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COMPARING GREEN CHEMICAL METHODS AND CHEMICAL METHODS FOR THE SYNTHESIS OF TITANIUM DIOXIDE NANOPARTICLES

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ABSTRACT: Metal nanoparticles are usually synthesized using various chemical methods such as chemical reduction, Solvo-thermal reduction, electrochemical techniques and photochemical reaction in reverse micelles. All these methods are divided into two categories chemical and green chemical. Each having certain advantages and disadvantages. In this study we have tried to give a few examples of these methods for the synthesis of Titanium dioxide nanoparticles and further discussed to compare the two.

INTRODUCTION: Recently, nanoparticles are issues in many fields such as industrial applications, environmental studies and human health impacts, due to their unique physical and chemical characteristics [1-2]. In the industrial field, nanomaterials made from metals such as titanium, silver, gold, cerium, and aluminum have been widely used for commercial purposes. Because of its high stability, anticorrosiveness, and photocatalytic properties, Titanium dioxide in nanoparticle form may be one of the most important materials for photocatalysts [3], cosmetics, and pharmaceuticals [4]. Traditionally most of the metal and metal oxide nanoparticles were routinely synthesized by various physical and chemical methods. Some of the commonly used synthetic methods are non-sputtering, solvothermal, reduction, sol-gel technique and electrochemical technique [5-8].

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But these methods are costly, toxic and potentially hazardous, not to mention the difficult separation procedure, high pressure and energy requirement [9]. Metal and metal oxide nanoparticles have high surface area and high fraction of atoms giving them their fascinating properties such as antimicrobial, magnetic, electronic and catalytic activity [10-13]. The synthesis of metal and metal oxide nanoparticles has attracted considerable attention in physical, chemical, biological, medical, optical, mechanical and engineering sciences where novel techniques are being developed to probe and manipulate single atoms and molecules.

Recently nanoparticle synthesis were achieved with bacteria, fungi, actinomycetes [14-16] and use of plant extract such as neem, camellia sinensis, coriandrum, nelumbo lucifera, ocimum sanctum and several others compatible with the green chemistry principles [17-19]. Among the various biosynthetic approaches, the use of plant extracts has advantages such as availability, safe handling and possess a broad viability of metabolites. The main phytochemicals responsible for the synthesis of nanoparticles are terpenoids, flavones, ketones, aldehydes amides etc. In continuation of the efforts for synthesizing Titanium dioxide nanoparticle, here we report a facile, green and one pot synthesis using the leaf extract of Nyctanthes. Nyctanthes was chosen because of its functional anti inflammatory, antioxidant, antifungal, antidiabetic, antimicrobial, antileishmanial, antipyretic and antinociceptive activity [20-22]. The biosynthetic route for preparing various nanoparticles has not yet been extended to the synthesis of Titanium dioxide nanoparticles especially with Nyctanthes. According to the aforementioned criteria, chemical and green chemical methods of synthesizin Titanium dioxide nanoparticles will be the focus of discussion in this paper.

Titanium dioxide nanoparticles:
Among the various metal oxide nanoparticles, Titanium dioxide nanoparticles have wide applications in air and water purification, DSSC due to their potential oxidation strength, high photo stability and non-toxicity [23-25]. Moreover Titanium nanoparticles possess interesting optical, dielectric, antimicrobial, antibacterial, chemical stability and catalytic properties which leads to industrial applications such as pigment, fillers, catalyst supports and photocatalyst [26-29].

Titanium dioxide nanoparticles are produced abundantly and used widely because of their high stability, anticorrosive and photo catalytic properties [30]. Some have attributed this increased catalytic activity of Titanium dioxide nanoparticles to their high surface area, while others attribute it to Titanium dioxide nanoparticles being predominantly anatase rather than rutile [31-32]. Titanium dioxide nanoparticles can be used in catalytic reactions, such as semiconductor photo catalysis, in the treatment of water contaminated with hazardous industrial by-products [33], and in nanocrystalline solar cells as a photoactive material [34]. Industrial utilization of the photocatalytic effect of Titanium dioxide nanoparticles has also found its way into other applications, especially for self-cleaning and anti-fogging purposes such as self-cleaning tiles, self-cleaning windows, self-cleaning textiles, and anti-fogging car mirrors [35].

In the field of nanomedicine, Titanium dioxide nanoparticles are under investigation as useful tools in advanced imaging and nanotherapeutics [36]. For example, Titanium dioxide nanoparticles are being evaluated as potential photosensitizers for use in photodynamic therapy (PDT) [37]. In addition, unique physical properties make Titanium dioxide nanoparticles ideal for use in various skin care products. Nano-preparations with Titanium dioxide nanoparticles are currently under investigation as novel treatments for acne vulgaris, recurrent condyloma accuminata, atopic dermatitis, hyperpigmented skin lesions, and other non-dermatologic diseases [38]. TiO_2 nanoparticles also show antibacterial properties under UV light irradiation [38-39].

