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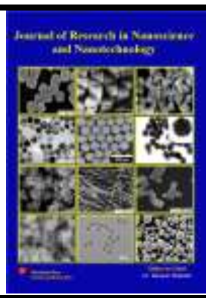

Advances in Synthesis Techniques and Environmental Applications of TiO_2 Nanoparticles for Wastewater Treatment: A Review

Hemra Hamrayev [1], Serdar Korpayev [2], Kamyar Shameli [3,*]

[1] Malaysia-Japan International Institute of Technology. Universiti Teknologi Malaysia, Jalan Sultan Yahya Petra, 54100 Kuala Lumpur, Malaysia
[2] Biotechnology Institute, Ankara University, 06135 Ankara, Turkey
[3] Institute of Virology, School of Medicine, Technical University of Munich, Munich, 81675, Germany
* Correspondence: kamyarshameli@gmail.com, kamyar.shameli@tum.de
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ABSTRACT

Worldwide, humanity has long grappled with the ongoing issue of environmental pollution, especially concerning water contamination. Contaminated water contains a range of pollutants, such as heavy metals, organic dyes, and pharmaceuticals, all of which pose harmful effects on both animals and humans due to their toxicity. As clean water sources continue to dwindle, there is an increasing demand for effective treatment methods for polluted water. In response to this pressing need, nanotechnology has emerged as a promising avenue and attracted significant global attention due to its multifaceted applications. Titanium dioxide nanoparticles (TiO_2-NPs) are commonly used in daily life and can be synthesized through a variety of physical, chemical, and environmentally friendly methods. Notably, TiO_2-NPs stand out for their high surface area-to-volume ratio and their ability to catalyse the degradation of pollutants through photocatalysis. In light of these advancements, this review explores recent progress in TiO_2-NPs synthesis and their environmental applications in wastewater treatment.

Keywords:
TiO_2 nanoparticles, environmental pollution, photocatalysis, wastewater treatment

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1. Introduction

Water pollution stands as a major global challenge affecting everyone universally. It is well established that access to clean water is necessary for human survival. Yet, due to the Industrial Revolution, accessing uncontaminated water directly from its natural source has become exceedingly difficult. Industries routinely discharge their wastewater directly into water bodies, leading to contamination and the pollution of once-pristine water sources. Pollutants that can be found in contaminated water include organic dyes, heavy metals, oils and solvents, medications, and other organic compounds [1]. These pollutants have detrimental effects on both animals and humans. Hence, there is a pressing need for non-selective removal techniques. Nanotechnology is one of the most cutting-edge techniques utilized for cleaning up contaminated water. Nanotechnology is an emerging field of modern science that has achieved notable advancements in creating and manipulating materials in the nanoscale dimension between 1 and 100 nm [2-5]. Nanomaterials exhibit unique properties and characteristics due to their exceptionally small size and larger surface area, rendering them more valuable than their bulk counterparts [6, 7]. Among various metal oxides, titanium dioxide (TiO_2) is one of the nanomaterials that has attracted the most interest lately because of its exceptional photocatalytic properties. TiO_2, an odorless, vividly white substance, typically exhibits hydrophobic properties in its natural state. In addition, TiO_2 is a semiconductor, that finds extensive use across diverse commercial sectors, including construction (e.g., in paints and coatings), healthcare, cosmetics, environmental applications, and energy [8]. This solid transition metal oxide is abundant and environmentally friendly owing to its lack of toxicity and non-flammable nature. In comparison to other metal oxides, TiO_2 NPs are highly favored for numerous applications because of their remarkable photocatalytic characteristics, affordability, abundance, self-cleaning capabilities, potent oxidizing properties, and superior chemical stability [9]. Furthermore, TiO_2 exhibits a unique trait: it remains undissolved in the majority of reaction environments, rendering it exceptionally suitable for photochemical processes and easily activated by UV radiation [10]. This combination of properties positions TiO_2 as a versatile and indispensable material across various fields, promising advancements and innovations in the realms of technology and sustainability.

A photochemical process known as photocatalysis uses light to activate a semiconductor or photocatalyst by producing charge carriers as a result of electronic excitation. These charge carriers have the potential to accelerate chemical reactions or alter their selectivity [11]. More precisely, photons of the right frequency that are shone on a photocatalytic material transfer photon energy to the electrons, causing band gap excitation—a process that excites electrons to the conduction band (CB) while leaving holes in the valence band (VB). These energized electrons can be effectively harnessed for various purposes, such as photovoltaics, breaking down organic pollutants like textile dyes, pharmaceutical wastes, and volatile organic compounds (VOCs), generating hydrogen (H_2), fixing nitrogen (N_2), reducing carbon dioxide (CO_2), and more [11].

Typically, TiO_2-NPs are synthesized through physical, chemical, and green methods. Physical methods include thermal decomposition [12], etching [13], ball milling [14], and laser irradiation [15]. In thermal decomposition, exceptionally high temperatures are required to conduct the synthesis process. Sputtering [16] and laser ablation [17] are two recent techniques used for producing and growing nanoparticles (NPs). Sputtering entails the expulsion of atoms from material surfaces through ion bombardment, while laser ablation employs high-powered laser beams to vaporize particles from a solid source. Although both methods are environmentally friendly, they require high initial setup costs and lack energy efficiency. Primarily, physical methods tend to be both energy and cost-intensive due to their reliance on expensive systems and equipment [18]. The chemical method of synthesizing NPs involves the use of expensive, hazardous, and toxic chemicals under specific conditions [18]. Releasing these chemicals into the atmosphere can lead to significant environmental problems, making it impractical to scale up nanoparticle synthesis using this approach. Moreover, several studies have shown that conventional chemical methods for synthesizing NPs with desired characteristics often pose hazards to test organisms, lacking environmentally friendly practices [18]. Hence, recent research has focused on adopting greener synthesis methods to reduce the toxicity associated with these NPs and focuses on recent advances in TiO_2-NPs as photocatalysts. Table 1 provides a summary of both the advantages and disadvantages of the various methods used to synthesize NPs.

