Green synthesis of titanium dioxide nanomaterials from drypetes sepiaria leaves extract and their application environment pollutant remediation

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Abstract: The current report, an eco-friendly to environment benign to fabrication of Titanium Di-Oxide Nanomaterials (TDO-NMs) using *Drypetes sepiaria Leaves* aqueous extract, during the procedure, rather than using noxious and persistent chemicals throughout the experiment. Characterization of Titanium Di-Oxide Nanomaterials was revealed using UV–Vis spectroscopy, X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR). The stability analysis of Titanium Di-Oxide Nanomaterials was determined through dynamic light scattering (DLS), which indicated that the Titanium Di-Oxide Nanomaterials were stability around -39.4 mV and with an average size of 16.78 nm. The synthesized Titanium Di-Oxide Nanomaterials were carried out for the degradation of industrial textile pollutants dyes, such as Methylene Blue. The synthesized Titanium Di-Oxide Nanomaterials acts as a remarkable catalytic activity for the removal of environment pollutants dye degradation and promising material for the dye degradation.

Keywords: Drypetes sepiaria Leaves; Titanium Di-Oxide Nanomaterials; MB; Photodegradation

1 Introduction

The controlling of matter at the nanoscale is known as nanotechnology. At this point, the increasing surface to volume ratio causes a drastic shift in the chemical and physical

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properties of the materials. With its application in science and technology to create novel materials at the nanoscale level, nanotechnology is emerging as a rapidly expanding discipline that has an impact on a variety of domains, including the environment, nanoscience [1]. Industrial, biological, and pharmaceutical medicine have all benefited greatly from the use of nanotechnology. Because of its outstanding chemical and thermal stability, titanium dioxide is one of the materials that has been investigated. Titanium Di Oxide Nanomaterials have garnered interest recently because of their potential uses in solar energy conversion, photo-catalysis, pigments, sensor devices, and biological activities like antifungal, antibacterial, and anticancer activity [2].

Excellent redox potential, outstanding chemical stability, low cost, and environmental friendliness are only a few of the several attributes of titanium dioxide oxide nanomaterials that are responsible for their broad range of applications. Numerous variables, including the crystal structure, crystallite size, particle shape, and specific surface area, influence these characteristics. The manufacturing technique of the nanoparticles also has a significant impact [3]. Regarding crystal structure, there are three major polymorphs of titanium dioxide nanomaterials: rutile (tetragonal), anatase (tetragonal), and brookite (orthorhombic). Anatase and brookite are metastable at all temperatures and can be transformed to rutile by heat treatment at high temperatures, although rutile is the sole stable phase [4]. Each crystalline phase has unique physical characteristics that are best suitable for particular applications, depending on structure and particle size. Due to their ability to avoid hazardous chemicals, high temperatures and pressures, log-term time periods, and environmental organisms, biological methods utilizing plant extracts and microbes have garnered greater attention in recent years than chemical and physical methods for the synthesis of nanoscale nanoparticles [5].

Dye is widely used to color products in printing, plastics, rubber, leather, textiles, cosmetics, and other sectors. They produce a significant volume of colorful effluent as a result. More than 10,000 dyes are available for purchase. The textile sector is the industry that uses the most dyes, producing about 7×10^7 dyes annually, and is projected to produce 2% of the dye effluents emitted from related industries [6]. A natural or synthetic chemical used to add a colour to or change the hue of something. Various varieties of dyes exist, including Natural dyes, Artificial coloring agents and colours added to food and Additional dyes akin to leather. An ecosystem's ability to function properly can be severely hampered by dye concentrations more than 1 mg/L that result from the direct discharge of textile effluents into water and soil. The environmental impact of dyes is significant; even a concentration of 1 part per million can have detrimental effects. Most dyes are non-biodegradable, carcinogenic, irritate skin, and cause various tissue alterations because of their synthetic origin and organic structures [7].

The green patch move towards, the current study focused on the bio-synthesis of nanoparticles from *Drypetes sepiaria* Leaves Extract. The current study was first-time report green synthesized Titanium Di-Oxide Nanomaterials by *Drypetes sepiaria* Leaves Extract. In the green synthesis of nanoparticles using varies application, however the prepared nanmaterials used for pollutant removal. The synthesized potential catalyst Titanium Di-Oxide Nanomaterials used for the environment pollutants remediation. The ability of synthesized Titanium Di-Oxide Nanomaterials as an efficient green catalyst for the degradation of Methylene Blue without using a reducing agent NaBH₄ has been investigated at room temperature.

