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Photocatalysis effect of a novel green synthesis gadolinium doped titanium dioxide nanoparticles on their biological activities

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ABSTRACT

We report the synthesis of gadolinium doped titanium dioxide (GdT) nanoparticles (NPs) via hydrothermal method using a novel bioreductant *Piper betel* leaf. The physiochemical properties of green synthesized GdT NPs were characterized using various techniques. The results reveal that GdT NPs have attached with the biomolecules which were supporting evidence for the reduction and capping agents. GdT NPs have crystalline in nature and their estimated grain size is of about 5.45 nm by the Scherer's formula. GdT NPs consist of well-dispersed agglomerates of grains with a narrow size distribution of about 4 nm to 7 nm and are having spherical in nature. GdT NPs are thermally stable. In addition, GdT NPs were subjected to antimicrobial and antioxidant assays. Herein, we observed that the GdT NPs show higher antibacterial activities against *Staphylococcus aureus* as compared with *Escherichia coli* and the minimum inhibitory concentration (MIC) value was found to be $25\,\mu g\,mL^{-1}$ under UV irradiation condition for both the cases. Furthermore, antioxidant activities of GdT NPs were evaluated in vitro, using the 1, 1- diphenyl-2-picrylhydrazyl (DPPH) radical cation decolorization test. Results suggest that GdT NPs give promising antioxidant activity as compared to standard ascorbic acid and *Piper betel* leaf. This novel and efficient strategy will show the route for avoiding the use of toxic solvents and a promising green route to restrict for drug discovery from natural products. Thus, the present findings may shine in the field of green pharmaceuticals industries.

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1. Introduction

Green chemistry is gaining tremendous importance in the day to day life due to its potentiality of synthesizing materials of various shapes and sizes using naturally occurring bioreductions [1]. There has been growing much interest for the creation of nanoscale materials using the greener route. In recent past, inorganic nanoparticles (NPs) whose structures exhibit significantly novel and improved physical, chemical and biological properties due to their nanoscale sizes have elicited much interest [2–4]. For the NPs, interesting optical, electronic and catalytic effects are expected on the nanoscale [5]. Various approaches are available for the synthesis of NPs [6–12].

In recent times, some attention has been focused on the green route for the synthesis of NPs. The use of environmentally benign plant materials like leaf [13], root [14] and fruit [15] for the synthesis of NPs offers numerous benefits because of their benign nature as well as safety and environmental concerns. The plant extract having the phytochemicals (Tannins, flavonoids and proteins etc.) are the key issue for the controlling the size and shape [1]. Therefore, the presence of stabilizers and various surfactants are desirable to have a control over the growth of the particles.

Titanium dioxide (TiO_2) NPs are well recognized as a versatile multifunctional material due to their superior chemical stability under physiological environment and non-toxicity etc. [16,17]. The TiO_2 material has been widely using in the area of solar cells [18], sensor [19], biological activities [20] and catalyst [21]. In view of these facts, we have chosen TiO_2 material for our study. Several researchers were successfully synthesized TiO_2 nanomaterials using plants leaf [22,23], fruit [24], root [25] and fungus [26] for the biological applications. Some transition elements and some rare earth materials were doped with the TiO_2 material in order to enhance the efficiency of solar cells [27,28] and biological activities [29].

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TiO_2 doping with rare earth material has been an effective method to enhance the photocatalytic activity because rare earth material having a special; electronic structure, which play an important role for the photo-generate charges transfer between 4f energy level and TiO_2 conduction band [30]. Doping of appropriate amount of La^{3+}, Ce^{3+}, Er^{3+}, Pr^{3+}, Gd^{3+}, Nd^{3+} and Sm^{3+} with TiO_2 can effectively enhance the photocatalytic activities, in further it is increased the adsorption capacity and also adsorption rate of TiO_2 catalysts. Here, it was found that gadolinium doping was most effective [31]. Zhou et al. reported that on doping gadolinium (Gd) with TiO_2 nanofibers their photocatalytic activity was enhanced [32]. Moreover, it is well known that half-filled electron configurations are more stable. Gd element has a half-filled 4f-shell containing only seven electrons and an empty 5d-shell, which are different from the other rare earth elements [33]. Therefore, the rare earth Gd^{3+} may serve as a superior co-catalyst for improving photocatalytic activity. According to the analysis, as discussed above, it can be deduced that Gd doping may be an ideal photocatalytic material with high photocatalytic activity.

The high degree of microbial diseases and their multidrug resistant properties make the researchers to develop a new class of antimicrobial agents [34–37]. A modern and innovative approach to drug development is the use of metallic NPs as new formulations of antimicrobial agents. Herein, the present study attempts to utilize the green route for the synthesis of gadolinium doped titanium dioxide (GdT) NPs for to study their optical, structural, morphology and thermal behaviors. Further, GdT NPs were tested for their antimicrobial and antioxidant activities. To the best of our knowledge, it is the first attempt to synthesize the Gd-doped TiO_2 NPs via hydrothermal method using *Piper betel* leaf extract as a capping and stabilizing agents also there are no reports on biological activities of GdT NPs.

