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Review

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A Review: Factors Affecting Particle Size of TiO_2 Nanoparticles Prepared by Sol-Gel Method and Their Use in Biology

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Abstract

Almost every aspect of life is impacted by nanotechnology. We covered the TiO_2 NPs sol-gel synthesis process in this paper, along with the variables that affect the particles, including water, temperature, time, and PH as well as the type and concentration of the precursor and the solvent. Based on alcohol and water, sol-gel is a wet chemical reaction. Because of their fascinating properties, titanium dioxide (TiO_2) nanoparticles (NPs) have a wide range of applications in Nanobiotechnology. TiO_2 NPs have been studied for their antiviral and antifungal properties against plant pathogenic species, particularly Ustilago tritici, which causes wheat rust. TiO_2 NPs in agriculture have been shown to enhance plant growth and germination.

Keywords: TiO_2; nanoparticles; sol-gel synthesis; factors affecting particle size; Solvent Type; biological application

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Introduction

As per the reports, titanium, the ninth most frequent element in the crust of the earth, is a white, sturdy, silvery, and corrosion-resistant metal that is stronger than steel but less dense than steel. It is found naturally in several oxide forms, the most common of which is titanium dioxide (TiO_2) [1,2]. TiO_2, also known as Titania, is the result of the chemical interaction of titanium atoms with oxygen atoms from groups VI-B and IV-B of the periodic table. Because of its interesting properties, such as stability, non-toxicity, biocompatibility, and optical and electrical qualities, titanium dioxide (TiO_2) is a well-known and widely studied photocatalyst. Three well-known crystalline forms of TiO2 are anatase, rutile, and brookite, and Figure 1 [3] depicts the structures of each. Although the third form of TiO_2, brookite, has occasionally been described [4–6], the active crystalline forms of TiO_2 are anatase and rutile [7,8]. TiO_2 NPs are remarkable and versatile oxides with a bigger output because of their strong oxidation capabilities, high refractive index, low cost, and impressive and adaptable oxidation qualities [9]. TiO_2 nanoparticles have been applied as food additives, photocatalysts, and antimicrobials to degrade pollutants from water waste [10–13]. TiO_2 NPs can help destroy cancer cells, viruses, bacteria, and fungi [14]. These NPs have been used as sensors and in electronic devices because of their special electrical and ionic properties [15]. Anticancer drugs such as temozolomide, cisplatin, doxorubicin, and daunorubicin have all used TiO_2 nanostructure as a drug delivery mechanism [16–18]. TiO_2 NPs have been used in dental applications because of their ability to enhance the mechanical properties of biomaterials without compromising their biocompatibility [19–21]. Furthermore, TiO_2 has been widely used as a coating that cleans and sterilizes a range of therapeutic instruments, such as sanitary napkins, dinnerware, cooking utensils, and hospital supplies.[22]. The American Food and Drug Administration (AFDA) has approved TiO_2 NPs for use in human food, pharmaceuticals, cosmetics, and chemicals in food contact items such as cutting boards and other surfaces in contact with unprotected food. One anticipated application for this new material technology is the regulation of food processing plants' hygienic design in the future [23]. In this review, we have outlined the factors affecting the particle size of sol-gel-produced TiO_2 nanoparticles and their biological applications.

