

Hydrothermally Processed Photocatalytic Nanomaterials for Wastewater Treatment: A Mini Review

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Abstract:

The sustainable development of every society is directly linked to the creation of new ways and technology for managing its environmental quality. The utilization of nanoparticles (NPs) has become a highly effective method for wastewater cleanup due to its inherent characteristics such as size, surface properties, and quantum effects. The inherent characteristics of these NPs have expanded their use in addressing the qualitative strain on water supplies. The objective of this review is to evaluate the application of hydrothermally synthesized nanoparticles in the removal of both organic and inorganic pollutants from wastewater. Hydrothermal processing is an essential method of thermochemical conversion which is utilized to transform biomass into biofuel or beneficial products. In general, the procedure is conducted under pressures and temperatures ranging from 4 to 22 MPa and 250 to 374°C, respectively. When the biomass is exposed to water, it is decomposed into minuscule constituents that can be repurposed. The selection of process parameters, including temperature, pressure, and duration, is determined by the desired products, which may include bio-oil, biogas, or bio-carbon. The current review provides an analysis of photocatalytic nanomaterials that have been employed in effluent treatment via hydrothermal processing. Consequently, current research has been focused on the development of an efficient method for degrading organic water pollutants for the past two decades. A significant proportion of the manuscript is dedicated to nanoparticles, nanostructures, and composites produced via hydrothermal means; all these components have been effectively implemented in the remediation of various types of dye wastewaters.

Keywords: Hydrothermal, Photocatalysts, Nanomaterials, Wastewater Treatment, Waste Treatment

1. Introduction

The use of nanotechnology and nanomaterials is a scientific decision that overcomes the limits of traditional macro-sized adsorbents widely employed in wastewater treatment. Nanosized adsorbents are more efficient at beating the intrinsic constraints of macro sized adsorbents. Hence, use of nanotechnology for treatment of wastewater has become a viable method. Nowadays, widespread usage of persistent organic pollutant (POPs) in variety of sectors, including pharmaceutical, textile,

and agricultural, has resulted in a substantial volume of wastewater containing complex, stable, and harmful contaminants (Gludge et al., 2021). The dyes, pharmaceuticals, pesticides, fertilisers, and detergents are often cited in this context (Lin et al., 2023, Oliveria et al., 2023, Alhali, 2023, Dovlatabadi et al., 2022, Khan, 2022) The carcinogen and toxic effects (Ismail et al., 2019, Pervez et al., 2020), high stability (Liu et al., 2020) and bio-accumulative (Kehrein et al., 2020) characteristics of these compound have also been documented. Among the different causes of pollution, the textile and printing industries are the primary contributors of organic pollutants, accounting for roughly 15% of global dye production. Because textile dyes, organic pollutants, and other industrial colourants are often persistent, extra attention must be paid to ensuring their breakdown in wastewater to compensate for the lack of fresh water.

Several techniques for wastewater treatment have been developed over the past decades, including the use of activated carbon (Azam et al., 2022), deionised water (Zhang & Liu, 2021) , sludge (Zhang et al., 2021), and sophisticated oxidative processes such as ionisation and photocatalytic degradation (Vieira et al., 2024, Singa et al., 2020, Dhiman et al., 2023). However, nontoxic, easy, stable, and eco-friendly photocatalysis-based process has the necessary features for treatment of wastewater containing dangerous POPs (Rueda-Marquez et al., 2020). Of late, nano-sized photocatalysts have garnered considerable interest in treatment of wastewater due to very high photocatalytic activity in degrading organic pollutants by visible light (Adhikari & Dharmarajan, 2022). The use of photocatalytic procedures is regarded to be less costly and to produce no polycyclic products in comparison to other traditional treatment methods yet photocatalytic processes have not been accepted widely as an option for industrial treatment because of the difficulties faced in development of improved catalytic materials, its recovery, toxicity, safe disposal, immobilisation of catalyst, and in the construction of reactor structures (Djurišić & Ng, 2020, Zakria et al., 2021). Types of processes for wastewater treatment is illustrated in Fig.1.