2. Experimental section

2.1 Materials and Methods

Drypetes sepiaria Leaves were collected in Seshachalam Hills at Tirupati region, Tirupati district, Andra Pradesh, India. Titanium tetra-isopropoxide (TIP), Methylene Blue dye to be a pure analytical grade, brought from Sigma-Aldrich and then employ without any auxiliary purification throughout the experiment. Double distilled (DD) water was utilized as solved right the way through the experiment.

2.2 Preparation of Aqueous Drypetes sepiaria Leaves extract

Drypetes sepiaria Leaves was collected washed tries with standard water followed by distilled water and dehydrated at room temperature for an overnight in a dust free chamber in the absence of sunlight radiation. The dried grass was grinned by electric mixer. 3 gm of the dehydrated grass powder was disseminated in 100 mL double distilled water and reserved in a hot plate at 80°C for 3h until deep green coloured solution was produced. The aqueous extract was filtered by utilizing whatman filter paper followed by centrifugation at 6000 rpm and the residue removed and liquid (supernatant) was stored in refrigerator at 4°C and used for the synthesis of Titanium Di-Oxide Nanomaterials

2.3 Synthesis of Titanium Di-Oxide Nanomaterials by using Aqueous *Drypetes sepiaria* Leaves Extract

0.05 M of Titanium tetra-isopropoxide (TIP) precursor was dissolved in 50 mL double distilled water. To the precursor solution, 50 mL of *Drypetes sepiaria Leaves* extract solution was added drop-wise at the ratio of 1:1 (v/v) utilizing dropper and kept under enthusiastic stirring for 3h until brownish yellow coloured colloidal precipitation appeared. Afterwards the solution was centrifuged at 6000 rpm for 30 min, and washed with double distilled water to eliminate any other impurities. The brownish green colour precipitate was dried at 80 °C for 3h in a hot air oven and grounded fine by mortar-pestle and calcinations at 600 °C for 3h.

2.4 Characterization of synthesized Titanium Di-Oxide Nanomaterials

The synthesized Titanium Di-Oxide Nanomaterials were characterized by using a UV-Visible spectroscopy (Jasco V-670 UV-Visible double beam spectrophotometer). The colloidal Titanium Di-Oxide Nanomaterials solution measured with the reference solution deionized water. The spectra were recorded in between wavelength range 200-800 nm, The Fourier Transform Infra Red instrument analyzed the existence of functional groups in the Drypetes sepiaria Leaves extract and Titanium Di-Oxide Nanomaterials. The purified dried Titanium Di-Oxide Nanomaterials using Shimadzu IR AFFINITY-1 against Drypetes sepiaria Leaves extract powder as control using JASCO FT-IR 4100 in the diffuse reflectance mode at a resolution of 4 cm in KBr pellets. In order to complete reduction of Titanium Di-Oxide Nanomaterials were separated from the colloidal medium by using centrifugation at 3000 rpm, 30 min and then fine pellet obtained was washed thoroughly with de-ionized water tries and dried oven at 60 °C, used for XRD and FT-IR analysis. The XRD analysis of purified Titanium Di-Oxide Nanomaterials was carried out by a Bruker D8 Advance diffractometer with Cu K α radiation (λ =1.54⁰). The diffractogram was recorded in the scanning range between 10^{0} - 90^{0} (20 value) with a scanning rate of 4^{0} /min and a step size of 0.02⁰. From (DLS) analysis to determine the stability of nanoparticles

expressed in term of Zeta value by the distribution of Titanium Di-Oxide Nanomaterials such as poly - dispersity indexes (PDI) using Horiba scientific nanoparticci (nanoparticle analyzer, SZ-100). The sample was interpreted in at 20 °C with a scattering angle of 173°. The synthesized Titanium Di-Oxide Nanomaterials were measured Zeta potential. The suspension was measured in cell culture medium at 25 °C, 150 V. Initially, the instrument was calibrated before sample with standard Malvern-50 V.

2.5 Photo catalytic activity of Titanium Di-Oxide Nanomaterials

The Photo catalytic activity was determined by synthesized Titanium Di-Oxide Nanomaterials on degradation of model pollutants dyes in an aqueous medium such as Methylene blue and Rhodamine B. The photo degradation was carried out by using Heber multi lamp photo reactor, the instrument set up the parameters with UV lamp irradiation at 125 W, λ_{max} 254 nm at low-pressure. 70 mL of 10 ppm solution of dye was taken in 100 mL capacity of quartz tube and then continuous stirring under the dark condition the dye solutions containing in the absence of a catalyst and then progress of reaction was monitor by VU-Visible spectroscopy. The secound set run the photoreactor with Titanium Di-Oxide Nanomaterials nanocatalyst (10 mg) under the UV lamp irradiation at same parameter condition. The catalytic degradation of organic pollutant dyes were observed by measuring UV-Visible spectra, the absorption spectra gradually degraded at regular time intervals at 200 -800 nm. The degradation was noticed by chaging colour and collected sample run the UV analyis at regular interval and then monitored UV-Visible spectroscopy.

