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Green synthesis of titanium dioxide nanoparticles using *Cassia fistula* and its antibacterial activity

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ABSTRACT

Green synthesis of titanium oxide nanoparticles has more advantages when compared with the chemical method. This work reports a green synthesis of titanium dioxide nanoparticles (TiO_2NPs) by the herbal plant extracts of *Cassia fistula*. Then the green synthesized NPs were characterized by UV-Vis spectroscopy, X-ray Diffraction (XRD), Fourier transforms infra-Red spectroscopy (FT-IR), atomic force microscopy (AFM), scanning electron microscopy (SEM), thermogravimetric analysis (TGA). The result of the SEM image shows that the nanoparticles are spherical in shape. The antibacterial activity was done on *Escherichia coli* and *Staphylococcus aureus.*

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INTRODUCTION

Last few decades, the researcher mainly focussing on the Nano-sized particle, because of its wide application (Wang *et al.,* 2017; Xu *et al.,* 2002; Chen *et al.,* 2004; Dawson *et al.,* 2001). The nanoparticles mainly used in optical, electrical, and thermal properties (Devi Baskar *et al.,* 2017; Park *et al.,* 2009). The nanomaterials are looked like a crystalline material with very fine and large gain limit (Nagaraju *et al.,* 2013; Chen *et al.,* 2007). Due to its appearance (like Nanocrystalline) exhibit huge properties and application in different fields (Morales *et al.,* 1998; Nagaraju *et al.,* 2012). Among all variety of inorganic materials, a researcher shows their interest mainly on metal oxide nanoparticles, due to their unique properties and application towards physical and chemical properties of TiO_2. Moreover, these properties may help to change their particle size, surface morphology, crystalline phase, and dimension (Nagaraju *et al.,* 2013; Gopalakrishna *et al.,*1995; DonyaRamimoghadam *et al.,* 2014).

Titanium oxide (Tio_2) is a metal oxide with the n-type wide band semiconductor material. It has a wide range of application in photochromic devices, lithium batteries, dye-sensitized solar cells, and wastewater treatment (Park *et al.,* 2009; Nagaraju *et al.,* 2013; Ghamsari et al, 2013; Yan et al, 2000; Iuchi et al, 2004; Wagemaker et al, 2001). The titanium oxide synthesized by using various methods like solution preparing, solvothermal synthesis, polyol reaction, sol-gel reaction (Noh *et al.,* 2016; Trentler *et al.,* 1999; Liao *et al.,* 2009; Allam *et al.,* 2009). The titanium oxide has more advantage due to its low toxicity, biocompatibility, chemical and thermal stability (Park *et al.,* 2009; Ghamsari *et al.,* 2013; Trentler *et al.,* 1999; Zhang *et al.,* 2012).

Nowadays, the biosynthesis of nanoparticle has been used in a wide range by a synthetic route using plants (Nasrollahzadeh *et al.,* 2016). The green synthesis of titanium oxide nanoparticles has more advantage due to their less consumption of chemicals (Devi Baskar *et al.,* 2017; Nasrollahzadeh *et al.,* 2016). The properties of Tio$_2$ nanoparticles like physical and chemical can be changed by particle size, surface morphology, crystalline phase, and dimension. Meanwhile, the preparation of nanostructured Tio$_2$ with the large surface area and crystallised size is very crucial for electric, optical and catalytic properties (Nagarajua *et al.,* 2013; Antonietti M *et al.,* 2004). Mainly, Tio$_2$ nanoparticles have been used as a catalyst in the organic reaction, decomposition of organic waste in water (Nasrollahzadeh *et al.,* 2016; Kundu *et al.,* 2013).

In the previous study, already reported that the green synthesis of metal oxide using *Cassia fistula* plant extract was a cost-effective and eco-friendly method. *Cassia fistula* leaves contain B2, bioflavonoids, triflavonoids, rhein, rheinglucoside, sennoside A, sennoside B, chrysophanol, and physcion, etc. Mainly, this plant is considered as medicinal plant due to their wide properties like a mild laxative for children and pregnant women, used to cure the unhealing disorder, also possess antipyretic, analgesic, anti-inflammatory and hypoglycemic activities (Suresh *et al.,* 2015).