Table 1
The advantages and disadvantages of the methods used to synthesize NPs

Synthesis Approaches	Advantages	Disadvantages
Physical Methods	○ No need for capping agents ○ Scalable ○ No by products ○ Producing nanomaterials with high purity	○ Expensive ○ Energy intensive ○ Producing nanomaterials using limited resources ○ High tech facilities
Chemical Methods	○ Producing nanomaterials using various materials ○ Control over size distribution, shape and crystallinity	○ Use of hazardous chemicals ○ Long reaction time ○ Environmentally harmful
Green Synthesis	○ Eco-friendly ○ Cost effective ○ Fabrication of biocompatible nanomaterials	○ Aggregation of NPs ○ Broad particle size distribution ○ Limited control over size distribution, shape and physical properties

2. Methods Used to Synthesize Nanoparticles

Different metallic and metallic oxide NPs can generally be synthesized using top-down and bottom-up methods. Top-down methods entail breaking down large or bulk molecules to produce the desired nanomaterials, while bottom-up methods involve assembling single atoms and molecules into larger nanomaterials, as illustrated in Figure 1. The comprehensive "bottom-up" and "top-down" approaches using a variety of methodologies for producing NPs are shown in Figure 2.0. Table 2 shows various methods used for the synthesis of TiO_2-NPs.

2.1 Physical Method

It was observed that all physical techniques used in nanoparticle synthesis were classified as top-down approaches. Physical methods, such as laser ablation [19], sputtering [20], and thermal evaporation [21] offer precise control over nanoparticle synthesis, crucial for tailoring properties to specific applications. Physical synthesis methods offer advantages such as precise control over nanoparticle properties, scalability, and minimal chemical contamination. However, they also present challenges such as high energy consumption and limited throughput compared to chemical methods. Despite their efficacy in color removal, physical methods do not degrade dye molecules, leading to their concentration and necessitating appropriate disposal measures.

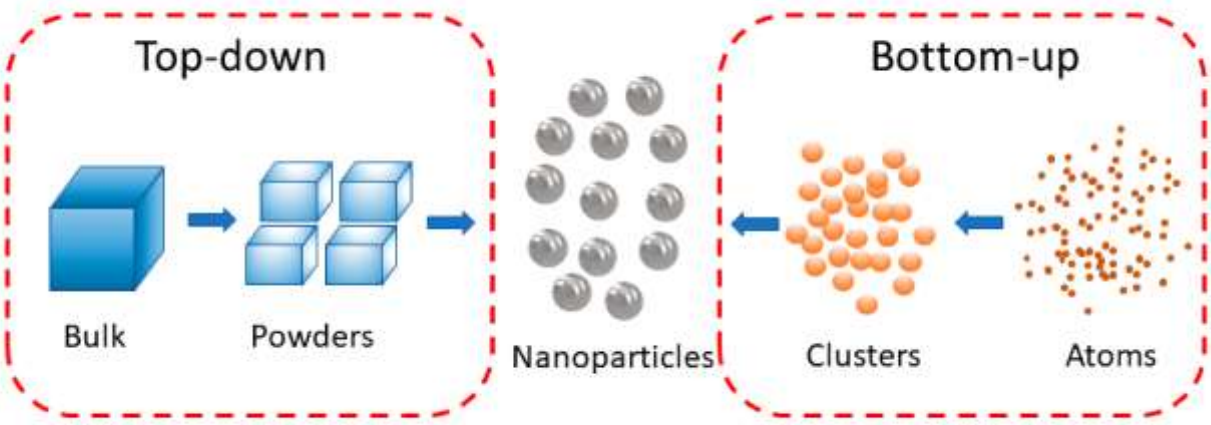

Fig. 1. "Top-down" and "bottom-up" synthesis of NPs [22]

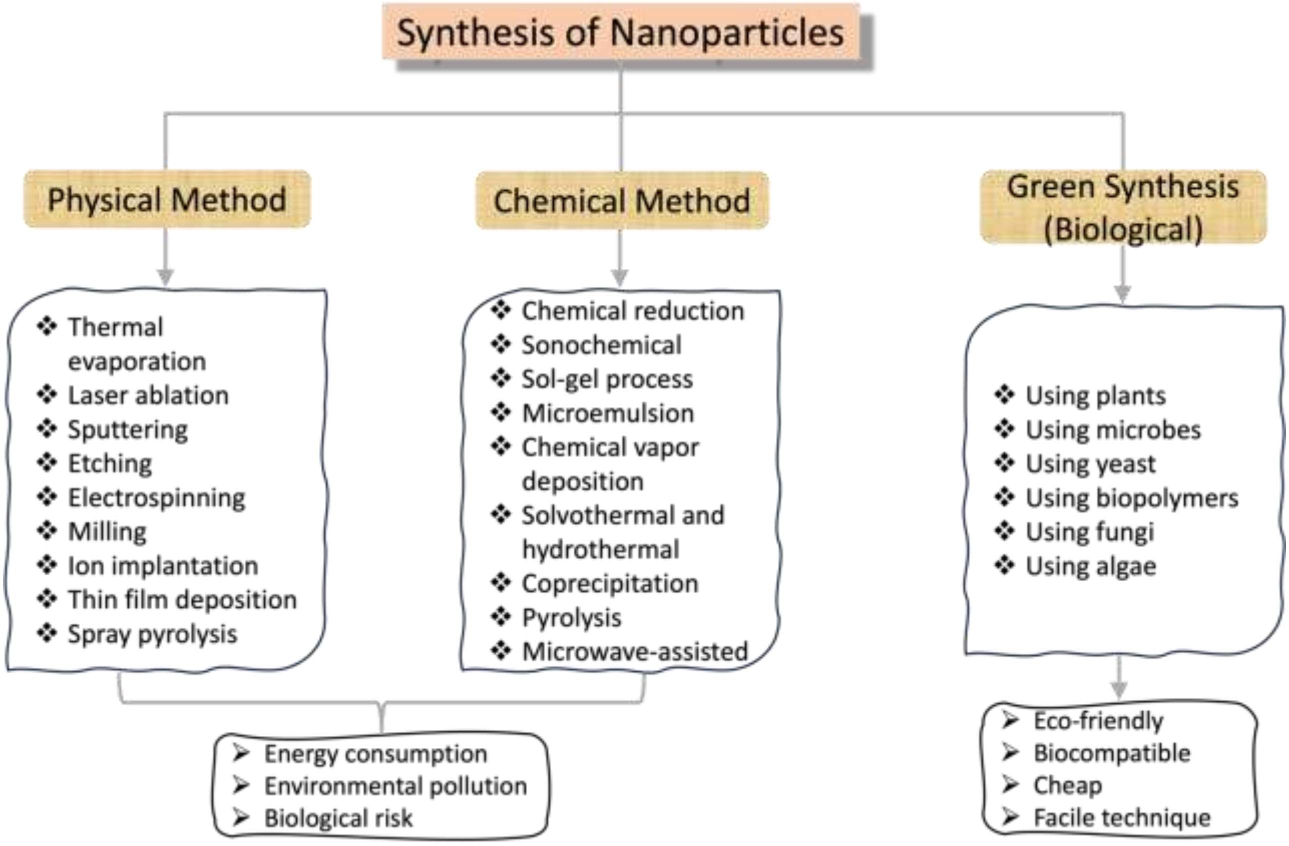

Fig. 2. Various methods for producing nanoparticles using "Top-down" and "bottom-up" techniques