3 Result and discussion

In the present study, we synthesized stable crystalline Titanium Di-Oxide Nanomaterials throughout the end using any external surfactant, chemical reducing agent in the process. Basically, plant extracts act as reduced metal ion to metal nano-materials and size controlling agents and prevents nucleation and coalescence of nanomaterials i.e. regulate the aggregation/agglomeration of nanomaterials. The following different host of analytical technique used for the synthesis of Titanium Di-Oxide Nanomaterials.

3.1 UV-Visible study of Titanium Di-Oxide Nanomaterials

In the present study, Titanium Di-Oxide Nanomaterials were synthesized using aqueous *Drypetes sepiaria* Leaves Extract, in the synthetic process double distilled water used as a solvent as an alternative of organic solvents. Hasty green progression biosynthesis Titanium Di-Oxide Nanomaterials using durva grass extract has been examine which is a simple easiest, cost well-organized, non-hazardous, eco-friendly for exploiting *Drypetes sepiaria* Leaves Extract. Plant extracts may take action both as reducing, stabilizing agents for the synthesis of Titanium Di-Oxide Nanomaterials [8].

The optical properties were resolute using UV–Vis spectroscopy. **Figure. 1** represents the distinguishing absorption spectrum of Titanium Di-Oxide Nanomaterials.



Fig. 1. UV-Vis Spectroscopy: Formation of the Titanium Di-Oxide Nanomaterials

The formation of Titanium Di-Oxide Nanomaterials noticeable band was noticed at λ_{max} 335 nm. The noticeable absorption coefficient for Titanium Di-Oxide Nanomaterials is resolute by the following equation.

$E_g = (hv) - (\alpha hv)^{1/2}$

Where, hv is indicating photon energy, α is the optical noticeable absorption coefficient, Eg is denoted as band gap energy. **Figure. 2** indicate the optical band gap of Titanium Di-Oxide Nanomaterials. The material band gap energy was deliberate by plotting $(\alpha hv)^{1/2}$ of Titanium Di-Oxide Nanomaterials Vs the photon energy (hv) Therefore, the band gap energy value of Titanium Di-Oxide Nanomaterials is around 3.21 eV respectively.



Fig. 2. UV-Visible absorption spectra: Band gap energy of Titanium Di-Oxide Nanomaterials

3.2 Powder X-ray Diffraction (XRD) examination of Titanium Di-Oxide Nanomaterials

XRD diffraction was used to analyze the structural properties of the synthesized Titanium Di-Oxide Nanomaterials and to categorize the phase and the purity of Nanomaterials. **Figure. 3** demonstrate the X-ray powder diffraction pattern recorded for Titanium Di-Oxide Nanomaterials. It shows the multi characteristic diffraction peaks lattice constants. The diffraction peaks corresponding to synthesized Titanium Di-Oxide Nanomaterials match with nanomaterials standard tetragonal structure. The obtained 20 and Miller indices are 25.12 (101), 37.71 (004), 48.56 (020), 54.53 (121), 62.85 (204), 75.96 (215), 82.34 (303). The obtained XRD pattern shows the sharp and narrow diffraction peaks indicate to crystalline nature of Titanium Di-Oxide Nanomaterials.



Fig. 3. The Powder XRD pattern of Titanium Di-Oxide Nanomaterials

The average diameter of crystallites was calculated by using Debye-Scherer equation.

$$D = 0.89\lambda/\beta \cos\theta$$

Where, λ is the X-ray wavelength of radiation (1.5406 Å), β is full-width half maximum (FWHM) of (101) plane and θ is Bragg's diffraction angle. From the above Scherer, equation calculated mean crystallite size of Titanium Di-Oxide Nanomaterials is 16.78 nm [9].

3.3 Fourier transforms infrared spectroscopy examination (FT-IR) of Titanium Di-Oxide Nanomaterials

FTIR examination was done, in order to determine the responsible formation of nanoparticles functional groups (phyto-chemicals) in *Drypetes sepiaria* Leaves extract and their role in the synthesis of Titanium Di-Oxide Nanomaterials. Representative spectrum of synthesized Titanium Di-Oxide Nanomaterials and *Drypetes sepiaria* Leaves extract (**Figure. 4**) spectra was recorded in the region of 500 to 4000 cm⁻¹ and **Figure. 4** represent the FT-IR spectrum of *Drypetes sepiaria* Leaves extract. In the spectra noticed peaks at 3358.07 cm⁻¹ is connected with hydroxyl –OH group stretching vibration suggesting the existence of phenols. The band located at approximately 2931.80 cm⁻¹ may possibly indicated to aromatic –C–H stretching vibration.



