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Titanium dioxide (TiO2) nanoparticles: A review on preparation, antimicrobial efficacy and industrial application.

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ABSTRACT:
With increase in multidrug resistance, antimicrobial resistance (AMR) remains as one of the major global health concerns responsible for millions of deaths. Nanotechnology, especially titanium dioxide nanoparticles known for its antimicrobial efficiency is widely preferred when compared to traditional antibiotics. Nanoparticles such as titanium dioxide are versatile, stable and highly effective in nature. Nanoparticles are widely prepared by biological, chemical and physical methods. These methods are widely preferred as they are inexpensive and produce high quality crystals. With efficient antimicrobial activity they are depend on the mechanism of photocatalysis. In photocatalytic mechanism, Reactive oxygen species (ROS) are generated which disrupts the membrane of cells such as cancer cells and pathogens. By inducing oxidative stress, they can be efficiently used against pathogenic bacteria, fungi and viruses. Apart from its antimicrobial activity, they are widely used in environmental remediation, energy technology and in biomedical application as passive coatings. This review aims to discuss the preparation, antimicrobial efficiency, environmental and biomedical application of Titanium dioxide nanoparticle, highlighting the versatile physical and chemical properties of titanium dioxide.

Keywords: Titanium dioxide nanoparticles, Antimicrobial efficiency, photocatalytic activity, environmental remediation.

I. INTRODUCTION:

Nanotechnology has altered material science with extensive research. One of the key nanoparticles namely titanium dioxide has made tremendous progress with research focusing on its versatility and inorganic materials [1]. Titanium dioxide is a naturally existing oxide and is also referred to as titania is found in three crystalline phases. Anatase, rutile and brookite make up the three crystalline phases of titanium dioxide [2]. For decades, the paper, paint, and cosmetic industry used the bulk form as white pigment. High surface to volume ratio, chemical stability, and photocatalytic activity which are the well-known physiochemical properties of TiO2 aided in its usage in nanotechnology [3]. The exceptional properties exhibited by Titanium dioxide nanoparticles makes it an ideal candidate to be employed in biomedical, environmental, energy, and healthcare sectors [4].

The unique electronic structure sparks widespread interest in TiO2. Upon interaction with UV, it gets highly activated and has wide gap due to it being a n-type semiconductor [5]. Reactive oxygen species such as hydroxyl radicals and superoxide ions are generated when exposed to UV light. Organic pollutants and pathogens are degraded as a result of oxidative reaction mediated by ROS [6]. This mechanisms exhibited by titanium dioxide nanoparticles is responsible in contributing to the environmental remediation and self cleaning properties of TiO2 [1]. Current studies focus on doping titanium dioxide with non metals or metals to narrow its band gap. By narrowing its bandgap, the nanoparticles can be easily activated by visible light, expanding its application[7].

The performance and safety of TiO2 nanoparticles are heavily dependent on the preparation methods of the nanoparticles. The morphology, crystalline phase and size of the nanoparticles are also influenced by the preparation methods [62]. Titanium dioxide nanoparticles are widely prepared by two distinct methods such as physical methods and chemical methods. Methods such as hydrothermal treatment, chemical vapor deposition, solgel are used to produce or generate nanoparticles [6]. Though the nanoparticles produced by these methods are of high purity, they are often associated with toxic precursors and increased energy consumption [1]. Another alternative method to produce nanoparticles include 'green synthesis' approach. Plant extracts and microorganism used in this method fulfil the role of reducing and stabilizing agent [6]. The nanoparticles produced by this approach are of high biocompatibilty, making them an ideal candidate for medical application such as enhanced wound healing and precision drug delivery. The green synthesis approach also aligns with sustainable chemistry [2].

The titanium dioxide nanoparticle possess antimicrobial properties that offers promising medical applications. Superbug remains as one of the major global healthcare concern as a result of multi drug resistance contributing to 10 million deaths per year[8]. To overcome this, TiO2 nanoparticles are used which minimizes antimicrobial resistance unlike other conventional methods [7]. The nanoparticles target specific pathways and destroys the microbes by generating ROS that damages the DNA, lipids, cell wall and proteins [4]. With its antimicrobial activity, it has increased efficency against a broad spectrum of pathogens namely Staphlyococcus aureus, E.coli and various fungal strains [5]. Apart from its application in healthcare associated infections, it is also used textile, food packaging, and in medical implants to reduce and prevent biofilm formation [3].

