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Revue on TiO_2-carbonaceous base materials for phothodegradation of pollutants in wastewater.

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Abstract: This review examines the various types of carbon-based materials utilized as supports for the immobilization of titanium dioxide nanomaterial catalysts in the degradation of organic pollutants found in wastewater. Different supports, including biochar, activated carbon, graphene, and carbon nanotubes, were employed based on the immobilization techniques used, such as sol-gel, precipitation, chemical vapor deposition, hydrothermal, solvothermal, and co-precipitation. Each of these methods has its own set of advantages and disadvantages. The various hybrid materials or composites were characterized using techniques such as X-ray diffraction, transmission electron microscopy, scanning electron microscopy, and UV spectroscopy. The findings from different studies highlighted the limitations, challenges, and potential new approaches for the preparation of these materials.

Keywords: Carbons materials, TiO_2, composites, Photodegradation, Pollutants, Wastewater.

1. Introduction

The rapid increase in water pollution is a pressing issue, driven by population growth, intensive industrialization, and urban expansion. Various contaminants, such as paints, toxic solvents, fertilizers, oils, dyes, and pharmaceuticals, are polluting water and damaging the environment (Haounati *et al.,* 2020). Among these, dyes pose a particularly significant problem due to their extensive use in industries; they can have toxic, mutagenic, and carcinogenic effects and are challenging to remove (Ouachtak *et al.,* 2020).

The treatment of hospital wastewater is an important concern because of its harmful impact on ecosystems and the environment (Lakrat *et al.,* 2017;). The widespread use of antibiotics like penicillin, diclofenac, diphenhydramine, and amoxicillin in healthcare facilities creates complex and hazardous challenges for wastewater disposal. These antibiotics can introduce toxicity and contribute to bacterial resistance in natural water bodies (Li *et al.,* 2019; Karimi-Maleh et al., 2021a, 2021b; Bergamonti *et al.,* 2019; Doan *et al.,* 2021). Furthermore, wastewater treatment plants (WWTPs) are not equipped to manage pharmaceutical residues, resulting in their release into sewage systems (Maryam *et al.,* 2020). Therefore, it is essential to address pharmaceutical waste in order to safeguard aquatic ecosystems.

Traditional methods for wastewater treatment, such as chemical precipitation, filtration, ion exchange, membrane technologies, and adsorption (El Haouti *et al.,* 2019), come with significant drawbacks and limitations. These include high operational costs, inefficiency in handling low concentrations of pollutants, poor selectivity, and the risk of releasing secondary toxic contaminants into the environment, which poses substantial threats to both human health and the ecosystem (Burakov *et al.,* 2018). Consequently, there is a need for highly efficient, cost-effective, and multifunctional approaches to effectively remove these pollutants from wastewater (Jalbani *et al.,* 2021; Hajipour *et al.,* 2021; Karimi-Maleh *et al.,* 2020a).

Recently, semiconductors such as TiO_2, ZnO, SnO_2, $SrTiO_3$, and Fe_2O_3 have become prominent as efficient photocatalysts (Zhang *et al.,* 2015). For a photocatalyst to be effective, it should be active, non-toxic, insoluble in water, and capable of utilizing visible and near-UV light (Abéga *et al.,* 2019).

Despite its potential, using TiO_2 alone with solar energy presents challenges. Its limited adsorption capacity and the dependency on UV radiation for activation restrict its effectiveness, primarily due to its wide band gap (Eg = 3.2 eV). Moreover, TiO_2 materials face challenges with low quantum yields caused by high recombination rates of photo-induced electron-hole pairs, which diminishes their efficiency under solar light (Hosseini-Monfared *et al.,* 2021). They also demonstrate poor separation efficiency of photocarriers, resulting in decreased photocatalytic activity. Doping has been recognized as an effective strategy to enhance light absorption and photoactivation of TiO_2 in the visible light spectrum, thereby improving the efficiency of solar energy utilization.
Composites are materials created by combining two or more parent substances. Numerous studies have investigated composites formed by merging carbonaceous materials with TiO_2, revealing changes in their band gap and pollutant removal efficiency. Biochar, a carbon-rich material produced through the pyrolysis of plant or animal biomass in an inert environment, is characterized by a high abundance of oxygen functional groups, an aromatic surface, and a large surface area with significant porosity due to its high specific surface area and rich functional groups (Tan *et al.,* 2016). Carbonaceous materials serve as excellent adsorbents for extracting organic pollutants from water. This review focuses on research concerning TiO_2 based composites combined with carbonaceous materials for the photodegradation of pollutants in wastewater. Its purpose is to provide insights for researchers on existing studies in this area and to encourage innovation in the development of new materials or synthesis techniques.

1. Types of Carbonaceous Materials

There are various types of activated carbonaceous materials employed in composite synthesis, with the most common being activated carbon and biochar.

1.1 Activated Carbon

Activated carbon is a fine, black, amorphous powder derived from any material that has a high carbon content and a low presence of inorganic materials. Suitable raw materials include wood, bark, coconut shells, peanut shells, olive pits, coal, peat, lignite, rice husks, as well as synthetic polymers or petroleum byproducts (Ould-Ifriss *et al.,* 2011).

Activated carbon encompasses a variety of carbonized materials characterized by a high degree of porosity and surface area. It has numerous applications in environmental and industrial fields for the removal, recovery, separation, and modification of various compounds in both liquid and gas phases. The choice of chemical activating agent is a crucial factor that affects the performance and suitability of activated carbon. It is produced through the carbonization of biomass, followed by an activation process that differentiates it from biochar, which does not undergo such activation (Delannoy et al., 2018). There are two primary activation methods: physical and chemical (Suhas *et al.,* 2016). Due to its microporous structure, activated carbon can obstruct larger organic dye molecules (Hao et al., 2018). Additionally, activated carbon tends to be more expensive than biochar because it requires further processing to activate the carbonized materials (Delannoy *et al.,* 2018).

1.2 Biochar

Biochar is a carbon-rich solid produced from the thermal treatment of biomass in an environment with little to no oxygen (Dai *et al.,* 2019). It features a large specific surface area, a highly porous structure, and functional groups such as carbonyl and hydroxyl, which make it an effective adsorbent for removing various contaminants from wastewater (Mayakaduwa *et al.,* 2015). The contaminants include organic dyes, heavy metals, and pharmaceuticals (Mayakaduwa *et al.,* 2015). Consequently, biochar is increasingly used in the textile industry to eliminate organic dyes because of its cost-effectiveness and environmentally friendly properties (Mui *et al.,* 2010). Typically, biochar is produced through slow pyrolysis at temperatures ranging from 200 to 700 °C with a heating rate of 10 °C/min (Li *et al.,* 2014). The fast pyrolysis method is unsuitable for biochar production due to its high temperature, rapid heating rate, and short residence time, which favor the formation of biofuels instead of biochar (Elkhalifa *et al.,* 2019).

