygen species such as singlet oxygen (¹O₂), •O₂[−], hydrogen peroxide (H₂O₂), and •OH when exposed to light [130]. However, further research is still needed to explore formation pathways and mechanisms. As shown in Table 3, the main ROS generated by the TiO₂-biochar-based photocatalysts during dye degradation are •OH and •O₂[−]. When the catalyst is exposed to light, photo-generated charge carriers in TiO₂ are generated and separated. Biochar provides electron transport pathways or pools electrons, thereby facilitating the separation of electrons and holes. The reduction in O₂ to form •O₂[−] is primarily driven by electrons migrating to the biochar interface. On the one hand, holes can undergo direct oxidation reactions with pollutants. On the other hand, holes react with OH[−] in water to form •OH. Furthermore, •O₂[−] generates H₂O₂ through subsequent redox reactions, which is then converted into •OH [130]. As shown in Table 2, TiO₂ adheres tightly to the biochar surface, forming an interfacial structure. ROS are more likely to form at this interface. Romero-Moran [131] reported that enhancing the electronic coupling between TiO₂ and interface-modifying materials makes the electron transport pathways easier to control. When the interfacial coupling between the two is weak, electrons are more likely to remain trapped within the TiO₂. The formation pathways of •O₂[−], H₂O₂, and •OH are influenced by the ability of interface-modified materials to capture electrons. This further suggests that ROS are more likely to form at the interfaces of composite materials.

The morphological characteristics of photocatalysts also influence their pollutant removal performance. The introduction of biochar results in a more uniform distribution of TiO₂ nanoparticles. Concurrently, the composite material's pore structure is improved, thereby enhancing dye removal efficiency [132]. Figure 8 presents the dye removal efficiencies reported in various publications. The preparation of wrinkled biochar/TiO₂ composites was prepared from macroalgae for the synergistic removal of MB. The adsorption capacity and photocatalytic degradation efficiency increased by 3.07- and 1.16-fold, respectively, compared with pure TiO₂ [64]. In this system, •O₂[−] was the primary ROS responsible for promoting the decolorization, degradation, and mineralization of MB. •O₂[−] and h⁺ react to form •OH. •O₂[−] can also recombine with holes in the TiO₂ valence band. This consumes the active sites, which is not conducive to the degradation of pollutants. A core-shell structured TA@BC heterojunction material was developed for the removal of Rh-B. The results showed that the removal efficiency of Rh-B at a concentration of 25 mg/L reached 98% within 4 h. This was an improvement on the removal efficiency of pure TiO₂, Al₂O₃, and the TiO₂/Al₂O₃ binary composite [49]. •OH was the primary ROS involved in attacking Rh-B molecules and mineralization. TiO₂-biochar was combined with Ag for the removal of MO. Compared with pure TiO₂, this composite achieved decolorization and mineralization rates of 97.48% and 85.38%, respectively [67]. •OH was the key ROS in this system, but h⁺ and •O₂[−] also contributed to the degradation of the pollutants. h⁺ decolorized MO through oxidation. •O₂[−] broke down the intermediate products. •OH carried out deep mineralization. A porous carbon-based material (TiO₂-BC) derived from coconut shells was used to degrade the anionic dye brilliant blue KN-R. After 1 h of UV irradiation, the composite achieved decolorization rates of 99.71% and 96.99% in strong acidic and alkaline conditions, respectively [44]. In this system, KN-R molecules were oxidized by h⁺, •OH, and •O₂[−]. Research has shown that when composite materials exhibit a well-developed porous structure, their dye removal efficiency is superior to that of TiO₂ aggregates. These aggregates adhere to carbon-based surfaces or structures with collapsed pore morphology [133]. Wang [97] prepared C/TiO₂ photocatalysts by combining gel and hydrothermal methods. The experimental results indicated that C/TiO₂ exhibits superior

photocatalytic degradation performance for $\text{NH}_3\text{-N}$ compared to pure biochar. This is because C/TiO_2 possesses a more developed pore structure and a larger specific surface area. The presence of small, spread-out TiO_2 particles and a porous biochar structure improves the effectiveness of dye removal.

Table 3. The dyes removal performance of TiO_2 -biochar-based photocatalysts.

Catalysts	Functional Group/Chemical Bond	Conditions	Dyes	ROS	Removal (%)	Activity	k/ min ⁻¹	TOC (%)	Cycles	Ref.
BC- TiO_2	-OH, C-O, Ti-OH, Ti-O-C	A 500 W visible light bulb; $T = 25 \pm 1$ °C; Time = 4 h, pH = 6.0–7.0; dosage = 100 mg; MB = 5 ppm; solution volume = 50 mL	MB	$\bullet\text{O}_2^-$	99.20%	0.010	-	-	-	[64]
BC/ TiO_2	C=O, C-O, O=C-O	A 10 mW·m ⁻² of UV irradiation; 180 min; pH = 5.5; $T = 298$ K; dosage = 100 mg; MB = 10 ppm; solution volume = 50 mL	MB	$\bullet\text{OH}$	83.00%	0.005	0.0081	-	3/77%	[72]
BSP 20	O-Ti-O, Ti-O, C-O, C=O, C=C, -CH ₃ , -CH ₂ -, -OH	A Philips UV sterilizer unit (power 6 W; voltage 220 V); 150 min; dosage = 100 mg; MB = 10 ppm; solution volume = 250 mL	MB	$\bullet\text{OH}$, $\bullet\text{O}_2^-$	100%	0.067	0.021	-	-	[54]
AC/ TiO_2	-OH, -COOH, C-H, -NH ₂ , -NH-CO-, C-O, C-O-Ti-O	Solar irradiation; 30 min; dosage = 100 mg; MB = 5 ppm; solution volume = 100 mL	Rh-B	h^+	91%	0.145	0.074	-	-	[65]
CS-BC/ ZnO/ TiO_2	C=O, N-H, C-H, O-H, Ti-O, Zn-O	A 300 W Xenon lamp ($\lambda > 420$ nm); 25 min; dosage = 100 mg; Rh-B = 10 ppm; solution volume = 200 mL	Rh-B	-	94.5%	0.756	0.099	-	5/~82%	[69]
TA@BC	-OH, -NH, C-H, C-O-C, C=C, Al-O, Ti-O-Ti	A 350 W xenon lamp; 4 h; dosage = 100 mg; Rh-B = 10 ppm; solution volume = 100 mL	Rh-B	$\bullet\text{OH}$	98%	0.408	-	-	-	[49]
$\text{TiO}_2/\text{AC}_{\text{y}_350}$	-OH, C=O, -COOH, C-O	4 UV lamps (18 W); 4 h; pH = 6; dosage = 100 mg; VBB = 15 ppm; solution volume = 200 mL	VBB	-	99%	0.124	-	-	4/~70%	[42]
Ni-T/AC	-OH, C-H, Ti-O, Ti-O-C	A 500 W Xe lamp (350 W/m ²); 2 h; $T = 25 \pm 1.0$ °C; dosage = 100 mg; dyes = 10 ppm; solution volume = 250 mL	CV BF MG	h^+	99.00% 94.85% 98.89%	0.206 0.198 0.206	0.060 0.029 0.048	-	5/~95%	[58]