2.1.1 Thermal evaporation

One of the commonly used methods for nanoparticle synthesis is thermal evaporation. Thermal evaporation synthesis involves the vaporization of a solid precursor material followed by its condensation onto a substrate, resulting in the formation of NPs. This method offers several advantages, including simplicity, scalability, and the ability to control the size and composition of NPs. Additionally, thermal evaporation synthesis allows for the production of NPs with high purity and uniformity. With its potential for large-scale production and precise control over particle properties, thermal evaporation continues to be a valuable method for nanoparticle synthesis.

2.1.2 Laser ablation

Laser ablation is a physical synthesis method used to produce NPs by irradiating a target material with a high-energy laser beam, causing rapid heating, vaporization, and subsequent nanoparticle formation. Recent advancements in laser ablation techniques have focused on enhancing control over particle size, shape, and composition. For instance, studies have investigated strategies such as pulse shaping and multi-beam irradiation to tailor nanoparticle properties precisely [23, 24]. However, it requires precise control over laser parameters and can be limited by high costs and low throughput. Furthermore, researchers have explored novel applications of laser ablation-synthesized NPs in fields such as biomedical imaging [25], drug delivery [26], and environmental remediation [27].

2.1.3 Sputtering

Sputtering is another widely used technique for the synthesis of NPs. In sputtering synthesis, a target material is bombarded with high-energy ions, typically from a plasma, causing atoms or molecules to be ejected from the surface of the target. These ejected particles, known as sputtered atoms or ions, can then condense and form NPs on a substrate placed near the target. Recent studies have highlighted its efficacy and versatility in nanoparticle fabrication. The research investigates the controlled growth of metal and metal oxide NPs via sputtering, emphasizing size and morphology control. Furthermore, another research explores the synthesis of complex nanostructures through sputtering, showcasing its potential for creating advanced materials with tailored properties. These studies underscore sputtering synthesis as a promising technique for applications ranging from catalysis to electronics, offering precise control over nanoparticle characteristics to meet diverse industrial and scientific demands.

2.1.4 Etching

Physical etching synthesis of NPs involves the controlled removal of material from a substrate through physical processes such as ion bombardment or plasma etching. This technique offers precise control over nanoparticle size, shape, and surface properties. Recent research has focused on enhancing the efficiency and scalability of physical etching methods for nanoparticle synthesis. For instance, studies demonstrate the fabrication of metal NPs via ion beam milling, showcasing the ability to achieve nanoscale precision and uniformity. Furthermore, the research explores the use of plasma etching for synthesizing semiconductor NPs with tailored electronic properties, paving the way for applications in optoelectronics and sensing. These advancements underscore the potential of physical etching synthesis as a versatile and robust approach for producing NPs with precise control over their characteristics.

2.1.5 Electrospinning

Electrospinning is a versatile technique for synthesizing NPs by using an electric field to draw a polymer solution or melt into ultrafine fibers. These fibers can be further processed to produce NPs with controlled size, morphology, and composition. Recent research has focused on enhancing the electrospinning process to fabricate NPs for various applications. For instance, studies demonstrate the synthesis of metal oxide NPs embedded within polymer fibers, showing promise for applications in catalysis and sensing. Additionally, another research explores the synthesis of biodegradable polymer NPs via electrospinning for drug delivery applications, highlighting the tunability of nanoparticle properties for enhanced therapeutic efficacy. These advancements illustrate the potential of electrospinning as a versatile and scalable approach for producing NPs with tailored characteristics for a wide range of applications.

2.2 Chemical Method

The process used in the chemical technique to produce NPs is essentially bottom-up. Small molecules or atoms join together to produce larger molecules through a process known as self-assembly in bottom-up approaches. Bottom-up strategies are the opposite of top-down strategies and resemble agglomeration. The chemical procedure entails the use of synthetic stabilizers, capping agents, and reducing agents, which are environmentally unfriendly and require elevated temperatures, pressures, and toxic substances [28]. Diverse chemical techniques like solvothermal synthesis [29], co-precipitation [30], microemulsion [31], chemical reduction [32], sol-gel processing [33], sonochemical [34], chemical vapor deposition [35], microwave-assisted synthesis [36], and hydrothermal synthesis [37] are employed in this context. The chemical approach effectively eliminates all dye molecules; however, it presents a difficulty with the accumulation of concentrated sludge, leading to challenges in disposal.

2.2.1 Sol-gel process

Sol-gel synthesis is a versatile method for producing TiO_2-NPs with controlled size, morphology, and surface properties. In this process, titanium alkoxides are hydrolyzed and condensed in the presence of a solvent and a catalyst under mild conditions, typically at room temperature. The resulting sol undergoes gelation to form a three-dimensional network, which can be dried to obtain TiO_2 NPs [18]. This method offers several advantages, including scalability, low processing temperature, and the ability to tailor nanoparticle properties by adjusting precursor concentration, solvent composition, and reaction conditions. Additionally, sol-gel synthesis allows for the incorporation of dopants or surface modifications to enhance the photocatalytic activity or improve the specific properties of TiO_2 NPs. For instance, doping with metals or non-metals can modify the band structure of TiO_2, extending its absorption range and enhancing photocatalytic performance for applications such as environmental remediation and solar energy conversion.

