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Preparation Method of Titanium Dioxide Nanoparticles and Its Application: An Update

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Abstract. Titanium dioxide (TiO_2) is widely used because of its good biocompatibility and stability. Various methods were used to prepare TiO_2 by using chemical, biological, and physical methods. In this paper, the currents methods to prepare TiO_2 were evaluated either with or without using plant extract. The average particle size produced for different methods of technique and its application was also analyzed. Chemical methods use toxic chemical while physical methods such as gamma produce high energy and using biological method with plant extract is more environmentally friendly. This review gives an up-to-date summary of TiO_2 produced using various synthesis methods with the focus on their particle sizes properties. Recent publications on this topic were extracted from related journals obtained from Science Direct online database from the year 2010 to 2021. Preparation methods, average particles sizes, and the applications of the synthesized TiO_2 were evaluated and further discussed in this review. It was found that TiO_2 of smaller particle sizes were obtained when synthesized without using plant extract. Furthermore, many researchers tend to combine TiO_2 with other materials or composites to be mixed with polymers for various purposes of application especially wastewater treatment application.

1. Introduction

Nanotechnology has become one of the most popular topics in the recent decade as it evolves into becoming a key technology in the 21st century. The development of nanotechnology has shaken the world as they witnessed its capabilities in numerous innovative applications in various fields. Generally, nanotechnology can be defined as the creation of nanoparticles with a dimension ranging from 1 to 100 nm by manipulating the atomic and molecular matter through new techniques or devices [1]. Nanoparticles are known to possess unique physical and chemical characteristics and the application of nanoparticles has been mainly concentrated in the fields of chemical, energy, health care, and cosmetics [2].

Among all kinds of nanoparticles, there are metal oxide nanoparticles such as AgO, ZnO, MgO, CuO, and TiO_2[3][4]. Each type of metal oxides has its own unique properties like electrical, magnetic, optical, thermal, effective against drug-resistant bacteria, viral, and fungal strain as well as catalytic activity[5][6][7].For example of silver nanoparticles, it is used due to its antimicrobial properties and can be applied in wide applications [4]. Compared to other metal oxide nanoparticles, TiO_2 is the most astounding nanomaterial because of its chemical stability, photocatalytic properties, high stability, biocompatibility, and non-toxicity [7][8]. Other than that, TiO_2 is an essential semiconductor

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photocatalyst that covers a broad range of applications in water treatment, dye-sensitized solar cells, cosmetics, pigments and for seed germination [9] [10]. In the application of pigment making, TiO_2 is used to provide opacity and whiteness to products such as paints, plastics, coatings, sunscreen, inks, medicines for most toothpaste and food [11].

TiO_2 can be synthesized through three approaches which are physical, chemical, and biological methods. Physical methods include thermal decomposition, laser irradiation, and electrolysis while chemical methods use chemicals such as sodium borohydride or sodium citrate as reducing agents. Physical methods typically use highly intensive costly equipment that involves vacuum and chemical methods are considered as the most commonly used technique to synthesize nanoparticles [12]. Usually, the shape, size, and other properties of TiO_2 can be controlled depending on the methods of synthesis [13]. Preparation through chemical and physical methods can lead to environmental problems upon discharge due to the usage of dangerous chemicals [13][7]. On the other hand, biological method or green synthesis is considered to be less poisonous and more friendly to the environment as the usage of toxic chemical can be removed and reduced because plant extract has a stabilizing and reducing agent properties during the reaction[14][15]. In addition, TiO_2 has been successfully synthesized using green synthesis by utilizing plant extract from different plants such as *Jatropha curcas*, *Azadirachta indica*, and *Psidium guajava* [14][7][16] whereby the extract is obtained from the leaves, roots, twigs and, the seeds.

In this review paper, comparative analysis of the preparation methods of TiO_2 nanoparticles using plant extract and without plant extract will be discussed. The scope of the referred journal is using a Science Direct online database and research papers published from the year 2010 until 2021 only. Throughout the study, the particle sizes for TiO_2 were compared based on the preparation method and presence or absence of plant extract during synthesis. Lastly, the potential application of TiO_2 is also discussed and explored in this paper.