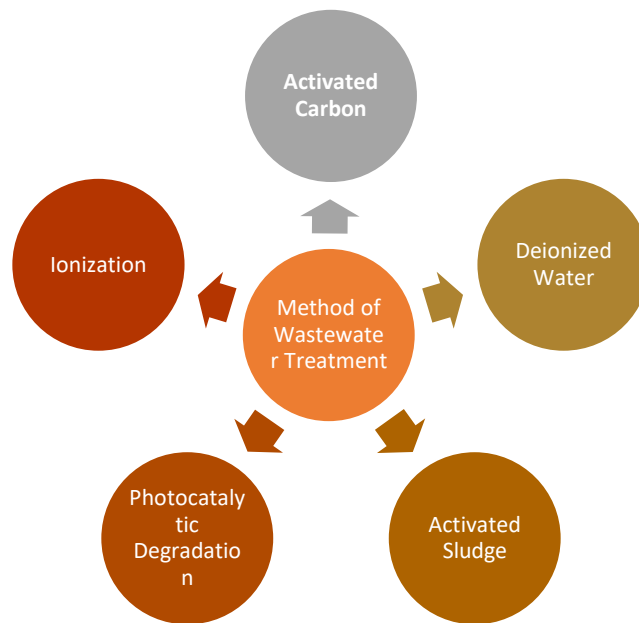


Fig.1. Methods of Wastewater Treatment

There is a clear need for eco-friendly methods to combat environmental degradation and its effects. Thus, over the last decades, the development of an effective approach for degradation of organic water contaminants has attained a lot of focus by the researchers. In current study, we discuss majorly about the nanoparticles, nanostructures and composites that have been prepared by hydrothermal synthesis and have been used to treat wastewaters containing dyes.

2. Wastewater Treatment Methods

Before the 20th century, microbes decomposed sewage over huge areas, but the urbanization reduced wastewater treatment and disposal land, and the population growth exponentially increased wastewater output. Thus, surface water couldn't filter this much sewage. Land and water bodies' self-purification capacities were crucial to these efforts, but also the nature is finite. Moreover, the early treatment focused on the suspended particle clearance, microbial infections, and biochemical oxygen demand but later the goals were expanded to include environmental and aesthetic concerns. Traditional wastewater treatment eliminated particles, organic material, and nutrients through multiple processes. The most prevalent wastewater treatment subprocesses include adsorption, coagulation-flocculation, photodegradation, ionic exchange, biological separation, and membrane separation (Pandey et al., 2023).

Wastewater treatment often involves physical separation, chemical breakdown, and biological degradation. Physical separation easily eliminates insoluble suspended pollutants but this unstable, slow-purifying process makes large-scale emission difficult. Chemicals are cleaned via sedimentation, membrane and depth filtration, and adsorption. These technologies can treat industrial wastewater, although sedimentation is most important for removal of toxins. Further, membrane

water filtration is cheap and effective, however foreign molecules hinder it (Egirani et al., 2020). Most chemical treatments involve condensation or oxidation where floated, filtered, flocculated, electro-floated, electrokinetic coagulated, ozone-oxidized, irradiated, or electrochemically treated colloidal particles precipitate the contaminants (Nidheesh et al., 2018). Fungal staining, microbial degradation, and bioremediation systems are also employed to treat industrial wastewater because bacteria, yeasts, algae, and fungus also absorb and degrade pollutants.