2.2.2 Microwave-assisted synthesis

Microwave-assisted synthesis has emerged as a rapid and efficient method for producing titanium dioxide TiO_2-NPs. In this approach, microwave irradiation is utilized to heat the reaction mixture, leading to accelerated nucleation and controlled growth of TiO_2-NPs. The use of microwaves allows for precise temperature control and uniform heating, resulting in reduced synthesis times compared to conventional methods. However, microwave-assisted methods, though requiring high-power microwave heating which is energy-intensive, are also not cost-effective and do not permit real-time monitoring of TiO2 particle growth. Furthermore, the mass synthesis of TiO_2-NPs is not possible with microwave-assisted synthesis [18, 38]. This technique offers several advantages, including higher yields, narrower size distributions, and enhanced crystallinity of the synthesized NPs. Moreover, microwave irradiation promotes the formation of highly reactive surfaces, which can improve the photocatalytic activity of TiO_2 NPs for applications such as pollutant degradation and solar energy conversion. Recent studies have explored various parameters, including microwave power, irradiation time, precursor concentration, and solvent composition, to optimize the synthesis of TiO_2 NPs with desired properties. Additionally, the microwave-assisted approach has been combined with other techniques, such as sol-gel or hydrothermal methods, to further enhance the control over nanoparticle morphology and crystalline structure.

2.2.3 Co-precipitation

The co-precipitation method is a widely employed technique for synthesizing TiO_2-NPs. In this method, titanium precursor compounds are typically dissolved in a solvent, and a precipitating agent is added to induce the formation of TiO_2 NPs through chemical precipitation. The reaction mixture is then subjected to appropriate thermal treatment to promote crystallization and obtain the desired phase of TiO_2-NPs. This method offers several advantages, including simplicity, scalability, and the ability to control the size and morphology of the synthesized NPs by adjusting reaction parameters such as temperature, pH, and precursor concentration. Moreover, the co-precipitation method enables the synthesis of TiO_2-NPs with high purity and uniformity, making them suitable for various applications including photocatalysis, sensors, and biomedical devices. Recent studies have focused on optimizing the co-precipitation process to enhance the structural, optical, and photocatalytic properties of TiO_2-NPs. Additionally, efforts have been made to explore novel precursor compounds and additives to tailor the properties of the synthesized NPs for specific applications.

2.2.4 Chemical vapor deposition

Chemical vapor deposition (CVD) is a prominent method for synthesizing TiO_2 NPs with precise control over size, morphology, and crystallinity. Vapor deposition encompasses any procedure where materials in a gaseous state are condensed to create solid-phase material. In CVD, a volatile precursor containing titanium is introduced into a reaction chamber along with a carrier gas under controlled temperature and pressure conditions. Upon decomposition or reaction, TiO_2 NPs are deposited onto a substrate within the chamber. This technique offers several advantages, including the ability to produce uniform coatings on complex shapes and substrates, the high purity of the synthesized NPs, and scalability for industrial applications. Moreover, CVD allows for the synthesis of TiO_2 NPs with tailored properties by adjusting parameters such as precursor composition, temperature, gas flow rates, and reaction time. Recent advancements in CVD have focused on improving the efficiency, reproducibility, and scalability of TiO_2 nanoparticle synthesis. Additionally, efforts have been made to explore novel precursor materials and innovative reactor designs to enhance control over nanoparticle properties and deposition processes.

2.2.5 Microemulsion

The microemulsion method stands out as a versatile approach for synthesizing TiO_2-NPs. In this method, a microemulsion system comprising water, oil, surfactant, and co-surfactant is utilized as a confined reaction medium for the formation of NPs. The hydrophobic and hydrophilic domains within the microemulsion facilitate the controlled nucleation and growth of TiO_2-NPs. This technique offers several advantages, including the ability to precisely control nanoparticle size, morphology, and crystallinity by adjusting parameters such as surfactant composition, reaction temperature, and precursor concentration. Moreover, the microemulsion method enables the synthesis of monodisperse TiO_2-NPs with narrow size distributions and high stability. Recent advancements in microemulsion synthesis have focused on enhancing the reproducibility, scalability, and efficiency of TiO_2 NPs production. Additionally, efforts have been made to explore the incorporation of dopants or surface modifications to tailor the properties of the synthesized NPs for specific applications.

2.2.6 Solvothermal and hydrothermal synthesis

The solvothermal and hydrothermal methods are widely utilized for synthesizing TiO_2-NPs with controlled size, morphology, and crystallinity. In both methods, a precursor solution containing a titanium source is sealed in a reaction vessel with a solvent and subjected to elevated temperature and pressure conditions. In solvothermal synthesis, organic solvents act as reaction media, while in hydrothermal synthesis, water serves as the solvent [38]. Within the hydrothermal method, temperatures can exceed the boiling point of water, reaching the pressure of vapor saturation. Conversely, in the solvothermal approach, temperatures can be elevated even higher than in hydrothermal processes, as a variety of organic solvents with high boiling points are available for use [39]. These reaction conditions facilitate the nucleation and growth of TiO_2 NPs, leading to crystalline structures with tailored properties. Solvothermal methods are regarded as the safest because they utilize organic solvents in terms of environmental toxicity, whereas hydrothermal uses surfactants, which are harmful to ecosystems, particularly aquatic life [18, 38]. Moreover, the solvothermal method typically offers superior control over the size, shape distributions, and crystallinity of TiO2 NPscompared to hydrothermal methods. These methods offer several advantages, including simplicity, scalability, and the ability to control nanoparticle size and morphology by adjusting reaction parameters such as temperature, pressure, and reaction time. Additionally, solvothermal and hydrothermal synthesis enables the production of TiO_2 NPs with high purity and uniformity, making them suitable for various applications, including photocatalysis, sensors, and energy storage.

2.2.7 Sonochemical approach

The sonochemical method is a powerful technique for synthesizing TiO_2 NPs with precise control over size, morphology, and crystallinity. The chemical impacts of ultrasound do not arise from direct interactions with molecular species. In this method, ultrasonic waves are used to create cavitation bubbles in the reaction mixture, generating localized high temperatures and pressures that facilitate the nucleation and growth of TiO_2 NPs. This method offers several advantages, including rapid synthesis kinetics, uniform dispersion of NPs, and the ability to produce high-quality TiO_2 NPs without the need for high temperatures or harsh reaction conditions. Moreover, sonochemical synthesis enables the synthesis of TiO_2 NPs with tailored properties by adjusting parameters such as sonication power, reaction time, and precursor concentration. Recent studies have focused on optimizing sonochemical synthesis parameters to enhance the efficiency, reproducibility, and scalability of TiO_2 NPs production. Additionally, efforts have been made to explore the incorporation of dopants or surface modifications to tailor the properties of the synthesized NPs for specific applications.