2. Preparation Method of Titanium Dioxide

Based on the findings from the referred journal, there are various techniques that can be adopted for the preparation of TiO_2 nanoparticles such as sol-gel method, instantaneous synthesis method, solvothermal method, microwave-assisted synthesis, simple mixing, and precipitation method. Figure 1 shows the illustration of these commonly used synthesis methods of preparing TiO_2. Meanwhile, Table 1 lists recently published research papers conducted on the preparation of titanium dioxide with different particle sizes and its application.

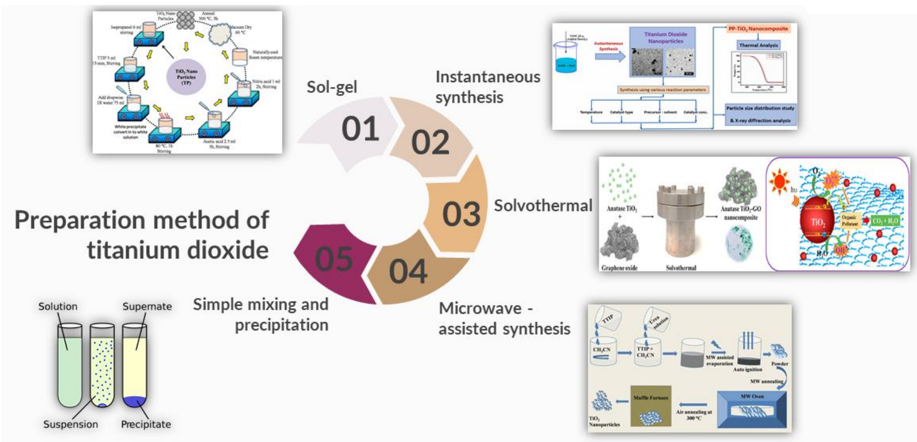

Figure 1. Preparation method of titanium dioxide

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Table 1. Previous research conducted to prepare TiO_2 using different techniques which produced different average particle size and its suitable application.

Nanocomposite	Plant Extract	Preparation Technique	Average particle size	Application	References
TiO_2/Carbon (C)/polyaniline (PANI)	-	TiO_2/C-Prepare from sol-gel method TiO_2/C/PANI - in situ chemical oxidative polymerization	TiO_2 - 40 nm TiO_2/C - 31 nm TiO_2/C/PANI - 20 nm	Provide optimum photocatalyst for degradation of Methylene Blue and Crystal violet	[17]
Polypropylene (PP)/TiO_2	-	TiO_2-instantaneous synthesis method	TiO_2 - 10-20 nm	Can be used as pigment in paints, cosmetics, and food additives product	[18]
Chitosan/polyethylene oxide-Ag/TiO_2	Ag- fresh leaves of *Chenopodium murale*	Ag- aqueous extract TiO_2 - sol-gel technique	Ag - 20 nm TiO_2 - 15 nm	Has a potential to use nanocomposites for food packaging application	[19]
poly(sodium styrene sulfonate)/ TiO_2	-	TiO_2 - solvothermal method previous hydrolysis of titanium (IV) isopropoxide (TTIP). Grafting method based on UV radiation	Nanosphere TiO_2 – 7 to 10 nm Nano rods TiO_2- 15 to 22 nm	Used for bio-polymer grafting and in nanomedicine applications	[20]
COP-Ps(Covalent porphyrin polymers)/TiO_2	-	in situ hydrothermal method	TiO_2- < 200 nm	Improved the photocatalytic activity to reduce CO_2	[21]
poly(3-hexylthiophene) (P_3HT)-TiO_2	-	TiO_2-microwave assisted synthesis P_3HT-TiO_2 - in-situ polymerization	TiO_2-7nm	Application in photovoltaic devices	[22]
Ag-TiO_2/porous polymer composite	-	Ag-TiO_2/porous polymer composite - in-situ solvothermal process. (Porous polymer - co-polymerization of divinylbenzene (DVB) and bismaleimide)	TiO_2- 10.3 nm TS -18.2 nm PPTS - 13.2 nm	It has a potential as a sustainable solution for pollution abatement	[23]