TiO ₂ -biochar	-OH, -COO ⁻ , C=O, C=C, Ti-O-Ti, Ti-O, O-Ti-C	A 19 W UV lamp; 2 h; pH = 11; dosage = 100 mg; dyes = 20 ppm; solution volume = 100 mL	BB41 EBT	•OH, h ⁺	98.4% 97.4%	0.164 1.623	0.067 0.018	-	5/ 96.14%, 77.2%	[55]
TiO ₂ /biochar	-OH, -C=O	A 500 W mercury lamp; 2.5 h; dosage = 100 mg; MO = 20 ppm; solution volume = 400 mL	MO	h ⁺ , •O ₂ ⁻ , •OH	96.88%	0.817	0.023	83.23	5/ 92.5%	[96]
Ag/TiO ₂ /biochar	C-C, C=C, C-O, C=O, O-C=O, -OH	A 500 W long arc mercury-vapor lamp; 1 h; dosage = 100 mg; MO = 20 ppm; solution volume = 400 mL	MO	•OH	97.48%	1.300	0.063	85.38	5/ 96.10%	[67]
TiO ₂ -BC	-OH, Ti-OH, Ti-O-C, Ti-O-Ti	A 500 W mercury lamp; 1 h; pH = 5; dosage = 100 mg; MO = 20 ppm; solution volume = 200 mL	MO	•O ₂ ⁻	100%	0.667	0.051	73.20	-	[73]
TiO ₂ @HC	-	4 UVA lamps (8 W); 5 h; pH = 2; dosage = 100 mg; AR97 = 20 ppm; solution volume = 400 mL	AR97	•OH	98.59%	0.263	0.032	-	5/ 88.80%	[71]
TiO ₂ /BC	-	A 300 W Xenon lamp; 1 h; pH = 1; T = 25 ± 2 °C; dosage = 100 mg; dyes = 30 ppm	RBB KN-R	•OH, •O ₂ ⁻	99.71%	-	-	-	5/ 73.98%	[44]
TiO ₂ /BC	O-H, C=C, C=O, SO ₃ ²⁻ , S=O	10 mW/m ² of UV bulb (15 W); 3 h; dosage = 100 mg; AO7 = 20 ppm; solution volume = 50 mL	AO7	•OH, •O ₂ ⁻	57.6%	0.064	0.009	-	6/~ 80.00%	[59]

Note: The removal efficiency represents photocatalytic degradation efficiency after dark adsorption. Activity = Pollutant removal capacity/(Catalyst dosage × Time) mg/(g·min).

The TiO₂-biochar-based photocatalyst demonstrates remarkable removal efficiency for both cationic and anionic dyes. The microstructure of this photocatalyst is a key factor in governing surface functional groups, ROS generation, and dye removal efficiency. The pore structure influences the distribution characteristics of oxygen- and nitrogen-containing surface functional groups. These functional groups can provide adsorption sites and photocatalytically active sites. The formation of Ti-O-C bonds in the composite material is a significant indicator of the success of the composite process. It contributes to charge transport and the inhibition of photogenerated electron-hole pair recombination. The porous structure promotes the concentration of functional groups, thereby creating conditions conducive to the generation of ROS. In TiO₂-biochar-based photocatalysts, the primary ROS are •OH and •O₂⁻. Furthermore, photocatalysts with specific morphologies, such as wrinkles, core-shell structures, and porous structures, perform better than pristine materials. Therefore, it is evident that controlling the morphology of photocatalysts significantly improves the efficiency of pollutant removal.

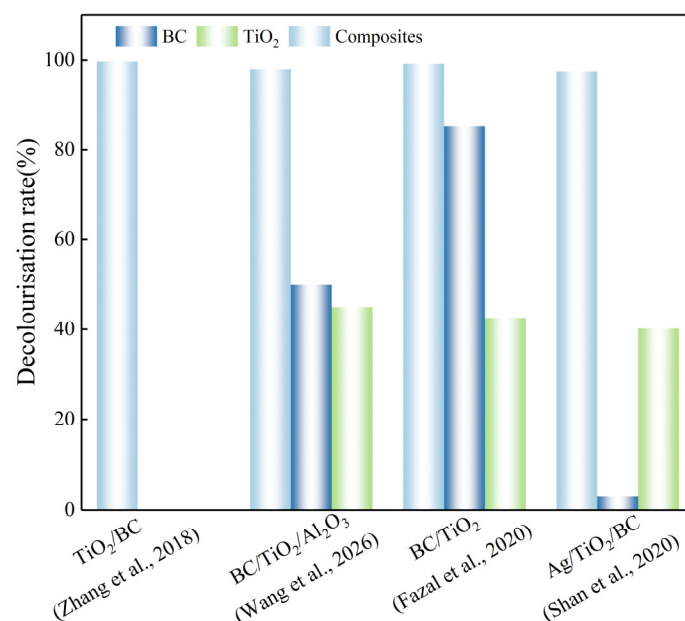


Figure 8. Adsorption-photocatalytic synergistic removal of dyes. Data was obtained from the literature [44,49,64,67].

Tables 2 and 3 reveal that TiO₂ nanoparticles that are uniformly dispersed within the porous biochar (pore-embedded) exhibit the best performance in degrading dyes. The advantages of this morphology include the enrichment of pollutants, the inhibition of TiO₂ agglomeration, and the provision of a continuous electron transport pathway. The Ti-O-C interface exhibits strong bonding properties, which broaden the range of light response and enhance the generation of ROS. Although layered, core-shell, and one-dimensional morphologies optimize charge transport, they are inferior to the pore-embedded type. This is due to their lower pore volume and pollutant enrichment capacity.

3.2. Antibiotics

The removal performance of TiO₂-biochar-based photocatalysts prepared via in situ synthesis for various antibiotics (e.g., SMX, SDZ, TC, and CIP) is summarized in Table 4. As shown in the table, the photocatalyst's removal efficiency for antibiotics is generally between 80% and 100%. This efficiency is slightly lower than its overall degradation performance for dyes. This is because the π structures of dye molecules are susceptible to attack by ROS. Furthermore, the dye's photochemical sensitization accelerates its degradation under light conditions [134–136]. In contrast, antibiotics (e.g., SMX) contain a p-aminobenzenesulfonamide group and a methyl substituent. ROS preferentially attack S-N bonds, which lead to the ring opening of oxazole rings and other reactions. This process enables antibiotics gradual mineralization [137,138]. The primary ROS generated by this photocatalyst during the degradation of antibiotics are $\bullet\text{O}_2^-$ and $\bullet\text{OH}$. This is essentially consistent with the ROS involved in dye degradation. The photocatalyst's ability to remove antibiotics can be further enhanced by modification. These results show that the photocatalytic system is highly effective at removing antibiotics from aquatic environments.

Table 4. The antibiotics removal performance of TiO₂-biochar-based photocatalysts.