Table 2
Various methods used for the synthesis of TiO_2-NPs

Material Precursor	Method	Shape	Size (nm)	Reference
Titanium isopropoxide	Sol-gel	Varying shapes	7.3 nm	[40]
Titanium tetraisopropoxide $[Ti(OCH(CH_3)_2)]_4$	Sol-gel	Spherical	15-20 nm	[41]
Titanium tetraisopropoxid	Green synthesis	Tetragonal	19-24 nm	[42]
Titanium tetraisopropoxide	solvothermal	Spherical	5 nm	[29]
Titanium tetraisopropoxide (TTIP)	Sol-gel	Amorphous	-	[43]
Titanium Chloride ($TiCl_4$)	Green synthesis	Tetragonal	20 nm	[44]
TiO $(OH)_2$	Green synthesis	Spherical	25 - 100 nm	[45]
Titanium tetrachloride	Hydrothermal method	Irregular	~ 17 nm	[46]
Titanium isopropoxide	Co-precipitation	Anatase-rutile structure	11.3 - 27.4 nm	[47]
Potassium hexafluorotitanate	Bio-mediated green synthesis	Spherical	10 – 30 nm	[48]
TiCl4	Bio-mediated green synthesis	Spherical	~14 nm	[49]
Titania bulk powder	Green synthesis	Spherical	80 - 140 nm	[50]
Titanium tetrabutoxide	Sol-gel method	Spheroidal	< 100 nm	[51]
Salt TiO_2	Green synthesis	Cubed and pentameric	1.5 - 30 nm	[52]
Titanium Butoxide	Green synthesis	Spherical	70–150 nm	[53]
Titanium oxalate complex	Hydrothermal	Flower-like	15 - 100 nm	[54]
Titanium isopropoxide (TTIP)	Ultrasound assisted sol-gel method	Spherical	~11 nm	[55]
Titanium tetra chloride	Green synthesis	Triangular	17.30 and 21.61 nm	[56]
Titanium isopropoxide (TTIP)	Sol-gel method	-	12 and 49 nm	[57]
Titanium trychloride ($TiCl_3$)	Green synthesis	Tetragonal	-	[58]
Titanium tetra-isopropoxide	Sol-gel method	Spherical	10–50 nm	[59]
$TiCl_4$	Sol-Gel Method	Tetragonal	3-30 nm	[60]
Titanium Tetrachloride ($TiCl_4$)	Sol- gel technique	Spherical spongy	-	[61]
Titanium (IV) isopropoxide (TTIP)	Sol–gel method	Irregular	<5 nm	[62]
Titanium tetra isopropoxide (TTIP)	Urea-assisted auto-combustion	Spherical	8–25 nm	[63]
Titanium oxy sulphate	Green synthesis	Spherical	20–90 nm	[64]
Bulk TiO_2	Biosynthesis	Spherical	62–74 nm	[65]
$TiO(OH)_2$	Green biosynthesis	Smooth, spherical and uneven	40.50 nm	[66]
Titanium trichloride ($TiCl_3$)	Co-precipitation	Spherical	12 nm	[67]
Titanium (IV) tetraisopropoxide	Hydrothermal	Elongated	5 - 10 nm	[68]
Titanium (IV) chloride	Sol-gel	Non-uniform	19–21 nm	[69]
Titanium Tetrachloride ($TiCl_4$)	Sol- gel technique	Spherical spongy	-	[61]

Titanium (IV) isopropoxide (TTIP)	Sol–gel method	Irregular	<5 nm	[62]
Titanium tetra isopropoxide (TTIP)	Urea-assisted auto-combustion	Spherical	8–25 nm	[63]
Titanium oxy sulphate	Green synthesis	Spherical	20–90 nm	[64]
Bulk TiO_2	Biosynthesis	Spherical	62–74 nm	[65]
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Titanium (IV) tetraisopropoxide	Hydrothermal	Elongated	5 - 10 nm	[68]
Titanium (IV) chloride	Sol-gel	Non-uniform	19–21 nm	[69]
Titanium (IV) chloride	Sol-gel	Uniform spherical	14-16 nm,	[70]
Titanium tetraisopropoxide	Sol-gel	Spherical	~26 nm	[71]
Titanium isopropoxide	Sol-gel	Spherical	1.52 nm	[72]
Titanium tetraisopropoxide	Green synthesis	Spherical	20–70 nm	[73]
$TiO(OH)_2$	Green synthesis	Spherical	36 - 68 nm	[74]
Titanium alkoxide	Sol-gel	Tetragonal	10–50 nm	[75]
Titanium (IV) butoxide	Sol-gel	Amorphous	700 nm	[76]
Tetrabutyl orthotitanate	Sol-gel	Spherical	20–80 nm	[77]
$TiO(OH)_2$	Phyto-synthesis	Spherical	23 ± 2 nm	[78]
$TiO(OH)_2$	Green synthesis	Spherical	32.58 nm	[79]
Titanium (IV) Chloride	Sol-gel	-	19 - 68 nm	[80]
Titanium tetra isopropoxide (TTIP)	Sol-gel	Spherical	73.99 nm	[81]
Titanium (IV) isopropoxide	Hydrothermal	Spherical	9 nm	[82]
Titanium tetra chloride ($TiCl_4$)	Green synthesis	Flower like	200 nm	[83]
Titanium tetra chloride ($TiCl_4$)	Facile solvothermal	Rhombohedral	-	[84]

2.3 Green Synthesis

The green synthesis of TiO_2-NPs involves the utilization of environmentally benign materials and methods, aiming to minimize the use of hazardous chemicals and energy-intensive processes. Various natural resources, such as plant extracts, microorganisms, and biocompatible polymers, have been explored as reducing and stabilizing agents in green synthesis routes for nanomaterials [85, 86]. One commonly employed approach involves using plant extracts rich in bioactive compounds such as polyphenols, flavonoids, and terpenoids. These phytochemicals serve as reducing and capping agents, facilitating the formation of TiO_2-NPs through eco-friendly routes. Green synthesis methods often involve simple procedures, such as mixing the plant extract with a titanium precursor solution, followed by gentle heating or exposure to sunlight. The advantages of green synthesis include sustainability, cost-effectiveness, and the absence of toxic by-products. Moreover, the phytochemicals present in plant extracts can impart additional functionalities to the synthesized TiO_2-NPs, such as enhanced photocatalytic activity or antibacterial properties. Recent research has focused on optimizing green synthesis conditions, exploring new plant sources, and elucidating the mechanisms underlying nanoparticle formation. Additionally, efforts have been made to scale up green synthesis processes for potential industrial applications while maintaining environmental sustainability.

