



Original Article

# A Comprehensive Review on TiO<sub>2</sub> Nanoparticles: Synthesis Strategies and Catalytic Roles in Organic Transformations

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## Abstract

Titanium dioxide (TiO<sub>2</sub>) nanoparticles have garnered considerable interest in both research and industry due to their unique physicochemical properties, environmental safety, and remarkable catalytic effectiveness in organic synthesis. This review highlights various techniques for producing TiO<sub>2</sub> nanoparticles, focusing on the core chemistry and reaction mechanisms responsible for their formation. Conventional methods such as sol-gel, hydrothermal, solvothermal, and precipitation techniques provide accurate control over crystallite dimensions, surface area, and morphology, all of which significantly influence catalytic performance. Recently, sustainable synthesis methods using plant extracts and biopolymers have emerged as promising alternatives to minimize the use of toxic reagents and energy requirements. The review also explores the catalytic characteristics of TiO<sub>2</sub> nanoparticles in diverse organic reactions, including oxidation, reduction, and coupling processes. Understanding the mechanisms of photocatalytic and thermal pathways reveals how surface defects, oxygen vacancies, and dopant modifications enhance charge separation and increase the density of reactive sites, thus boosting selectivity and efficiency in reactions. The relationship between synthesis conditions and catalytic performance is thoroughly examined to reveal the structure-activity connections crucial for the informed design of catalysts. Lastly, current challenges, such as limited activity under visible light and the recyclability of catalysts, are addressed, along with potential solutions involving surface modifications, heterojunction formation, and doping techniques. This comprehensive overview establishes a foundation for the continued development of TiO<sub>2</sub>-based nanocatalysts aimed at producing greener and more efficient organic synthesis.

**Keywords** Titanium dioxide nanoparticles, sol-gel technique, hydro-thermal, nanocatalyst, heterogeneous catalysis, organic synthesis

## Introduction

Titanium dioxide (TiO<sub>2</sub>) stands out as one of the most extensively studied metal oxides due to its outstanding physical, chemical, and electrical properties. It typically exists in three crystalline forms—anatase, rutile, and brookite—with anatase exhibiting superior photocatalytic and catalytic performance due to its wider band gap (3.2 eV), higher surface area, and effective charge carrier separation. The combination of chemical durability, non-toxicity, and cost-effectiveness has made TiO<sub>2</sub> a crucial material in environmental remediation, solar energy applications, and especially in heterogeneous catalysis [1].

In recent times, the movement towards eco-friendly chemistry and green catalysis has stimulated investigations into nanostructured TiO<sub>2</sub> materials. TiO<sub>2</sub> nanoparticles display distinctive physicochemical characteristics such as a high surface-to-volume ratio, tunable morphology, and abundant active sites, making them proficient catalysts for various organic transformations [2]. These transformations include oxidation, reduction, condensation, and coupling reactions performed under mild or light-assisted conditions. TiO<sub>2</sub> can function as a photocatalyst when subjected to ultraviolet or visible light or serve as a thermally activated heterogeneous catalyst, depending on its configuration and synthesis approach [3]. The method of synthesis plays a pivotal role in determining the structural and catalytic attributes of TiO<sub>2</sub> nanoparticles. Traditional methods like sol-gel, hydrothermal, solvothermal, and precipitation techniques have been extensively optimized to manage particle size, shape, crystallinity, and surface defects.

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For instance, the sol-gel technique allows for precise control over stoichiometry and particle shape at relatively low temperatures through the hydrolysis and polycondensation of titanium alkoxides such as titanium isopropoxide (TTIP) [4]. Conversely, hydrothermal and solvothermal techniques utilize high-temperature and pressure conditions to generate highly crystalline nanoparticles with customized morphologies such as nanorods, nanotubes, and nanospheres [5].

Lately, "green synthesis" methods that leverage biological resources like plant extracts, microorganisms, and biopolymers have emerged as sustainable substitutes for conventional chemical processes. These biological systems act as reducing and stabilizing agents, decreasing the dependence on harmful precursors and solvents [6]. The presence of phytochemicals such as flavonoids and terpenoids encourages the nucleation and growth of TiO<sub>2</sub> nanoparticles, facilitating the production of biocompatible materials suitable for environmental and catalytic applications [7]. The catalytic efficiency of TiO<sub>2</sub> is closely related to its electronic structure and surface chemistry. It stands as a fundamental element of modern catalytic materials due to its adaptable structure, eco-friendliness, and versatility in organic synthesis. The pathway of their synthesis directly impacts their physicochemical properties and catalytic performance, emphasizing the necessity for thoughtful design and mechanism-oriented strategies. This review comprehensively explores the various synthesis methods for TiO<sub>2</sub> nanoparticles, elucidates their mechanistic details, and evaluates their extensive catalytic applications in organic transformations. Additionally, it highlights emerging trends in the modification of TiO<sub>2</sub> and the creation of hybrid catalysts, aiming to establish sustainable and effective catalytic systems for green chemistry.