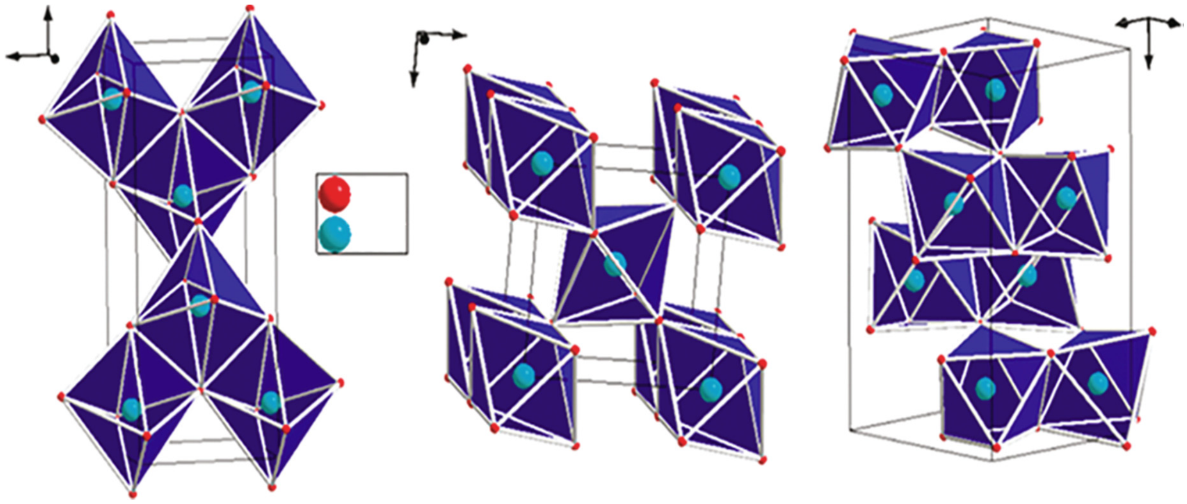

Figure 1. Different crystalline form Titanium Dioxide NPs [3].

Sol-Gel Method for TiO_2 Synthesis

In 1960, innovative materials such as thin films, dense powders, porous structures, and thin fibers were first created using the sol-gel technique. The sol-gel process involves the transformation of a liquid solution (sol) into a solid phase (gel). [24,25]. In material science and ceramic engineering, the sol-gel wet chemical process is the most often used method. An inorganic solid is created by employing water to start the polymerization processes that convert a precursor solution. The fundamental ingredients used in the production of sol are inorganic metal salts and metal alkoxide. a sol form following a series of polymerization and hydrolysis procedures. After that, the sol is treated to produce ceramic materials in different forms. Pouring the soul into a mold will result in a wet gel. Sol undergoes further drying and heat treatment to become solid ceramic materials. An aerogel that is extremely porous and low in density is produced when liquid from wet gel is removed under supercritical circumstances. [26]. After adjusting a sol's viscosity to come within the necessary range, ceramic fibers can be removed from it.[27]. The detailed procedure of sol-gel is depicted in Figure 2. TiO_2 nanostructure was produced through hydrolysis of titanium precursor using the sol-gel method. This method usually involves hydrolyzing titanium (IV) alkoxide condensational while an acid is present [28–41]. This technique consists of four steps. The first four steps are hydrolysis, polycondensation, drying, and heat deterioration. Equations (1) hydrolysis and (2) condensation indicate [28] that gels are produced by the condensation polymerization reaction, which is triggered by the hydrolysis of metal alkoxides with water in the sol-gel process.

$$Ti(OR)_4 + 4H_2O \rightarrow Ti\,(OH)_4 + (ROH)_4 \qquad \text{1. Hydrolysis}$$

$$Ti(OH)_4 \rightarrow TiO_2 + 2H_2O \qquad \text{2. Condensation}$$

Where R stands for isopropyl, ethyl, etc. Solvent loss occurs during the entire polymerization process, which transforms the liquid sol into a solid gel phase. The soil particle size is significantly influenced by temperature, pH, and solution chemistry [29]. TiO_2 NPs can be synthesized by using different precursors such as titanium trichloride ($TiCl_3$) [30], titanium tetra isopropoxide $Ti[OCH(CH_3)_2]_4$ [31], titanium tetrachloride ($TiCl_4$) [32] and titanium tetra butoxide $Ti(OBu)_4$ [33].