3. Application of TiO_2 in Wastewater Treatment

Wastewater treatment is crucial for mitigating environmental pollution and preserving water resources. Wastewater treatment is one of the most popular uses for nanostructured TiO_2-NPs since it exhibits superior photocatalytic efficacy compared to bulk-sized TiO_2-NPs (Table 3). It is typically discovered that traditional wastewater treatments are insufficient to eliminate pollutants and color from the wastewater. TiO_2-NPs have gained significant attention due to their excellent photocatalytic properties, making them promising candidates for wastewater treatment applications. The application of TiO_2-NPs in wastewater treatment represents a promising avenue for addressing the growing challenges of water pollution. TiO_2-NPs exhibit remarkable photocatalytic properties, which have been extensively studied for their potential to degrade organic pollutants and remove various contaminants from wastewater. Organic dyes are frequently studied for their role in pollutant degradation, given their widespread use in industrial and pharmaceutical sectors, often resulting in their discharge into wastewater. The major industrial contributors to environmental pollution include the fuel and energy sectors, as well as the chemical, metallurgy, wood, and paper industries. In this section, we delve into the diverse applications of TiO_2-NPs, exploring their mechanisms of action, effectiveness in pollutant removal, and potential drawbacks. By examining the current research and advancements in this field, we aim to provide insights into the practical implications and prospects of utilizing TiO_2-NPs for sustainable wastewater treatment solutions.

3.1 Photocatalytic Mechanism of TiO_2-NPs

In wastewater treatment, TiO_2-NPs function as photocatalysts, harnessing solar or artificial light to initiate the oxidation and degradation of organic pollutants. This process begins with the valence band, which hosts filled energy levels containing electrons, and the conduction band, characterized by vacant energy levels, is distinctly separated from the valence band. When light interacts with TiO_2-NPs, it triggers the creation of electron-hole pairs, as illustrated in Figure 3. These pairs consist of electrons moving from the valence band to the conduction band, leaving behind positively charged holes. As this photocatalytic dance unfolds, and leads to the formation of reactive oxygen species (ROS) such as hydroxyl radicals (•OH) and superoxide radicals ($\bullet O^{2-}$), ultimately aiding in the pollutant degradation [10, 85, 87]. These ROS react with organic contaminants, breaking down complex molecules into smaller, less harmful by-products. In the "Photocatalytic Mechanism of TiO_2-NPs " section, we delve into the intricate processes by which TiO_2-NPs harness light energy to initiate chemical reactions. Upon exposure to light, TiO_2-NPs generate electron-hole pairs, which subsequently undergo redox reactions with water and organic pollutants present in wastewater given in Fig. 4. This leads to the degradation of organic compounds into harmless byproducts and the generation of reactive oxygen species, contributing to the disinfection of water. Through a comprehensive examination of the photocatalytic mechanism, including factors such as surface properties, particle size, and light intensity, we aim to provide a deeper understanding of how TiO_2-NPs can be optimized for efficient and sustainable wastewater treatment applications. Additionally, we explore current research trends and challenges in harnessing TiO_2-NPs photocatalytic activity to develop innovative solutions for water pollution mitigation.

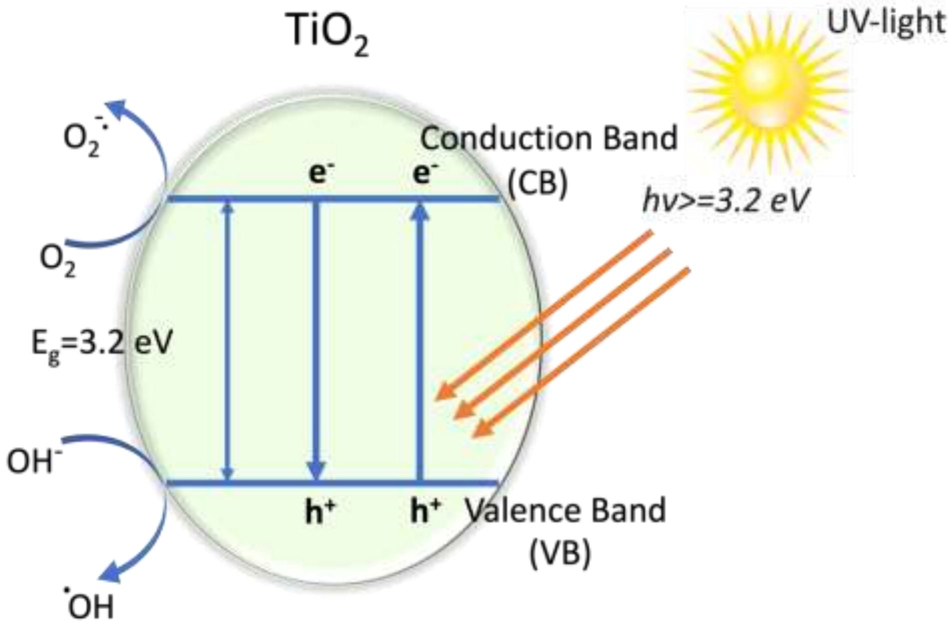
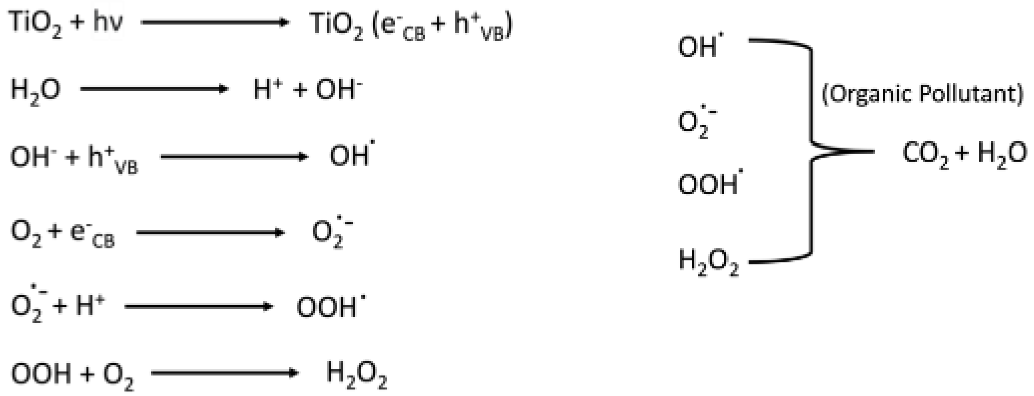


Fig. 3. Schematic graphic depicting the various reactions that occur on the surface of TiO_2 when exposed to UV light



Fig, 4. Various free radicals are produced on the surface of TiO_2, and these free radicals then mineralize organic contaminants

Table 3
TiO_2 NPs as photocatalysts for wastewater treatment.