### 1. Synthesis Techniques of TiO<sub>2</sub> Nanoparticles

The technique employed for the synthesis of titanium dioxide (TiO<sub>2</sub>) nanoparticles is essential in influencing their morphology, crystallinity, surface area, and catalytic efficiency. Over the past few decades, researchers have developed numerous chemical and eco-friendly synthesis methods to tailor TiO<sub>2</sub> nanoparticles for specific uses. The most widely used synthesis methods include sol-gel, hydrothermal/solvothermal, precipitation, and green synthesis techniques. Each approach varies in its mechanism, cost, environmental impact, and the degree of control it offers over the characteristics of the particles [8].

### 2. Sol-Gel Technique

The sol-gel method is one of the most frequently utilized techniques for generating TiO<sub>2</sub> nanoparticles, as it allows for molecular-level control over composition and uniformity. This process involves the hydrolysis and polycondensation of titanium alkoxide precursors such as titanium tetra-isopropoxide (TTIP), titanium butoxide (Ti(OBu)<sub>4</sub>), or titanium ethoxide (Ti(OEt)<sub>4</sub>) in alcoholic solvents. The procedure begins with the formation of a sol, a colloidal suspension of hydrolyzed titanium species, which subsequently transforms into a gel through polymerization and condensation reactions. Following drying and calcination, the gel framework converts into crystalline TiO<sub>2</sub> nanoparticles. Factors such as pH, temperature, precursor concentration, and solvent composition play significant roles in determining the resulting phase and morphology. Acid-catalyzed hydrolysis typically produces smaller, more uniform anatase particles, while base-catalyzed conditions usually promote rutile phase formation. The advantages of the sol-gel method include low synthesis temperatures, excellent compositional control, and high purity of the final product. However, challenges such as

agglomeration during drying and the need for precise regulation of the hydrolysis rate can limit scalability [9].

### 3. Hydrothermal and Solvothermal Techniques

The hydrothermal method is an efficient synthesis technique that employs high-pressure, high-temperature aqueous conditions to crystallize TiO<sub>2</sub> nanoparticles from precursor solutions. Typically conducted in sealed Teflon-lined autoclaves, this method allows for careful control over nucleation and crystal growth, resulting in highly crystalline nanoparticles with specific shapes such as rods, sheets, and spheres [10]. The solvothermal method is a variation that employs organic solvents rather than water, which affects the dielectric constant and surface energy, thus influencing the crystal structure and morphology of the resulting TiO<sub>2</sub>. In hydrothermal synthesis, titanium precursors like titanium chloride (TiCl<sub>4</sub>), titanium isopropoxide (TTIP), or titanium sulfate (Ti(SO<sub>4</sub>)<sub>2</sub>) interact with water or alkaline substances (NaOH, NH<sub>4</sub>OH) at elevated temperatures (150–250 °C). This process can lead to the formation of metastable anatase or brookite phases, depending on the duration of the reaction and pressure conditions [11]. The resulting nanoparticles exhibit high crystallinity and fewer defects, which is advantageous for photocatalytic and catalytic uses. The solvothermal technique allows for the alteration of reaction media using alcohols, amines, or ketones as solvents, which function both as reaction media and capping agents, improving dispersion and preventing agglomeration [12]. Furthermore, the hydrothermal method is scalable and environmentally friendly, as it does not necessitate surfactants or templates.