3. Advanced Oxidation Process (AOP)

According to the definition of "Advanced Oxidation Process" also known as AOP, it is a method of treating water at ambient temperature and pressure, in which a significant quantity of reactive radicals such as hydroxyl radicals are created to achieve water purification. During the process of chemical oxidation, the contaminants that are present are oxidised by several different reagents, including ozone, hydrogen peroxide, oxygen, and air. During the oxidation process, potentially harmful compounds are broken down into less dangerous products such as water, carbon dioxide, and more straightforward substances. AOP has been the subject of a significant amount of research since it offers more benefits than the other wastewater treatment procedures that are typically employed. The main advantage of this process is that it is faster and has more intense response rate, as well as its ability to completely eradicate all traces of mineralization or organic elements. Furthermore, the AOP process does not yield sludge as a byproduct. However, it is important to note that this approach entails a substantial expense due to the intricate chemical reactions involved and the necessity to tailor the process to the specific application (Ponnusami et al., 2023).

Mechanism of Action of Photocatalysts

Substrates that can absorb light and function as catalysts for chemical processes are known as photocatalysts. In general, semiconductors make up the whole of photocatalysts. When photocatalysts are subjected to light with an energy level high enough, a process known as photoexcitation takes place. This causes the electron to get excited and migrate from one orbital to another. The electron moves up to the conduction band, moving it up from the valence band. The creation of a gap in the valence band can be attributed to the absorption of photons with energies that are either equal to or larger than the band gap. Both the electron and the band gap will eventually make their way to the surface of the photocatalytic semiconductor, where they will perform their functions as a reducing and oxidising agent, respectively. The pores on the surface of the catalysts oxidise the water molecules that have been adsorbed into hydroxide radicals. This process oxidises the existing organic matter and converts it into products that are safer for the environment, such as carbon dioxide and water (Jabbar et al., 2023). The illustrative mechanism is given in Fig.2.

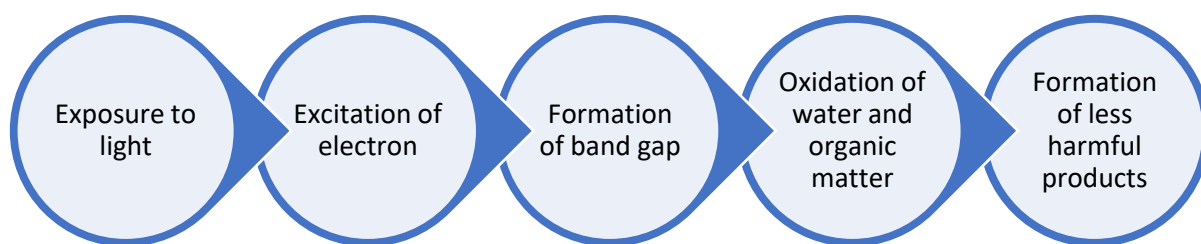


Fig.2. Mechanism of Action of Photo catalysts

4. Hydrothermally Processed Photocatalytic Nanomaterials for Wastewater Treatment

A study by Arvind et al., 2023 produced Ag–TiO₂ nanofibers using green synthesis. The Ag–TiO₂ nanofibers (NFs) XRD pattern showed anatase TiO₂ and FCC Ag nanoparticles. SEM image showed nanofiber-like surface morphology. The optical band gap energy of Ag–TiO₂ nanofibers (NFs) is 2.5 eV. Synthesized Ag-modified TiO₂ nanofibers (NFs) were tested for photocatalytic performance under direct sunlight using MB, MG, CR, and CV dye aqueous solutions. The effects of catalyst size on MB dye breakdown efficiency were also examined. The best catalyst concentration was 0.02 mg/mL. MB dye had the highest photosynthetic degradation efficiency of 94% at 120 min under direct sunshine. Ag–TiO₂ nanofibers (NFs) have shown excellent antibacterial action against Gram-positive bacteria like *Staphylococcus aureus* and Gram-negative bacteria like *E-Coli*. Due to these properties, Ag–TiO₂ nanofibers (NFs) may be a good photocatalyst for wastewater dye pollutants.