Thus, temperature and residence time are critical factors influencing biochar yield during pyrolysis. Inadequate conditions can lead to the gasification of biochar, which generates gaseous fuels and results in decreased biochar production (Kong *et al.,* 2014).

1.3 Carbon Nanotubes

Carbon nanotubes (CNTs) typically possess a high specific surface area to mass ratio (ranging from 75 to 1020 $m^2\ g^{-1}$) and exhibit exceptional sorption capabilities (Czech and Buda, 2016). Unlike activated carbon and biochar, CNTs tend to aggregate due to hydrophobic interactions, van der Waals forces, and π-π stacking, creating four distinct types of adsorption sites: inner cavities, interstitial channels, external grooves, and outer surfaces (Apul and Karanfil, 2015). CNTs offer many advantages, including a large electron-storage capacity, superior metallic conductivity, and the ability to absorb light across a wide range of wavelengths (Woan *et al.,* 2009). Overall, CNTs are a promising option as support materials to enhance photocatalytic activity due to their remarkable characteristics.

1.4 Graphene

Graphene is recognized as a two-dimensional sheet of carbon atoms linked by sp^2 bonds, forming an aromatic π electron system (Novoselov *et al.,* 2004). It features a higher density of potential adsorption sites compared to CNTs, owing to its open-plane structure, which includes three primary types of adsorption sites: 1) open surfaces, 2) longitudinally parallel external surfaces, and 3) interstitial channels (Ersan *et al.,* 2017). Additionally, graphene possesses numerous unique properties, such as high electron mobility, exceptional mechanical strength, high thermal conductivity, and a large specific surface area (Hu *et al.,* 2013).

2. Metal Oxide Semiconductors

Numerous researchers have explored the application of metal oxide semiconductors in heterogeneous photocatalysis for the removal of persistent organic dyes. Among the various metal oxides utilized in this process are titanium dioxide (TiO_2), zinc oxide (ZnO), tungsten trioxide (WO_3), tin dioxide (SnO_2), iron (III) oxide (Fe_2O_3), and others. Several criteria are considered when selecting semiconductors for photocatalysis, including their catalytic activity, which depends on properties such as band gap energy, surface morphology, surface area, and pore volume (King Saud University, 2015).

The band gap energy of metal oxides is a crucial property in photocatalysis, as it represents the minimum energy required to excite electrons (Gomis-Berenguer *et al.,* 2016). The promotion of electrons from the valence band (VB) to the conduction band (CB) can only occur if the energy of photons from ultraviolet (UV) irradiation or visible light is equal to or greater than the band gap energy of the specific metal oxide. Additionally, band gap energy influences the recombination rate of electron-hole pairs, which can reduce the effectiveness of the photocatalytic reaction between radicals and organic dyes, consequently diminishing photocatalytic activity (Gomis-Berenguer *et al.,* 2016).

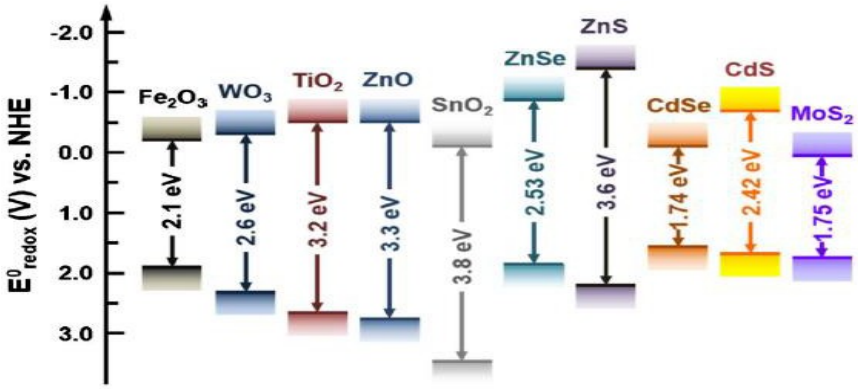


Figure 1: Band Gap Energy of Some Metal Oxide Semiconductors on Potential Scale (V) against Normal Hydrogen Electrode (NHE) (Lee, Jung and Kang, 2017).

Titanium Dioxide

Titanium dioxide (TiO_2) is the most widely utilized metal oxide photocatalyst, thanks to its abundant availability, affordability, low toxicity, photocatalytic efficiency, environmental friendliness, chemical inertness, wide band gap, and resistance to corrosion (Antonopoulou *et al.,* 2017; Zhao *et al*., 2018a; Abouri *et al*., 2024).

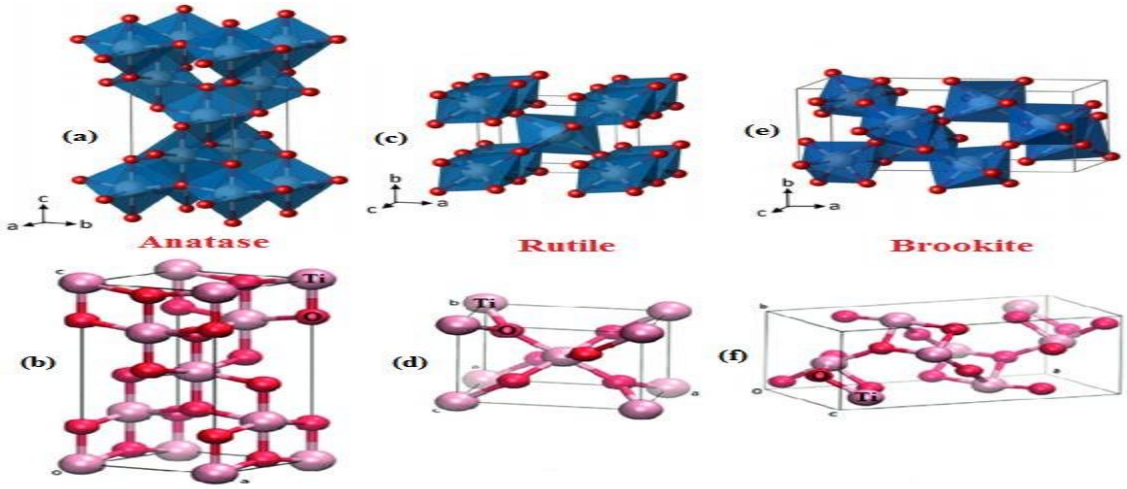


Figure 2: Differences crystalline forms of TiO_2 anatase (a, b), rutile (c, d), brookite (e, f)

Among the three crystalline structures of titanium dioxide, anatase TiO_2 is regarded as the most suitable candidate for photocatalytic degradation (Allen *et al.,* 2018; Elafia *et al.,* 2023). Although anatase has a larger energy band gap than rutile, which limits its light absorption capacity, it possesses a higher electron oxidation potential and facilitates the transfer of electrons to adsorbed molecules from TiO_2 (Zangeneh et al., 2014). However, the relatively low solar photon conversion efficiency due to TiO_2's large band gap energy remains a significant challenge in photocatalytic reactions. Specifically, the band gap energies of anatase and rutile are 3.2 eV and 3.0 eV, respectively (Colmenares, Varma, and Lisowski, 2016). This indicates that only UV light with a wavelength shorter than 380 nm can excite electrons from the valence band to the conduction band, which constitutes less than 5% of the solar energy available (Colmenares, Varma, and Lisowski, 2016). In contrast, Cheng et al. (2015) noted that visible light comprises 46% of the solar spectrum.

