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Green synthesis of titanium dioxide (TiO_2) nanoparticles by *Trigonella foenum-graecum* extract and its antimicrobial properties

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ABSTRACT

In recent years, biosynthesis of nanoparticles has received considerable attention due to the growing need to develop clean and nontoxic chemicals, low-cost approaches, eco - friendly solvents and renewable materials. In the current study, the biosynthesis of TiO_2 nanoparticles (TiO_2NPs) was attained by a chemical and biosynthesized method by using the aqueous leaf extract of *Trigonella foenum-graecum* (TF-TiO_2NP). TiO_2 NPs were characterized by FTIR, UV, XRD, HR-TEM and HR-SEM methods. The X-ray diffraction displayed the existence of TF-TiO_2NPs which is confirmed by the incidence of peaks at 25.28 corresponds to 101 anatase form. HR-SEM perceptions revealed that synthesized TiO_2NPs were spherical in shape and the size of individual nanoparticles as well as a few aggregates was found to be 20–90 nm. The antimicrobial activities of biosynthesized nanoparticles (TF-TiO_2NPs) were examined using Kirby-Bauer method. The TF-TiO_2 nanoparticles showed significant antimicrobial activity against all the tested microorganisms.

1. Introduction

An overwhelming interest on the studies with nanomaterials has been noticed in the recent days. Nanobiotechnology is an empowering technology that transacts with nanometer-sized materials in different fields of science such as nanotechnology, biotechnology, materials science, physics and chemistry. Besides several chemical and physical methods which have been settled for preparing metallic nanoparticles, nanobiotechnology also assists as an important method in the development of hygienic, safe and eco-friendly events for fusion and gathering of metallic nanoparticles [1]. In last few years, biosynthesis of nanoparticles have obtained significant consideration owing to the emerging necessity in nontoxic chemicals, antibacterial, antiviral, diagnostics, anticancer, targeted drug delivery environmental amenable solvents and renewable materials [2].

Earlier studies exhibited that the use of chemical reducing agent caused the production of larger particles which consumes extra energy. Moreover it was also stated that additional side effects were made by chemical methods that are not ecological. Also, the chemically synthesized nanoparticles were described to exhibit less stability and added agglomeration [3–5]. Henceforth, there is a necessity to improve an eco-friendly protocol that will create dispersible and stable nanoparticles of manageable size which consumes less energy.

The biosynthesis of nanoparticles has attracted consideration of several scholars due to their chemical and physical processes being costly and radical reaction conditions. As a result, quest for new low-cost paths for nanoparticles synthesis, researchers used microorganisms and the plant extracts [6]. Not many works have been reported on the biosynthesis of rare metal nanoparticles such as lanthanum and titanium. Titanium dioxide (TiO_2) is steady in aqueous media and accepting both alkaline and acidic solutions. TiO_2 nanoparticles were used in the field of photocatalysts, cosmetics, and pharmaceuticals [7]. The titanium nanoparticles have the capability of reacting with –OH and O_2, which are adsorbed upon the surface for obtaining oxygen and hydroxyl free radical [8]. Due to increased surface area of nanoparticles, the area of interaction with pathogenic bacteria increases resulting in making them suitable as antimicrobial agent. The small size of the particles enables them to easily enter bacterial surface and capable of harming them [9]. Titanium nanoparticles are prominent to several researchers owed to its low cost linked to noble metals such as Au, Pt and Ag. Earlier studies reported the use of TiO_2 in life science [10] and stated that the bactericidal and catalytic properties of TiO_2 can be developed by increasing particles of noble metals (Au, Cu or Ag) above its surface [11].

TiO_2 is a valuable semiconducting transition metal oxide material and shows special features such as easy control, reduced cost, nontoxicity; good resistance to chemical erosion, then it can be used in solar cells, chemical sensors and environmental distillation applications [12]. These nanoparticles have good electrical, magnetic and optical properties which are different from bulk counterparts [13]. Titanium

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Table 1
Nanoparticles synthesized from different plants.

S.no.	Plant materials used	Metal nano particles	Particle size (nm)	References
1	*Calotropis gigantea*	TiO_2	10.52	[15]
2	*Psidium guajava*	TiO_2	32.58	[16]
3	*Aloe Barbadensis Miller*	TiO_2	20	[17]
4	*Ageratina alttissima.L*	TiO_2	60–100	[18]
5	*Vitex negundo Linn*	TiO_2	10	[19]
6	*Psidium guajava*	TiO_2	32.12	[20]
7	*Curcuma longa*	TiO_2	43.88	[21]
8	*Vigna unguiculata*	TiO_2	11.55	[22]
9	*Eclipta prostrata*	TiO_2	36	[23]
10	*Moringa Oleifera*	TiO_2	12.22	[24]

dioxide produces amorphous and crystalline forms and primarily occurs in three crystalline polymorphous that is anatase, rutile and brookite [14]. Several researchers have described the usage of natural products from medicinal plants such as *Calotropis gigantean, Psidium guajava, Aloe barbadensis Miller*, *Ageratina alttissima L*, *Vitex negundo, Psidium guajava, Curcuma longa, Vigna unguiculata, Eclipta prostrata* and *Moringa oleifera* for the formation of TiO_2 nanoparticles (Table 1). Huge amount of nanoparticles can be effortlessly made from the plants and majority of synthesized products are nontoxic. The biocompatible and non-toxic possessions of TiO_2NPs can be used for biomedical science such as bone tissue engineering along with pharmaceutical industries. Therefore, there is possibility to improve new approaches for the production of Titanium nanoparticles. In this work, titanium nanoparticles are synthesized by using the leaf extract of *Trigonella foenum graecum*.