Titanium dioxide nanoparticles have also spread its root to energy and environmental application. They serve an important role in dye sensitized solar cells and in lithium-ion batteries using their exceptional charge-transport property, thereby fulfilling its application in energy sector [1]. TiO2 nanoparticles are considered as standard in advanced oxidation process used for effluent treatment. The nanoparticles are capable of mineralizing pollutants such as harmful dyes and pharmaceutical compounds, thereby contributing to successful remediation of effluents [3]. However, with increased effluent production, it is necessary to weigh the pros and cons of TiO2 exposure [3].

This review provides an overview on the properties of nanoparticles, the preparation methods employed for its production of nanoparticles, the antimicrobial efficiency of TiO2 nanoparticles against various pathogens and application of nanoparticles in medical and environmental sectors.

II. TITANIUM DIOXIDE NANOPARTICLES:

Nanoparticles are found to have dimensions ranging between 1 to 100 nm. They are widely preferred for their increased catalytic activity as a result of increased surface area. They are said to have the ability to control light propagation and bandgap, highly favoured for their quantum effects [9]. Nanoparticles are characterized based on various criteria including origin, morphology, toxicity, chemical constituents and many more. They are classified as natural, engineered and incident nanoparticles based on their origin. They are also classified as metal oxide nanoparticles, semiconductor nanoparticles, ceramic nanoparticles and core shell nanomaterials based on their chemical composition [10]. Therapeutic approaches widely employ metallic and metal oxide nanoparticles such as liposomes, quantum dots, polymeric nanoparticles, and titanium dioxide [4].

Titanium dioxide is an insoluble, colourless, white pigment that is not primarily found in the atmosphere and has molecular weight of 79.9 g/mol, density of 4.26 g/cm3 at 25°C. It exists in three forms such as rutile, brookite, and anatase. Anatase is chemically active as it produces Reactive Oxygen Species when exposed to UV light and the rutile form is considered chemically inert. Titanium dioxide nanoparticles have increased surface area that contributes to its biological and toxicity activity [11]. Known for its chemical stability, non-toxicity and catalytic activity, titanium dioxides are widely studied and are deemed to be functional building blocks, giving rise to nanotubes, nanorods and nanosheets [12]. The three forms of titanium dioxide such as anatase, brookite and rutile influence the behaviour of the nanoparticles. The bulk properties of titanium dioxide do not apply in case of nanoscale. It resorts to structural relaxation and distinct coordinating context to maintain its stability as to not fall apart [13]. As the particle size decreases, the bandgap increases as result of quantum size effect. Because of this, it requires high energy light that typically falls under shorter wavelength (blue/ultraviolet) leading to blue shift [14].

The activity of the nanoparticles is heavily influenced by its synthesis method. The most used methods include solgel and hydrolysis method with TiCl4 as precursors. Variables or factors such as choice of precursor, pH, calcination temperature and concentration influence the purity, particle size, phase transition, and operational cost of the nanoparticles produced [15][16]. The electron hole pair generated influences the application of nanoparticles. While the hole reacts with moisture content to form hydroxyl radicals, the electrons form superoxide radicals when it reacts with oxygen [17]. Titanium dioxide nanoparticles are widely employed bioremediation and against microbes resulting usage in medical and environmental aspects [15][17].

III. METHODS EMPLOYED IN PREPARING TITANIUM DIOXIDE NANOPARTICLES:

There are three main methods used for the synthesis of titanium dioxide nanoparticles. Physical, chemical and biological methods are widely used for

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preparation of nanoparticles. The thermal decomposition, electrolysis and laser irradiation methods make up the physical methods employed for nanoparticles preparation. The key disadvantage associated with employing physical methods is that the method is energy extensive and has increased operational cost. Reduction by sodium borohydride and sodium citrate make up the chemical methods which are perceived as toxic and limits the clinical usage. On the other hand, biological methods include utilization of plants, microbes and enzymes which are deemed safer and as an alternative method compared to the traditional methods [18]. The widely used preparation methods employed for synthesis of nanoparticles are discussed below.

a. Sol Gel method:

Based on the type of precursor used, the preparation method for Titanium dioxide nanoparticles can be categorized into two types . It is classified as alcohol-based process and aqueous based process [19]. In aqueous based process, the inorganic metal salts like chloride are dissolved in water. This occurs through a process widely known as hydrolysis- condensation, where the metal ions are converted to solid oxides. The hydrolysis is initiated as a result of increasing temperature or pH, where the water molecules become hydroxyl group or oxo group by losing protons. This is followed by condensation, where the species link with each other by means of olation and oxolation [20]. The alcohol-based process of synthesis involves the usage of organic metal compounds such as metal alkoxides. The precursors used under sol gel method are highly reactive due to the bond between the titanium and oxygen that is highly polar. It is characterized by two reactions that occur simultaneous forming a continuous network called gel [19]. Sol gel method is widely preferred due to several reasons. They form gel that can be easily moulded into complex geometries before undergoing hardening. Compared to traditional methods, it is a low temperature process carried around 200 to 600 °C. However, it is also associated with limitations such as ineffective control over porosity, cost and time involved in the process [21].

b. Instantaneous synthesis:

One of the methods used to synthesize titanium dioxide nanoparticle is instantaneous synthesis method. In instantaneous method, the nanoparticles are formed within few seconds, and the process is relatively short when compared to traditional methods such as sol gel . Sol gel method occurs at 80 °C for four hours to form gel, whereas instantaneous synthesis method occurs at room temperature and requires no external energy. As the name suggest, this method is simple and the entire process occurs in a single step, producing easily dispersible nanoparticles. Titanium tetraisopropoxide is typically used as precursor instead of $TiCl_4$ as the later produce small crystallite and are comparatively expensive and difficult to handle [22].

c. Solvothermal synthesis:

Solvothermal method is deemed as one of the gold standard methods to be employed to produce titanium dioxide nanoparticles from non-aqueous at low temperatures. Unlike traditional methods like sol gel, use of organic solvents increases the reaction temperature and pressure which creates a beneficial situation for creating highly active catalyst [23]. The main advantage in utilizing this method is because of the ability to tune the orderly structure of the nanoparticles. When the temperature of the solvothermal method is altered, the TiO_2 NPs through transitions, form 3D structures that resemble roses, chrysanthemum and sea urchins thereby increasing light harvesting and photo catalytic efficiency [24]. The textural properties of nanoparticles are heavily determined by the solvent, and the choice of solvent is important. By using toluene based medium, ultra-high surface area anatase can be produced.This reduces the requirement of post synthesis calcination, that is well known to cause particle sintering [25]. In order to regulate the size and dispersibility, surfactants such as lauric acid are introduced into the toluene system which act as capping agents that inhibits the growth of crystal, making sure they are small as 4 nm [26]. A synergy between solvent polarity, reaction temperature, and surfactant that exist in solvothermal method contributes to precision and accuracy at morphological and structural level that is difficult to be observed in conventional methods.

d. Hydrothermal method:

Hydrothermal method is similar to solvothermal method, but the major difference is the use of water in hydrothermal method and use of organic solvent in solvothermal method. Titanium nanoparticles were produced by heating titanium sulphate in the presence of ammonium hydrogen fluoride. Ammonium hydrogen fluoride was added due to its ability to regulate the final shape of the particle. As a result, titanium dioxide, in its purest form was formed. The pure titanium dioxide had better photocatalytic activity, than those that were prepared using conventional methods [27].

e. Microwave assisted synthesis:

Compared to conventional methods such as hydrothermal method, the titanium dioxide nanoparticles produced by microwave synthesis are sustainable and efficient. The process is comparatively rapid. The microwaves heat the liquid at a relatively faster rate, producing high quality crystals within a short period of time [28][29]. As a result of the process being quick, the particles formed are very small and leads to increase in surface area for an increased photocatalytic activity. The superior photocatalytic activity of titanium dioxide nanoparticle produced by microwave assisted synthesis have shown increased NO removal compared to commercially available TiO2 photocatalyst [28]. This method allows modification of the material based on their application. This involves creating specific surface states and defects leading to their application in electrochemical sector [30]. They are widely considered as “green method” since they require less energy and the encourages simple water-based mixtures. They are widely preferred for solar power due to their ability to make it cheaper by allowing TiO2 to easily mix with materials such as graphene, making low-cost solar cells [31]. Thus, titanium dioxide nanoparticles produced by means of microwave assisted method of synthesis, are widely preferred for their employment in bioremediation and renewable energy contributing to sustainable practices.