Catalysts	Functional Group/ Chemical Bond	Conditions	Pollutants	ROS	Removal (%)	Activity	k/ min ⁻¹	TOC (%)	Cycles	Ref.
PMBC@TiO ₂	-OH, C-O, C=O, O-C=O	25 W UV lamp; 2.5 h; pH = 6.46; dosage = 100 mg; SDZ = 20 ppm; solution volume = 50 mL	SDZ	•O ₂ ⁻ , •OH	94.60%	63.070	-	-	-	[43]
BC/TiO ₂ /g-C ₃ N ₄	-OH, C=O, C-H, C-N, Ti-O/Ti-O-Ti	solar irradiation; 1 h; dosage = 100 mg; SDZ = 30 ppm; solution volume = 170 mL	SDZ	•O ₂ ⁻ , h ⁺ , •OH	98.13%	0.818	-	-	5/ 68.85%	[48]
TBC-2	C=O, C-O, O=C-O	An 8 W UV lamp; pH = 8; 2 h; dosage = 100 mg; solution volume = 50 mL	ST	•OH	226.7 mg·g ⁻¹	-	0.029	-	6/ 167.2 mg·g ⁻¹	[51]
biochar/TiO ₂	-	A 15 W UV lamp; 6 h; pH = 4; t = 293 ± 2 K; dosage = 100 mg; SMX = 10 ppm; solution volume = 20 mL	SMX	•OH	90%	0.006	-	-	-	[47]
TiO ₂ /pBC	C=O, -COOH, C-O, -OH	A 50 W xenon lamp; 3 h; t = 25 °C; pH = 4; dosage = 100 mg; SMX = 10 ppm; solution volume = 80 mL	SMX	•OH, h ⁺	91.27%	0.041	0.013	-	-	[52]
Zn-TiO ₂ /pBC	-	A 50 W Xenon lamp; t = 25 °C; pH = 5.03; dosage = 100 mg; SMX = 10 ppm; solution volume = 80 mL	SMX	-	81.21%	0.036	0.009	-	5/ 77.41%	[53]
GAC-TiO ₂	-	4 submersible UV lamps; 80 min; dosage = 100 mg; SMX = 100 ppm; solution volume = 180 mL	SMX	-	83.60%	1.522	-	-	5/ ~40%	[57]
Alg-PSB@TiO ₂	-OH, -COO ⁻ , C-O, -CH/-CH ₂ , C-H, Ti-O, Ti-O-Ti, Ti-O-C	A 600 W solar simulator; 1.5 h; pH = 6.07; t = 25 °C; dosage = 100 mg; SMX = 15 ppm; solution volume = 100 mL	SMX	h ⁺ , SO ₄ • ⁻ , e ⁻	99.9%	0.167	0.050	73.4	-	[46]
TiO ₂ @AC-STM	-OH, C-H, C-C, C-O, Ti-O-Ti, Ti-O-C	UV light (254 nm, 35 W/cm ²); pH = 6–9; 2 h; dosage = 100 mg; TC = 20 ppm; solution volume = 400 mL	TC	•O ₂ ⁻	88%	0.587	0.014	-	4/ ~55%	[60]
TiO ₂ /AC	-	The 18 W germicide lamp;	TC	-	100%	0.333	0.043	-	-	[45]

		pH = 3; 75 min; dosage = 100 mg; TC = 50 ppm; solution volume = 20 mL								
Au/TiO ₂ /BCP	-OH, C=O, O-C, C-C/C=C, C-N, C-Ti, N-Ti, N-O, π - π , Ti-O	A long-arc mercury lamp; 3 h; t = 25 °C; dosage = 100 mg; TC = 40 ppm; solution volume = 1.25 L	TC	•O ₂ ⁻	98.4%	2.733	0.019	-	4/90%	[68]
A-BC@g-C ₃ N ₄ /La-TiO ₂ (A)	-OH, -NH, C-N, C-O, Ti-O	A 300 W xenon lamp; 1.5 h; t = 25 °C; dosage = 100 mg; TC = 30 ppm; solution volume = 200 mL	TC	•OH, •O ₂ ⁻	100%	0.333	0.028	-	4/~98%	[74]
N-doped bio-char/TiO ₂	-	A 150 W xenon lamp; 2 h; CEPs = 200 ppm; solution volume = 200 mL	CEPs	-	31.5%	-	0.021	-	4/~90%	[62]
AC-TiO ₂	Ti-O-C, C-O, C-O-C, Ti-O, Ti-O-Ti	LED bulb (50 W); 4 h; dosage = 100 mg; CEF = 100 ppm; solution volume = 100 mL	CEF	•OH, •O ₂ ⁻	99.6%	0.415	-	-	5/81.65%	[66]
N-doped TiO ₂ /BC	C-O, N-H, Ti-O-Ti, N-Ti-O, Ti-C, C=O, -OH	19 W UV light/natural sunshine; 2 h; pH = 6.9; dosage = 100 mg; CIX = 50 ppm; solution volume = 20 mL	CIX	•OH	98.9%/96.9%	-	0.023	-	5/~90%	[56]
BC/TiO ₂	-	10 UV-C (15 mW/cm ²); 110 min; dosage = 100 mg; CIP = 10 ppm; solution volume = 200 mL	CIP	-	95%	-	0.020	-	-	[63]

From Tables 2 and 4, layered structures exhibit greater efficacy in antibiotic degradation. Antibiotics contain benzene rings and heterocyclic structures. The aromatic structures in antibiotics can establish π - π stacking interactions with the π -conjugated structure of layered carbon. Furthermore, because antibiotics are highly chemically stable and difficult to degrade, efficient heterojunctions need to be introduced into the layered structure to enhance charge separation. For irregular and tubular morphologies, severe agglomeration of TiO₂ can cover some of the active sites. This results in a faster recombination rate of photogenerated charge carriers. Therefore, these morphologies demonstrate poor antibiotic degradation.

The impact of morphology on antibiotic removal performance is being analyzed. Figure 9 illustrates the degradation performance of photocatalysts with different microstructures in relation to antibiotics. Photocatalysts with spherical or tubular microstructures have been shown to be more effective at removing pollutants. Tubular and granular TiO₂-biochar-based photocatalysts achieved antibiotic removal efficiencies ranging from 81.2% to 95% [139]. During the degradation of SDZ, •O₂⁻ and •OH were the principal ROS. Although •OH participated in the decolorization process during the photocatalytic degradation of ST, the adsorption process remained predominant during the removal of pollutants. In the photocatalytic degradation of CIP, •OH induced the defluorination reaction of CIP. •O₂⁻ oxidized the C7-N bond. This ultimately led to the complete mineralization

of CIP. In contrast, the pollutant removal efficiencies of layered photocatalysts and those with other morphologies generally range from 98.13% to 100%. In the BC/TiO₂/g-C₃N₄ system, •O₂[−] can trigger the oxidative cleavage of the H-N bond in SDZ molecules. •OH and •O₂[−] attacked the S-N bond synergistically. •OH and •O₂[−] were the main ROS in the A-BC@g-C₃N₄/La-TiO₂(A) photocatalytic degradation of TC, facilitating ring opening and mineralization. h⁺ can directly oxidize TC molecules and assist ROS in the reaction. In the coupled activation of Vis/Alg-PSB@TiO₂ by PMS for the degradation of SMX, the degradation of the pollutant was driven by the joint action of SO₄^{•−}, h⁺ and e[−]. This synergistic effect enhances the performance by fostering the separation of photo-generated charge carriers. Previous studies have shown that, following successful composite formation between TiO₂ and biochar, the resulting material typically exhibits a rougher surface. The resulting composite also typically has a higher specific surface area compared to pure biochar [140].