In this present investigation, we synthesized titanium oxide nanoparticles using herbal plant *Cassia fistula* aqueous extracts. The synthesized nanoparticles were characterized using UV-Vis spectroscopy, X-ray Diffraction (XRD), Fourier transforms infra-Red spectroscopy (FT-IR), atomic force microscopy (AFM), scanning electron microscopy (SEM), thermogravimetric analysis (TGA). Meanwhile, the synthesized Tio$_2$ NPs were tested in the antibacterial activity.

MATERIALS AND METHODS

Plant extracts preparation

Cassia fistula leaves were collected and dried. After that 1mg of *Cassia fistula* leaves were weighed and boiled with 100ml of distilled water. Then the extracts were filtered by using Whatman N. 1 filter paper, stored and used it for further analysis.

Synthesis of Tio$_2$ nanoparticles

1mm of Tio$_2$ was weighed and mixed with 80ml of distilled water. In that, 20ml of the prepared extract was added. After that, the sample was kept for observation in a shaker at 24hours. Color changes were noted and are showed in fig 1. After 24 hours of incubation, the prepared nanoparticles were centrifuged, and the powder was collected by using a hot air oven.

Characterisation of Tio2 NPs

The prepared green based Tio$_2$ nanoparticles were characterised by using UV-Visible spectroscopy, FT-IR, X-ray Diffraction, SEM, EDAX, TGA results. We absorbed the UV- visible spectrum range from 300- 800nm. We analyse the crystallise size of nanoparticles using X-ray diffractometer (PAN analytical X-Pert PRO) operating at 30 kV and 40 mA. The FT-IR spectroscopy is used to record the FT-IR spectrum of at a resolution of 4cm^{-1}. The SEM is used to analyse the shape and size of the molecule and EDAX are used to analyse the elemental composition of nanoparticles (J.Santhoshkumar *et al.,* 2017).

Antibacterial activity of TiO2 NPs

The antibacterial study of Tio$_2$ nanoparticles was done by using agar well diffusion method. Here we tested our nanoparticle against one gram-positive and one gram-negative bacteria. Finally, we predict the result based on the zone of inhibition.

RESULTS AND DISCUSSIONS

Visual observation and UV-vis spectroscopy

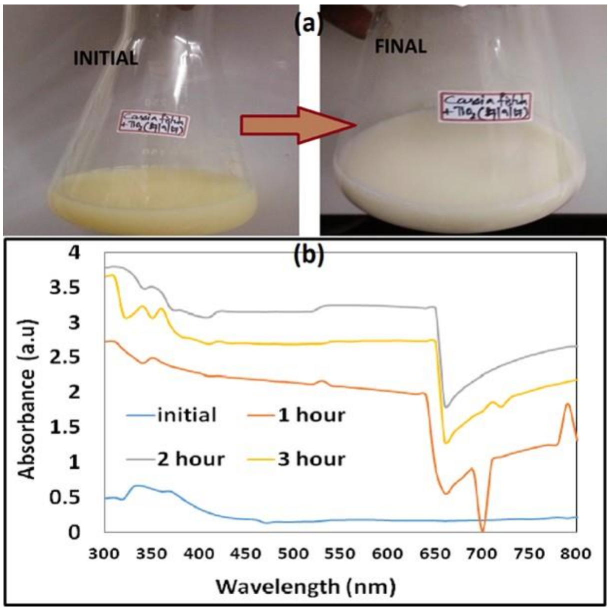

Figure 1: Green synthesis of TiO2 NPs (a) Visual observation (b) UV-Visible spectroscopy

The visual observation of ZnO nanoparticle was the pale yellow color turns to pure white milky color and represented below in (fig.1a). Then the UV-Visible spectra of Tio$_2$ NPs using *cassia fistula* are shown on (fig. 1b). It shows the peaks at different intervals. The spectral image displays the absorption peaks of Tio$_2$ NPs at a wavelength of 350nm. The UV image of *cassia fistula* already done on ZnO NPs with different concentration and with the wavelength of 370nm (Suresha *et al.,* 2015).