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Table 1. Continued

Nanocomposite	Plant Extract	Preparation Technique	Average particle size	Application	References
TiO_2	-	Sol gel reaction from variable amounts of waste derived polymeric biosurfactant.	TiO_2 Anatase + Brookite - 18 nm Anatase - 10-10.5 nm	Photocatalytic. Also, can be used for the remediation of aqueous effluents contaminated by organics	[24]
Polypyrrole(PPy)/Ag-TiO_2	-	Ag/TiO_2 - Sol-gel method PPy/Ag-TiO_2 – In situ oxidative polymerization.	PPy/Ag- TiO_2 - 1 nm	Used for sensor application to detect ammonia gas at room temperature	[25]
TiO_2	-	Modified sol gel methods using hyperbranched polyethyleneimine polymer template (HPEI)	TiO_2 - 200 nm	Applied in environmental application in fields such as solar cells, water treatment and air purification.	[13]
Cellulose/ TiO_2	-	Coagulation in sodium hydroxide-thiourea-urea aqueous solution medium by precipitation method.	FESEM Images TiO_2 - 2 nm cellulose/ TiO_2- 1 nm TEM Images TiO_2 - 0.2 nm cellulose/ TiO_2- 50 nm	Application in many variable fields - especially in pharmacology and biotechnology	[26]
TiO_2	Extract of *Azadirachta indica* leaf	TiO_2-Precipitation	Spherical shaped TiO_2- 15 to 50 nm (TEM) SEM image – 25 to 87 nm	Used to fight against vast range of pathogen due to its antibacterial properties	[7]
Poly (methyl methacrylate) (PMMA)/2-aminopyridine/ 2-nitroaniline doped with TiO_2	-	TiO2- precipitation	TiO_2- 5 nm	Synthesized nanocomposites are low-cost material for water treatment, industrial effluents- Congo Red removal and preservatives for food	[27]
TiO_2	Extract of *Aazadirachta indica* twigs, *Ficus benghalensis* and *Syzygium aromaticum*	TiO2 - precipitation	TiO_2-20 nm (tetragonal shapes)	Application as antibacterial, antibiofilm, antifogging and mosquitocidal activity	[28]

Table 1. Continued

Nanocomposite	Plant Extract	Preparation Technique	Average Particles Size	Application	References
borneol-based polymer/ TiO_2 composite film	-	Borneol-based polymer - prepared by free radical polymerization. Composite film - borneol- based polymer dispersed with TiO_2 particles and polytrifluoro chloroethylene (PCTE) binder in tetrahydrofuran to form dispersion	TiO_2 - 5 to 10 nm	Suitable for wastewater treatment	[29]
TiO_2	-	Sol-gel procedure with certain ratio of brookite phase	BJH Method TiO_2- 8.36 nm pei-TiO_2 - 8.85 nm dt-TiO_2 - 8.93 nm	Photodegradation of methylene blue in water and as basic heterogeneous catalyst carbon-carbon forming reactions (act as a catalyst).	[30]
TiO_2	*Jatropha curcas L. latex* (Hedge castor oil)	Precipitation method	TiO_2- 10-20 nm spherical in shape	Used as pigment in paints, removal of toxins from air and water, biomedical sciences such as bone tissue engineering and pharmaceutical industries.	[14]
TiO_2	*Psidium guajava* (Aqueous leaves extract)	Precipitation method	TiO_2 spherical and cluster - 32.58 nm average.	Used as antibacterial coatings and wastewater disinfection	[16]
TiO2	*Glycyrrhiza glabra (licorice) (roots extract)*	Simple mixing and precipitation method	TiO2- 69 nm	Used as multifaceted therapeutic agent demanding in vivo studies	[31]

First of all, sol-gel method is one of the routes for the synthesis of nanoparticles and nanocomposites. Under mild conditions, the sol-gel method allows the mixing of two or more dissimilar phases to form hybrids [32].In sol-gel methods, the production of homogenous nano-sized materials of complex composition can be easily produced with a cost-effective production [33]. From the name of the method, the sol represents the chemical transformation of the liquid into a gel phase and subsequently transition into solid oxide material [34]. The three-dimensional network structure of sol-gel is prepared by freeze-drying and calcination [35].The synthesis of TiO_2 NPs from sol gel method is a wet chemical technique that mostly used in ceramic engineering and material sciences [15] Heshmapour and his co-researcher use sol-gel method for the synthesis of TiO_2 and then mixing with ceramic and PANI. From their experiment, spherical shaped and relatively agglomerated large particles of TiO_2 were produced. After the addition of ceramics and PANI, the particles gradually become smaller and show the disappearance of agglomeration. The reason for the disappearance of agglomeration is due to the porous surface of ceramics that increase the number of catalytic active sites. For this reason, the photocatalytic activity of TiO_2 can be amplified when it is modified with ceramic and polyaniline (PANI) which leads to the least agglomeration and clustered TiO_2. [17].