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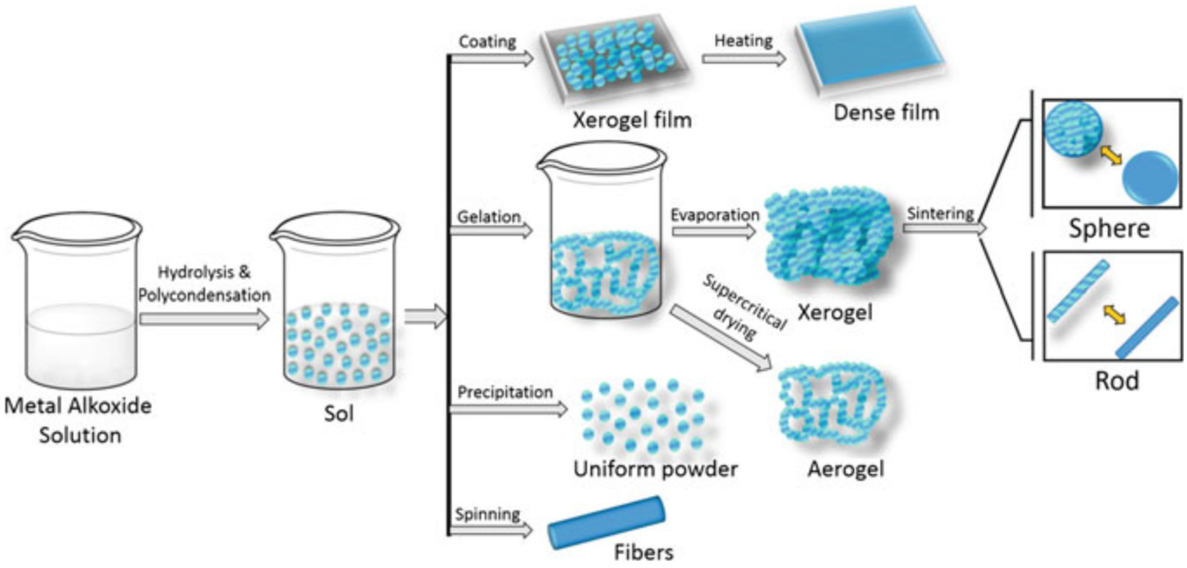

Figure 2. Schematic representation of sol-gel process [34].

The initial stage in the alcohol-based Sol-gel synthesis of TiO2 begins with metal alkoxide, whereas the initial step in the aqueous-based Sol-gel synthesis is inorganic metal salt.

Alcohol-Based Process: Three important metal alkoxide components of TiO_2 are $Ti(OC_2H_5)$, $Ti(OC_3H_7)_4$, and $Ti(OC_4H_9)_4$. These alkoxides have a metal-oxygen bond, and because Ti and O have very different electro-negativity levels, the bond becomes highly polar and extremely reactive. As a result, the process continues by adding water while simultaneously causing condensation and hydrolysis, which ultimately leads to the creation of a gel [35].

Aqueous Based Process: $TiOSO_4$ and $TiCl_4$ are significant aqueous-based process precursors [36,37]. The hydrolysis and condensation process can also be used for aqueous-based sol-gel synthesis [38]. The water-based phases of the sol-gel process are precipitation and peptization. The inorganic metal salt must be hydrolyzed to undergo precipitation; once this is done, the salt rapidly becomes a gelatinous precipitate with the addition of base. Next, the extra electrolyte is removed by washing. Peptization is the process of re-dispersing a colloid to stop coagulation [35]. The process of directly separating material into colloid-sized particles by adding a peptizing chemical is known as peptization [39]. Peptizing a colloid that has reached a main potential minimum is especially difficult.

Factors Affecting Particle Properties of TiO_2 NPs

PH: With an increase in the pH of the sol [40], the TiO_2 NPs typically grow larger. When making TiO_2 NPs using the sol-gel technique, pH is crucial for determining the final particle size and shape [41]. High concentrations of hydrogen ions interfere with the reaction and slow down nucleation, which causes the particles to expand quickly to form big grains. It will take the new nucleus some time to develop into sizable TiO_2 particles [42]. Both the size of nanoparticles and the stability of sol are dependent on the pH level, which also controls acidity [43]. By aging a highly acidic solution of $TiCl_4$ at high temperatures for 6-47 hours, Matijevic et al. [44] reported the production of TiO_2 spherical particles of a narrow size.

Precursor Concentration: The number of TiO_2 nuclei formed at high titanium (IV) isopropoxide (TTIP) precursor concentrations causes' enhanced coagulation and sintering, which causes the size of the particles to increase as the precursor concentration rises [45]. Moreover, higher precursor concentration enhances the anatase phase's crystallinity and increases the probability that it will change into the rutile phase [46].