Titanium dioxide nanoparticles possess different physicochemical properties compared to Titanium dioxide fine particles (FPs). These properties likely influence bioactivity. Based on this fact, adverse health effects and environmental bio-safety of Titanium dioxide nanoparticles should be carefully evaluated even if Titanium dioxide fine particles have been demonstrated to have low toxicity. It is recommended that researchers carefully characterize the physicochemical properties of Titanium dioxide nanoparticles not only in the bulk form but also as delivered to the test system.

Green chemistry for the synthesis:
Green chemistry for chemical synthesis addresses our future challenges in working with chemical processes and products by inventing novel reactions that can maximize the desired products and minimize by-products, designing new synthetic schemes and apparatus that can simplify operations in chemical productions, and seeking greener solvents that are inherently environmentally and ecologically benign. Over the past two centuries, fundamental theories and reactivity in chemistry have been soundly established. Such theories and reactivity have provided the foundations for the chemical enterprise that generates critical living needs such as food for the world's population, achieves various medical wonders that save millions of lives and improve people's health, and produces materials essential to the present and future needs of mankind. Just less than two centuries ago, organic compounds were believed to be only accessible through biological processes under the influence of vital forces [40]. Today, many molecules of great complexity can be synthesized

readily. The total syntheses of natural products with extremely high complexity such as Vitamin B_{12} [41] and polytoxin [42] in the laboratory are testimonials of achievements comparable to the construction of the great pyramids at the molecular scale. However, despite such enormous achievements, we are facing great challenges in future chemical synthesis. The present state-of-the-art processes for synthesizing chemical products are highly inefficient. The concept of atom economy [43] was created to emphasize the importance of this inefficiency. The E factor [44] provided a quantifiable measure of such inefficiency and showed that, for every kilogram of fine chemical and pharmaceutical products produced, 5–100 times that amount of chemical waste is generated. Such low efficiency in state-of-the-art organic syntheses presents great challenges in resource conservation and draws environmental and health concerns related to the chemical wastes.

The application of the twelve principles of green chemistry in nanoparticle synthesis is a relatively new emerging issue concerning the sustainability. This field has received great attention in recent years due to its capability to design alternative, safer, energy efficient, and less toxic routes towards synthesis. These routes have been associated with the rational utilization of various substances in the nanoparticle preparations and synthetic methods, which have been broadly discussed in this review paper. This paper is not meant to provide an exhaustive overview of green synthesis of nanoparticles, but to present several pivotal aspects of synthesis with environmental concerns, involving the selection and evaluation of nontoxic capping and reducing agents, the choice of innocuous solvents and the development of energy-efficient synthetic methods.

Green chemistry for the synthesis of Titanium dioxide nanoparticles:

Santhoshkumar et al (2014) introduced a paper titled Green synthesis of Titanium dioxide nanoparticles using Psidium guajava extract and its antibacterial and antioxidant properties. Synthesized TiO_2 NPs were tested by disc diffusion method against human pathogenic bacteria. The total antioxidant activity and phenolic content (Folin-Ciocalteau method) of synthesized TiO_2 NPs and aqueous plant extract were determined. The scavenging radicals were estimated by DPPH method. The synthesized TiO_2 NPs were characterized by XRD, FTIR, FESEM and EDX. Results: FTIR spectra of synthesized TiO_2 NPs exhibited prominent peaks at 3 410 cm^{-1} (alkynes), 1 578 cm^{-1}, 1 451 cm^{-1} (alkanes), and 1 123 cm^{-1}(C-O absorption). The morphological characterization of synthesized TiO2 NPs was analyzed by FESEM which showed spherical shape and clusters with an average size of 32.58 nm.

The synthesized TiO_2 NPs showed more antibacterial activity than the standard antibiotic disk, tetracycline which drastically reduces the chances for the development of antibiotics resistance of bacterial species. The plant aqueous extract and synthesized TiO_2 NPs were found to possess maximum antioxidant activity when compared with ascorbic acid. The content of phenolic compounds (mg/g) in leaf aqueous extract and synthesized TiO_2 NPs were found to be 85.4 and 18.3 mgTA/g, respectively [45].

Hojatkashani (2013) introduced a paper titled green synthesis of Titanium dioxide nanoparticles. Synthesis of chemical compounds using materials which are compatible with environment, non-toxic and safe, is one of the principles of green chemistry. In this paper, a vast veriety of green chemistry principles along with two new methods for the green synthesis of Titanium dioxide nanoparticles has been collected. Titanium dioxide is a photo catalyst which has various applications especially in green chemistry. This paper is about synthesis of TiO_2 nanoparticles first by using Nyctanthes Arbor-Tristis leaves extract and then by a bacteria called Planomicrobium sp. Then the size and morphology of the synthesized nanoparticles by two methods are compared by their scanning electron microscopic (SEM) images [46].