In addition to ceramic and PANI, modification of TiO_2 other nanocomposites such as waste-derived polymeric biosurfactant and hyperbranched polyethyleneimine polymer template (HPEI) have also been conducted using sol-gel method [24][36]. The sol-gel method can produce highly pure crystalline nanoparticles powder at low synthesis temperature without requiring a washing step since it is a waste-free synthesis and [37][34]

Another alternative method to synthesize TiO_2 is through the instantaneous synthesis method. As the name of the process suggests, the synthesis of TiO_2 is only in one-step process. [18]. The process reaction time of a few seconds, which is 3-5 seconds, was performed at 25 °C with no external added energy. Prakash and his co-researcher conducted their research using TiO_2 compounded with polypropylene and the time taken for production of TiO_2 only took around 5 seconds at 25 °C [18]. This technique is proven faster than the sol-gel method which takes at least 4 hours of stirring and further drying at room temperature to prepare TiO_2 [38].

The third method used for TiO_2 synthesis is by using the solvothermal method. The solvothermal method has the ability to direct precipitate arbitrary phases from the liquid phase [39] and it also appraises as a very appealing method to produce materials with different morphology [40]. Besides, this method is called the solvothermal method because it uses organic solvents in the synthesis procedure. Based on Faline-Daudre and teams, the fabrication of TiO_2 were prepared by a solvothermal method that uses the hydrolysis of titanium (IV) isopropoxide in the presence of benzyl alcohol and acetic acid at 180°C which resulted in a different shape and size of TiO_2 [20]. Alternatively, there is another reaction which is called hydrothermal. The hydrothermal method is analogous to that solvothermal method whereby it uses water to act as solvents instead of organic solvents [40]. According to Zhang and teams, nanoparticles were prepared by heating titanium sulfate as metal sources with ammonium hydrogen fluoride as a shape control agent. From this technique, the relatively pure TiO_2 is obtained which exhibits the improvement of photocatalytic activity [21].

Microwave-assisted synthesis is included as one of the methods for the synthesis of TiO_2. In accordance with this process, the reactants are directly heated by microwave. The chemical reaction is faster because electromagnetic radiation occurs at a relatively low value [41]. Moreover, due to dipole rotation, microwave irradiation offers instant localized superheating in which the transfer of energy occurs in 10^{-9} seconds per cycle of electromagnetic energy. Thus, the molecules' movement becomes more energetic which leads to a quicker reaction in less time [42]. According to Almeida and teams, the precipitation obtained from the previous step will be inserted into a microwave reactor for 4 minutes at 80 °C [22]. Thus, on a lab-scale, microwave-assisted heating is suitable and easy to be used as the outcomes will increase the reaction rate, less reaction time, increase in the product conversion, and less formation of undesired products. For this reason, this technique has gained importance due to its efficiency compared to the traditional heating route [22].

The simple mixing and precipitation method is also one of the methods that can be chosen to produce TiO_2. These methods are very common and convenient. The conversion of solution into solid by converting the substances into an insoluble form or making the solution supersaturated is called precipitation [43]. Furthermore, the precipitation method does not require any complicated process and complex preparation conditions in addition to its usefulness and attractiveness that can easily control the particles' size and shapes [44].As stated by Achudhan and the co-workers, plant extract is used for the synthesis of TiO_2 nanoparticles. $TiCl_4$ is added into the plant extract under continuous stirring until the color changes from transparent to pale white. The pale white precipitation indicates the formation of TiO_2 [28]. Other than that, simple mixing and precipitation were also being conducted without plant extract which according to Singh and the teams whereas titanium IV isopropoxide was stirred with deionized water for 35 minutes at 40 °C . During the stirring process, the mixture underwent hydrolysis, and precipitation of hydrous titanium oxide was formed. By centrifugation and washing with deionized water and methanol, white precipitation of TiO_2 was obtained [27]. Hence, using this method, the precipitation is able to calcined at a lower temperature without using any harmful surfactant [43].