Precursor Types: In their investigation of the impact of different alkoxides on TiO_2 NP size, Vorkapic and Matsoukas discovered that at 25 ^{o}C, the final size reduces as the alkoxy group's length increases. [43]. Their findings demonstrated that the order of decreasing particle size was ethoxide,

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isopropoxide, isopropoxide, and butoxide, which corresponded to the order of lowering the alkoxides reactivity and, thus, the hydrolysis rate.

Solvent Types and Concentration: Generally, molecular addition causes the particles to grow in size after nucleation; however, the kind of solvent used affects this growth since different solvents have different potentials for particle interaction. Researchers found that the size of the particles increased and the size without alcohol dropped when the amount and molecular weight of alcohol decreased [43]. This is because an increase in molecular weight and concentration results in a decrease in the solvent's dielectric constant, which in turn leads to an increase in the rate of re-aggregation, a decrease in stability, and larger particle sizes. Park et al. [47] examined the creation of TiO_2 NPs through the thermal hydrolysis of titanium tetrachloride ($TiCl_4$) in mixtures of water and n-propanol. The results of the study show that when the powders were dissolved in various solvents, the degree of aggregation increased in the order of methanol, ethanol, and propanol, suggesting that colloidal destabilization was the primary mechanism by which these alcohols changed particle size. In another investigation by Xu et al. [48], the photo-catalytic activity of unsupported TiO_2 steadily rose with the rise in the chain of the solvent used in manufacture due to an increase in anatase content and a decrease in particle size. They discovered this after changing the solvent from methanol to 2-propanol. At a particle size reduction of 11.6 nm to 10.5 nm, the anatase content increases to 91%. A significant amount of amorphous TiO2 can be discovered in the final sol, and the rate of hydrolysis can slow down as the concentration of alcohol in the sol-gel reaction mixture increases.

Temperature: Temperature has a significant role in regulating the characteristics and particle size of TiO2 NPs during sol-gel production. Vorkapic and Matsoukas investigated how the temperature of the hydrolysis process affected the particle size [43] They examined temperature effects between 0 and 50 ^{o}C and discovered that lower hydrolysis temperatures promoted the production of bigger particles. The size reduces with increasing temperature, reaching its minimum between 25 and 50 ^{o}C. High temperatures reduce the solvent's viscosity and dielectric constant, increasing the thermal energy of colloids and lowering the electrostatic barrier to aggregation, which leads to bigger particle sizes [49].

Water: The hydrolysis reaction is significantly influenced by the water (H_2O) content. According to Xiaobo [50], the formation of Ti-O-Ti chains through alkoxylation is encouraged when the reaction mixture has an abundance of titanium alkoxide and has a low water content and low hydrolysis rates. Water concentration shouldn't be too low otherwise the alkoxide will only partially hydrolyze with the water, which will cause condensation between the $(OH)_X$ $Ti(OR)_{4-X}$ monomers [51]. According to some researchers, the equation R= $[H_2O]$/[TEOT] larger than 2.5 indicates that the water-to-alkoxide ratio needed for particle production should be greater than 2.5 [52]. Particles having an average size of 300 nm were obtained from the greatest R-value known, 7, which was. A greater nucleophilic interaction between water and alkoxide molecules happens as the amount of water is increased, leading to more alkoxyl groups being replaced by the OH group of water. The resulting monomers then engage in intermolecular interactions to create a three-dimensional network structure. The hydrolysis is more complete and more alkoxide converts to the corresponding metal hydroxide $M(OH)_Z$ when the R-value is over a critical value, whereupon the two react to create polymer-like particles [51]. High hydrolysis rates brought on by a lot of water favor Ti $(OH)_4$ production.

The molar ratio of H_2O/alkoxides (RW) utilized in the sol-gel method, as shown in Figure 5(a), has a significant impact on the properties of the resulting oxides, according to a study by Yu and Wang [51]. They proposed that the RW utilized would affect the reaction mechanism for sol-gel conversion, and they proposed three distinct ways, as illustrated in Figure 5(b). All mechanisms might operate simultaneously, but one would predominate [51]. Case I: Condensation reaction between the monomers happens when RW is less than four (RW 4). This is because the hydrolysis between the alkoxide and water is incomplete. Case II: A greater nucleophilic reaction between water and alkoxide molecules and the three-dimensional network occurs when RW is raised between two and four (2 RW 4). Case III: The hydrolysis will be complete when RW is used over the crucial value. The matching metal hydrate will be formed from the alkoxide. The monomers will react with one

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another to create polymer-like particles. The effects of various parameters on the characteristics of TiO_2 NPs are shown in Table 1.

