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GREEN CHEMISTRY PERSPECTIVE OF TIO_2 NANOPARTICLES

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ABSTRACT
Titanium dioxide (TiO_2) nanoparticles have attracted significant scientific and industrial interest owing to their outstanding optical, electrical, and catalytic properties. Driven by the growing need for sustainable, environmentally friendly, and economically viable materials, extensive research has focused on the development and application of TiO_2 nanostructures in diverse fields such as photocatalysis, organic synthesis, environmental remediation, and biomedical engineering. TiO_2 occurs naturally in three main crystalline phases-anatase, rutile, and brookite-each possessing distinct physicochemical characteristics. Among them, the anatase phase is particularly favored due to its high surface area, superior photocatalytic activity, and excellent chemical stability.

This review provides a comprehensive overview of the synthesis strategies employed to control the size, morphology, and crystallinity of TiO_2, nanoparticles. Conventional methods, including sol-gel, hydrothermal, solvothermal, sonochemical, and co- precipitation techniques, are discussed alongside emerging green synthesis approaches that utilize plant extracts and biological resources. Green routes have gained prominence as they reduce the use of hazardous chemicals while improving environmental compatibility and biocompatibility.

The article also highlights key characterization techniques such as X-ray diffraction, FTIR, UV-visible spectroscopy, SEM, TEM, EDX, and BET analysis, which are essential for understanding the structure-property relationships of TiO_2, nanoparticles. Typically, anatase TiO_2, nanoparticles with sizes ranging from 10 to 50 nm exhibit enhanced surface reactivity and charge transfer behavior.

Furthermore, the catalytic role of TiO_2 nanoparticles in organic transformations-including Biginelli, Mannich, Hantzsch, Friedel-Crafts, and multicomponent reactions-is critically reviewed. Strategies such as metal/nonmetal doping and composite formation to enhance visible-light activity are also discussed. Overall, TiO_2 nanoparticles emerge as a versatile and promising material for advancing sustainable chemical, environmental, and biological technologies.

Keywords: *Titanium dioxide (TiO_2) nanoparticles (NPs), heterogeneous catalysis, photocatalysis, green synthesis, semiconductor, metal/nonmetal doping, nanocomposites, antimicrobial activity*

INTRODUCTION
Titanium dioxide (TiO_2) nanoparticles have emerged as a cornerstone material in nanoscience, owing to their exceptional chemical stability, tunable electronic structure, and multifunctional properties that support wide applications[1-10] in - *photocatalysis, environmental remediation, energy conversion, sensors, and biomedical systems*. The performance of TiO_2 at the nanoscale is critically influenced by its *structural attributes*, including crystallite size, phase composition (anatase, rutile, brookite), surface defects, and morphology. These attributes, in turn, are governed by the specific synthesis route employed, making the choice of preparation technique a key determinant of properties and practical utility.

Among the various chemical synthesis techniques, the **sol–gel method[11]** is one of the most broadly utilized approaches for the fabrication of TiO_2 nanoparticles due to its simplicity, low processing temperatures, and capacity to produce highly homogeneous materials with controlled particle size and phase composition. In sol–gel processes, titanium alkoxide precursors undergo *hydrolysis and polycondensation* to form a colloidal "sol," which evolves into a gel that can be further dried and calcined to produce nanocrystalline TiO_2 powders (e.g., anatase phase with size tuning capabilities). Studies have demonstrated effective sol–gel synthesis yielding anatase TiO_2 with enhanced structural quality for applications such as photocatalysis and sensor technologies.

The **hydrothermal and solvothermal methods**[12] represent another widely used class of wet chemical techniques, in which reactions are conducted in aqueous or organic solvents under elevated temperatures and pressures within sealed autoclaves. These conditions facilitate controlled nucleation and crystal growth, producing TiO_2 nanoparticles with well-defined crystalline phases, narrow size distributions, and high surface area. Hydrothermal/solvothermal routes are particularly valued for tailoring distinct nanostructures—from spherical particles to nanorods and hierarchical forms—by adjusting parameters such as temperature, solvent, pH, and time.