To enhance visible light absorption, various methods, including doping with different materials such as metals (e.g., Cu, Fe) and non-metals (e.g., carbon, sulfur, nitrogen, phosphorus, boron, etc.), have been employed (Marschall and Wang, 2013; Dong et al., 2015). Metal-doped TiO_2 exhibits lower band gap energy compared to pure TiO_2, which plays a crucial role in improving photocatalytic degradation efficiency. Additionally, non-metals introduce impurity states that are closer to the valence band edge of TiO_2, which can lead to charge carrier recombination (Dong et al., 2015). Just as with metal doping, non-metal doping of TiO_2 shifts the absorption range from the UV spectrum to visible light, resulting in a reduction of band gap energy that enhances catalytic performance.

3. Different Synthetic Methods

There are various methods for synthesizing carbon-based composite materials, including sol-gel, hydrothermal, solvothermal, co-precipitation, and ultrasonic techniques.

3.1 Sol-Gel Method

The sol-gel method is comparatively low-cost and allows for the manipulation of various sizes and shapes of the resulting morphology (Shan *et al.,* 2010). This method involves the transformation of a ceramic or inorganic polymer solution from a liquid precursor to a sol, and eventually into a gel network structure (Danks *et al.,* 2016). According to Danks *et al.* (2016), metal alkoxide precursors undergo hydrolysis and condensation during sol formation. Examples of such precursors include

titanium alkoxide, titanium tetrachloride, tetrabutyl titanate, and titanium tetra-isopropoxide. The gel is then calcined at high temperatures to create oxygen bridges between the hydroxyl groups of the carbon material and TiO_2 (Shan *et al.,* 2010), thus enhancing the adhesion between the two components. The sol-gel method has been successfully applied to fabricate cellulose biochar/TiO_2 composites using tetrabutyl titanate as a precursor, as reported by Zhang *et al.* (2018) and Zhang *et al.* (2017). Additionally, Abéga *et al*. (2019) prepared a composite using carbon nanotubes and titanium(III) chloride ($TiCl_3$) via the sol-gel method, demonstrating a good distribution of TiO_2 particles on the surface of the nanotubes.

3.2 Hydrothermal Method

The hydrothermal method employs water as a reaction medium to synthesize TiO_2 composites under a temperature-controlled environment (Tan *et al.,* 2011). An autoclave is typically used in this method to provide controlled temperature and pressure conditions (Tan *et al.,* 2011). Alhaji *et al.* (2017) emphasized that temperature is a critical factor in the hydrothermal process, as it influences the immobilization of TiO_2 on the surface of the biochar.

3.3 Chemical Vapor Deposition Method (CVD)

The Chemical Vapor Deposition (CVD) process typically involves injecting single or multi-component volatile gaseous precursors onto a support at high temperature and pressure within a vacuum chamber (Alhaji *et al.,* 2017). When these gaseous precursors decompose, thin films of material are deposited on the surface of TiO2 (Alhaji et al., 2017). One of the key advantages of CVD is that TiO_2 can be deposited onto biochar regardless of the substrate's shape or size within a short time frame (Shan et al., 2010). Additionally, CVD provides high thermal stability and strong adhesion between biochar and TiO2 (El-Sheikh *et al.,* 2004). Various types of CVD techniques exist, including Metal Organic Chemical Vapor Deposition (MOCVD), Atmospheric Pressure Chemical Vapor Deposition (APCVD), and Hybrid Physical Chemical Vapor Deposition (HPCVD). For example, Lu et al. (2018) successfully prepared a TiO_2/biochar composite using this method.

3.4 Solvothermal Method

The solvothermal method allows for the control of crystal structure, size, and surface morphology of the composite. It generally requires lower calcination temperatures compared to the hydrothermal process; however, there are some drawbacks associated with this method. The use of solvents such as ethanol or methanol, high calcination temperatures and pressures, extended synthesis times, and the necessity for highly concentrated NaOH (basic conditions) can limit its practicality (Zhou *et al.,* 2010).

3.5 Co-Precipitation Method

The co-precipitation method is a widely used technique in chemistry for the simultaneous precipitation of multiple compounds from a solution. This method is both convenient and cost-effective for preparing various materials, including nanostructures like TiO_2 and magnetite nanoparticles. In co-precipitation, substances that would typically be soluble under certain conditions are induced to precipitate. While this approach offers advantages such as high product purity, high yield, and scalability for industrial production, it also has some downsides, including limited control over particle size, potential impurities in the final product, and the need for complex washing steps.

3.6 Ultrasonic Method

Ultrasonic methods can be employed to prepare a variety of composites, including diamond/Cu composites, carbon nanotube-graphene/polydimethylsiloxane (PDMS) composites, and polymer concrete composites. This approach utilizes high-frequency sound waves to consolidate and process materials, leading to enhanced properties and performance.

3.7 Impregnation Method

The impregnation method offers several advantages, including the ability to achieve a uniform coating of TiO_2 on the surfaces of carbonaceous materials, as well as control over the crystal structure and surface morphology of the composite. Additionally, it is a continuous process that is relatively easy to scale up for industrial applications. However, this method also has limitations, such as the requirement for high calcination temperatures and prolonged synthesis times (Gar Alalm *et al.,* 2016; Xu *et* al., 2010).

Table 1 : Comparison of Common Carbonaceous-TiO_2 Composite Synthesis Techniques (Awfa *et al.,* 2021)

Synthesis Method	Advantages	Disadvantages
Sol-Gel	- Provides homogeneous distribution and strong chemical bonding of TiO_2 on carbonaceous substrates. - Allows control over process stoichiometry. - Yields high purity nano-sized crystallized TiO_2 powder.	- Requires high calcination temperature. - Involves high consumption of various costly reagents. - Has a long synthesis time.
Impregnation	- Achieves uniform TiO_2 coating on carbonaceous surfaces. - Facilitates control over crystal structure and surface morphology of the composite. - Supports continuous processes that are relatively simple for industrial scaling.	- Involves high calcination temperatures. - Requires long synthesis times.
Ultrasonic	- Offers higher penetrating power, allowing detection of extremely small flaws. - Provides greater sensitivity and accuracy than other non-destructive testing techniques.	- Limited access to the surface being tested. - Difficult to inspect certain materials like cast iron.