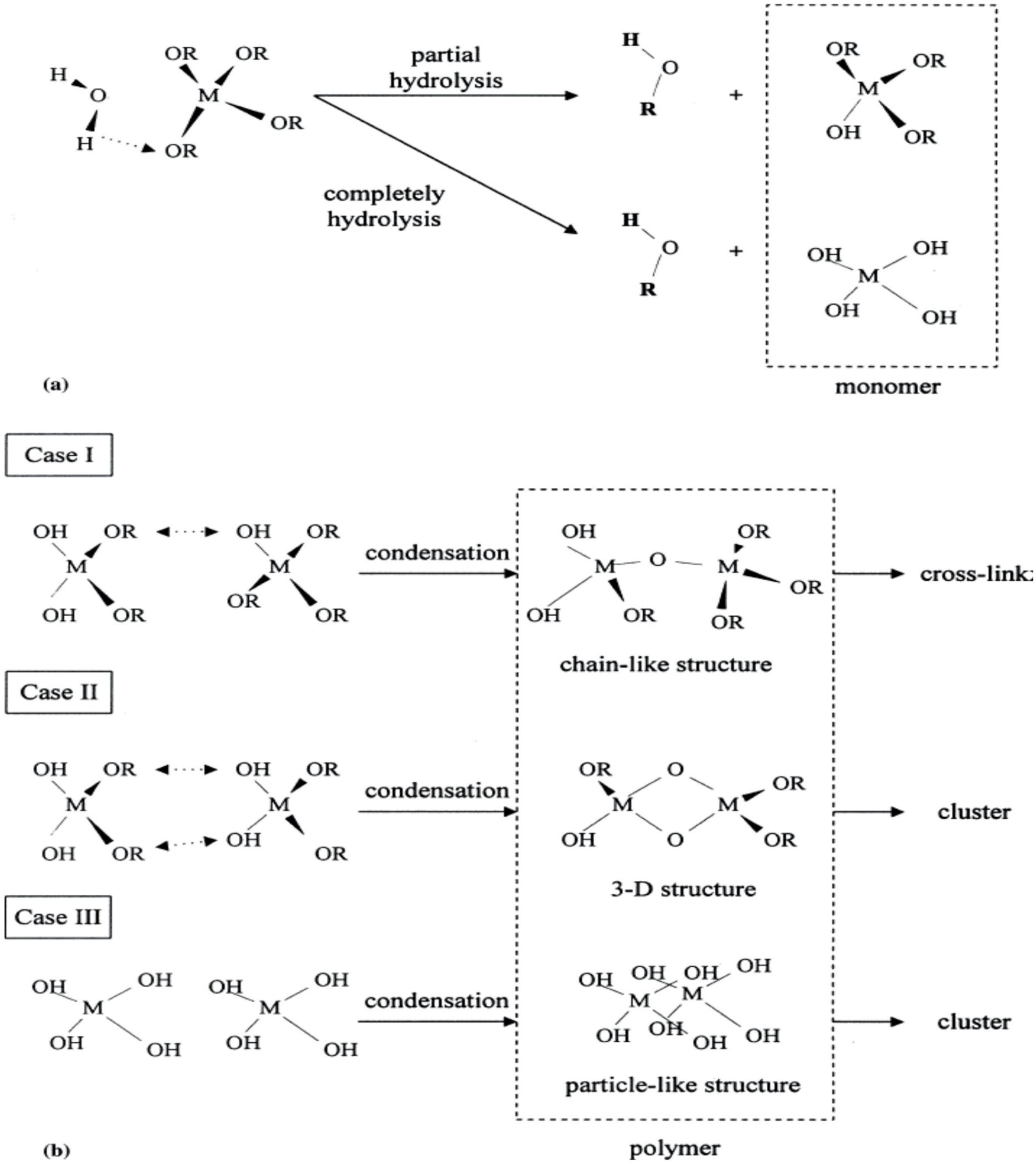

Figure 5. (a) (b) Sol-gel process with different water content [51].