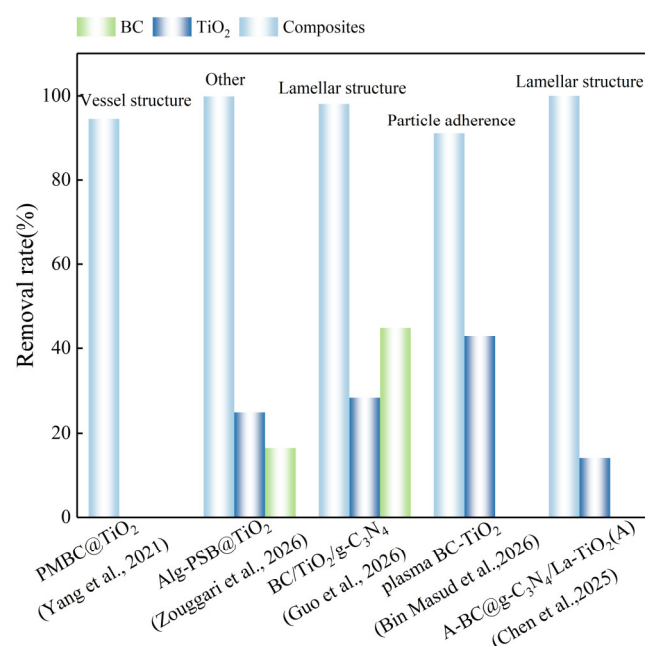


Figure 9. Adsorption–photocatalytic synergistic removal of antibiotics. Data was obtained from the literature [43,46,48,63,74].

The deep mineralization of dyes involves the further oxidation of intermediate products by ROS. The mineralization and decolorization rates of MO are high in the Ag/TiO₂/biochar system. Due to their distinct molecular structures, antibiotics and dyes follow different metabolic pathways. The CIP mineralization is achieved through bond breaking and defluorination. TC mineralization is promoted through heterocyclic ring opening. Research into dyes and antibiotics primarily uses removal and decolorization efficiency as indicators of performance. It is difficult to assess whether the pollutants have been completely mineralized because there has been insufficient analysis of TOC and intermediate products.

The differences in the molecular structures of dyes and antibiotics determine variations in their degradation behaviors [141]. Based on the above analysis, the primary ROS involved in the photocatalytic degradation of dyes and antibiotics are •OH and •O₂[−]. These ROS attack their weak chemical bonds, thereby facilitating the mineralization of pollutants. The fundamental principles are consistent during the adsorption–photocatalytic synergistic degradation process. However, there are significant differences in degradation pathways. ROS primarily attack the azo bonds and anthraquinone structures of dye molecules. This breaks the conjugated chromophores. The most obvious feature is discoloration. Subsequently, the intermediate products are mineralized by ROS [142]. The intermediate products formed are

predominantly aromatic amines. Antibiotics contain numerous functional groups and heterocyclic structures. ROS initially target the chemical bonds within molecules, particularly C-N, C-F, and amide bonds. This can trigger bond cleavage, decarboxylation, and ring-opening reactions [143]. Multiple intermediate products are formed during this process. Although some antibiotics undergo significant degradation, the toxicity of their intermediate products is higher than that of the original antibiotics [144]. Furthermore, intermediate products that are even more toxic are produced by the photocatalytic degradation of antibiotics. By regulating ROS, these toxic intermediates can be converted into less toxic products [145,146]. Both their toxicity and their degradation rate should be considered. In summary, the molecular structures of dyes and antibiotics primarily determine their degradation pathways and the way chemical bonds are broken. They also determine the toxicity of their intermediate products. The morphology of different photocatalysts and the ROS generation can be controlled. This enables pollutants to be degraded and intermediate products to be converted into harmless substances.

Additionally, the type of ROS is closely related to the structure of the catalyst. The uniform dispersion of TiO₂ particles on the surface of the biochar results in weak interfacial bonding. The holes in the TiO₂ valence band can readily participate in oxidation reactions with water molecules or -OH. This facilitates the generation of •OH. When TiO₂ nanoparticles are confined within biochar, the bonding strength at the Ti-O-C interface is enhanced. The concentration of electrons and dissolved oxygen within the pores makes it more favorable for the formation of •O₂⁻. The layered structure features exposed channels and displays strong charge separation capabilities. It can engage in redox reactions with water molecules, -OH and dissolved oxygen simultaneously. The synergistic promotion of pollutant degradation is achieved through the combined roles of •OH, •O₂⁻ and h⁺.

3.3. PFASs and MPs

Based on existing research, the range of applications for TiO₂-biochar-based photocatalysts is gradually expanding to include other areas involving recalcitrant pollutants. These pollutants include MPs and PFAS [119,147] (Table 5). Previous research reported that after 40 h of light exposure, BC/CST reduced the particle size of PE from 500 µm to 70 µm [148]. The degradation rate of PE reached 67.58% when g-C₃N₄ was integrated with TiO₂/WCT-AC to form a heterojunction. Furthermore, the photocatalytic system retained a degradation rate of approximately 65% even after five cycles [149]. It should be noted that differences in the performance of various TiO₂-biochar-based photocatalysts in the treatment of PE MPs are influenced by a combination of factors. These factors include the light source, precursors, reaction time, and the type of MPs.

PFAS contain strong carbon-fluorine (C-F) bonds and are known as forever chemicals. Liu [150] synthesized TNTs@biochar, which can be used for synergistic adsorption and photocatalysis to efficiently remove PFOA. In optimal conditions, both the adsorption rate and the photodegradation rate can reach 99%. Li [151] prepared Fe/TNTs@AC composites, achieving PFOA adsorption and defluorination rates of 99% and 62%, respectively, when optimized conditions were used.

PFAS and MPs have more stable chemical bonds and are more resistant to oxidation than dyes and antibiotics. They take more time to degrade. As shown in Table 5, both h⁺ and •OH are extremely reactive oxidizing species. They play a dominant role in the degradation of both types of pollutants.

Table 5. Degradation of PFAS and MPs by TiO₂–biochar-based photocatalysts.