Sonochemical synthesis[13] exploits high-intensity ultrasound to induce acoustic cavitation, creating localized hot spots with transiently elevated temperatures and pressures that accelerate chemical reactions. When applied to TiO_2 nanoparticle preparation, sonochemical methods can promote rapid nucleation and the formation of highly dispersed nanoparticles with modified surface characteristics. Such processes have been successfully integrated with co-precipitation strategies to produce doped TiO_2 nanocatalysts for advanced oxidation processes, demonstrating the adaptable nature of sonochemical routes for tailoring material properties.

The **co-precipitation method**[14,15] offers a simple, scalable pathway for TiO_2 nanoparticle synthesis through the controlled precipitation of titanium hydroxide or oxide from solution by adjusting pH and reaction conditions, followed by filtration, drying, and heat treatment. Variations in precursor type, pH, and calcination influence the crystalline phase and average particle size of the final TiO_2 product. Co-precipitation is a cost-effective approach widely used for bulk production and composite material preparation, though additional heat treatment is typically required to enhance crystallinity.

In summary, these diverse synthesis methods—sol–gel, hydrothermal/solvothermal, sonochemical, and co-precipitation—provide flexible platforms to engineer TiO_2 nanoparticles with tailored structures, morphologies, and functionalities[16]. Elucidating the mechanisms and effects of synthesis parameters on nanoparticle properties remains critical for optimizing performance across current and emerging applications.

Green synthesis[17] using plant extracts, microorganisms, or natural polymers has gained attention for its eco-friendliness, simplicity, and cost-effectiveness, producing biocompatible nanoparticles (10–50 nm) with high purity and crystallinity. Characterization through XRD, FTIR, UV–Vis, TEM, EDX, and BET analysis provides insights into structure, morphology, and surface area, which govern photocatalytic and biological activities. TiO_2 NPs act as efficient heterogeneous nanocatalysts in organic transformations such as Biginelli, Mannich, Hantzsch, and Friedel–Crafts reactions, offering high yields under mild, solvent-free conditions with excellent recyclability. They also exhibit superior photocatalytic degradation of pollutants like methylene blue, rhodamine B, and congo red, along with strong antibacterial and antioxidant properties. Green-synthesized TiO_2 NPs from sources like Aloe vera and Azadirachta indica show effectiveness against both Gram-positive and Gram-negative bacteria.

The scope of titanium dioxide (TiO_2) catalysts has greatly expanded from 2000 to 2025, evolving from simple UV-driven photocatalysts to multifunctional nanomaterials applied in environmental purification, organic synthesis, fuel production, and biomedical fields. Research has focused on surface modification, metal/oxide doping, and nanostructuring to enhance visible-light activity, stability, and recyclability. Presently, TiO_2-based catalysts play a vital role in sustainable energy conversion, green chemistry, and advanced pollution control technologies.

ADVANTAGES OF TIO_2 CATALYST:

1. High Stability: Chemically inert and resistant to corrosion, heat, and photodegradation.
2. Strong Oxidizing Power: Generates reactive radicals for effective degradation of organic pollutants.
3. Non-Toxic and Environmentally Friendly: Safe for large-scale environmental and biological applications.
4. Low Cost and Abundant: Economically feasible and widely available material.
5. Reusability: Can be easily recovered and reused in heterogeneous catalytic systems.

DISADVANTAGES OF TIO_2 CATALYST:

1. Wide Bandgap: Active mainly under UV light, limiting efficiency under visible light.

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2. Low Quantum Efficiency: Rapid recombination of photoinduced electron–hole pairs reduces activity.
3. Limited Surface Area: Reduces adsorption and catalytic reaction sites.