Nanoparticles	Synthesis Method	Applications	Removal Efficiency (%)	Ref.
TiO_2	Green synthesis	Removal of chemical oxygen demand (COD) and chromium (Cr)	82.26% (COD), 76.48% (Cr)	[88]
TiO_2	Green synthesis	Degradation of toxic Methyl Orange (MO) dye.	~99%	[89]
TiO_2	Sol-gel	Removal of Cadmium	89.45%	[90]
TiO_2	Hydrothermal	Degradation of methylene blue dye	97.6%	[91]
TiO_2	Green synthesis	Removal of lead (Pb)	82.53%	[92]
TiO_2	Hydrothermal	Photodegradation of MO dye	81% - 88 %	[93]
TiO_2	Electrospinning	Removal of methylene blue dye	87.36%	[94]
TiO_2	Sol-gel	Photocatalytic degradation of methylene blue	68 %	[94]
TiO_2	Green synthesis	Reduction of oil	75.56%	[95]
TiO_2	Green synthesis	Reduction of phosphate	94.67%	[95]
TiO_2	Solvothermal	Photo catalytic degradation of MO and Methylene blue (MB) dyes	96.3 % (MB) and 97 % (MO)	[29]
TiO_2	Green synthesis	Rhodamine B (RhB) dye	96.59%	[96]
TiO_2	Hydrothermal	Degradation of Orange II, MO and RhB dyes	99%, 98% and 99%, respectively	[97]
TiO_2	Sol-hydrothermal	Photocatalytic degradation of X-3B and X-BR solutions	99.5% and 96.08%, respectively	[98]
TiO_2	Precipitation	Photocatalytic degradation of MO	99.08%	[99]
TiO_2	Hydrothermal	Sonocatalytic degradation of ciprofloxacin	65.01%	[100]
TiO_2	Green Synthesis	Photocatalytic for RhB dye	90%	[101]
TiO_2	Green Electrosynthesis	Photocatalytic Degradation of Phenol	88%	[102]

3.2 Types of Wastewaters Treated Using TiO_2-NPs

3.2.1 Organic dyes

Dyes, colorful organic pollutants, are discharged into water bodies from textile and printing industries. Organic dyes pose environmental and health risks due to their persistence, non-biodegradability, and potential toxicity [87]. The presence of dyes in water inhibits the intake of oxygen and sunshine, which has an impact on aquatic life. Some of the dyes cause water to become unfit for human consumption by anaerobic decolonization to carcinogenic amines [87]. TiO_2-NPs offer a promising solution through their photocatalytic activity, which initiates the degradation of organic dyes upon exposure to light. The photocatalytic degradation process involves the absorption of photons by TiO_2-NPs, leading to the generation of electron-hole pairs. These photoinduced charge carriers then react with water and oxygen molecules adsorbed on the TiO_2 surface, forming highly reactive oxygen species such as hydroxyl radicals. These radicals effectively oxidize the organic dye molecules, breaking them down into smaller, less harmful compounds or ultimately into CO_2 and H_2O. Several factors influence the efficiency of TiO_2-NPs in dye degradation, including the specific dye characteristics, TiO_2-NPs' properties such as particle size and surface area, as well as the intensity and wavelength of the light source. By exploring the mechanisms and optimization strategies for dye degradation using TiO_2-NPs, this section aims to shed light on the potential of this technology for mitigating the environmental impact of organic dye pollutants in wastewater.

3.2.2 Pharmaceuticals

Over the past two decades, there has been growing concern about micro-pollutants from pharmaceuticals and personal care products, including antibiotics, anti-inflammatory medications, and insect repellents, because they persist in the environment and are biologically active in both humans and animals. Antibiotics, crucial for safeguarding against bacterial infections, are now recognized as a growing category of pharmaceutical pollutants, posing significant challenges for their removal from both soil and aquatic settings [103]. Pharmaceuticals, including antibiotics, hormones, and pain relievers, enter wastewater systems through various routes, posing environmental and public health concerns due to their potential adverse effects on aquatic organisms and ecosystems. As a result, advanced oxidation processes (AOPs), including semiconductor photocatalysis, are emerging as promising advanced methods for purifying water contaminated with pharmaceuticals [104]. TiO_2-NPs offer a promising approach for pharmaceutical degradation through their photocatalytic activity. When exposed to light, TiO_2-NPs generate electron-hole pairs, which initiate redox reactions with water and oxygen molecules adsorbed on their surface. This leads to the formation of highly reactive oxygen species, such as hydroxyl radicals, which oxidize and degrade pharmaceutical compounds into simpler, less harmful substances. The effectiveness of TiO_2-NPs in treating pharmaceuticals in wastewater depends on various factors, including the properties of the pharmaceutical compounds, TiO_2-NPs characteristics such as particle size and surface area, as well as the light source's intensity and wavelength. Understanding these factors and optimizing conditions are crucial for enhancing the efficiency of TiO_2-NPs in pharmaceutical degradation.

3.2.3 Heavy metals

Heavy metal ions in water sources have become a significant public concern due to rising industrial activities that contaminate water bodies with toxic metals like nickel and cadmium. These pollutants adversely affect the structure and stability of aquatic ecosystems because of their toxicity, bioaccumulation, persistence, and non-biodegradable nature [105]. TiO_2-NPs can also assist in the removal of heavy metals through adsorption or photocatalytic reduction processes, reducing the toxicity of wastewater. In the section "Treating heavy metals in wastewater using TiO_2-NPs," we investigate the promising application of TiO_2-NPs for the remediation of heavy metal pollution in wastewater. Heavy metals, such as lead, cadmium, mercury, and chromium, are highly toxic contaminants that pose serious environmental and health risks due to their persistence and bio-accumulative nature [38]. Small concentrations of heavy metals, when present in food, air, or water, can lead to acute or chronic toxic effects upon entering the human body [38]. TiO_2-NPs offer a sustainable solution through their photocatalytic properties, which enable the removal and detoxification of heavy metals from wastewater. Under light irradiation, TiO_2-NPs generate electron-hole pairs, initiating redox reactions with water and oxygen molecules. This results in the formation of reactive oxygen species, such as hydroxyl radicals, which interact with heavy metal ions, leading to their oxidation and subsequent immobilization or precipitation as less toxic forms. The effectiveness of TiO_2-NPs in treating heavy metals depends on various factors, including the specific heavy metal species, TiO_2-NPs characteristics like surface area and crystal structure, and the presence of coexisting ions in the wastewater matrix. Optimization of these parameters is essential to enhance the efficiency and selectivity of heavy metal removal using TiO_2-NPs.