Hydrothermal synthesis of bismuth vanadate (BiVO₄) was done by Mansha et al., 2023 by changing the pH of concentrated H₂SO₄. With increasing pH (from 06 to 10), the produced material became nano-spheres and cubes between 50 and 60 nm. The lateral effect tuned BiVO₄'s bandgap from 2.47 to 2.50 eV, which is important for this investigation. Desirous bandgap corresponds to the abundant visible spectrum of solar light and has many real-world applications. BiVO₄ was studied by UV–Vis, X-ray, scanning electron, and energy-dispersive X-ray (EDX) spectroscopy. Synthesized BiVO₄ was investigated as a photocatalyst for Leather Field Industry pollution degradation. The BiVO₄ catalyst decomposed the industrial pollutant in 3 h under sun light.

A simple hydrothermal method was used by Abhilash et al., 2019 to synthesize metal oxide nanoparticles (MONPs). XRD, XRF, FTIR, DLS, HR-TEM, EDS, PL, AFM, and BET were used to characterize the material. The inimitable chemical and physical properties of Fe₂O₃ metal oxide

nanoparticles (MONPs) may highlight its medical and biological uses today. Rhombohedral Fe_2O_3 was employed for photocatalytic breakdown of rhodamine-B (RB) which was recycled and used for toxicity testing. This study examined Fe_2O_3 's impact on albino Wistar rat cardiac tissue histology. High-dosage manufactured compounds were stable and toxic to cutaneous melanoma cells (B16-F10), human embryonic kidney (HEK), and 293 cells depending on dose. Finally, *Escherichia coli* (MTCC 7410) cell wall damage tests were performed to determine rhombohedral nanomaterial behavior. Combining these biocompatibility studies allows for future eco-friendly applications of these materials.

Effect of different conditions on photocatalytic degradation of methylene blue was studied by Hamed et al., 2022 by utilizing TiO_2 films synthesised by hydrothermal technique was examined in this study. The data demonstrated that the catalyst shape, oxygen vacancy, Ti^{3+} surface defects affect the photocatalytic degradation of dye. It is also affected by initial pH, concentration, and active species. This work demonstrated that optimal concentration of Ti foundation was attained at 0.10 M and accomplished 42 percent methylene blue dye degradation.

This study by Bhagwat et al., 2021 used hydrothermal approach to fabricate tungsten trioxide/titanium dioxide nanohybrid structures. SEM inspection revealed the nanosheet-like shape of the produced $\text{WO}_3\text{-TiO}_2$ nanohybrid. UV-Vis-DRS measurements revealed that absorption edge of $\text{WO}_3\text{-TiO}_2$ nanohybrid shifted to the visible range due to the lowered bandgap. Under visible light, the photocatalytic activity of $\text{WO}_3\text{-TiO}_2$ nanohybrid was determined by photocatalytic degradation of Orange G dye in wastewaters. In the presence of $\text{WO}_3\text{-TiO}_2$ nanohybrid, 94 percent of Orange G dye was destroyed in 210 minutes at neutral pH, indicating improved photocatalytic activity. These findings indicate that generated nanohybrid material is highly effective photocatalyst for pollutant degradation in wastewater treatment applications.

Li and co-workers also investigated the influence of hydrothermal constraints on morphology, absorption of light, crystal structure, efficiency of photogenerated charge carriers for separation and photocatalytic elimination of Bisphenol A (BPA) in titanium-based nanomaterials. Increase in the concentration of NaOH, hydrothermal temp. and time lead to an increase in development of TiO_2 . UV absorption by nanomaterial varied greatly by shape. The optimum concentration of NaOH, hydrothermal time and temperature, along with HCl washing solution were 1 M, 28 hours, 170°C and 0.1 M respectively. Ti-based TiO_2 nanosheets removed most of the BPA (92.7 percent) under these circumstances, which was attributed to its highly ordered nanosheet structure, appropriate UV absorption, high crystallinity and excellent electron-hole pair separation efficiency.