OBJECTIVES

The primary objective of this review is to provide a comprehensive and coherent overview of the synthesis, modification, and application of titanium dioxide (TiO_2) nanoparticles in organic chemistry and biological systems. The review aims to systematically analyze the major preparation strategies, encompassing physical, chemical, and environmentally benign (green) synthesis routes. Particular emphasis is placed on understanding how synthetic parameters—such as precursor type, reaction temperature, pH, and dopant incorporation—influence particle size, morphology, crystallinity, surface characteristics, and overall reactivity.

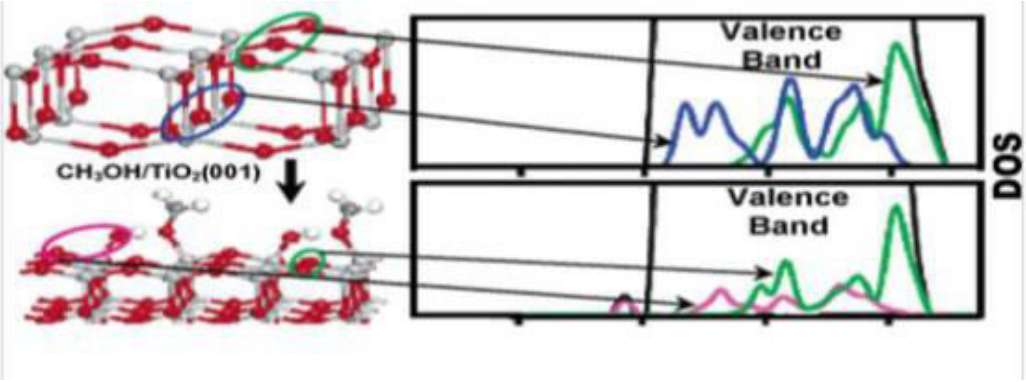
Another key objective is to critically assess the catalytic performance of TiO_2 nanoparticles across a broad range of organic transformations, including oxidation, reduction, coupling, condensation, and multicomponent reactions. This review seeks to correlate catalytic efficiency with reaction mechanisms, selectivity, reusability, and sustainability, thereby highlighting the role of TiO_2 nanomaterials as versatile and recyclable catalysts.

In addition, this review aims to explore the expanding biological and environmental applications of TiO_2 nanoparticles. Special attention is given to their antimicrobial, antioxidant, anticancer, and photocatalytic degradation activities, with a focus on linking these functional properties to their physicochemical features.

Finally, the review intends to identify current limitations and research gaps in the field, such as improving visible-light activation, minimizing nanoparticle aggregation, enhancing catalytic selectivity, and ensuring biocompatibility. By addressing these challenges, the review provides perspectives on the rational design of safer, more efficient, and environmentally sustainable TiO_2-based nanomaterials for future catalytic and biomedical applications.

LITERATURE REVIEW

Xue-Qing Gong and co-workers (2005)[18] carried out a detailed investigation into the interaction of methanol with anatase TiO_2 surfaces to better understand surface adsorption phenomena relevant to TiO_2 nanoparticles. Their findings showed that methanol exhibits a strong tendency to adsorb on anatase TiO_2 through a dissociative mechanism. Notably, this adsorption remains favorable even under hydrated conditions, indicating that methanol can effectively displace pre-adsorbed water molecules from the TiO_2 surface. This behavior highlights the higher surface affinity of methanol compared to water and provides valuable insight into the surface reactivity of TiO_2. The theoretical results reported in this study were found to be in good agreement with experimental observations on TiO_2 nanoparticles, thereby reinforcing the relevance of surface chemistry in determining the catalytic and adsorption properties of TiO_2-based nanomaterials .