Synthesis Method	Advantages	Disadvantages
Co-Precipitation	- Results in high product purity and yield. - Eliminates the need for organic solvents - Scalable for industrial production.	- Poor control over particle size. - Limited product purity. - Challenges in achieving uniform size distribution. - Requires complex washing steps.
Solvothermal	- Allows control over crystal structure, size, and surface morphology of the composite. - Has lower calcination temperatures than hydrothermal methods.	- Utilizes ethanol or methanol as solvents. - Requires high calcination temperature and pressure. - Long synthesis times. - Needs highly concentrated NaOH (basic conditions) during synthesis.
Chemical Vapor Deposition (CVD)	- Enables simultaneous reduction and activation of the catalyst - Supports continuous processes suitable for industrial applications.	- Requires high temperature and pressure conditions. - Necessitates supply of high-purity inert gas - Produces gaseous by-products during the process.
Hydrothermal	- Allows control over crystal structure, size, and surface morphology of the composite. - Uses water as a solvent.	- Involves high calcination temperatures and pressures. - Has long synthesis times. - Requires highly concentrated NaOH (basic conditions) during synthesis.
Dip-Coating	- A technique for generating thin film layers of composites.	- Requires high calcination temperatures - Consumes various costly reagents. - Long synthesis times. - Exhibits lower photocatalytic activity compared to powder composites.

Synthesis Method	Advantages	Disadvantages
Pyrolysis	- Yields high purity nano-sized powder composites. - Allows control over stoichiometry in multi-component systems. - Supports continuous processes that are relatively simple for industrial scaling.	- Requires high energy input to generate flame in the combustion chamber. - Necessitates high temperature conditions.

4. TiO2-Non-Metal-Based Composites

Numerous studies have investigated the photodegradation of pollutants using TiO_2-non-metal-based composites. This research will specifically focus on carbonaceous materials such as activated carbon (AC), biochar, carbon nanotubes (CNTs), and graphene, which are the primary carbonaceous materials tested for water and wastewater treatment (Santhosh *et al.,* 2016). These materials are characterized by their large specific surface areas and high charge carrier mobility.

Due to their low cost, stability, high porosity, and functional groups, along with ease of separation, carbonaceous materials are excellent candidates for TiO_2 doping. Incorporating biochar with TiO_2 particles can enhance the availability of electrons in the conduction band, facilitating the generation of both oxygen and hydroxyl radicals through reduction and oxidation processes, respectively. The presence of free electrons on the surface of the carbon material allows for the formation of a carbon material-TiO_2 hybrid composite, providing a synergistic effect that combines adsorption and photocatalysis to effectively degrade emerging pollutants.

In 2019, Lu and colleagues conducted a study on the photodegradation of methyl orange using a TiO_2 (P25)/biochar (walnut) composite, prepared via the impregnation method. Their findings demonstrated an improvement in the removal efficiency of methyl orange, indicating an increase in photocatalytic activity compared to TiO_2 P25 alone. Scanning electron microscopy (SEM) results revealed the agglomeration of TiO_2 particles on the surface of the biochar.

Similarly, Lazarotto *et al.* (2020) synthesized biochar-TiO_2 composites using biochar derived from coffee waste and TiO_2 through impregnation and carbonization processes. They employed a 1:1 and 2:1 ratio of biochar to TiO_2 at a temperature of 650 °C for one hour. The characterization of the obtained composites included various methods such as SEM, which confirmed the presence of TiO_2 particles on the biochar surface. Moreover, their study observed a significant reduction in the band gap from 3.37 eV to 1.75 eV. The incorporation of biochar into the TiO_2 matrix notably enhanced the efficiency in removing diclofenac. Additionally, biochar contributed to reducing the recombination of photogenerated electron-hole pairs. Acting as an electron donor, biochar facilitated the movement of electrons away from the hole of the e^-/h^+ pairs, thus preventing their recombination and increasing photocatalytic efficiency.

Furthermore, Wang *et al.* (2020) focused on synthesizing a composite made from biochar (derived from corn stalks carbonized at 773 K for three hours) and TiO_2 (using butyl titanium) through the carbonization impregnation method. The resulting composite was characterized using several

techniques, including SEM. Their analysis indicated that the high carbonization temperature adversely affected the photocatalytic efficiency of the material. Specifically, at elevated temperatures, the internal walls of the carbon material crumbled and blocked the pores, consequently reducing the effectiveness of the composite (see Figure 3). In 2020, Ahmed et al. synthesized TiO_2-biochar hybrid nanocomposites aimed at removing 3,4-Dimethylaniline. The composites were produced via the sol-gel method using tetrabutyl titanium as a precursor. Scanning Electron Microscopy (SEM) images revealed small TiO_2 grains and agglomerations that were uniformly distributed across the surface of the composites. When compared to the SEM images of the biochar alone, it was evident that TiO_2 had been successfully immobilized on the biochar surface.

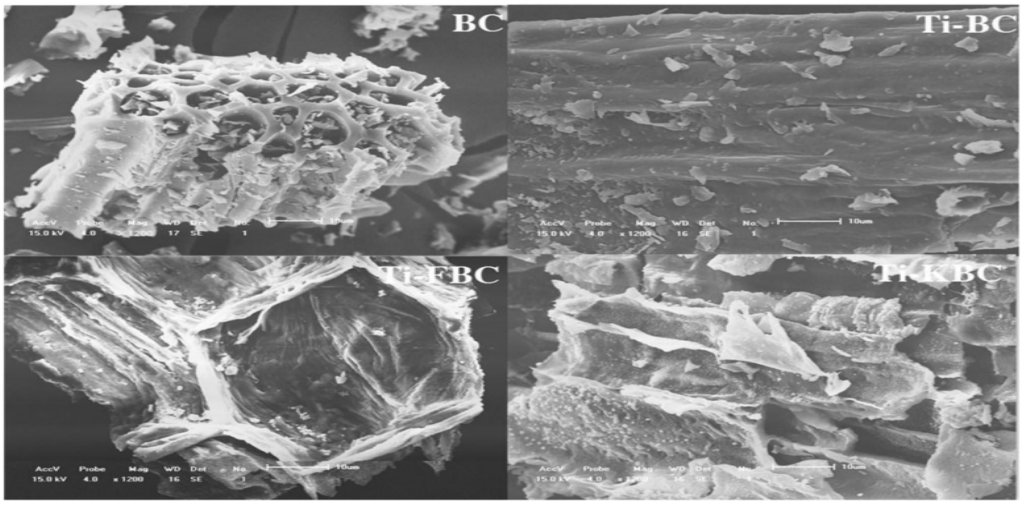


Figure 3: Scanning Electron Microscopy (SEM) images of biochar (BC), TiO_2-biochar composite (Ti-BC), fine TiO_2-biochar composite (Ti-FBC), and potassium-treated TiO_2-biochar composite (Ti-KBC), magnified 1200 times (Lazarotto *et al.*, 2020).