2. Materials and methods

2.1. Chemicals

Titanium (IV) *n*-butoxide (TNB, $C_{16}H_{36}O_4Ti$) [99 Wt% liquid analytical grade] was purchased from Alfa Aesar chemicals and gadolinium (Gd) nitrate was purchased from Sigma-Aldrich chemical company. Ascorbic acid, ethanol and methanol were purchased from Spectrochem Laboratories Pvt. Ltd India and 1, 1-diphenyl-2-picrylhydrazyl (DPPH) was purchased from HiMedia Laboratories Pvt. Ltd. India and were used without any further purification. De-ionized water (DW) is used in the preparation of all suspensions and solutions. All glass wares were washed before use with dilute nitric acid and dried in an oven.

2.2. Biological materials

The leaves of *Piper betel* were purchased from local market Dharwad, Karnataka, India. Antimicrobial activities were tested using *Staphylococcus aureus* as a Gram-positive bacterium, *Escherichia coli* as a Gram-negative bacterium and *Candida albicans* as a fungus organism.

2.3. Preparation of leaf extract

20 g of chopped *Piper betel* leaves were added into 250 ml Erlenmeyer flask along with 100 ml of DW and later stirred using a magnetic heating stirrer at 80 °C for 30 min. The obtained supernatant solution was filtered with the Whatman filter paper No. 1, stored below 20 °C and used within a week.

2.4. Green synthesis of GdT NPs

GdT NPs were synthesized according to a method described in previous report [23], with little modifications. Briefly, 0.1 M of gadolinium nitrate was taken in 10 ml of DW in 25 ml beaker, stirred for 10 min at room temperature. Then 5 ml of *Piper betel* leaf extract and 1 ml of TNB were added to that mixture and stirred for 30 min, it turns to the brown color solution from transparent solution. The brown colloidal solution was then transferred to a 25 ml Teflon-lined stainless steel autoclave, the autoclave was sealed and placed in an oven and heated upto 180 °C for 3 h, then the autoclave was cool down to room temperature. Under the ambient conditions, the reaction mixture was centrifuged to collect the product; the product was washed continuously with DW and ethanol. The final product was dried in an oven at 50 °C for 1 h. The obtained product is in powder form and it was used for various characterizations. Further, it is used for the biological activities.

2.5. Instrumentations and characterizations

UV–vis spectrophotometer (Model- V-670 JASCO at USIC, K. U. Dharwad, India) was used to record the absorption spectrum. Fourier transform infrared spectroscopy (FT-IR) (Model- Nicolet 6700 at USIC, K. U. Dharwad, India) was used for to analyze the absorption bands. X-ray diffraction (XRD) (Model- Bruker AXS D8 Advance at STIC, Cochin, India) analysis was carried out for the crystal structure and grain size estimation. Transmission electron microscopy (TEM) (Model- JEOL/JEM- 2100 at STIC, Cochin, India) and scanning electron microscopy (SEM) (Model- JEOL/JSM-6390LV at STIC, Cochin, India) analysis were examined for the

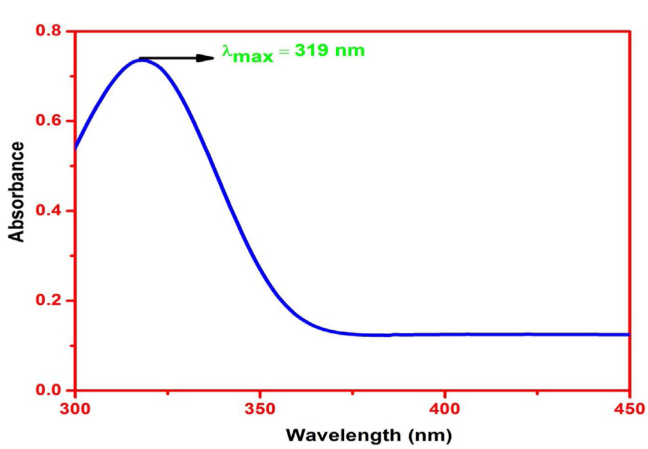

Fig. 1. UV–vis absorption spectrum of GdT NPs.

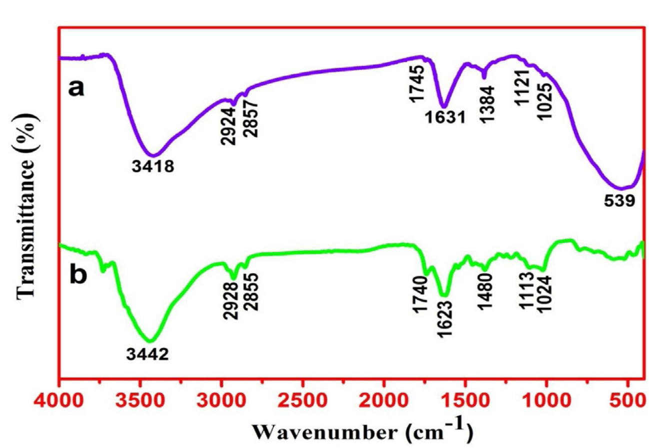

Fig. 2. FT-IR spectra **(a)** GdT NPs and **(b)** *Piper betel* leaf.

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