Nanocrystalline titanium (IV) dioxide (TiO_2) has emerged as a versatile and efficient heterogeneous catalyst in organic synthesis, particularly for carbon–carbon bond-forming reactions. Lakshmi Kantam and co-workers demonstrated the catalytic potential of nanocrystalline TiO_2 in the conjugate 1,4-addition of indoles to α,β-unsaturated ketones, affording β-indolyl ketones in excellent yields under mild conditions[19]. The high activity of the catalyst was attributed to its large surface area and Lewis acidic sites, which facilitate effective substrate activation. Notably, the same catalytic system was also employed for a one-pot 1,2-addition of trimethylsilyl cyanide (Me_3SiCN) to various carbonyl compounds. This protocol showed remarkable tolerance toward diverse

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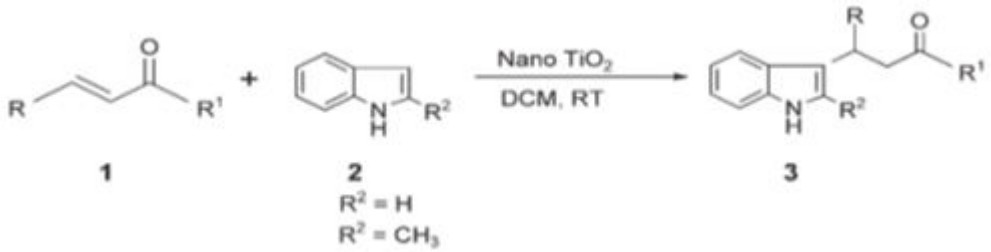
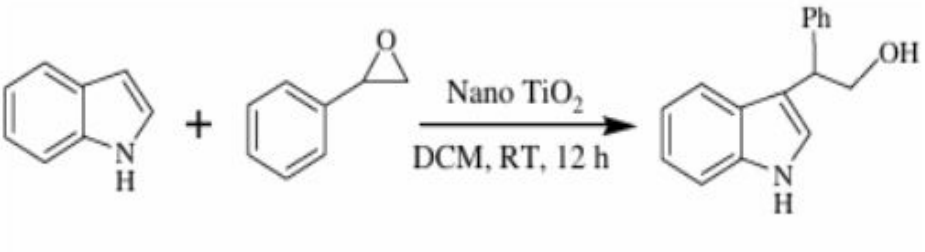
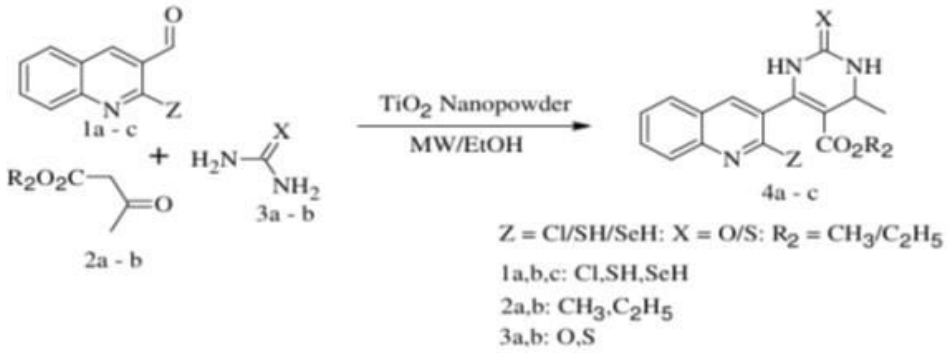
functional groups and strong resistance to moisture, underscoring the robustness of TiO_2 nanoparticles and their suitability for multistep and practical synthetic applications.



In a related study, Lakshmi Kantam et al. further explored the catalytic efficiency of nanocrystalline TiO_2 in Friedel–Crafts alkylation reactions[20]. The reaction of indoles with epoxides at room temperature resulted in the formation of 3-alkylindole derivatives in moderate to good yields with excellent regioselectivity. This work highlighted the ability of TiO_2 nanoparticles to effectively promote electrophilic aromatic substitution reactions under environmentally benign conditions, reinforcing their role as an attractive alternative to conventional homogeneous acid catalysts.