Catalysts	Functional Group/ Chemical Bond	Conditions	PFAS	ROS	Removal/ Defluorination	k/ min ^{−1}	Cycles	Ref.
Fe/TNTs@AC	-	UV irradiation; 22 ± 1 °C; 4 h; pH = 7.0; PFOA = 100 µg/L; dosage = 100 mg; solution volume = 10 mL	PFOA	h ⁺	91.3%/~60%	-	5/ ~60	[151]
In/TNTs@AC	-OH, -COOH, C=O	UV irradiation; 25 °C; 4 h; pH = 7; dosage = 100 mg; PFOA = 0.1 mg/L; solution volume = 400 mL	PFOA	h ⁺	100%/60%	-	4/ ~45%	[152]
TNTs@biochar	-OH, C=O, C=C, C-O, Ti-O, C-H	a 254 nm UV source; 7 h; pH = 7.0; 23 ± 2 °C; PFOA = 100 mg/L; dosage = 1.5 g/L; solution volume = 65 mL	PFOA	h ⁺ , •OH	99%/99%	-	-	[150]
			MPs		Mass loss/ TOC			
BC/CST	Ti-O, C-O-C, C-O, C=O	Four light bulbs; 40 h; PE = 50 mg; catalyst = 100 mg; solution volume = 100 mL	PE	•OH	The particle size reduced to 430 µm	-	-	[148]
g-C ₃ N ₄ /TiO ₂ /WCT-AC	C=C; N=C=N; C=N=C; Ti-O	PE = 50 mg; 500 W xenon lamp; 200 h; solution volume = 50 mL; 25 °C	PE	•O ₂ [−] , •OH	67.58%	-	5/ ~65%	[149]

4. Synergistic Mechanism of Adsorption and Photocatalysis for TiO₂–Biochar-Based Photocatalysts

In TiO₂–biochar-based photocatalysts, the removal of pollutants primarily involves three processes: (1) The adsorption of pollutants by biochar. The mechanisms primarily encompass electrostatic interactions, pore filling, hydrogen bonding, hydrophobic interactions, π – π interactions, and n – π interactions [153]. (2) Upon light irradiation, TiO₂ is excited and generates electron–hole pairs. Photogenerated carriers are transferred to the surface of the biochar, where they react with dissolved oxygen to form •O₂[−]. The hole reacts with H₂O/hydroxyl to form •OH [154]. (3) The TiO₂–biochar photocatalyst generates ROS that participate in the further degradation of pollutants. [11] (Figure 10). When biochar is combined with TiO₂, carbon atoms can be incorporated into the TiO₂ lattice, forming Ti–O–C bonds. This interaction can effectively reduce the bandgap energy and improve light utilization [155]. During the entire adsorption–photocatalytic synergy process, biochar not only serves to concentrate pollutants. It also provides a support framework for TiO₂. It acts as both a carrier medium and a storage area for electrons, thereby promoting the separation and migration of electron–hole pairs [156].

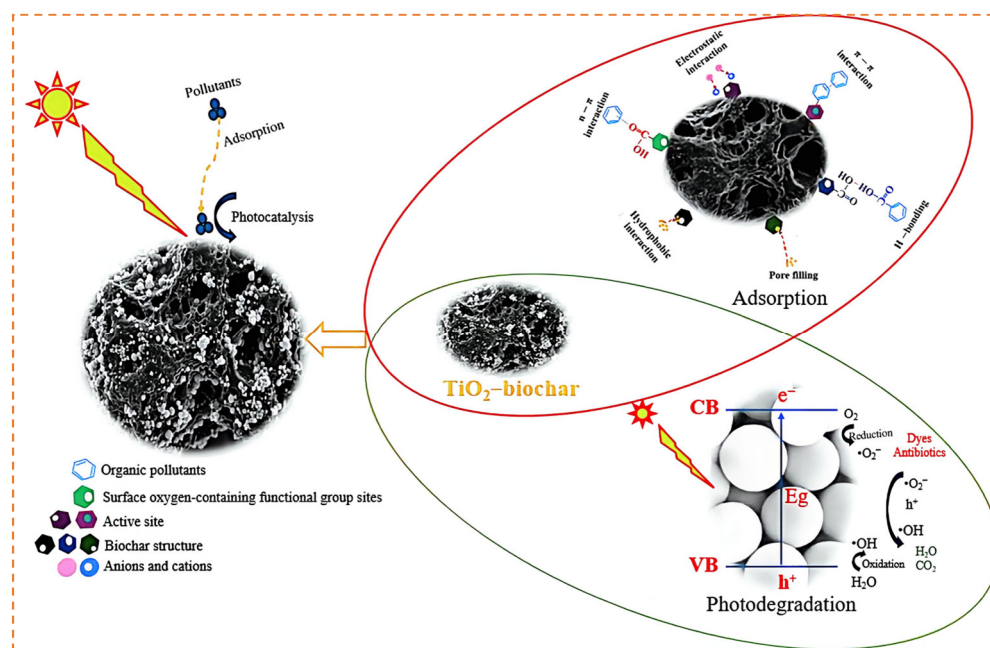


Figure 10. Mechanism of adsorption–photocatalytic synergy.

The morphology of TiO₂–biochar-based photocatalysts holds significant implications for adsorption behavior and charge transfer pathways. This photocatalyst has three main microstructural configurations: surface-adhered, pore-embedded, and interlayer-distributed. Surface-adhered composites primarily depend on physical adsorption and interfacial interactions [32]. Only a small number of Ti–O–C bonds are formed at the interface. Electrons migrate from the conduction band of the TiO₂ to the surface of the biochar. As an electron acceptor, or electron sink, biochar attracts electrons to its outer surface [157]. In a redox reaction, electrons react with dissolved oxygen to primarily generate $\bullet\text{O}_2^-$ [158]. Fewer water molecules are adsorbed at the interface because the biochar surface is directly exposed to air. This indicates that only a limited number of water molecules are available to interact with the holes in the TiO₂ valence band. The amount of $\bullet\text{OH}$ produced is low. The results of the C 1 s and O 1 s peak deconvolution can be used to analyze the bonding strength at the Ti–O–C interface. The O–H characteristic peak for surface-adhered catalysts has a higher relative peak area than the peaks for pore-embedded and interlayer-distributed catalysts. It has been shown that the surface is enriched in hydroxyl groups. Upon light irradiation, these hydroxyl groups are likely to produce significant amounts of $\bullet\text{OH}$. The EPR analysis shows that as the duration of illumination increases, the characteristic intensity of $\bullet\text{OH}$ also increases [46,61,68]. This structure has the following advantages: high chemical stability, pronounced adsorption–photocatalytic synergy, and low energy consumption [37,159–161]. However, repeated use may reduce the efficiency of removing pollutants or the stability of surface-adhered materials due to the depletion of active sites. Consequently, confining photocatalysts within the pores of biochar represents a novel strategy (Figure 11b).

Pore-embedded composites contain an interconnected carbon-based conductive skeleton. These continuous channels enable light to penetrate deep within the material. The presence of TiO₂ makes it possible for excited electrons to be transported rapidly. This happens along the conductive framework within the biochar pores [162]. The holes retained on the TiO₂ react with the water in the pores to form $\bullet\text{OH}$. When electrons migrate to the surface of biochar, they react with the dissolved oxygen within the pores to form $\bullet\text{O}_2^-$. This is because the pore-embedded variety absorbs more water and dissolved oxygen within its pore channels. This confined environment minimizes the diffusion of reac-

tants and enhances ROS generation. Thus, the concentrations of $\bullet\text{OH}$ and $\bullet\text{O}_2^-$ generated are higher than in the surface-adhered composites.