Table 1. Effect of different factors on the properties of TiO_2 nanoparticles.

Precursor	Solvent	Catalysis	pH	Temperature oC	Crystalline phase	Particles size	Reference
$Ti(OBu)_4$ 5 cc	Isopropyl Alcohol 10 cc	HNO_3	2	400 600	Anatase Mix (anatase + rutile)	6 nm 25.8 nm	[53]

				800	Rutile	33 nm	
TTIP 0.4 M	Ethyl alcohol 0.1 M	NaOH	8.5	450 900	Anatase Rutile	21-24 nm 69-74 nm	[54]
TTIP 5.5 ml	Ethanol 54.8 ml	NH_4OH Or HCl	1 7 10	500	Anatase with rutile Anatase Anatase	14 nm 19 nm 20 nm	[55]
TTIP 5 mL	Deionized water 100 mL	NaOH	7 9	400	Anatase	14 nm 16 nm	[56]
TTIP 76.7 ml	Isopropanol 76.4 ml	HNO_3	1.5	400 500	Anatase	5-10 nm	[57]
$TiCl_4$ 3.5 ml	Ethanol 35 ml	NH_4OH	1.1	250 400 600	Anatase	9.22 nm 14.33 nm 36.72 nm	[58]

Uses of TiO_2 NPs in Biology

Titanium dioxide (TiO_2) NPs have a wide range of uses in Nanobiotechnology as a result of their intriguing features [59]. The effects of several metallic NPs on various bacterial strains have been investigated. Due to their strong oxidizing potential, titanium dioxide (TiO_2) NPs exhibit eco-friendly biocidal capabilities. Numerous bacterial strains, endospores, fungi, algae, protozoa, viruses, microbial toxins, and prions are just a few of the infectious microbes that titanium dioxide (TiO_2) NPs are used to treat [60]. In the next subsections, a few of these common uses for titanium dioxide (TiO_2) NPs will be briefly explored.

***Antiviral Activity of TiO_2 NP*:** Nanotechnology advancements have proven to be successful therapeutic solutions for treating a variety of ailments, including viral infections. Nano conjugates including silver, TiO_2, and carbon have been demonstrated to directly interact with the target viruses in various ways. The capsid protein coat and genetic material (DNA or RNA) make up the virus. Among infectious diseases like influenza, hepatitis B and C, HIV, herpes, and the present catastrophic coronavirus, viruses pose the greatest threat to humankind. One of the key strategies for combating viruses is the physical destruction or photo-catalytic oxidation of the viral protein sheath by nanomaterials. Viral particles are broken up by TiO_2 NPs by an unidentified interaction with the viral envelope. In another study, it was demonstrated that TiO_2 interacts with the capsid proteins, which is followed by their oxidative destruction and deactivation. Examples of this kind of devastation have been described in MS2 bacteriophage viruses and influenza viruses [61–63].

In another work, TiO_2 Nano conjugates were found to have direct interaction-mediated antiviral activity against the H3N2 influenza virus [64]. Additionally, TiO_2 NPs have shown antiviral efficacy against the H9N2 avian influenza virus. Utilizing DNA-tagged TiO_2 NPs prevented H5N1 and H1N1 viral proliferation in addition to the influenza virus [65]. At a minimum concentration of 6.25 g/ml, TiO_2 Nano colloids also exhibit antiviral action against NDV, which may be caused by the lipid breakdown of the viral coat [66]. To ascertain the antiviral activity of TiO_2 nanostructures on BBSV, which damages the fava bean plant, a study was conducted [67]. The broad bean stain virus (BBSV) was combated in faba bean plants using TiO_2 nanostructures. The faba bean with TiO_2 nanostructure treatment compared to untreated plants, showed a greater decline in the illness in the two weeks after infection with the broad bean stain virus (BBSV) [68]. The study demonstrated that the PR1 gene, or the defensive regulatory gene in the salicylic acid signaling pathway, was strongly expressed following the application of TiO_2 nanostructures, reducing the severity of the disease. Due to their small size and high zeta potential, TiO_2 NPs most likely interacted with virus particles to accomplish this. Shortly, titanium NPs would make an excellent platform for the treatment of New Castle disease virus infections as well as an excellent option for the evaluation of novel antiviral medications [66].