Additionally, Brunauer-Emmett-Teller (BET) analysis indicated a reduction in specific surface area, decreasing from 979.5 m^2/g for the biochar to 767 m^2/g for the composite. This reduction is attributed to the precipitation of TiO_2, which occupies a portion of the mesopores and micropores. Nevertheless, the composite exhibited enhanced adsorption capacity due to a more uniform distribution of adsorption sites and improved pore size compared to the biochar.

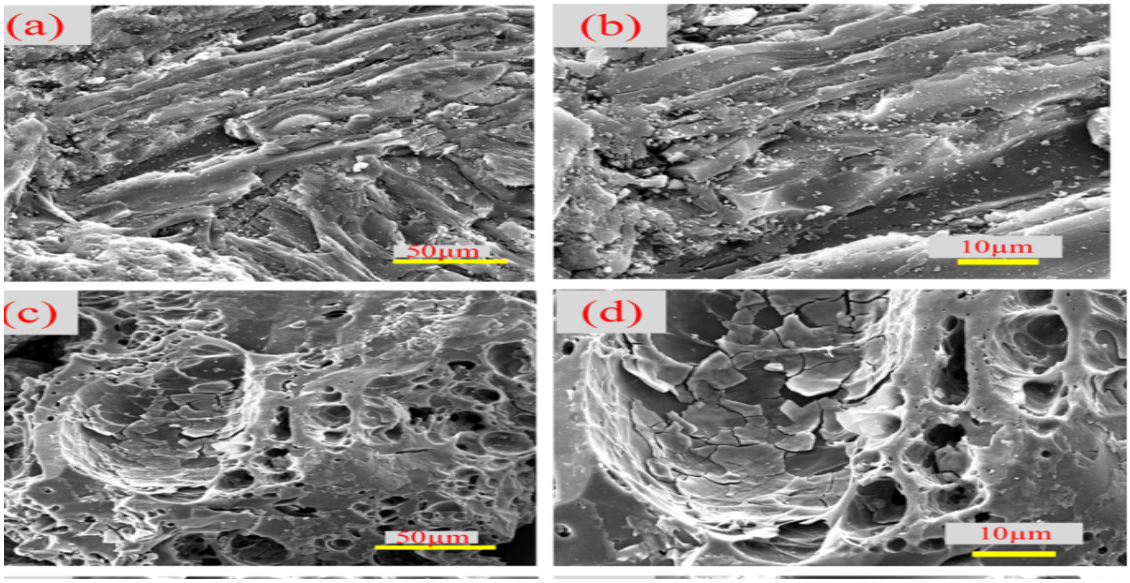


Figure 4: Scanning Electron Microscopy (SEM) images illustrating (a, b) biochar and (c, d) TiO_2-biochar composite, as reported by Ahmed et al. (2020).

In 2017, Lisowski and colleagues demonstrated that composites derived from granulated miscanthus straw with TiO_2—using titanium (IV) isopropoxide—and similar composites from softwood straw could be synthesized effectively through ultrasound-assisted methods. The SEM analysis of these composites showed irregular, plate-like structures, which are believed to enhance photocatalytic efficiency by providing increased active surface areas.

Furthermore, Wang *et al.* (2020) conducted a study on the preparation of TiO_2-modified biochar aimed at the photodegradation of Enrofloxacin. They employed an impregnation technique to synthesize the composites and characterized them using multiple analytical methods. The BET results indicated a significant increase in surface area: by 4.34, 10.43, and 11.52 times for TiO_2-biochar (Ti-BC), TiO2-ironized biochar (Ti-FBC), and TiO_2-alkaline biochar (Ti-KBC), respectively. SEM observations revealed that the biochar surface contained some impurities, with debris particles occupying many of its pores. Notably, after high-temperature pyrolysis, the internal walls of the biochar collapsed, causing debris to fall inside the pores and resulting in blockages, which could hinder its overall performance.

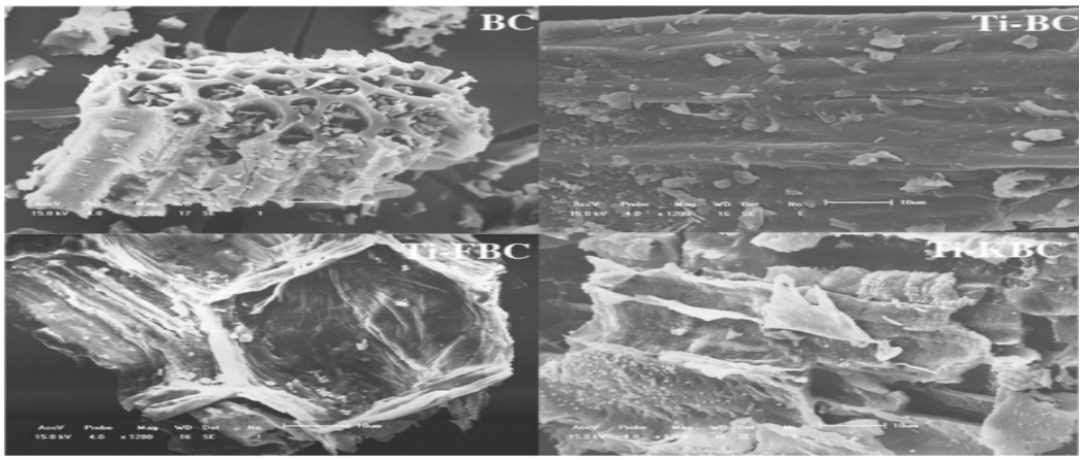


Figure 5: Scanning Electron Micrograph (SEM) images of biochar (BC), Ti-BC, Ti-FBC, and Ti-KBC at 1200× magnification.

In addition to SEM, various analytical techniques such as Energy Dispersive X-ray Spectroscopy (EDS), Fourier Transform Infrared Spectroscopy (FT-IR), X-ray Diffraction (XRD), and X-ray Photoelectron Spectroscopy (XPS) confirmed that TiO_2 was successfully supported on the biochar surface in its anatase phase. The UV-vis Diffuse Reflectance Spectroscopy (DRS) results demonstrated that the Ti-KBC composite had the narrowest bandgap, correlating with the highest catalytic activity. Reusability testing revealed that after five cycles, the Ti-KBC composite retained a degradation rate of 77.14% for enrofloxacin, outperforming biochar (BC), Ti-BC, and Ti-FBC.