X-ray diffraction

The XRD analysis was done to confirm the crystalline nature and particle size of the biologically synthesised Tio$_2$ NPS. The fine sample of nanoparticle was placed on the XRD grid, and the crystallite size was determined and represented below in the (fig.3). Different Bragg's reflection is shown in the XRD pattern of Tio_2 NPS, which corresponds to (101), (102), (110), (103), (112), (201), (004), (202) on the set of lattice planes. Depends on Bragg's reflection, the synthesized Tio_2 NPS is face-centred cubic (FCC) and essentially crystalline in nature. The (112), (201) and (202) shows very weakly broadened wurtzite structure (Vijayakumar *et al.*, 2017).

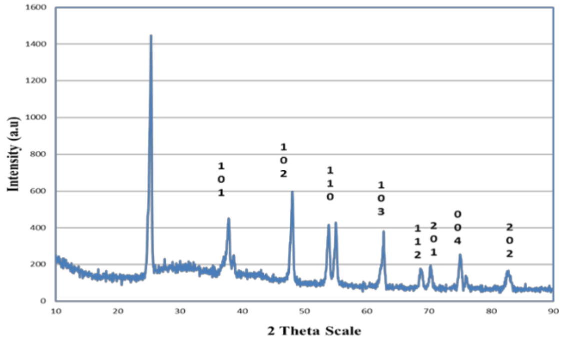

Figure 2: The XRD image of Tio2¬ NPs

Scanning electron microscopy (SEM) and Elemental dispersive analysis (EDX)

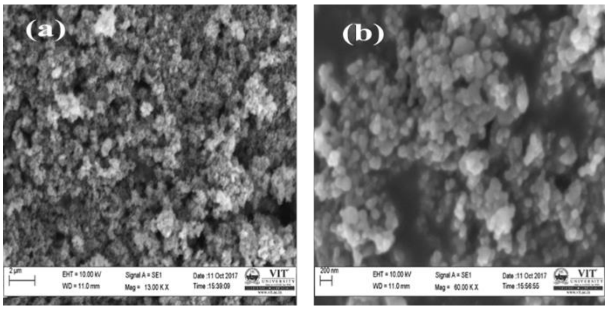

Figure 3: Tthe SEM image of Tio2¬ NPs (a) 2µm (b) 200nm

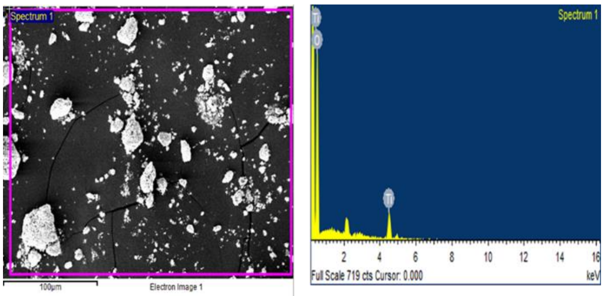

Figure 4: The EDAX image of Tio2 NPs

The SEM image of Tio_2 NPs is shown in (fig.4). It clearly shows that the particles consist of agglomerated and nearly spherical in shape. Therefore, the previous researcher reported that this kind of results only comes in metal oxides (D. Suresha *et al.*, 2015). In fig 4, the first image (a) displays the 2µm with 13000 K X magnification and the second image (b) displays the 200nm with 6000 K X magnification. The EDAX spectrum image shows the elemental composition, which is present in the Tio_2 NPs and is showed in fig 5. It displays three strong peaks which are identified as titanium and oxide molecules (Dhaneswar Das *et al.*, 2013).