Expanding the scope of TiO_2-catalyzed transformations, Prakash Naik and co-workers reported a modified Biginelli reaction using non-powdery TiO_2 as a catalyst under microwave irradiation[21]. The method enabled rapid synthesis of dihydropyrimidinones under solvent-free conditions, significantly reducing reaction times while improving product yields. The combined use of microwave assistance and a recyclable TiO_2 catalyst offered a cleaner, energy-efficient, and sustainable approach to multicomponent reactions, aligning well with the principles of green chemistry.



Wu and co-workers reported an interesting hydrothermal strategy for fabricating core–shell Au–TiO_2 nanoparticles featuring a distinctive wedge-like TiO_2 shell structure[22].The epitaxial growth of the TiO_2 shell over the gold core was clearly evidenced through detailed structural analyses using TEM, HRTEM, and XRD techniques. The presence of fluoride ions derived from TiF_4 played a crucial role in directing the anisotropic shell growth and stabilizing the core–shell architecture. Owing to the strong interfacial interaction between Au and TiO_2 and the unique shell morphology, these nanostructures demonstrated remarkable photocatalytic efficiency in the oxidation of acetaldehyde under both ultraviolet and visible light irradiation. The enhanced activity was primarily attributed to improved charge separation at the Au–TiO_2 heterojunction and effective light utilization arising from the tailored nanostructure.

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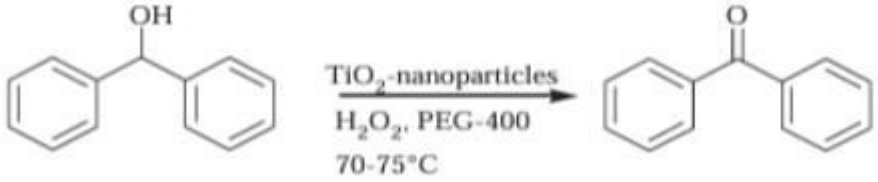
The catalytic potential of titanium dioxide nanoparticles has been widely explored in sustainable organic synthesis. In this context, Mirjalili and co-workers demonstrated the effectiveness of nano-TiO_2 as a green and reusable catalyst for the synthesis of quinoxaline derivatives through the condensation of 1,2-diamines with diketones [23]. The reaction proceeded smoothly under mild, ambient conditions and delivered high product yields, highlighting both the catalytic efficiency and environmental compatibility of TiO_2 nanoparticles. This study emphasizes the growing importance of TiO_2-based nanocatalysts as eco-friendly alternatives to conventional catalytic systems in heterocyclic chemistry.

Kidwai and co-workers reported a green and efficient strategy for the oxidation of secondary alcohols to the corresponding ketones using nanocrystalline TiO_2 as a heterogeneous catalyst in combination with hydrogen peroxide as the oxidizing agent [24]. In this method, TiO_2 nanoparticles played a dual role by enhancing catalytic activity while allowing easy recovery and reuse, thereby improving the overall sustainability of the process. The oxidation reactions were carried out under mild temperature conditions and did not require pressurized oxygen, making the protocol safer and more economical compared to conventional oxidation methods. The use of hydrogen peroxide further contributed to the environmental compatibility of the process, as it generates water as the only by-product. This study highlights the potential of TiO_2 nanoparticles as versatile and eco-friendly catalysts for selective oxidation reactions in organic synthesis.



Mahmood Tajbakhsh and co-workers (2012)[25] demonstrated the effectiveness of nanoscale TiO_2 as a heterogeneous catalyst in the one-pot Hantzsch condensation reaction for the synthesis of 1,4-dihydropyridine and polyhydroquinoline derivatives. The use of TiO_2 nanoparticles enabled the reaction to proceed under environmentally benign and mild conditions, delivering the desired products in excellent yields within a relatively short time. A key advantage of this catalytic system was the ease of catalyst separation from the reaction mixture, allowing its recovery and reuse over multiple cycles without noticeable loss of catalytic efficiency. These findings underline the potential of TiO_2 nanoparticles as a sustainable and practical catalyst for green organic synthesis.