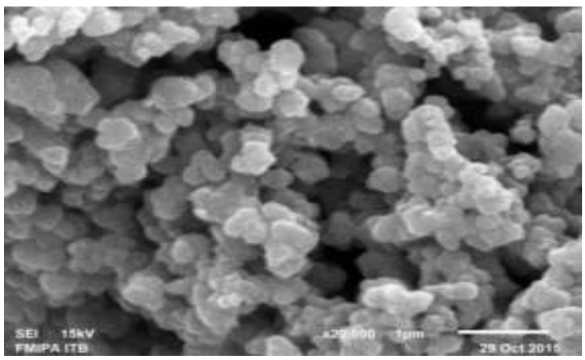
Fazal *et al.* (2019) prepared a hybrid biochar-TiO_2 composite (BCT) through a wet precipitation technique. This composite was characterized by several methods—including XRD, SEM, BET surface area analysis, FT-IR, and UV-Vis spectroscopy—to evaluate its crystallinity, morphology, and functional groups. The results suggested that the hybrid displayed superior charge separation, with reduced electron-hole recombination, and enhanced light absorption compared to pure TiO_2 and biochar alone. Notably, the optimized hybrid achieved a 99.20% photodegradation efficiency for dye-laden wastewater, significantly higher than the 85.20% and 42.60% efficiencies observed for pure

biochar and TiO_2, respectively. They concluded that this hybrid material holds promise for industrial wastewater treatment (Wang *et al.,* 2020).

4.2 TiO_2-Activated Carbon Composites

Activated carbon, widely used as a support material in photocatalysis, has been extensively studied in conjunction with TiO_2 and other catalysts. Here, we review some notable research on TiO_2-activated carbon composites designed for photodegradation applications.

Sheilatina et al. (2021) investigated the synthesis and photocatalytic activity of TiO_2 supported on coconut shell-based activated carbon. The activated carbon was prepared via coconut shell activation, and the composite was produced using the sol-gel method, with titanium tetraisopropoxide serving as the TiO_2 precursor. The composite's characterization included XRD, FT-IR, SEM coupled with EDX, and surface area measurement via BET analysis. SEM and EDX investigations revealed an uneven distribution and shape of TiO_2 particles on the activated carbon, with the particles forming spherical clusters exhibiting some agglomeration (see figure). The EDX analysis indicated that the composite contained approximately 15.81% carbon and 85.19% titanium. Photodegradation experiments using UV light to degrade indigo carmine dye demonstrated the photocatalytic potential



of the TiO_2/activated carbon composite.

Figure 5: Surface morphology of TiO_2/activated carbon observed at 20,000× magnification.

The FTIR spectrum of the TiO_2/activated carbon composite did not display an absorbance band between 1200-1600 cm^{-1}, which is typically associated with Ti–O–C bonds. This absence suggests that the interaction between TiO_2 and activated carbon in this photocatalyst is primarily through physical adhesion rather than chemical bonding. BET analysis showed that the surface area (S_BET) of the composite was slightly smaller than that of pure activated carbon, measuring 286.36 m^2/g compared to 291.61 m^2/g. This reduction is likely due to the impregnation process, where TiO_2 particles occupy the pores of the activated carbon, leading to pore blockage.

The photocatalytic performance was notably effective, with the highest degradation percentage (91.79%) achieved at pH 3 and an initial dye concentration of 5 mg/L after 25 minutes of irradiation with sunlight, targeting indigo carmine dye. Interestingly, TiO_2/activated carbon demonstrated higher photocatalytic activity under sunlight compared to UV light, highlighting its potential for solar-driven applications (Sheilatina et al., 2021).

In another study, Anh et al. (2022) prepared TiO_2-activated carbon (AC) composites via a sol-gel method, using $TiCl_4$ as the TiO_2 source and commercially available activated carbon as support. Material characterization included SEM, EDX, FT-IR, XRD, and diffuse reflectance spectroscopy.

SEM images confirmed successful loading of TiO_2 onto activated carbon, with many TiO_2 particles filling the pores and embedding into the cavities rather than merely coating the surface (see figure 6). UV-Vis diffuse reflectance spectra showed an absorption edge shift: TiO_2 alone at about 370 nm, while TiO_2–AC extended to around 420 nm, indicating a red-shift into the visible region, which enhances visible-light activity. EDX analysis revealed that the composite contained approximately 49.32% carbon, 35.26% oxygen, and 15.42% titanium. The bandgap energy of TiO_2–AC was calculated at 2.61 eV, lower than pure TiO_2's 3.15 eV, further supporting improved visible-light absorption. Photoelectrochemical (PEC) tests with TiO_2 and TiO_2–AC-based photoanodes demonstrated that the composite exhibited superior photocatalytic activity, evidenced by higher current density and more efficient phenol degradation compared to the pure TiO_2 electrode.

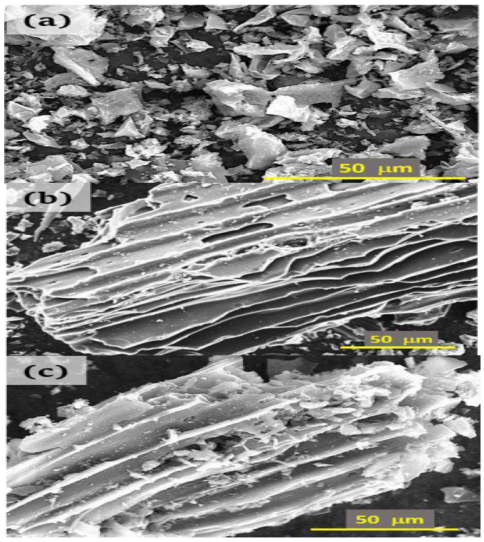


Figure 6: Scanning Electron Microscopy (SEM) images of: (a) TiO_2 (titanium dioxide), (b) AC (activated carbon), and (c) the TiO_2–AC (titanium dioxide–activated carbon) composite, as reported by Anh et al. (2022)

In 2018, Noorimotlagh et al. investigated the removal of Nonylphenol (NP), a non-biodegradable compound harmful to the endocrine system, using a visible-light-active carbon-doped TiO_2 (CDT). This TiO_2 was supported on the surface of granular activated carbon (GAC), with an anatase/rutile (A/R) ratio tailored to optimize photocatalytic activity. The composite was prepared via the sol-gel method, employing Titanium (IV) isopropoxide as the precursor. The physicochemical properties of the resulting material were thoroughly characterized using techniques such as X-ray diffraction (XRD), SEM-EDX, transmission electron microscopy (TEM), BET surface area analysis, and UV–Vis spectroscopy. SEM images confirmed the presence of TiO_2 particles dispersed on the GAC surface, while EDX analysis evidenced the incorporation of carbon into the TiO_2 lattice. The A/R ratio of approximately 53.06/46.94 was identified as beneficial for enhancing visible-light absorption, which was corroborated by optical measurements showing a reduction in the band gap from 3.17 eV to 2.72 eV due to carbon doping. Furthermore, increasing the calcination temperature from 475°C to 600°C lowered the band gap marginally to 2.66 eV, suggesting improved light utilization (Noorimotlagh et al., 2018).