Fig. 4. FT-IR spectroscopy of Drypetes sepiaria Leaves extract

The peak which is represent to 1614.42 cm^{-1} can be recognized to (-C=O) carbonyl stretching vibration. The peak to be found at 1390.75 cm⁻¹ which is corresponding to (-C-N) carbon & nitrogen stretching trembling. In the current spectrum, there was also the strong permutation band at in the region of 1330.88 cm⁻¹ which is analogous to carbon & oxygen (-C-O-C-) stretching vibration. The peaks arise at 1238.37 cm⁻¹ can be endorsed to aromatic carbon and nitrogen and carbon (C-N-H) stretching pulsation. Similar way, Mahesh et al. [10] proposed various functional groups in sunflower leaf extract from FTIR analysis.



Fig. 5: FT-IR spectroscopy of Titanium Di-Oxide Nanomaterials

Figure 5 demonstrate the FT-IR spectrum of Titanium Di-Oxide Nanomaterials. in the spectrum one band was recognised it is approximately at 3365.78 which is analogous to

stretching vibration of carbonyl group on the surface of the Titanium Di-Oxide Nanomaterials and the peak at arises at 1631.78 which is corresponding to functional of carbonyl (-C=O) stretching band vibration. The peak at noticeable around 428.20 that is consequent to metal & oxygen (Ti–O) stretching vibration of Titanium Di-Oxide Nanomaterials, respectively, which is represents the **Figure 5**.

3.4 Stability examination of Titanium Di-Oxide Nanomaterials by Dynamic light scattering (DLS)

Zeta potential indicates valid key indicator for the constancy stability of the colloidal Titanium Di-Oxide Nanomaterial's dispersion medium. The constancy stability of Titanium Di-Oxide Nanomaterials was resolute by Dynamic light scattering technique. Constancy Stability of Titanium Di-Oxide Nanomaterials expressed in terms of Zeta value. **Figure 6** responsible the stability of colloidal Titanium Di-Oxide Nanomaterials dispersion medium, it was noticed to -39.4mV its is straight result based on the moving of the charged particles and the enormity of the zeta potential indicates the degree of electrostatic disgust between adjacent, correspondingly charged particle's in dispersion medium. The results was exposed in negative value indicate the good stability of Titanium Di-Oxide Nanomaterials due to the superior the zeta potential value could be impressive to higher the electric charge on the surface of the Titanium Di-Oxide Nanomaterials.



Fig. 6.DLS spectrum: Stability analysis of Titanium Di-Oxide Nanomaterials

3.5 Photocatalytic activity of fabricated Titanium Di-Oxide Nanomaterials

A Titanium Di-Oxide Nanomaterials was one of the significant applications for the photocatalytic environmental pollutant degradation. For the photo-catalytic applicationsmethylene blue (MB) has been preferred as model pollutant dyes for photo-catalytic degradation by multi lap photo reactor under UV light presence.

3.6 Photocatalytic activity of Titanium Di-Oxide Nanomaterials for the degradation of Methylene blue dye

The photo-catalytic activity of *Drypetes sepiaria* Leaves based mediated synthesized Titanium Di-Oxide Nanomaterials were study in removal of pollutant methylene blue dye. In order to recorded the degradation of methylene blue under high energy (λ_{max} =254 nm) UV light bombarding with irradiation. **Figure 7** demonstrates the absorbance spectra monitored at 200 to 800 nm for the customary time intervals for the degradation of methylene blue pollutant under the ultra violet light. The absorption noticeable band at 482 nm indicates the greatest wavelength of the pollutant methylene blue dye. It obviously represents that; reduce the absorbance of Titanium Di-Oxide Nanomaterials under the Ultra violet-light bombard nation. In correspondingly, Banoth Pravallika et al. [11] proposed dye removal of the Methylene blue dye in the existence of UV-light irradiation. Preliminary, recorded without Titanium Di-Oxide Nanomaterials, pollutant methylene blue did not transform comprehensively only less than 10 % small entities degradation species produced and the reaction components was monitored up to 90 min.

In analogous way in the occurrence of Titanium Di-Oxide Nanomaterials the reaction components was denoted by UV-Vis spectroscopy and then the dye removal of degradation was extraordinary decreased that was wan noticed in the figure 10 and removal of % of MB dye degradation was represent by following formula.