Shahrzad Abdolmohammadi and co-workers demonstrated the catalytic potential of titanium dioxide (TiO_2) nanoparticles in multicomponent organic transformations by developing an efficient one-pot, three-component reaction under solvent-free conditions[26]. In their study, arylmethylidenepyruvic acids, 1,3-cyclohexanediones, and ammonium acetate were combined in the presence of TiO_2 nanoparticles to synthesize 5-oxo-4-aryl-1,4,5,6,7,8-hexahydro-2-quinolinecarboxylic acids. The reaction proceeded smoothly with high efficiency, affording excellent product yields within a short reaction time. The use of TiO_2 nanoparticles not only enhanced the reaction rate but also eliminated the need for hazardous solvents, highlighting the environmentally benign nature of the protocol. This methodology offers a simple, practical, and green synthetic route for the construction of biologically relevant heterocyclic frameworks, thereby underscoring the versatility of TiO_2 nanoparticles as sustainable catalysts in modern organic synthesis.

Shu-Juan Bao and co-workers [27] demonstrated an environmentally benign, biomimetic route for synthesizing TiO_2 nanomaterials with tunable crystal phases and morphologies by employing naturally derived biological templates . In their study, the crystal structure of the synthesized TiO_2 was found to be strongly influenced by the choice of biotemplate. Organic matrices such as yeast cells and egg albumen predominantly promoted the formation of the anatase phase, whereas dandelion pollen acted as an effective template for directing rutile phase formation. This template-guided synthesis strategy enabled controlled phase engineering under mild conditions, eliminating the need for aggressive reagents or energy-intensive processes. The work highlights the potential of biomimetic and green chemistry approaches for producing phase-selective TiO_2 nanostructures in a sustainable and scalable manner.

METHODOLOGY:

Titanium dioxide (TiO_2) nanoparticles were prepared following a sol–gel approach, which is widely recognized for producing uniform and catalytically active metal oxide nanomaterials. In this method, tetra-*n*-butyltitanate was used as the titanium precursor and mixed with deionized water in a 1:6 volume ratio under constant stirring to ensure controlled hydrolysis and condensation. The synthesis protocol was adapted from the work reported by Kassaee *et al.* (2011)[28]. The resulting sol gradually transformed into a gel, which was subsequently dried at 105°C to remove residual solvents. Calcination of the dried gel at 350 °C led to the formation of crystalline TiO_2 nanoparticles.

The synthesized TiO_2 nanoparticles were then evaluated for their catalytic efficiency in multicomponent organic transformations. TiO_2 catalyst was employed in the one-pot synthesis of β-aminocarbonyl compounds by the reaction of benzaldehyde, aniline, and cyclohexanone. The reaction proceeded smoothly at ambient temperature and reached completion within 2 h, highlighting the effectiveness of TiO_2 as a heterogeneous catalyst under mild conditions. Upon completion, the reaction mixture was filtered to recover the catalyst, followed by aqueous workup. The desired products were finally purified using column chromatography.

The application of TiO_2-based solid acid catalysts in multicomponent organic transformations has been demonstrated through the Mannich reaction using sulfate-functionalized titanium dioxide (SO_4^{2-}/TiO_2). As reported by Yao and co-workers (2017)[29], this catalyst system offers an efficient and reusable platform for C–C bond formation under mild conditions.

The reaction was investigated using both three-component and two-component strategies. In the three-component protocol, an aromatic aldehyde (20 mmol), a primary amine (16 mmol), and dimethyl malonate (22 mmol) were combined in dry toluene in the presence of 10 mol% SO_4^{2-}/TiO_2. The reaction mixture was maintained under an inert argon atmosphere and stirred at room temperature for 24 hours to facilitate product formation.

Alternatively, a stepwise two-component approach was employed, wherein the aromatic aldehyde and primary amine were initially allowed to react to generate the corresponding imine intermediates. These imines were isolated and subsequently subjected to reaction with dimethyl malonate under identical catalytic and solvent conditions.