### 4. Precipitation Technique

The precipitation technique is a simple, economical, and scalable method for producing TiO<sub>2</sub> nanoparticles. It entails the regulated hydrolysis of titanium precursors such as titanium tetrachloride (TiCl<sub>4</sub>), titanium isopropoxide (TTIP), or titanium sulfate (Ti(SO<sub>4</sub>)<sub>2</sub>) in the presence of a base like NH<sub>4</sub>OH or NaOH, resulting in the formation of titanium hydroxide. Upon heating, this hydroxide converts into crystalline TiO<sub>2</sub>. Factors such as pH, temperature, and precursor concentration significantly influence the crystal phase (anatase or rutile), particle size, and surface area. This approach facilitates the straightforward addition of dopants or surfactants during the synthesis process, improving photocatalytic performance and controlling the morphology. Advantages include affordability, ease of use, and high purity, though issues like particle agglomeration and the formation of amorphous phases can occur if synthesis conditions are not carefully managed. Recent studies have indicated enhanced catalytic and photocatalytic activities of TiO<sub>2</sub> obtained through precipitation in oxidation and degradation reactions [13].

### 5. Green Synthesis

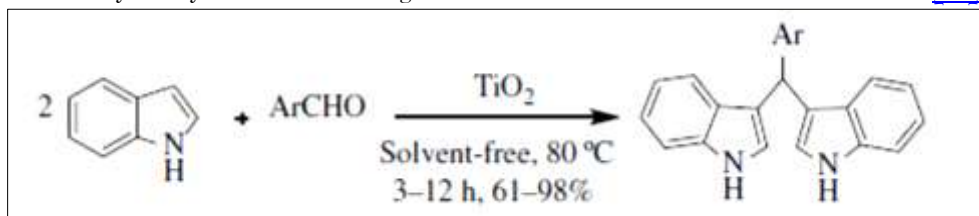
In accordance with sustainable development goals, the eco-friendly production of TiO<sub>2</sub> nanoparticles has emerged as a viable, environmentally responsible alternative to conventional chemical techniques. This method utilizes biological materials such as plant extracts, microorganisms, algae, or biopolymers as reducing and stabilizing agents [6]. Phytochemicals like polyphenols, terpenoids, and flavonoids can reduce titanium precursors such as TiCl<sub>4</sub> or TTIP to TiO<sub>2</sub> nanoparticles under mild conditions. For instance, extracts from Aloe vera, Azadirachta indica (neem), and green tea have been successfully employed to produce anatase TiO<sub>2</sub> nanoparticles with outstanding photocatalytic characteristics [14]. This technique is environmentally friendly, cost-efficient, and appropriate for large-scale production. Additionally, biomolecules act as

natural stabilizers, preventing particle agglomeration and enhancing stability. Green synthesis also offers the benefit of reduced toxicity and improved biocompatibility for biomedical and catalytic applications [7]. However, challenges persist in achieving uniform particle size and reproducibility due to inconsistencies in biological precursors.

#### 6. Organic Reactions Catalyzed by TiO<sub>2</sub> Nanoparticles

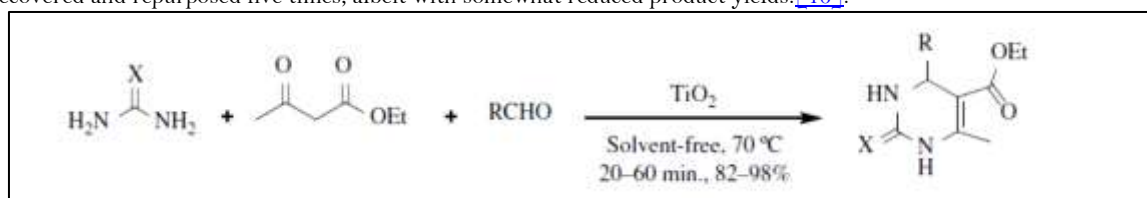
Due to its robust metal-oxygen bonds, exceptional redox properties, chemical stability, and non-toxic nature, titanium dioxide (TiO<sub>2</sub>) has emerged as one of the most versatile and eco-friendly catalysts for various organic

transformations. The key driver of TiO<sub>2</sub>'s catalytic effectiveness is its semiconducting properties, which allow it to engage in redox reactions both when heated and under light exposure. TiO<sub>2</sub> proves to be highly beneficial in heterogeneous catalysis and sustainable organic synthesis, as it can act as a Lewis acid, photocatalyst, or a support for active metal species, contingent on the reaction conditions. This paper provides a brief overview of the applications of TiO<sub>2</sub>, nanoscale TiO<sub>2</sub>, and modified nanoscale TiO<sub>2</sub> as effective catalysts in facilitating specific organic processes. Hosseini-Sarvari claims that bis(indolyl)methane's can be easily produced in the presence of TiO<sub>2</sub> under mild reaction conditions without the use of a solvent [15].