Continuing this vein of research, Radhi et al. (2023) synthesized TiO_2-loaded activated carbon nanoparticles with two different TiO_2 loadings, aiming to use these composites for deep desulfurization of dibenzothiophene (DBT) in fuel. The composites were prepared via the sol-gel

method using TTPS as the titanium source. Characterization through XRD, BET, SEM, and EDX revealed that the resulting material had an average particle size of approximately 48.9 nm and a remarkably high surface area of around 710.12 m^2/g. SEM images showed non-uniform crystal distribution with surface agglomerates on the activated carbon, indicative of nanoparticle clustering. A TiO_2 content of 6.33 wt.% was achieved, leading to a sulfur removal efficiency exceeding 99% at an initial sulfur level of 100 ppm, with a maximum sulfur capacity of 24.08 mg S/g adsorbent, demonstrating promising potential for fuel purification (Radhi et al., 2023).

In another approach, Azhar et al. (2018) developed TiO_2/AC composites functionalized with specific groups to act as pollution degradation centers. The synthesis involved first preparing nano-sized TiO_2 via a microemulsion method, followed by depositing it onto functionalized activated carbon through impregnation. The structural and surface properties were characterized using techniques such as thermogravimetric analysis (TGA), BET surface analysis, and field emission SEM (FESEM). BET results indicated mesoporosity, especially at P/Po ratios between 0.6 and 1.0, while the hysteresis loop suggested nanoparticle aggregation, leading to reduced porosity. FESEM images revealed that the activated carbon surface was porous and sponge-like, whereas the TiO_2 particles formed uniform spheres with sizes between 15 and 18 nm. The small particle size and high surface area observed in this synthesis rated it superior to commercial TiO_2 P25. Notably, increasing the support material (AC) ratio improved degradation rates of the target molecule, bmimCl, with the 10 wt.% TiO_2/AC composite achieving an overall removal efficiency of 18.47%. Excessive support, however, curtailed the removal efficiency (Azhar et al., 2018).

In summary, these studies highlight diverse synthesis strategies and extensive characterizations aiming to optimize TiO_2-based composites for environmental remediation, particularly through improved light absorption, surface area, and pollutant interaction.

In 2014, Rajamanickam et al. synthesized a TiO_2–CAC (carbon-activated carbón) composite using a sol-gel method, employing commercial activated carbon (CAC) and titanium isopropoxide as the titanium source. The composite was prepared with various ratios of CAC to TiO_2 to optimize its properties. The catalysts were thoroughly characterized using several techniques, including X-ray diffraction (XRD), high-resolution scanning electron microscopy (HR-SEM), energy dispersive spectra (EDS), diffuse reflectance spectra (DRS), photoluminescence spectra (PL), X-ray photoelectron spectroscopy (XPS), and Brunauer–Emmett–Teller (BET) surface area analysis. The XRD results revealed that the TiO_2 particles appeared well-defined and spherical, while the TiO_2/CAC composites showed very small, uniform spherical structures dispersed over a smooth, homogeneous background, although some agglomeration was observed. Such structures are particularly useful in catalysis due to their high specific surface area.

EDS analysis confirmed the presence of titanium, oxygen, and carbon in the composites. The photocatalytic activity of these TiO_2/CAC composites was tested for degrading Sunset Yellow (SY) dye in aqueous solutions under UV-A light. The composites demonstrated higher efficiency compared to pure TiO_2 and TiO_2-P25 at pH 7, particularly in mineralizing the dye. The enhanced activity was attributed to the synergistic effect between TiO_2 and CAC, as well as the increased surface area provided by CAC. The study also investigated the effects of operational variables such as catalyst amount, dye concentration, and initial pH on the photocatalytic process. Maximum dye removal was observed at pH 7, with degradation efficiency decreasing from 86.7% to 37.4% over 60 minutes as

dye concentration increased from 3 × 10^{-4} M to 8 × 10^{-4} M. The degradation mechanism involves reactive oxygen species like hydroxyl radicals (OH) formed on the catalyst surface, facilitated by charge separation where valence band holes produce OH radicals from water, and conduction band electrons generate superoxide radicals ($O_2^{\cdot -}$) by reacting with molecular oxygen. Both radicals are highly reactive and contribute significantly to dye degradation. Chemical oxygen demand (COD) measurements confirmed the mineralization of SY, and the catalyst was shown to be reusable (Rajamanickam et al., 2014).

In 2018, Gu et al. explored the removal of ibuprofen using a composite made of activated carbon impregnated with TiO_2. This composite was prepared via the sol-gel method, utilizing titanium isopropoxide as the titanium source and commercially available Filtrasorb 400 activated carbon as support. The characterization revealed that the AC90T10 composite comprising 90% activated carbon and 10% TiO_2 achieved the highest removal efficiency, reaching 92% degradation of ibuprofen under UV light within 4 hours. The synergistic effect of adsorption and photodegradation explained this high performance, with adsorption playing a dominant role. The study also found that the weight ratio of composite to ibuprofen had limited impact within concentrations of 5–25 mg/L, whereas the reaction time under UV light (4 hours) and the solution pH (favoring acidity) were critical factors for effective removal (Gu et al., 2018).

4.3 – TiO_2-Carbon Nanotubes (CNTs) and Graphene

In 2018, Wang et al. synthesized TiO_2/CNT nanocomposites aimed at degrading methyl orange via photodegradation. The composite was prepared using the sol-gel method, with tetrabutyl orthotitanate (TBOT) serving as the titanium precursor, and carbon nanotubes (CNTs) acting as support material. Characterization of the synthesized composite was performed using several techniques, including scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS), and Fourier transform infrared spectroscopy (FTIR). SEM images revealed that TiO_2 nanoparticles were uniformly distributed on the surface of the CNTs, indicating that the support provided an increased surface area beneficial for photocatalytic processes, consistent with findings by Liu et al. (2011).

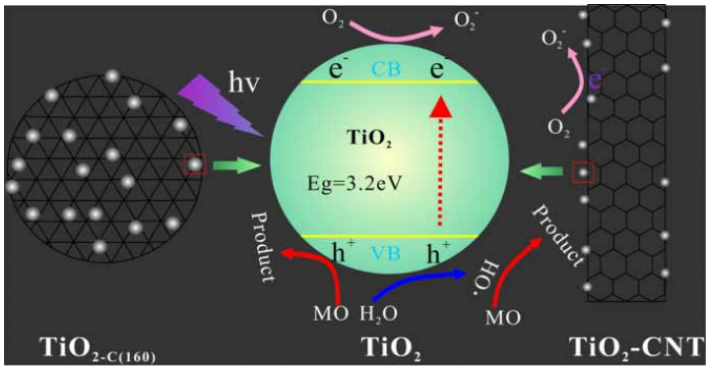


Figure 7: proposed mechanism of methyl orange (MO) photocatalytic degradation on photocatalytic composites.