Atomic force microscopy

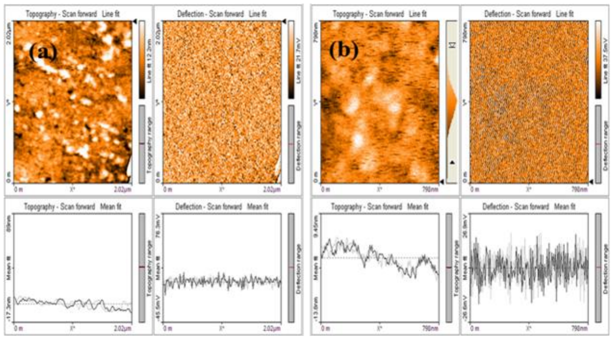

Figure 5: the AFM images of Tio2 NPs (a) 2.03µm (b) 798nm

The AFM imaging was conducted in 2.02µm and 796nm. The image displays that the phytochemicals are capped on the surface of the nanoparticle. Therefore, it gives us the best understanding of topography and roughness of nanoparticle (Santhoshkumar *et al.*, 2017). To validate the surface morphology, drop coated images were taken in a non-contact mode, which is represented in (fig. 6).

Thermogravimetric analysis

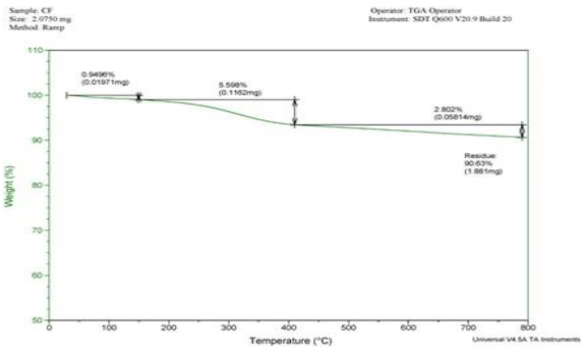

Figure 6: the TGA-DTA image of Tio2 NPs

The thermogravimetric and derivative thermogravimetric analysis was predicted on the synthesised Tio_2 NPs. The TGA-DTA curve of Tio_2 nanoparticles shown in (fig.7). It can be seen that the TG curve decreases till 400̊C. The TG-DTA traces show three different regions. The first weight loss is shown on below 150̊C due to their dehydration of water. The second weight loss shows that the decomposition of compounds in the Tio_2 NPs from 150-400̊C. The previous researcher already reported this work for rice sample (Ramimoghadam *et al.*, 2014). Therefore, the TGA curves represent that the proteins were degraded, and the organics were burned during the heating process.

Antibacterial activity

The antibacterial activity of synthesised Tio_2 NPs was tested against two pathogenic strains namely

Escherichia coli and Staphylococcus aureus. The agar well diffusion method was performed to test the Tio_2 NPs. It shows the zone of inhibition on the Tio_2 NPs was shown in fig 8. Here we used tetracycline as an antibiotic and water as a control (D. Suresha *et al.,* 2015).

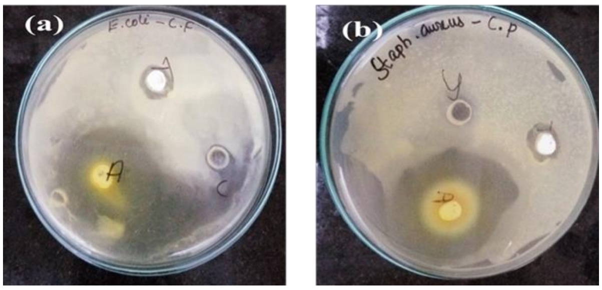

Figure 7: The antibacterial image of Tio2 NPs (a) E. coli and (b) S. aureus

CONCLUSIONS

In summary, the synthesis of Tio_2 NPs was done by using a green synthesis method. The UV spectral results show that the nanoparticles synthesised properly, XRD results predict that the particles are crystalline, the SEM and EDAX results display that it has nearly spherical in shape and the elemental composition Tio_2 NPs in the sample. TGA-DTA shows the heat liability of the NPS and AFM shows the roughness of the NPs. The antibacterial activity shows the effect of NPs. Therefore, the above results were predicted by using green synthesised Tio_2 NPs.

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