Trigonella foenum graecum (L.) generally called as fenugreek and it is an aromatic leguminous plant which is widely distributed in many Middle Eastern, Asian and European countries. The habit of *Trigonella foenum* was shown in (Fig. 1). Fenugreek seeds are main source for phosphorus, iron and sulphur. In Indian traditional medical system, Ayurveda, it is used as an aphrodisiac for rejuvenation, bronchial and digestive complaints, arthritis and gout. It can be used as an expectorant, diuretic, laxative, anti-tumour and anti-parasitic effects. The seeds of fenugreek can be used to treat liver cancer, low blood pressure and diabetes. These seeds and leaves have been used extensively in various medicinal preparations [25]. In the present study, TiO_2 nanoparticles were biosynthesized using aqueous leaf extract of *Trigonella foenum graecum* and categorized with FTIR, UV, XRD, HR-SEM, EDX and HR-TEM. Henceforth, this method could be appropriate for evolving a biological method for mass scale generation of nanoparticles.

Fig. 1. Habit of *Trigonella foenum.*

2. Materials and method

2.1. Plant materials

The leaves of *Trigonella foenum graecum* were gathered from Annanur, Chennai, Tamil Nadu, India in June 2000. The plant was taxonomically recognized and authenticated by Botanical Survey of India, Coimbatore, Tamil Nadu, India.

2.2. Chemicals

All the chemicals (AR grade) were purchased from Alfa Aesar, Hyderabad, India and Millipore water was used.

2.3. Preparation of plant extract

Leaves of *Trigonella foenum* were washed with distilled water to eradicate soil and dirt. Around 15 g of leaves of *Trigonella foenum* were weighed and grained using mortar and pestle. The crushed leaves were dispersed in 50 mL of millipore water and stirred it using magnetic stirrer for about 20 min. Then, the solution was filtered as plant extract using Whatman No 1 filter paper.

2.4. Synthesis of titanium dioxide nanoparticles

15 mL of leaf extract of *Trigonella foenum* was added to 0.5 M solution of Titanium oxy sulphate and stirred for 15 min for synthesis of TiO_2 nanoparticles. Further, 1 M sodium hydroxide was added drop by drop to this solution until the pH reaches 8. The precipitate was frequently washed by deionized water to eradicate the scums excess sodium hydroxide and then the washed precipitate was filtered and sintered at 700 °C for 3 h. The sintering at high temperature provides the formation of well crystalline nano TiO_2. The sintered TF-TiO_2NPs was characterized using FTIR, UV, XRD, HR-SEM, EDX and HR-TEM. Meanwhile, TiO_2NPs was prepared by the same method above without using the leaf extract of *Trigonella foenum* and characterized using FTIR, UV analysis, XRD, HR-SEM, EDX and HR-TEM.

2.5. Fourier transform infrared spectroscopy

The binding properties of TiO_2NPs manufactured by *Trigonella foenum* leaf extract were examined by FTIR study. FTIR measurements were taken on Bruker vertex 70. Powdered and dried TiO_2NPs were pelleted with Potassium bromide (KBr) (1:3 ratio). The spectra were chronicled in the wavenumber range of 400 cm^{-1} - 4000 cm^{-1} and analysed by deducting the pure KBr spectrum.

2.6. UV–Vis absorbance spectroscopy

The UV–Vis absorption spectra of the TiO_2NPs and TF-TiO_2NPs were chronicled at room temperature by Pharmaspec UV-1700 (Shimadzu Corporation, Tokyo, Japan) UV–Visible spectrophotometer. The scanning range of the samples was 200–900 nm in a resolution of 1 nm at a scan speed of 200 nm/min.

2.7. XRD

X-Ray Diffraction (XRD) is an analytical technique which is used to identify the phase formation and crystallinity of TiO_2NPs. XRD analysis was conducted via the PW 1148/89-based X-ray diffractometer employing nickel filtered Cu α radiation (k = *1.54056* Å) at 298 K. The tool was armed with graphite monochromator and then functioned at 30 mA and 40 kV. The diffractogram was attained in the 2θ range of 10–80°. The obtained raw data were processed in origin 8 software and compared with the standard JCPDS database card no. 21-1272 (Anatase) and 21-1276 (Rutile).