In both methodologies, the heterogeneous SO_4^{2-}/TiO_2 catalyst was easily separated from the reaction mixture by simple filtration, highlighting its recyclability. The crude products were further purified by column chromatography to obtain the desired Mannich adducts in high purity. This methodology underscores the effectiveness of TiO_2-based solid acid catalysts in promoting sustainable and efficient organic synthesis.

In the green synthesis approach reported by Gautam *et al.* (2017)[30], *Jatropha curcas* leaves were used as a natural reducing and stabilizing agent for the preparation of TiO_2 nanoparticles. Fresh leaves were initially washed thoroughly with distilled water to remove surface contaminants, followed by fine chopping and sun-drying. An aqueous leaf extract was prepared by boiling 20 g of the dried leaf material in 100 mL of distilled water at approximately 80°C for 40 minutes. The resulting solution was allowed to cool and then filtered to obtain a clear extract.

The prepared plant extract was subsequently mixed with a 0.50 M titanium tetrachloride ($TiCl_4$) solution in an equal volume ratio (1:1) under continuous stirring at room temperature. The gradual development of a whitish-brown coloration served as a visual indication of TiO_2 nanoparticle formation. To facilitate complete precipitation, aqueous ammonia was added dropwise to the reaction mixture. The precipitated nanoparticles were collected by filtration and repeatedly washed with ethanol to eliminate residual impurities. The obtained material was air-dried and subjected to calcination at 450°C for 3 hours to improve crystallinity. Finally, the calcined product was gently ground to obtain TiO_2 nanoparticles in fine powder form.

Titanium dioxide nanoparticles were synthesized following a controlled hydrolysis–precipitation route as reported by Mya Hhit and Zm Min Myat (2019)[31]. Initially, a mixed acidic medium was prepared by combining hydrochloric acid with deionized water and cooling the solution in an ice bath. Titanium tetrachloride was then introduced gradually under continuous stirring, ensuring that the reaction temperature remained below 10°C to

prevent premature hydrolysis. After complete addition, the mixture was stirred for an extended period to allow uniform dispersion of titanium species.

The reaction mixture was subsequently heated moderately, followed by the addition of excess hydrogen peroxide, which induced a noticeable color change, indicating the formation of peroxo-titanium complexes. The pH of the solution was then carefully adjusted to alkaline conditions using aqueous ammonia, leading to the formation of a yellowish precipitate. This precipitate was subjected to an aging process at elevated temperature to enhance particle growth and structural stability.

The resulting product was repeatedly washed with deionized water to remove residual ions, separated by centrifugation, and dispersed using ultrasonic treatment to minimize agglomeration. Drying was carried out at low temperature, and the dried powder was finally calcined at high temperature for a fixed duration to obtain crystalline TiO_2 nanoparticles with improved phase purity and thermal stability.

CONCLUSION

Titanium dioxide (TiO_2) nanoparticles have gained considerable attention as green and effective heterogeneous catalysts for a variety of organic transformations, including one-pot Mannich reactions. Their ability to promote the synthesis of β-aminocarbonyl compounds under mild, solvent-free conditions makes them promising substitutes for conventional homogeneous catalytic systems. Furthermore, TiO_2-based catalysts are known for their excellent thermal stability and ease of recovery, allowing repeated use. However, literature reports highlight certain limitations, such as moderate product yields, insufficient control over stereochemistry, and gradual loss of catalytic activity upon recycling. To overcome these drawbacks, researchers have explored modified TiO_2 materials incorporating metal or non-metal dopants, including Pd, Co, and N, which have been shown to enhance surface reactivity, improve charge transfer, and increase selectivity. Despite these developments, systematic studies focusing on doped TiO_2 catalysts in Mannich reactions remain scarce. Accordingly, the present work was aimed to evaluate doped or hybrid TiO_2 nanoparticle systems to achieve improved catalytic performance, selectivity, and reusability toward sustainable β-aminocarbonyl synthesis.

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