f. Precipitation method:

Titanium dioxide nanoparticles produced by precipitation method are of high-quality crystals and are easily scalable. This method is widely preferred as the method is inexpensive. Titanium (IV) isopropoxide can be easily used to produce high quality anatase nanoparticles via simple precipitation method. The optimal calcination temperatures play an important role. It influences the surface area that governs the photocatalytic performance. The baking temperature (calcination) is considered to be 400°C [32]. The temperature influences the phase of the nanoparticles. At lower temperature, the titanium dioxide nanoparticles are at anatase phase which are highly preferred for its higher reactivity and at higher temperatures such as those towards 800°C, the nanoparticles are at rutile phase which are less reactive [33]. There are two types of approach in precipitation method namely direct precipitation and homogenous precipitation. Direct precipitation is fast and straightforward however, is messy forming uneven clumps. On the other hand, homogenous precipitation using a precipitating agent such as urea are relatively slower but produces superior quality of nanoparticles with enhanced catalytic activity [24]. By employing homogenous precipitation, complex heterostructures such as ZnO-SnO2 can be produced for enhanced chemical reactivity [35].

IV. ANTIMICROBIAL EFFICACY OF TITANIUM DIOXIDE:

The reactive oxygen species generated by TiO2 upon exposure to light marks the antimicrobial activity of Titanium dioxide. They are responsible for oxidative stress within the microbial cells. Because of photoexcitation, electrons jump from valence band to conduction band and forms electron-hole pairs. This serves as the key mechanism behind antimicrobial activity. This leads to the production of hydroxyl and super oxide, highly reactive radicals with reaction with respect in the presence of environmental moisture and oxygen [36]. These ROS are responsible for facilitating oxidative stress in cells, damaging the membrane lipids and essential proteins [37]. Titanium dioxide nanoparticles doped with metals such as copper have the ability to maintain ROS even in the dark [38]. They have widely been used in agriculture as nano pesticides against agricultural pathogens and are known to stimulate the antioxidant pathway of the plant, proving it to be smart nano pesticide [39]. The modern green synthesis method, a widely favoured approach enhances the accumulation of ROS, contributing directly to genotoxic damage [40][41]. On the worst case, generation of excess ROS contributes to DNA damage and apoptosis in human cells especially human epidermal cells posing a major healthcare concern [42].

Titanium dioxide has the ability to cause genotoxicity, damaging the DNA of micro-organisms. It is also said to disrupt the enzyme activity of the pathogens contributing to its demise. The biofilm that coats the bacteria and protects it is easily broken-down by the nanoparticles making the pathogens vulnerable. Some titanium dioxide nanoparticles are known to interrupt or target the communication process through which the microbes communicate. The efficacy of antimicrobial activity of TiO2 Nanoparticles is influenced by regulating or monitoring certain factors. Shorter wavelengths are known to increase the activity of microbes. Radiation power also plays a vital role in governing the minimum inhibitory concentration. High power leads to low minimum inhibitory concentration leading to a smaller number of nanoparticles to kill the number of pathogens [43]. Moreover, the shape of the nanoparticles influences the antimicrobial properties of TiO2 nanoparticles. It is said that when irregular and spherical shaped nanoparticles are compared, the antibacterial efficiency of TiO2 nanoparticle is widely favoured in irregular shaped nanoparticles [44]. Addition of doping metals and plant extracts also heavily influence the antimicrobial activity of nanoparticles [40]. Below are the tables that summarize the antibacterial, antifungal and antiviral efficiency of nanoparticles.

a. Antibacterial activity of titanium dioxide nanoparticles:

SI.NO	Material Used	Particle size (nm)	Microorganism	Antimicrobial efficiency (%)	Reference
1	A-TiO2, R-TiO2, M-TiO2	300	S.sanguinis, A.naeslundii, F.nucleatum	99 to 99.9	[45]
2	Ag-TiO2	10 to 20	E. coli, S.aureus	>99.9	[46]
3	Ag-TiO2	TiO2- 20, Ag- 5 to 10	E. coli, S.aureus	99.9	[47]
4	Fe-TiO2	5 to 15	E. coli, S.aureus	99.99	[48]
5	TiO2-SiO2	10 to 50	E. coli, S.aureus	>99	[49]
6	Ni-TiO2	12 to 25	E. coli, B.subtilis	95 to 99	[50]
7	Mn-TiO2	15 to 20	E. coli, S.aureus	99.9	[51]
8	F-N doped TiO2	10 to 15	E. coli	100	[52]
9	Ni-TiO2, N-TiO2	10 to 22	E. coli, S.aureus	99.9	[53]
10	Cu-TiO2	15 to 25	E. coli, S.aureus	99.99	[54]

b. Antifungal activity of titanium dioxide nanoparticles:

SI.NO	Material	Particle size (nm)	Microorganism	Antimicrobial efficiency (%)	Reference
1	Commercial Tio2	21	C.albicans	100	[55]
2	Biosynthesized TiO2	20 to 90	U.tritici	75 to 80	[56]
3	Green synthesized TiO2	20 to 40	C.albicans	90	[57]
4	NCDs/TiO2	TiO2 - 2o to 30 NCD- 3 to 5	B.cinerea	99.9	[58]
5	Nanomaterials supported TiO2	TiO2- 21 Metal clusters - 2 to 5	A.niger spores	100	[59]
6	Pd- N-TiO2	12 to 15	F. glaminearum	>90	[60]

c. Antiviral activity of titanium dioxide nanoparticles:

SI.NO	Material	Particle size (nm)	Microorganism	Antimicrobial efficiency (%)	Reference
1	Cu/TiO2	20 to 30	H1N1(Influenza A) Ad40 HSV-2	70 to 85	[61]
2	Ag/TiO2	TiO2- 25, Ag- 5 to 8	H1N1(Influenza A)	99.99	[62]
3	TiO2 NPs functionalized with secondary metabolites	15 to 25	SARS-CoV 2	>99	[63]
4	TiO2 thin films	200	Influenza (type A)	99.9	[64]

V. APPLICATIONS OF TiO2 NANOPARTICLES:

a. Environmental application:

Titanium dioxide nanoparticles are widely employed in environmental activities such as wastewater treatment and purification of drinking water. With the aid of its superior photocatalytic activity, it efficiently removes contaminants such as dyes, pesticides and antibiotics. Titanium dioxide is widely regarded as metal oxide nanoparticle catalysts and are used in advanced oxidation process, along with other effluent treatment methods [65]. Apart from their photocatalytic activity that aids in environmental cleanup, the antimicrobial aspects of titanium dioxide nanoparticles help in removal of pathogenic microorganisms known for causing infection that are primarily found in wastewater [66]. In photocatalysis, in the presence of UV light, titanium dioxide nanoparticles generate electron-hole pairs. The reactive oxygen species are produced as a result of electron-hole pairs. The polluntants are inturn degraded by the reactive oxygen species (ROS) [67]. Additionally, it's role as photocatalyst is exploited as it is widely involved in heterogenous oxidation and complete mineralization of organic compounds in aqueous solutions. As a result, carbon dioxide and water are produced when the organic compounds are broken down into inorganic compounds because of the above reaction[68]. Apart from its role in treating water pollution, they are also employed in treating soil pollution, facilitating the removal of dyes, heavy metals, agricultural chemicals, pathogens and many more. Nanoparticles produced by green synthesis approach are widely used as nano pesticides and nano fertilizers. High surface area, stability, UV utilization and photocatalytic activity contribute to the diverse properties of TiO2 nanoparticles. With versatile properties, This makes titanium dioxide nanoparticles a perfect candidate for environmental application [69]. The application of TiO2 nanoparticles extend to remediating air pollution from remediating water and soil pollution. Like in water and soil pollution, they are used in photocatalysis for degradation of gas pollutant. Known for their low cost, strong oxidative power and chemical stability, titanium dioxide nanoparticles are widely prefered advanced method to counter pollution. They are employed in air purification technology where the gas pollutant including volatile organic compounds (VOC) are decomposed into harmless CO2 and H2O [70].

b. Biomedical application:

Known for their exceptional biocompatibility, chemical stability, and photocatalytic activity, titanium dioxide has spread its roots into medicine as nanomedicine. The integration of nanotechnology with medicine, broke the limits from fundamental physics and paved its way into clinical solutions, serving a key role in combining therapy with diagnostics [71]. Orthopaedics and dentistry remain as one of the key areas where nanotubes and other nanostructures are used. They are widely used in implants. In implants, they functionalize the surface of the implants. Due to this, the nanomodification, aids in enhancing adhesion of osteoblasts thereby mimicking the natural bone matrix [71]. Apart from structural support, one of the important applications of titanium dioxide in medicine lies as a potential agent in photodynamic therapy. When exposed to light, typically a UV radiation, it gets activated. By inducing oxidative stress and disrupting the cell membrane, the activated titanium dioxide nanoparticles destroy the cancer cells and multi drug resistant pathogens by releasing reactive oxygen species (ROS)[72]. Furthermore, the antimicrobial efficiency of titanium dioxide poses itself as an ideal candidate for self-disinfectant coatings typically employed for surgical instruments, overall reducing healthcare associated infections [73]. Apart from other various roles of titanium dioxide nanoparticles, they are widely used as sensitive biosensors. Because of its high surface to volume ratio, they are used for measuring glucose and other proteins. The versatile application also includes usage as drug delivery vehicles where the drugs are released upon environmental triggers [72]. Overall, titanium dioxide plays a versatile role in multi-purpose biomaterials, and regenerative medicines.

VI. CONCLUSION:

Titanium dioxide nanoparticles have evolved from simple industrial additive to a potential agent capable of solving complex challenges in healthcare, significantly reshaping its application in modern medicine. The exceptional biocompatibility, chemical stability, and semiconducting properties of titanium dioxide nanoparticles set it apart from other nanoparticles allowing its application in biological systems, all the while performing active therapeutic applications. Titanium dioxide nanoparticles are widely used as passive coatings in orthopaedic and dentistry industry. The employment of TiO2 has moved from passive coatings to nanostructures implemented on the surface of implants. Using advanced fabrication technologies like advanced anodic oxidation, highly ordered arrays of TiO2 nanotubes are created which mimics the mineralized matrix of the bone. This creates an environment where the osteoblast cells to firmly adhere and rapidly proliferate unlike their activity in smooth surfaces. They are widely preferred for successful long term joint replacement and as dental anchors as they can reduce implant loosening and surgical failure by minimizing micro motions.

Aside from its usage for structural support, the electronic configuration of TiO2 contributes to its photocatalytic activity enabling its role in targeted cancer therapy. When the titanium dioxide nanoparticles enter the malignant cells, they are exposed to a specific wavelength of light, making them as photosensitizers. The electrons get excited and jumps from the valence band to conduction band when the titanium dioxide nanoparticles are exposed to light. This creates the electron-hole pair. The water and oxygen molecules in the surrounding reacts with the electron-hole pairs. Reactive oxygen species such as hydroxyl radicals and superoxide anions are generated due to the above process. These ROS attacks the tumour by damaging the DNA, proteins and lipid membranes leading to programmed cell death. Due to the therapy being activated and dependent on light, healthy tissues are not targeted unlike other conventional therapies. Recent studies suggest that doping titanium dioxide nanoparticles with other metals showed increased penetration with the help of near infrared light when compared with exposure to Ultraviolet light.

The oxidative mechanism of TiO2 can be exploited against pathogens, especially against ‘superbug’. Antibiotic resistance continues to be one of the major concerns of global health. By implementing TiO2 due to its exceptional physical and chemical properties, the challenges can be overcome which are not addressed by traditional pharmacology. These nanoparticles create a self-disinfecting environment, making them an ideal candidate for disinfecting hospital surfaces, surgical equipment, drapes or wound dressings. Upon exposure to light, the TiO2 Nanoparticles attack the pathogens by disrupting the cell walls of bacteria and degrading the viral capsids, thereby neutralizing the pathogens. As a result, reducing the risk of pathogens to develop resistance.

Titanium dioxide nanoparticles are now utilized for controlled drug delivery. The anti-inflammatory or genetic materials are loaded onto the nanoparticles. The loaded molecules are released as a result of being triggered by external environment. This allows precise drug release with increased efficiency all the while, reducing side effects. Titanium dioxide nanoparticles known for its increased efficiency are widely used to develop smart medical devices and personalized medicines.

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