CaWO₄ nanoparticles were synthesized by Nobre et al., 2021 by utilising the hydrothermal technique at temperatures of 100, 120, 140 and 160 °C for 1 hour. The principal active modes were identified using FTIR spectroscopies. For all generated samples, FE-SEM pictures revealed irregular polyhedral nanoparticles with an EDX spectrum including all the calcium tungstate's typical atomic peaks. Finally, these samples exhibited an extraordinary photocatalytic response when placed in a solution of Rhodamine B dye (RhB). The CaWO₄ nanoparticles performed better than the other produced samples, which is because of their smaller particle size and high dislocation density.

Ferric vanadate NPs (FeVO₄-NPs) were manufactured by Sajid et al., 2021 by using single pot autoclave hydrothermal technique (HTL). The powder was thoroughly studied for physico-chemical characteristics using several techniques. The size of nanoparticles was determined to be between 80 nm-0.12 μm. Under visible light, the photocatalytic reaction of catalyst was investigated for degrading Methylene Blue (MB) dye. The photoluminescence spectrum analysis demonstrated the potential of FeVO₄-NPs in the visible red area. FeVO₄-NPs catalysed the reaction initiator effectively to create strong radical with enhanced photocatalytic degradation performance, 98.74 percent in 180 minutes. This study demonstrates the feasibility of rapidly preparing FeVO₄-NPs for Fenton-like photocatalytic destruction of water contaminants.

Chu et al., 2020 created a systematic technique for the rapid and efficient synthesis of photocatalysts by using loaded nano-TiO₂ on carbon fibre using microwave hydrothermal process. Several analytical methods were used to analyse the produced TiO₂/CF. When used as a photocatalyst, TiO₂/CF is more readily recycled than pure TiO₂ particles. The oxidation of CF with nitric acid resulted in formation of polar groups, which increased the bonding characteristics. The loading rate of TiO₂ increased with increasing duration of CF treatment from 0 and 4 hr. Additionally, the experimental findings revealed that TiO₂/CF has good photocatalytic activity, degrading Rhodamine B at a rate of 97 percent during 1 hour of UV irradiation. Additionally, the photocatalyst yielded an 88 percent degradation rate even after ten testing cycles.

Table 1: Hydrothermally processed Catalysts

Type of Nano catalyst	Photocatalytic Action Agent	Degradation Percentage	Reference
Ag-TiO ₂	Methylene Blue	94%	Aravind et al.,2023
BiVO ₄	Industrial Pollutant	82%	Mansha et al., 2023
Fe ₂ O ₃	Rhodamine-B	86%	Abhilash et al., 2019

TiO ₂ films	Methylene Blue	42%	Hamed et al., 2022
WO ₃ -TiO ₂	Orange G	94%	Bhagwat et al.,2021
TiO ₂	Bisphenol A	92.7%	Li et al., 2021
CaWO ₄	Rhodamine-B	92%	Nobre et al., 2021
FeVO ₄ -NPs	Methylene Blue	98.74%	Sajid et al., 2021
TiO ₂ /CF	Rhodamine-B	88%	Chu et al.,2020

5. Conclusion

The existing water issue globally will become worse over the next few decades. Treatment of wastewater, particularly industrial effluents as well as drinking water, will become significant in current and future water management. Wastewater regulation frameworks will become increasingly stringent to mitigate environmental damage and for reusing water. These can be accomplished only through development of better, more efficient, and sustainable technologies for water treatment. Nanomaterials will undoubtedly play a major role in future water management systems and these nanomaterials will sustainably improve the performance of current technologies by consuming fewer resources and implementing more environmentally friendly catalytic processes. The brief review discussed the applications of hydrothermally processed different nanoparticles, nanocomposites, and nanostructures that have been utilised to treat wastewater, with a particular emphasis on dye wastewater treatment techniques. As a result of this analysis, we can conclude that hydrothermally treated nano catalysts are something to keep an eye out for in the future since they possess a very high proclivity to remediate wastewater.

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