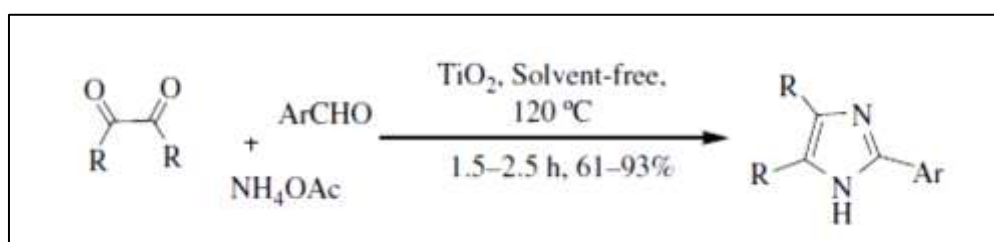
#### Scheme 1. Synthesis of bis(indolyl)methanes in the presence of TiO.

Kassaei used TiO<sub>2</sub> for the Biginelli reaction of ethyl acetoacetate, urea/thiourea, and aldehydes. During the optimization trials, they found that the best results were obtained when the reaction was conducted at 70 °C without the use of a solvent. The results demonstrated that although the method is quite successful in converting aromatic and heteroaromatic aldehydes to the corresponding contained DHPMs, aliphatic aldehydes do not react well in the same reaction conditions. Furthermore, this catalyst was recovered and repurposed five times, albeit with somewhat reduced product yields. [16].



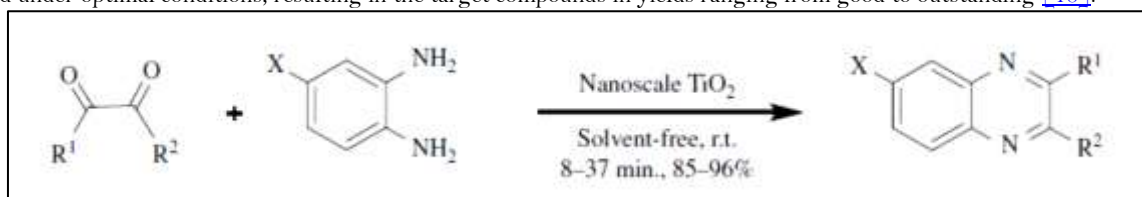
#### Scheme 2. Biginelli reaction catalyzed by TiO.

Brahmachari and associates reported an efficient three-component synthesis of 2,4,5-triarylimidazoles from the reaction of aldehydes, ammonium acetate, and 1,2-diketones using TiO<sub>2</sub> as the catalyst [17].



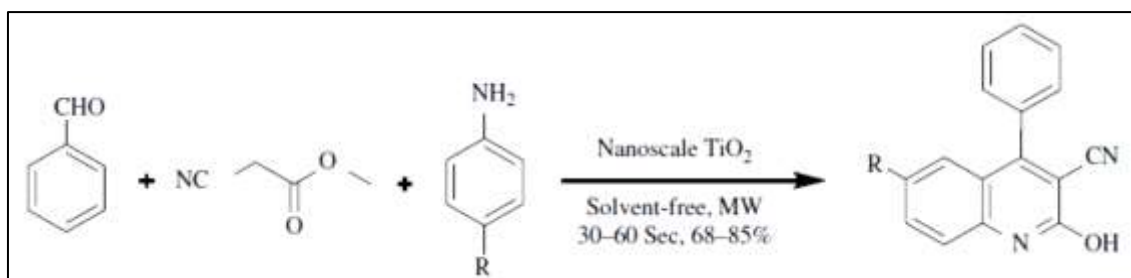
#### Scheme 3. Synthesis of 2,4,5-triarylimidazoles using TiO<sub>2</sub> as the catalyst.

A simple and efficient approach for synthesizing quinoxaline derivatives using catalytic amounts of nanoscale TiO<sub>2</sub> at ambient temperature without solvents was introduced by Mirjalili and his team. They found that various diamines effectively reacted under optimal conditions, resulting in the target compounds in yields ranging from good to outstanding [18].