Antifungal Activity of TiO_2 NPs: Wheat crops were treated with NPs to test the antifungal activity of TiO_2 NPs against the plant pathogenic species Ustilago tritici that causes wheat rust. TiO_2 NPs were tested against the U. triciti fungus at three different concentrations (24, 50, and 75 μL) in comparison to a control. The third concentration (75 μL) demonstrated the strongest and most significant effects, which resulted in a 62% growth reduction against U. tritici, even though all three concentrations had outstanding antifungal activity [69].

Use of TiO_2 NPs in Agriculture: According to Chao and Choi's findings, TiO_2 NPs boost plants' ability to photosynthesize and their resistance, which results in a 30% rise in crop yield [70]. Onion spinach showed an increase in photosynthetic rate [71]. According to a different study, using TiO_2 nanostructures increased plant growth and germination [72]. The proposed process includes an increased stimulation of the creation of carbohydrates and increased photosynthesis since it has light-absorbing characteristics [73]. TiO_2 can also control nitrogen-metabolizing enzymes such as glutamate dehydrogenase, nitrate reductase, glutamine synthase, and glutamic-pyruvic transaminase by absorbing nitrates. These enzymes can change inorganic nitrogen into proteins and chlorophyll [69,74,75].

It was discovered that Zea mays sprayed with Nano TiO_2 during the crop's productive stages had greater pigmentation [76], which is what increases crop output [74]. TiO_2 NPs may be sprayed onto leaves or added to the soil. Different crops' photosynthetic rates can be accelerated with TiO_2 NPs, which can also increase agricultural productivity. When applied to tomato plants, TiO_2 NPs in aerosol form were found to be more effective at boosting photosynthesis and lycopene content [77,78] than TiO_2 NPs applied as a soil additive. Strong irradiance TiO_2 NP exposure caused numerous phenotypic and physiological responses in tomato plants, including an increase in fruit and flower output, augmentation of anthocyanin and carotenoids, and an increase in enzyme activity. High fruit output was the most notable finding with Nano TiO_2, which was presumably caused by a stress-induced reaction that accelerated propagation [79]. Among the many other known NPs, TiO_2 NPs have been proven to be effective and can be widely employed in agriculture, especially for the remediation of soil contaminated with heavy metals.

According to earlier research [80,81], NPs have both antagonistic and synergistic effects on heavy metal accumulation and plant growth under varied environmental conditions. In a different study, the impact of TiO_2 NPs on the morphological alterations of wheat plants was explored. White plants were treated with TiO_2 NPs in increasing doses, however, they did not exhibit any visual signs like chlorosis or necrosis. White plants' roots and shoots' dry weight (DW) were not significantly impacted by the TiO_2 NP treatment. Plant growth was unaffected by an increase in TiO_2 NP dosage [82]. White seed germination or root elongation was not statistically altered as a result of TiO_2 NP treatment [83].

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Conclusion

Due to its intriguing features, including stability, non-toxicity, biocompatibility, and optical, and electrical qualities, titanium dioxide (TiO_2) is a well-known and extensively explored photocatalyst. The American Food and Drug Administration (AFDA) has approved that TiO_2 NPs are used in human food, medications, cosmetics, and compounds in food contact materials such as cutting boards and other surfaces in contact with unprotected food. The sol-gel process was originally used to synthesize TiO_2 NPs considering different parameters for controlled particle size. TiO_2 NPs were used for antiviral and antifungal activities showing the best result of inhibition for both virus and fungus. TiO_2 NPs are also used in agriculture, Plant growth was unaffected by an increase in TiO_2 NP dosage. According to a different study, using TiO_2 nanostructures increased plant growth and germination. In this review, we discussed the factors affecting particle size and their uses in biology.

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