Fig. 7. UV–Vis absorption spectra of Methylene blue dye degradation in the presence of Titanium Di-Oxide Nanomaterials under UV light irradiation

Here, MB dye of C_0 is the initial concentration of the absorbance value in Ultra violet plot and C_t is the removal of MB dye degradation time taken for the time interval absorbance in the Ultra violet spectra. The obtained consequence result represents the 97.55 % of the pollutant methylene blue dye removal within 70 min in the presence in the presence of Ultra light irradiation (bombard nation). **Figure 8** demonstrates the overall percentage of pollutant methylene blue removal, which is degradation of small non toxic molecules degradation in the presence of Titanium Di-Oxide Nanomaterials. Therefore, the obtained final result resemble with other existence reported literatures of the pollutant methylene blue dye degradation [12].



Fig. 8. Percentage of Methylene blue dye degradation in the presence of Titanium Di-Oxide Nanomaterials catalyst (black line) and without catalyst (red line)

3.7 Feasible mechanism of Methylene blue removal

The practicable noticeably demonstration mechanism was removal of methylene blue dye degradation by means of Titanium Di-Oxide Nanomaterials, its semiconductor proceed as a photo-catalyst for the methylene blue dye which is represented as the following formulas (equeations). The feasible mechanism demonstrates the about the various reaction succession of removal pollutant dye degradation and where a bombarding with UV light in being there of Titanium Di-Oxide Nanomaterials to produce surplus of spontaneous oxygen species (ROS). In the same way photocatalytic dye removal of probable mechanism anticipated by Narasaiah et al. [13] being there of ZnO NPs for the removal of Methyl Orange and Cong red dye. A characteristic procedure, mechanism was anticipated by the subsequent formulas. When a UV light bombarding with Titanium Di-Oxide Nanomaterials, then leading to electrons from valence to conduction band transfer and the equivalent energy is superior than the band gap energy of Titanium Di-Oxide Nanomaterials (3.21 eV) in the procedure endorsing the production of holes in valence band & electron in conduction band. The produced ROS was reactive with methylene blue dye molecules to most important to water molecules and carbon dioxide.

In this methodology when a UV light is bombarding with Titanium Di-Oxide Nanomaterials, then the outside surface on the Titanium Di-Oxide Nanomaterials electrons move from valence-conduction band as exposed in formula (1). The conduction level of electron (e^-_{CB}) interact with oxygen to engender the super oxide ions (\overline{O}_2) formula (2) and then foremost to super oxide radicals interact with hydrogen ions (H⁺) to generate the – OOH formula (3). The valence level holes (hv ⁺_{VB}) interact by way of the molecules (water) to discharge the –OH then ions on the loose to hydroxide (('OH)) radicals equation (4, 5). The cleavage of hydrogen peroxide to releasing photoelectrons, hydroxyl radical ('OH) and then reduced the oxygen (O₂) adsorbed on the surface of the photo-catalyst to generated superoxide radicals (\overline{O}_2). Ultimately, methylene dye molecules were disintegrating by the producing highly vigorous hydroxyl radicals ('OH) and superoxide active radicals (\overline{O}_2) to releasing water molecules, carbon dioxide and mineral acids which is represented formulas (6-9) [14].



4. Conclusion

Successfully synthesized Titanium Di-Oxide Nanomaterials (Anatase phase) by a greenassisted method route using the Drypetes sepiaria Leaves extract, on a single step procedure at reasonable temperature. The XRD patterns confirmed the assembly of Titanium Di-Oxide Nanomaterials and crystalline nature, the average crystalline size was establish to be 16.78 nm. Stability of Titanium Di-Oxide Nanomaterials denoted as in expressions of zeta value, that was establish to be -39.4 mV. From the FT-IR advanced technique confirmed the occurrence of phyto-chemicals (poly-phenols), and even after the Temperatures (calcinations) process, only three bands presence due to presence of biomolecules capped on the surface of the Titanium Di-Oxide Nanomaterials and lower peak that is less than 1000 cm⁻¹ one peak arises that around 428 cm⁻¹, that is endorsed to metal & oxygen (Ti-O) band stretching vibration correspondingly. The synthesized Titanium Di-Oxide photo-catalyst (3.21 eV) exhibits outstanding photo-catalytic demonstrate for the catalytic removal (degradation) of Methylene blue which is the percentage of dye degradation reaching 98.12 % within 50 min correspondingly under the UV light bombard nation. From the results concluded that the synthesized plant based-Titanium Di-Oxide Nanomaterials exhibits excelling photocatalytic activity and promising materials for the environmental pollutants removal.

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