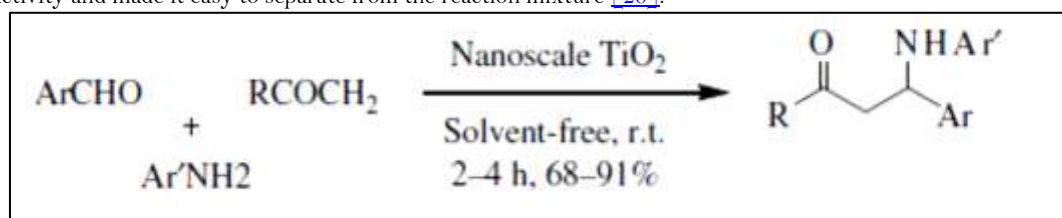
#### Scheme 4. Synthesis of quinoxaline derivatives under solvent free conditions catalyzed by nanoscale TiO<sub>2</sub>.

Using nanoscale TiO<sub>2</sub> as the catalyst, Bhojya Naik and colleagues described a tandem reaction method for the synthesis of various quinoline/benzo[h]quinoline-3-carbonitrile that involves a Michael addition and a Knoevenagel condensation under microwave irradiation. The aromatic aldehydes and amines interacted well with this technique, producing the expected products in high yields over incredibly short reaction times [19].



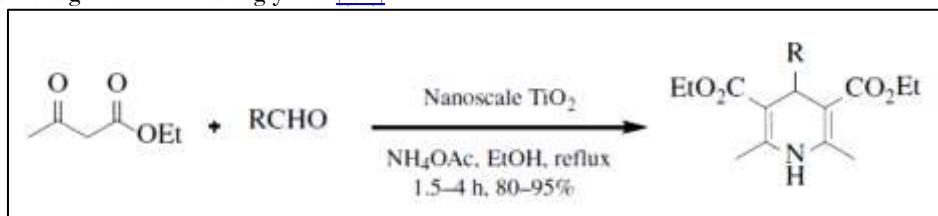
**Scheme 5. Preparation of various quinoline/benzo[h]quinoline-3-carbonitrile in the presence of nanoscale TiO<sub>2</sub>.**

Furthermore, in a three-component Mannich process, this catalyst might be an efficient catalyst. When different aldehydes, amines, ketones, or cyclic ketones reacted without a solvent, the  $\alpha$ -amino carbonyl adducts were generated in good to high yields with good anti-selectivity. Observations showed that nanoscale TiO<sub>2</sub> outperformed the other nano metal oxides in terms of related product selectivity and yields. This method allowed the catalyst to be employed four times without appreciably lowering its catalytic activity and made it easy to separate from the reaction mixture [20].



**Scheme 6. Preparation of various quinoline/benzo[h]quinoline-3-carbonitrile in the presence of nanoscale TiO<sub>2</sub>.**

Tajbakhsh et al. reported a successful three-component method for producing 1,4-dihydropyridine derivatives using nanoscale TiO<sub>2</sub> in ethanol under reflux conditions. With this reagent present, a number of aliphatic, aromatic, and heterocyclic aldehydes smoothly condensed in good to outstanding yields [21].



**Scheme 7. Synthesis of 1,4-dihydropyridine derivatives using nanoscale TiO<sub>2</sub>.**

## Conclusion

Titanium dioxide (TiO<sub>2</sub>) nanoparticles have shown themselves to be highly efficient and ecologically safe catalysts for a range of organic processes. Because of their excellent surface activity, adaptable shape, and semiconducting nature, they can function as both heterogeneous catalysts and photocatalysts under mild and sustainable circumstances. Important organic reactions, such as the reduction of nitroarenes to amines, the oxidation of alcohols to aldehydes and ketones, and the production of imines, sulfoxides, and coupling products via oxidative or reductive routes, are effectively catalyzed by TiO<sub>2</sub>. In condensation processes like the Knoevenagel, Aldol, and Biginelli syntheses, TiO<sub>2</sub> acts as a Lewis acid, activating carbonyl compounds and promoting the high-yield production of C-C or C-N bonds. TiO<sub>2</sub>'s low toxicity, chemical stability, and recyclability make it ideal for green chemistry applications. Despite limitations like charge recombination and restricted visible-light absorption, its catalytic potential is continually being increased by ongoing advancements in band-gap engineering, surface modification, and heterojunction design. TiO<sub>2</sub> is still essential for the creation of energy-efficient and sustainable catalytic systems in modern chemical synthesis.

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## Synthesis Strategies and Catalytic Roles in Organic Transformations."

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## Conflicts of interest

The authors declare that there are no conflicts of interest regarding the publication of this paper

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