The interlayer distribution of the composites forms a continuous conductive network between the layers, which is useful for electrical conductivity (Figure 11c). Charge transfer resistance is reduced, allowing electrons to migrate in a specific direction [163]. The formation of $^1\text{O}_2$ is dependent on the microenvironment of interlayers [164]. The rapid migration of electrons along this structure reduces the recombination rate of photogenerated carriers. Simultaneously, the interlayer gaps accumulate water molecules and dissolved oxygen, thereby promoting the formation of $\bullet\text{OH}$, $\bullet\text{O}_2^-$, and $^1\text{O}_2$. Organic pollutants are oxidized more effectively by this structure. Tai [165] developed several electron transfer pathways within layered biochar derived from Caragana, which was used to confine zero-valent iron particles. This structure achieved a 100% degradation rate of TC within 80 min under the action of persulfate. Jing [166] used stonewort biochar to construct a 'scallop-shaped' confined structure for encapsulating $\text{Fe}_3\text{O}_4/\text{ZnO}$. The formation of the confined space regulates the crystal morphology of the photocatalyst. It also enables the efficient degradation of PFOA. At present, the main application of biochar in confined spaces is in adsorption, with its use in photocatalytic degradation being comparatively limited.

In pore-embedded and interlayer-distributed composites, the confinement effect allows for the formation of more oxygen vacancies. The oxygen-containing functional groups in biochar combine with TiO_2 , generating more Ti-OH . The increased intensity of the $-\text{OH}$ peak further indicates the formation of a stronger Ti-O-C interface. Consequently, the $\text{O } 1s$ characteristic peak shifts and broadens [46]. Compared with surface-adhered composites, pore-embedded and interlayer-distributed composites have shorter electron migration paths. The recombination of photogenerated charge carriers is effectively suppressed. Numerous studies have shown that good charge transport properties are typically associated with low photoluminescence (PL) intensity, a small Nyquist half-radius in the electrochemical impedance spectroscopy (EIS) curve, and a high transient photocurrent response [96,167]. In contrast, surface-adhered composites have lower bonding strength and struggle to form oxygen vacancies. This results in higher resistance to electron transport.

The differences in interfacial charge transport and ROS levels resulting from different morphologies are key factors. These factors contribute to the variations in the performance in removing dyes and antibiotics. The charge transfer mechanism depends not only on the microstructure of the composite material. It also relates to surface functional groups and electron transport behavior. Tables 3 and 4 show that the biochar surface contains oxygen-containing functional groups. These groups include $-\text{OH}$, $-\text{COOH}$, and C=O . These are the key functional groups that influence surface polarity, surface charge, and electron transfer capacity [168]. The electron shuttle mechanism is achieved through indirect electron transfer between the electron acceptor C=O and the electron donor $-\text{OH}$ [169,170]. The morphology of composite materials also influences electron migration pathways, recombination probability, and the exposure of active sites [171,172]. Electronic transport behavior reflects charge transfer mechanisms. The transfer of charge is effectively mediated by the material's microstructure, composition, and heterogeneous interfaces [173]. The recombination rate of photo-generated carriers and the production of ROS are collectively moderated by microstructure, surface functional groups, and electron transport pathways, thereby regulating photocatalytic activity.

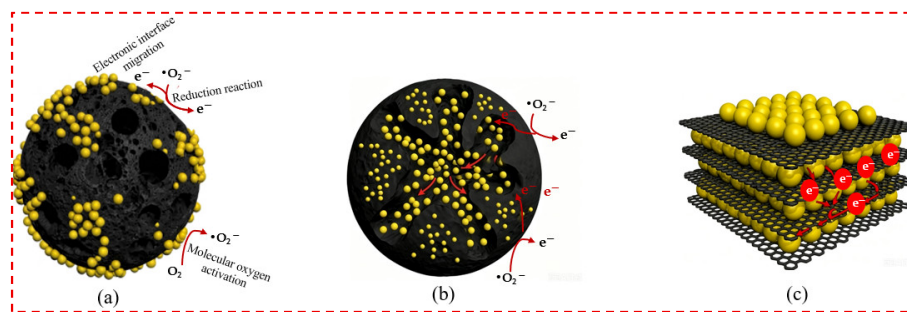


Figure 11. The influence of morphological features on the path of electron transport at the interface. (a) surface-adhered, (b) pore-embedded, and (c) interlayer-distributed.

5. Comparison of TiO₂-Based Adsorption-Photocatalysts

TiO₂-biochar-based photocatalysts have great potential for use in removing pollutants. Compared with other synergistic adsorption-photocatalytic systems (e.g., TiO₂/g-C₃N₄, TiO₂-MOFs, and TiO₂-zeolites), TiO₂-biochar-based systems offer certain advantages. These include cost-effectiveness, chemical stability, environmental sustainability, and simple preparation. This section discusses the performance of different TiO₂-based adsorption-photocatalysts under comparable TiO₂-based photocatalytic conditions.

In terms of cost, the commercial prices per 100 g of biochar, cellulose, chitosan, g-C₃N₄, MOFs, and zeolites are approximately 0.18, 3.4, 13.1, 127, 12,700, and 33.9 USD, respectively. Among these, biochar is the most cost-effective.

The surface of biochar comprises oxygen-containing functional groups. The pH can determine the degree of ionization of oxygen-containing functional groups. It also influences the characteristics of their surface charge distribution [174]. Biochar has a strong adsorption capacity for cationic pollutants when the pH of the aqueous solution is high. This is because of the deprotonation of surface functional groups [175]. Conversely, when the pH level is low, the surface functional groups become protonated. As a result, biochar captures anionic pollutants through electrostatic effects [176,177]. Furthermore, biochar possesses a stable aromatic carbon skeleton. It has highly effective adsorption properties for both cationic and anionic pollutants. Kataya [178] produced biochar from kitchen waste. The adsorption capacities for MB and MO were 30.40 and 25.15 mg/g, respectively, which are higher than those reported for other biochars. Reddy [179] used biochar to remove heavy metals, polycyclic aromatic hydrocarbons, and *E. coli* simultaneously. The removal rates for the three pollutants averaged 35.83%, 68%, and 27%, respectively. Tohdee [180] conducted five cycles to determine the adsorption stability of biochar derived from oil palm empty fruit bunches. The results revealed that the adsorption efficiency exceeded 45% for both pure biochar and composite materials. Its aromatic conjugated conductive framework not only has adsorption properties but also acts as an electron transport channel. This channel suppresses the recombination of photogenerated charge carriers. Nworie [181] synthesized the composite material Ag@RLEBN using rice leaf derivatives. The band gaps of the pure biochar and the composite materials were reduced to 1.9 and 1.8 eV, respectively. Ag@RLEBN achieved a degradation rate of 99.7% for MB. This rate remained at 90% even after five reuse cycles of use. Regarding environmental sustainability, Ashmita Patro [182] and Osman A [183] have systematically discussed the multifaceted applications of biochar.