3.2.4 Oil and solvent

Industrial, oily wastewater has been identified as a major cause of water pollution [106]. This kind of oily wastewater is typically generated by petrochemical, pharmaceutical, metallurgical, and food industries, with oil fields being a particularly significant source. Oil spills and solvent discharges pose significant environmental threats, leading to soil and water pollution, as well as detrimental effects on aquatic ecosystems and human health. TiO_2-NPs present a promising solution due to their unique photocatalytic properties, which enable the degradation and detoxification of oil and solvent compounds in wastewater. When exposed to light, TiO_2-NPs generate electron-hole pairs, initiating redox reactions with water and oxygen molecules adsorbed on their surface. This results in the production of highly reactive oxygen species, such as hydroxyl radicals, which interact with oil and solvent molecules, breaking them down into simpler, less harmful substances. The efficiency of TiO_2-NPs in treating oil and solvent contamination depends on various factors, including the nature and concentration of the contaminants, TiO_2-NPs characteristics such as particle size and crystallinity, and the intensity and wavelength of the light source. Optimization of these parameters is essential to maximize the degradation rate and minimize the formation of harmful byproducts.

3.2.5 Pathogen inactivation

The rising resistance of phytopathogenic microbes has made controlling plant pathogens in crop production increasingly challenging, necessitating the development of new antimicrobial materials [107]. Photocatalytic active nanomaterials could provide an alternative solution to combat these plant pathogens. TiO2-NPs exhibit antimicrobial properties and can be employed for the disinfection of water by inactivating bacteria, viruses, and other pathogens. Pathogens, including bacteria, viruses, and protozoa, pose significant public health risks when present in untreated wastewater, leading to the spread of waterborne diseases and infections. TiO2-NPs offer an innovative solution through their photocatalytic activity, which facilitates the inactivation of pathogens in wastewater. Upon exposure to light, TiO2-NPs generate electron-hole pairs, initiating redox reactions with water and oxygen molecules adsorbed on their surface. This results in the production of highly reactive oxygen species, such as hydroxyl radicals, which exhibit strong antimicrobial properties. These radicals interact with the cellular components of pathogens, disrupting their structure and function, and ultimately leading to their inactivation. The effectiveness of TiO2-NPs in pathogen inactivation depends on various factors, including the type and concentration of pathogens, TiO2-NPs characteristics such as surface area and crystallinity, and the intensity and wavelength of the light source. Optimization of these parameters is crucial to achieve high levels of pathogen removal and ensure the safety of treated wastewater.

4. Challenges and Future Perspectives

While TiO_2-NPs has long been recognized as a top material for commercial applications, it encounters challenges both today and potentially in the future, like any other photocatalyst. During photocatalytic degradation, TiO_2-NPs may aggregate together, potentially diminishing their photocatalytic activity, especially smaller particles, which scatter light more than larger ones [10]. Despite the promising potential of TiO_2-NPs in wastewater treatment, several challenges remain, including the need for cost-effective synthesis methods, the scalability of photocatalytic reactors, and the potential environmental impacts of nanoparticle release. Future research efforts should focus on addressing these challenges while exploring novel TiO_2-NPs formulations and photocatalytic reactor designs to enhance their efficiency and applicability in real-world wastewater treatment scenarios. The review paper "Advances in Synthesis Techniques and Environmental Applications of TiO_2-NPs for Wastewater Treatment" illuminates significant progress in TiO_2-NPs synthesis and their application in wastewater treatment. However, challenges persist. Optimizing synthesis methods for scalability and cost-effectiveness while maintaining nanoparticle quality poses a formidable task. Future research should focus on refining existing techniques or devising novel approaches to bridge this gap. Moreover, understanding the environmental implications of TiO_2-NPs is paramount. Efforts must be made to explore their fate, transport, and potential toxicity in aquatic environments. This knowledge will inform regulatory frameworks ensuring the safe and sustainable deployment of TiO_2-NPs in wastewater treatment. Integrating TiO_2-NPs with conventional treatment processes presents both challenges and opportunities. Optimizing integration strategies to enhance treatment efficiency requires further investigation. Additionally, future research should prioritize exploring synergies between TiO_2-NPs and advanced treatment technologies. Collaborative efforts between researchers, policymakers, and regulatory bodies are essential in establishing comprehensive guidelines for TiO_2-NPs use. By addressing these challenges, the field can harness the full potential of TiO_2-NPs in revolutionizing wastewater treatment, mitigating pollution, and advancing toward a sustainable water management paradigm. Challenges in synthesizing TiO_2-NPs for wastewater treatment include scalability, cost-effectiveness, and precise control over nanoparticle properties. Environmental concerns regarding nanoparticle toxicity and energy-intensive synthesis methods also persist. However, future perspectives suggest innovative green synthesis techniques, advanced characterization methods, and integration with existing treatment technologies. Developing comprehensive regulatory frameworks and fostering interdisciplinary research efforts are crucial for optimizing TiO_2-NPs synthesis and maximizing their environmental application in wastewater treatment.

5. Conclusion

In conclusion, the review paper "Advances in Synthesis Techniques and Environmental Applications of TiO_2-NPs for Wastewater Treatment" underscores significant strides made in TiO_2-NP synthesis and their environmental utilization. While challenges such as scalability, cost-effectiveness, and environmental impact persist, promising future perspectives emerge. Innovations in green synthesis methods, advanced characterization techniques, and integration with existing treatment technologies offer avenues for improvement. Moreover, comprehensive regulatory frameworks and interdisciplinary collaboration are crucial for ensuring the safe and sustainable deployment of TiO_2-NPs in wastewater treatment. By addressing these challenges and embracing future research directions, the field can harness the full potential of TiO_2-NPs to mitigate water pollution, protect public health, and promote environmental sustainability in wastewater treatment practices.

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