3. Average Particle size

Based on the journal findings, two methods were identified for the preparation of titanium dioxide nanoparticles which are using plant extract and without using plant extract. First of all, using plants requires the preparation of the extract either from the twigs, leaves, fruit, or the roots of the plant. According to Achudhan and team, the synthesis of titanium dioxide nanoparticles was green synthesized from multiple plant extracts of *Azadirachta indica* twigs, *Ficus benghalensis* and *Syzygium aromaticum*. The resulting nanoparticle morphology obtained from the synthesis with plant extract are of crystalline and tetragonal shapes. Meanwhile, through HR-TEM images, the particle sizes show the average sizes of 20 nm [28]. Plant leaves can also be used to produce green extract for the preparation of TiO_2 nanoparticles. Based on Subhapriya and partner, the synthesis for TiO_2 nanoparticles is by using aqueous leaf extract of *Trigonella foenum-graecum*. The preparation of TiO_2 nanoparticles resulted with an average particle size of 20 nm based on HR-TEM images that also displayed the spherical shapes of polydisperse nanoparticles [45]. Besides, using leaves as the extract is also mentioned by Santhokumar and co-workers whereas aqueous leaf extract of *Psidium guajava* is used for the synthesis of TiO_2 nanoparticles. Through the FESEM images, the nanoparticles are considered smooth and spherical in shape with an average particle size of 32.58 nm [16]. After that, the extraction also used the plant latex as one of the materials for the synthesis. As stated by Hudlikar and teams, 0.3% of aqueous extract *Jatropha curcas L.* latex is used for the green synthesis of TiO_2 nanoparticles. In TEM images, it shows that the average particle size of TiO_2 obtained is in the range of 25 to 100 nm. Another source of plant extracts can be obtained from the plant roots [46]. Based on Bavanilatha and co-workers, the roots extracts from *Glycyrrhiza glabra* are used for the preparation of TiO_2 nanoparticles. Based on the FESEM images, it shows a polydisperse grain size of nanoparticles with an average size of 69 nm [31].

Furthermore, according to findings, another method for the synthesis of TiO_2 nanoparticles is without using any plant extract. There are several techniques that can be used to prepare the TiO_2 nanoparticles. First, Heshmatpour and co-workers produced the TiO_2 by performing a sol-gel method. In addition to that, the TiO_2 nanoparticles also will be composited with ceramics also through the sol-gel method and polyaniline (PANI) by in situ chemical oxidative polymerization to attain n-TiO_2/C/PANI nanocomposite. Through the TEM studies, the nanoparticles of pure TiO_2 and n-TiO_2/C/PANI nanocomposite have demonstrated the average size of 40 nm and 15 nm respectively [17]. Another technique used to acquire TiO_2 nanoparticles is via the instantaneous synthesis method. As stated by Prakash and co-workers based on the TEM images, TiO_2 in small clusters demonstrated particle sizes within the range of 10 nm to 20 nm while the individually rounded shape particle sizes are range within 5 nm to 12 nm [18]. The third method is the solvothermal method as the TiO_2 nanoparticles are fabricated along with hydrolysis of titanium (IV) isopropoxide in the presence of benzyl alcohol and acetic acid at 180 °C into different shapes and sizes. This method has resulted in two shapes of nanosphere and nanorods for TiO_2 nanoparticles whereas the size is 7 nm to 10 nm and 15 nm to 22 nm respectively based on TEM images [20]. The microwave-assisted synthesis method also is included as one of the techniques to synthesize TiO_2 nanoparticles without using plant extract. Almeida and colleagues use the microwave-assisted synthesis method. Before using the microwave, precipitation is obtained when titanium tetrachloride is mixed with distilled water under vigorous stirring along with the addition of ammonia hydroxide. After that, the precipitation is then inserted into the microwave reactor in order to acquire the TiO_2 nanoparticles. Based on HRTEM images, it indicates that the particle size for the TiO_2 nanoparticles is 20 nm [22]. Another method used to procure TiO_2 nanoparticles without plant extract is by the simple mixing and precipitation method. As mentioned by Singh and teams TiO_2 was obtained by precipitation. After that, TiO_2 is used to act as doping for poly (methyl methacrylate). Thus, based on the result of SEM images, synthesized TiO_2 nanoparticles are somehow spherical and consist of clusters and agglomerates with a size of 5 nm. Meanwhile, through TEM images, it is revealed that the shapes of TiO_2 nanoparticles are spherical, cubic, and were well dispersed. Also, the particles sizes obtained are in the range of 20 to100 nm and the average particles sizes of 65 nm [27].