Paskalis (2014) introduced a paper titled a validation for Green Synthesis Protocol using Hibiscus Flower. In this study TiO_2 nanoparticles have been synthesized from Titanium Oxysulfate solution using Hibiscus flower extract. The synthesized nanoparticles were characterized using x-ray diffraction (XRD), scanning electron microscopy (SEM) and FTIR. The XRD pattern with sharp peaks describes the crystallinity and purity of Titanium dioxide nanoparticles. The shape and morphology of TiO_2 nanoparticles were studied by SEM analysis, the results clearly represent that the flower extract capped Titanium dioxide nanoparticles were dispersed and disaggregated. FTIR spectrum discloses the information about the interaction between the functional groups of the phytochemicals in the flower extract and the TiO_2 [47].

Chemical synthesis:

Chemical synthesis is a purposeful execution of chemical reactions to obtain a product, or several products [48]. This happens by physical and chemical manipulations usually involving one or more reactions. In modern laboratory usage, this tends to imply that the process is reproducible, reliable, and established to work in multiple laboratories. A chemical synthesis begins by selection of compounds that are known as reagents or reactants. Various reaction types can be applied to synthesize the product, or an intermediate product. This requires mixing the compounds in a reaction vessel such as a chemical reactor or a simple round-bottom flask. Many reactions require some form of work-up procedure before the final product is isolated.

The amount of product in a chemical synthesis is the reaction yield. Typically, chemical yields are expressed as a weight in grams (in a laboratory setting) or as a percentage of the total theoretical quantity of product that could be produced. A side reaction is an unwanted chemical reaction taking place that diminishes the yield of the desired product.

Chemical synthesis of Titanium dioxide nanoparticles:

García (2012) introduced a paper titled Synthesis of Titanium oxide nanoparticles by plasma. This work presents a study about the evolution of Titanium dioxide particles synthesized with glow discharges of water and Tetra Titanium Propoxide (TTP). The syntheses involved sequential changes of TTP from solid, liquid and vapor phases under resistive glow discharges at 13.56 MHz, 0.5-0.8 mbar and 100 W with reaction times between 60 and 240 min. The reaction of TTP and water between the electrodes originated Titanium dioxide powder composed of white particles with diameter between 106 and 695 nm and different geometries depending on the time of synthesis. These materials may have environmental applications in the sorption of toxic contaminants [49].

Sharmila Devi (2014) introduced a paper titled Synthesis of Titanium Dioxide Nanoparticles by Sol-Gel Technique. Nanosized Titanium dioxide (TiO2) powder was synthesized via sol-gel method using Titanium Tetra isopropoxide (TTIP) as the precursor. It prepared nano powder was used for further characterization. The phase transformation was investigated by an X-ray diffractometer (XRD). The anatase structure of Titanium dioxide was obtained after calcination. The microstructure was characterized using a Scanning Electron Microscope (SEM) [50].

Jiye (2016) introduced a paper titled a facile synthesis of rutile-rich Titanium dioxide nanoparticles using reverse micelle method and their photocatalytic applications. In this work flower-like shaped rutile-rich TiO2 nanoparticles were synthesized by the reaction of HCl with Titanium diisopropoxide bis(acetylacetonate) immobilized in reverse micelles composed of oleic acid, water, and xylene. Brunauer Emmett Teller (BET) analysis showed large surface area of the synthesized TiO_2 nanoparticles of $177.8 m^2/g$. It investigated the effect of Ti precursor concentration and oleic acid's role in the formation of TiO_2 nanoparticles. Rutile-rich TiO_2 nanoparticles with large surface area showed better photocatalytic activity in decomposing methyl orange under visible-light irradiation than anatase and rutile mixed phase TiO_2 particles.

Conclusion: Alternate methods are also adopted for the synthesis of metal and metal oxide nanoparticles utilizing bacteria, fungi and plant extracts as reducing agents. In recent years, the development of metallic and metal oxide nanoparticles in an eco-friendly manner using plant materials has attracted considerable attention. The biogenic reduction of metal ions to the base metal is quite rapid and more efficient than conventional methods. This method is eco-friendly, can be conducted readily at room temperature under sunlight conditions, and is also scaled up easily. The reducing agents involved include various water-soluble metabolites (e.g., alkaloids, terpenoids, polyphenolic compounds) and coenzymes. Noble metals (Silver and Gold) have been the main focus of plant-based synthesis. These green synthesized nanoparticles have a range of shapes and sizes compared to those produced by other organisms. The advantages of using plant-derived materials for nanoparticle synthesis have attracted the interest of researchers to investigate the mechanisms of metal ions uptake and bio-reduction by plants. Moreover, green synthesis generates nanoparticles with high disparity, high stability and narrow size distribution.

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