Cellulose is a natural polymeric material that is hydrophilic and biodegradable. It consists of repeating units of D-glucose linked together. It is a homopolysaccharide rich in hydroxyl groups and possesses a linear, rigid backbone [184]. Hydroxyl groups are the primary functional groups responsible for influencing the adsorption properties of cellulose [185]. Zhuang [186] applied cellulose-based materials for the adsorption of MPs. The

results showed that the adsorption capacity of MPs under neutral conditions was 144.85 mg/g. The adsorption capacity decreased to 120 mg/g at pH 5 and 9. The adsorption capacity declined markedly after three cycles. Olorunnisola [187] investigated the adsorption behavior of cellulose towards antibiotics. It was explicitly stated that the adsorption capacity peaked at a temperature of 30 °C under neutral conditions. Following five cycles of stability testing, the antibiotic removal efficiency decreased by 10–15%. Cellulose does not possess photocatalytic degradation capabilities. When combined with the active components, it provides an efficient dispersing structure for their dispersion. This enhances both adsorption and photocatalysis. Hong [188] prepared the cellulose-TiO₂ composite for removing Rh-B. Research has found that this material achieved a 100% removal rate for Rh-B within 30 s over a temperature range of 15–55 °C and a pH range of 2–12. The removal rate remained at 72% even after reusing cycles. Furthermore, studies have also reported on the use of cellulose-TiO₂ in the field of antibiotics [189]. At present, researchers most commonly treat cellulose with alkali. However, large quantities of alkaline waste liquid are generated, which can easily cause secondary pollution of water bodies.

In addition to cellulose, chitosan is also a natural polysaccharide polymer. Its molecular surface contains abundant amino (-NH₂) and -OH, which has led to its widespread application in the field of pollutant removal [190,191]. Al-Mur [192] used chitosan to remove MO and MB. The removal efficiencies of MO and MB after 30 min were 98.80% (pH = 3, dosage = 0.5 g) and 90.65% (pH = 9, dosage = 1.0 g), respectively. The removal efficiency decreased by 17–18% after five cycles. Ait [193] developed DCPD-CS and DCPA-CS adsorbents through the combination of chitosan and calcium hydrogen phosphate. They were applied to remove TC. Research has found that their adsorption capacities reached 223.84 mg/g and 205.92 mg/g at pH = 11, respectively. With five desorption cycles of 500 mM (NH₄)₂HPO₄, the adsorption efficiency was maintained at 98%. By combining TiO₂ with chitosan, the recyclability of the material can be improved. Rosdiana [194] reported the preparation of TiO₂-chitosan microspheres for the removal of MB. After 5 min of exposure to light at pH = 11, the degradation rate of 2 ppm MB reached 92.74%, with $k = 0.0132 \text{ min}^{-1}$. However, acidic solvents and cross-linking agents are used in the preparation of composite materials. This may increase ecological risks to the environment.

Zeolites are a class of microporous materials. They are characterized by a large specific surface area, high chemical stability, and a negatively charged framework. These properties enable them to exhibit strong adsorption properties towards cationic pollutants [195]. They include both natural and synthetic zeolites. Alsuhybani [196] evaluated the potential of natural zeolites for removing MB. The removal efficiency for MB was 98.9% under optimal conditions (pH = 7, dosage = 0.01 g/L, T = 25 °C). After four adsorption-desorption cycles using 0.1 M NaOH, the zeolite's efficiency in removing MB decreased by around 13%. When zeolite is combined with TiO₂, not only is the dispersion of TiO₂ improved, but the photocatalytic and adsorption properties are also synergistically enhanced. Zhang [197] constructed a TiO₂/MoS₂@zeolite photocatalyst via ultrasound-assisted hydrothermal synthesis for the degradation of MO. The degradation rate of MO by the composite material was 95.00% after 60 min of photocatalytic degradation, with $k = 2.304 \text{ h}^{-1}$. After four cycles, the degradation rate still reached 90%. However, there are issues with the zeolite regeneration process. These involve high consumption of regenerant, which leads to increased production costs and difficulties in treating wastewater.

Both g-C₃N₄ and MOFs possess adsorption and photocatalytic properties. g-C₃N₄ is usually obtained by the high-temperature pyrolysis of thiourea or urea. g-C₃N₄ is a polymeric photocatalyst with a two-dimensional layered structure, a narrow bandgap, and good chemical stability [198]. When combined with TiO₂, it can construct heterojunctions for the degradation of pollutants. Vijayan [199] developed a Z-type heterojunction g-C₃N₄/TiO₂ photocatalyst via impregnation and used it to degrade MB. The results indicated that, after

120 min of light irradiation, the photocatalyst achieved a degradation rate of 94.44% for MB. The k was 0.023 min^{-1} . After four cycles, the degradation rate remained at 90%. Zhang [149] prepared a $\text{g-C}_3\text{N}_4/\text{TiO}_2/\text{WCT-AC}$ S-type heterojunction photocatalyst for the removal of PE. Under the optimal reaction conditions (time = 200 h, dosage = 50 mg, pH = 7), the material achieved a PE removal rate of 67.58%. $\text{g-C}_3\text{N}_4$ is regarded as an environmentally friendly photocatalyst. At present, few studies have explored its long-term impact on the ecological environment [200]. Therefore, further investigation is required.

Combining porous MOFs with TiO_2 promotes charge separation and broadens the photoresponse range. The composite can also absorb pollutants efficiently. This enhances the capacity for pollutant removal [201]. However, MOFs are prone to hydrolysis in aqueous environments. The structure of ligand molecules may be disrupted by water molecules, causing the framework to collapse. This is one of the key issues that limits their widespread application [202]. The preparation of MOFs relies on expensive metal salts and organic ligands [203]. During the synthesis process, some of the organic ligands (e.g., 2-methylimidazole) may be irritating and toxic to living organisms. It is difficult to ensure safe handling during subsequent processing [204]. Additionally, MOFs often contain metal ions at the center. In aquatic environments, this can result in the leaching of metal ions, posing a potential environmental risk.

Figure 12a shows an evaluation table based on quantitative data from relevant reports. Each indicator is scored on a scale of 0 to 5. A score of 5 represents the best performance, and 0 indicates the worst. The assessment criteria include raw material cost, chemical stability, recyclability, pollutant removal performance, and environmental impact. Based on Figure 12a,b, the TiO_2 -biochar-based composite exhibits the best performance.