The comparison of TiO_2 nanoparticles sizes using two methods which are using plant extract and without plant extract, shows that the average particle sizes of nanoparticles obtained from the synthesis of plant extract are relatively larger than without plant extract which is 15 nm - 69 nm and 5 nm - 40 nm respectively. However, the size is still acceptable because all the nanoparticles are still less than 100 nm. There are two mechanisms to drive the growth of colloids: the dissolution of smaller and soluble particles to ensuing precipitation and growth of larger particles. The second mechanism is via the agglomeration of smaller particles to form larger particles [31]. According to Santoshkumar and his teams, the TiO_2 nanoparticles with an average size of 32.58 nm still have their adsorption capability even though the size is not relatively small. It is proven when there are extracellular organic moieties on the surface of the metallic nanoparticles adsorbed when carbon, oxygen, magnesium, and chlorine are present [16]. After that, the particle sizes were found smaller by not using any plant extract than using plant extract. Based on Heshmatpour and co-workers, particle size with high surface energy tends to agglomerate more. This usually happens on pure TiO_2 nanoparticles that exhibit cluster growth. Thus, by adding ceramic and polyaniline the agglomeration diminishes, and the particle size becomes smaller. For this reason, the porous surface area of ceramic increases the number of catalytic sites, and with the formation of polyaniline, the smallest clusters arise from repulsion forces between the nanoparticles. Hence, smaller particle sizes can be produced through the modification of ceramic nanocomposite of TiO_2 by polymer without using any plant extract [17].

4. The Application of Titanium Dioxide

Figure 2. The application of titanium dioxide nanoparticles

The application of titanium dioxide is based on the findings journal in the tabulated data is shown in Figure 2. There are four major applications of TiO_2 which are applied in water treatment or wastewater treatment, biomedical, commercialization and sensor application. Based on the findings journal, water treatment or wastewater treatment has the largest application compared to other applications. Commonly TiO_2 was combined with other polymers such as PANI, or borneol-based polymer for pollution abatement and easy recovery as well as water purification. As an instance for water and wastewater treatment, TiO_2 is used for the removal of organic contaminants such as methylene blue, Congo red, and crystal violet. Then, the other application of TiO_2 is in commercialization. TiO_2 is used as pigment for paints and cosmetics. TiO_2 is also used for food additives or preservatives and along with for food packaging applications. In addition, it is also being used as the biofilm for photovoltaic devices such as solar cells for the generating of electricity. Another application is in biomedical and as well in pharmaceuticals. For this application, it is used as a therapeutic agent for the treatment of diseases such as infectious disease and cancer. Apart from that, TiO_2 has antibacterial properties that make it possible to use for grafting in nanomedicine for demanding in vivo studies. Then, TiO_2 is also used for employment in sensor applications. In sensor application, it is often used for the removal of toxins. For example, TiO_2 is used for the removal of ammonia gas at room temperature.

5. Conclusion

In conclusion, the preparation of TiO_2 by using different methods has resulted in different particle sizes and shapes. Besides, the time taken to obtain the synthesized product also varies depending on the methods and equipment used. Of all the methods, sol-gel method took the longest time to synthesize TiO_2 nanoparticles. Apart from that, the particle size is also affected by whether it is produced in the presence of plant extract or without plant extract, and upon the evaluation from the journal, it is discovered that the synthesis of TiO_2 without plant extract has resulted in smaller particle size as compared by using plant extract. The reason for this is because using plant extract tends to make the particles agglomerate and clustered together. In addition, through the application of TiO_2 nanoparticles, it is revealed that TiO_2 is mainly used for water treatment followed by biomedical, sensor and, commercialization such as pigments in paints and as well as cosmetics.

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