In 2007, Luo et al. prepared multi-walled carbon nanotube (MWCNT)/TiO_2 nanocomposites using short MWCNTs as a starting material. These short nanotubes offered advantages such as ease of suspension, sorting, and manipulation, which enabled light to penetrate effectively into their inner tubes. The enhanced photocatalytic performance was demonstrated through the degradation of

reactive brilliant red X-3B dye. The results showed that the MWCNT/TiO_2 composite outperformed pure TiO_2, mainly due to the large surface area of CNTs, which increased the adsorption of pollutants and facilitated their photodegradation. However, the study also noted that excessive amounts of CNTs could cause photon scattering, thereby reducing the photocatalytic reaction rate. Additionally, the calcination temperature was found to influence the activity of the composite, with optimal performance depending on this parameter (Natarajan et al., 2017).

In 2016, Zhang et al. synthesized reduced graphene oxide (RGO)-decorated TiO_2 composites using a hydrothermal method. The composites were further improved by calcining at 450 °C for 30 minutes under an air atmosphere. The calcined samples exhibited superior photocatalytic efficiency compared to those without calcination. This enhancement was attributed to better crystallization of TiO_2 particles and the oxidation of residual organics in the composite post-calcination. The improved performance also depended on factors such as adsorption capacity, light absorption, charge transfer, and separation properties of the photocatalyst. The two-dimensional π-conjugated structure of graphene contributed to more effective pollutant degradation due to its high surface area and excellent electron transport capabilities (Yang et al., 2017; Huang et al., 2013).

Yang et al. (2012) studied the surface modification of CNTs and the enhanced photocatalytic activity of TiO_2 coated onto hydrophilically modified CNTs. Their findings indicated that the improved photocatalytic performance was mainly due to the high dispersion of TiO_2 on CNTs and the intimate contact at their interface, which resulted in dense heterojunctions facilitating charge transfer. CNTs helped promote interfacial charge transfer and prevented charge recombination by providing pathways for electrons, which was especially effective in the n-CNT-TiO_2 nanocomposites. The presence of CNTs created new interband energy states, delaying electron-hole recombination and thus enhancing environmental remediation efforts (Sharma et al., 2021).

5-Comparison of Biochar-TiO_2, AC-TiO_2, CNT-TiO_2, and Graphene-TiO_2

Table 2

Composites	Advantages	Limits	References
Biochar-TiO_2	- Cheaper initial cost than AC, CNT, and graphene.	- Limited interaction between biochar and TiO_2, resulting in less extension of absorption bands into longer visible wavelengths.	Wang et al., 2020; Ahmed et al., 2020; Lu et al., 2019
AC-TiO_2	- Cheaper initial cost than CNT and graphene.	- Limited interaction with TiO_2, with less extension into longer visible wavelengths.	Omri et al., 2014; Sheilatina et al., 2021; Anh et al., 2022; Radhi et al., 2023

Composites	Advantages	Limits	References
CNT-TiO_2	- Improved absorption into visible wavelengths due to interactions with CNTs. - High mechanical strength, thermal stability, large electron storage, and excellent electrical conductivity.	- Higher initial cost than AC, but cheaper than graphene. - More prone to aggregation than AC.	Wang et al., 2018; Ateia et al., 2017b; Apul and Karanfil, 2015
Graphene-TiO_2	- Enhanced visible light absorption owing to strong interactions - High mechanical strength, thermal stability, substantial electron storage, and conductivity.		

Table 3: Photocatalytic Degradation of Pollutants Using Carbonaceous-TiO_2 Composites

Compound	pH	Wavelength (nm)	Initial Conc (mg/L)	Irradiation Time (min)	% R	Ref
Amoxicillin (AC-TiO_2)	3-10	Sunlight	50	180	60-100%	Gar Alalm et al., 2016
Ibuprofen (AC-TiO_2 co-doped N)	N/A	254–420	20	120	11–100%	El-Sheikh et al., 2017
AC-TiO_2	6	320–400	1	10	>90%	Rosa et al., 2017
1-Butyl-3-methylimidazolium chloride (AC-TiO_2)	N/A	212	5	240	18.87%	Zawawi et al., 2018
CNT/TiO_2	2	>420	15	180	40%	Abega et al., 2019
CNT-TiO_2 (Aniline)	5.6	366, 436, 546	93	15	>95%	Orge et al., 2016

Compound	pH	Wavelength (nm)	Initial Conc (mg/L)	Irradiation Time (min)	% R	Ref
CNT-TiO_2 (Urea & Ibuprofen)	2–11	410	5	120	50–90%	Yuan et al., 2016
CNT-TiO_2 (4-Chlorophenol)	9	365	15	300	70%	Zouzelka et al., 2016
CNT-TiO_2 (Tetracycline)	3–11	240	0.5–30	120	20–100%	Ahmadi et al., 2016
TiO_2-AC (Methylen Blue)	N/A	664	30	60	99.47%	Xu et al., 2020
3D rGO-TiO_2 (Carbamazepine)	N/A	365	10	90	>99%	Nawaz et al., 2017
AC-TiO_2 (Indigo Carmine)	3	610	5	25	91.97%	Sheilatina et al., 2020
TiO_2/Biochar (Methyl orange)	N/A	464	20	150	92.45%	Lu et al., 2019
TiO_2-Biochar (Enrofloxacin)	5	254	100	60	60.66%	Wang et al., 2020
Biochar-TiO_2 (Methylen Blue)	N/A	660	5	240	>99%	Fazal et al., 2019

Conclusion and Future Perspectives

This review highlights the advantages of integrating carbonaceous nanomaterials with TiO_2 to enhance photocatalytic activity. These materials possess high surface area, excellent electrical, optical, thermal, and chemical properties, providing a solid foundation for improved photocatalytic performance. Their good electron-accepting and transport capabilities reduce electron-hole recombination and extend light absorption into the visible range.

However, challenges remain. Effective solid and direct contact between the carbonaceous nanomaterial and TiO_2 is crucial for efficient charge transfer and substrate adsorption. Reduction of TiO_2's band gap through bonding with structured carbonaceous materials is promising, but most research emphasizes activity enhancement over the practical issues of recovery and reusability. After use in water treatment, these catalysts can themselves become pollutants, underscoring the need to address their lifecycle.

Future research directions should focus on optimizing parameters such as material ratio, temperature, and synthesis conditions to improve efficiency, selectivity, and reusability, especially under real wastewater treatment conditions. Analyzing mineralization products is also essential to ensure treatment safety and environmental health.

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AUTHORS' CONTRIBUTIONS

All working authors were involved in this work from data collection to the final version of the manuscript. Conceptualization, methodology, investigation and writing of the original draft preparation, Mouafo Mouafo; validation, writing and revue, Abega Aimé Victoire, Kouadio Essaie Appiah, Brigton Henri, Soro Doudjo , Supervision of Daouda MAMA and YAO Kouassi Benjamin. All authors have read and agreed to the published version of the manuscript.

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