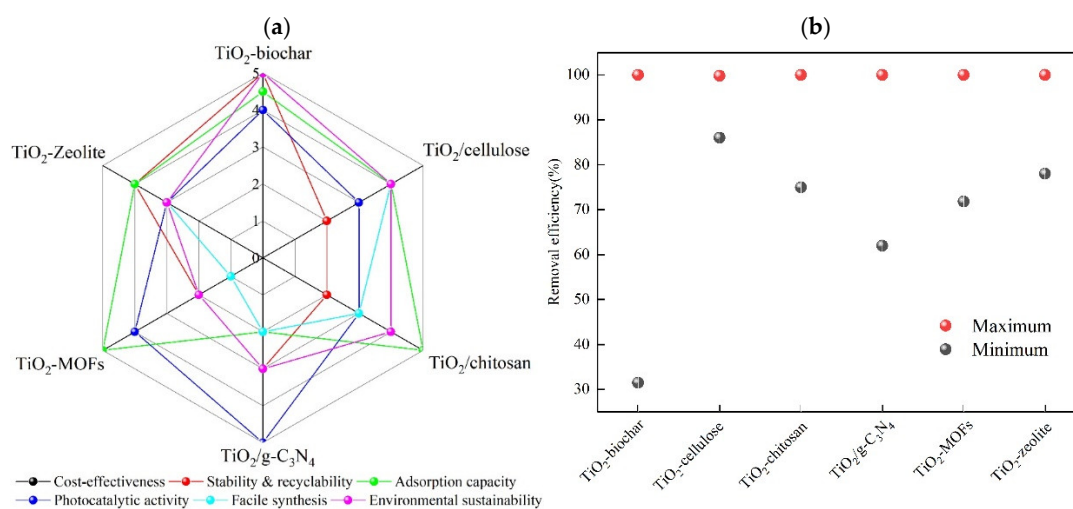


Figure 12. (a) Radar chart comparing the overall performance of different TiO_2 -based adsorption-photocatalysts (scores range from 1 to 5, with 5 representing the best performance), (b) comparison of adsorption-photocatalysts.

6. Current Limitations and Future Research Requirements

TiO_2 -biochar-based photocatalysts have been extensively studied for aquatic environmental remediation. A great deal of research has been conducted on the preparation, performance, and mechanisms of various photocatalysts. However, there remain limitations. These limitations include a lack of practical applications, photocatalyst stability, and challenges in meeting the requirements of actual wastewater treatment.

In terms of application scale, the adsorption-photocatalytic synergistic removal of pollutants remains at the laboratory stage. The preparation of high-performance photocatalysts is affected by many factors. These factors include the concentration of the tita-

nium source, the biomass ratio, the stirring rate, and the reactor size [205]. It is difficult to ensure stability and high catalytic activity during large-scale production. Meanwhile, the chemical reagents used in the preparation process are mostly expensive analytical-grade chemicals. This would increase manufacturing costs. Furthermore, due to restrictions in the preparation conditions, there may be variations in the properties of materials produced in different batches. This restricts the reproducibility of the material's preparation and its potential for industrial application.

TiO₂-biochar-based photocatalysts have demonstrated great potential for the removal of organic pollutants from water. However, photocatalytic reactions are usually conducted in aqueous systems under continuous stirring. During this process, the catalyst experiences constant collisions and friction, which may affect its stability and durability. A decrease in bond strength at the composite interface causes the TiO₂ to separate from the biochar matrix. This results in the biochar matrix polluting the aqueous solution a second time.

Current studies have largely focused on the photocatalytic degradation of individual pollutants. However, actual wastewater environments contain a variety of pollutants and natural organic matter. There may be competition among pollutants for the consumption of ROS [206,207]. This further reduces the activity of photocatalysts.

In addition, TiO₂-biochar-based photocatalysts are mostly in powder form. The process of photocatalytic degradation causes the substance to move easily in water, which is dangerous for the environment. This consequently may threaten marine ecosystems and human health.

Finally, the primary indicator currently used to evaluate photocatalytic degradation performance is pollutant removal efficiency. However, this parameter is significantly influenced by experimental conditions. These include the initial concentration of pollutants, the catalyst dosage, the type of light source, the irradiation time, the pH value, and the water matrix. It is insufficient to compare the intrinsic performance of different photocatalysts using this indicator alone. In the future, a multidimensional evaluation should be adopted. This approach should incorporate indicators such as contaminant removal efficiency, TOC mineralization rate, apparent rate constants, cycling stability, catalyst dosage-normalized activity, and quantum efficiency.

Consequently, future research should focus on six key areas. (1) Developing large-scale synthesis technologies. (2) Designing catalysts that exhibit high stability and catalytic activity. (3) Constructing large-scale reaction apparatus. (4) Improving solid-liquid separation technologies. (5) Conducting research on multiphase pollutants. (6) Formulating multi-dimensional evaluation criteria. The gradual advancement of photocatalytic systems towards practical application is being achieved.

7. Summary

This review summarizes the progress over the past decade in the in situ synthesis of TiO₂-biochar-based photocatalysts for the removal of organic pollutants. It outlines the preparation processes of composite materials and their pollutant removal performance. The review examines the influence of preparation parameters on morphological characteristics, as well as the role of morphology in regulating surface functional groups, ROS, and pollutant degradation efficiency. It reveals the intrinsic mechanisms linking preparation parameters, morphological characteristics, surface functional groups, ROS, and removal efficiency. This demonstrates that biochar enhances the potential of TiO₂-based materials for pollutant removal in water treatment systems.

An analysis of the relationship between preparation parameters and the microstructure of the photocatalyst reveals that crystal growth and the microstructure of TiO₂ can be effectively regulated by variations in titanium precursors. The use of titanium alcoholates generally results in a more uniform TiO₂ morphology. The development of "green"

sources of titanium and the implementation of the “treating waste with waste” principle still face numerous challenges. The biochar produced can be categorized into different types based on variations in carbon sources. These types include layered structures, honeycomb-like porous structures, and other complex architectures. Porous biochar produced using cellulose as a carbon source facilitates the loading and dispersion of TiO₂ nanoparticles. An optimal titanium-to-carbon mass ratio not only regulates the photore-sponse characteristics and electron transport pathways of the composite material but also effectively suppresses rapid electron–hole recombination. The preparation method and solvent influence the microstructure, particle dispersion, and pore structure of the photo-catalyst, thereby modulating its interfacial behaviors. The pyrolysis temperature and dop-ing modification strategies can induce confinement effects to a certain extent, thereby re-ducing the leaching of metal ions into water. On the other hand, these strategies regulate optical properties, thereby enhancing photodegradation performance. In summary, the morphology of TiO₂–biochar-based photocatalysts is influenced by the synergistic regula-tion of the preparation parameters.

The relationship between morphological characteristics and pollutant removal perfor-mance is investigated. The microstructure of TiO₂-biochar-based photocatalysts can be con-trolled, thereby regulating the distribution of oxygen- and nitrogen-containing surface func-tional groups. This regulation affects the formation of ROS. The photocatalyst achieves highly efficient degradation of dyes and antibiotics primarily mediated by •OH and •O₂^{•−}.

The electron transport mechanism can be influenced by the three distribution config-urations of TiO₂-biochar-based photocatalysts: surface-adhered, pore-embedded, and in-terlayer-distributed structures. In surface-adhered systems primarily rely on π -conju-gated structures as electron transport pathways. In pore-embedded and interlayer-distrib-uted systems, electrons are transferred to the carbon framework primarily via interfacial charge transfer. The morphology of TiO₂-biochar-based photocatalysts is influenced by the synthesis parameters. Furthermore, the structure influences the distribution of surface functional groups, the ability to generate ROS, and interfacial charge transfer behavior.

A comparison with other TiO₂-based adsorptive photocatalysts reveals that TiO₂-bi-ochar-based photocatalysts offer distinct advantages in terms of cost-effectiveness, envi-ronmental sustainability, and pollutant control. These photocatalysts demonstrate signif-icant potential for practical applications.

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