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ORIGINAL ARTICLE

Plant-mediated Synthesis of *Alternanthera philoxeroides* Titanium Dioxide Nanoparticles (APTiO_2NP)

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ABSTRACT

Background and Aim: Physical and chemical methods of synthesizing metal nanoparticles have been the focus for the last decade as researchers have broadly exploited them. These methods produce harmful by products. The biological synthesis of metal nanoparticles was found to be easy and economical. Green synthesis is a significant tool for reducing the destructive effects associated with the traditional methods for nanoparticle synthesis. The present work focuses on an eco-friendly approach for the green synthesis of titanium dioxide nanoparticles based on *Alternanthera philoxeroides* plant extract.

Objective: The advantages of the green synthesis method include biocompatibility, cost-effectiveness, sustainable practices, nontoxic, environmentally benign precursors, and simple procedures with time-saving.

Material and Method: Titanium dioxide, sterile distilled water, What's man filter paper No. 1, and *Alternanthera philoxeroides.* The green synthesis of titanium dioxide nanoparticles is carried out in simple steps. The extract of *Alternanthera philoxeroides* was used for the biological synthesis of the titanium dioxide nanoparticles. Nanoparticles were characterized by UV-vis spectrophotometer, Fourier-transform infrared spectroscopy (FTIR), and X-ray powder diffraction (XRD).

Results: The UV-Vis spectral pattern established the colloidal APTiO_2NP. The surface plasmon resonance (SPR) of APTiO_2NP showed maximum absorption at 372.8nm, and its energy bandgap of APTiO_2NP was calculated to be 3.326 eV. FTIR spectrum confirmed the involvement of Protein, Tannins, Saponins, Phenols, flavonoids, Glycosides, and Terpenoids, which are present in *Alternanthera*

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philoxeroides that would act as capping agents for TiO_2 NPs. The XRD result confirmed that the products were highly crystalline. The prominent peak was shown at 25.58°, and some other peak positions characterize the anatase phase of TiO_2NPs.

Conclusion: The synthesized APTiO_2NP could be used for environmental remediation regarding antimicrobial activity, catalytic activity, removal of pollutants dyes, and heavy metal ion sensing.

KEYWORDS

• *Alternanthera philoxeroides* • Nanoparticles • UV-Vis spectrophotometer • XRD • FTIR, surface plasmon resonance

INTRODUCTION

Nanotechnology is a growing branch of science and technology that produces or manufactures nano-sized materials that are very useful across all fields of science, including synthetic and biological chemistry. Two main approaches are used to synthesize nanoparticles: the top-down and bottom-up methods. The top-down approach involves breaking down large bulk materials into small nanoparticles through various mechanical, chemical, or physical methods, such as mechanical milling, laser ablation, high-pressure torsion, chemical etching, and sputtering. In contrast, the bottom-up approach entails building or growing nanoparticles from individual atoms or molecules. This method allows for precise control over the size, shape, and composition of nanoparticles, utilizing techniques like the sol-gel method, template-assisted synthesis, chemical precipitation, microwave-assisted synthesis, and self-assembly method. Nanoparticles (10^{-9} m) are identified as small objects that behave as complete units in terms of their transport properties**.** The small-sized nanoparticles have a large surface area relating to their volume at the time of interaction. Nanoparticles have unique catalytic, optical, magnetic, and electrical properties due to their nano-scale dimensions[2]. Different protocols havebeen considered for the production of metallic nanoparticles. Environmental toxicity is the major concern with physical and chemical synthesis methods. These methods are costly, toxic, high-pressure, high-energy requirements, difficult to separate, and potentially hazardous. The green nanoparticle synthesis method is a bottom-up approach where the main reaction is reduction/oxidation. The green chemistry view for nanoparticle synthesis is the choice of the solvent medium, reducing agent, and non-toxic material, respectively, for the stabilization of nanoparticles.[3] Developing clean, biocompatible, non-toxic, and eco-friendly methods using plant extract, microorganisms, and enzymes are gaining importance in nanotechnology. Titanium dioxide nanoparticles are natural mineral oxides widely used in pharmaceuticals, cosmetics, food coloring, and implantable biomaterials. Biological synthesis of nanoparticles by bacteria, fungi, yeast, and plant extract is the best alternative to develop nanoparticles. The green approach is a cost-effective, less labor-intensive, non-toxic, and environmentally friendly process for nanoparticle synthesis to avoid adverse effects in many non-material applications.[4] These biological sources can reduce the metal salts to their corresponding metal or metal oxide nanoparticles and stabilize them.

Titanium dioxide nanoparticles show diverse physio-chemical properties such as optical, dielectric, photocatalyst, antimicrobial agent, chemical stability, low cost, easy handle, eco-friendly, nontoxicity, and preservatives[5]. Tio_2 nanoparticles are extremely used in many areas like wastewater treatment, silk reproduction, reduction of dyes toxicity, food industries, pharmaceutical drugs, biomedicines, cosmeceutical,[6] tissue engineering drug delivery, diagnostics, imaging,sensing, artificial implants, gene delivery, and pest management[7;8;3]. UV-vis spectroscopy is a technique used for the characterization of nanoparticles and confirming the formation of nanoparticles by obsessing the characteristic surface plasmon resonance band. FTIR technique analyses the functional groups in the range of 500-4000cm^{-1} by crushing a small amount of sample APTiO_2NP and plant extract with KBr powder. To understand the binding interactions between the molecules in synthesized nanoparticles. The XRD pattern was examined for structural characterization (Crystalline phase identification) of the sample at 40 kV within the 2θ range with intensity Cu-Ku radiation (λ =1.5406 Å).

A wide variety of plant species, such as *Cynodon Dactylon*[25], *Sesbania grandiflora*[5], *Phyllanthus niruri*[10], *Trigonella foenum graecum*[11], *Aloe Vera*[12], *Tulbhagiaviolacea*[5], and many others which follow the principles of green chemistry for nanoparticles synthesis. *Alternanthera philoxeroides* is a member of the *Amaranthaceae* family and is native to South America and it is invasive to India[14]. It is an herbaceous perennial, a nonwoody summer plant and considered anenergetic invader in many regions of the world due to its ability to adjust to different ecosystems[15]. It showed the presence of glycosides, carbohydrates, tannins, steroids, flavonoids, tannins, Saponin, cortisones, amino acids, proteins, alkaloids, and diuretic steroids[16], *Alternanthera philoxeroides* contains ferulic acid, syringic acid, terpenoids, salicylic acid, kaempferol, and chlorogenic acid, one monoterpene (safrole), and one phenylpropane (myrcene)[16]. It shows anti-inflammatory activity, antiarthritic activity[7], anti-nociceptive, anti-hyperglycaemic effects, antifungal activity[14], antibacterial activity[20,21], a-glucosidase inhibitory activities[9], antiarthritic activity[19] anticancer activity[25] The occurrence of reducing sugar and amino acids in *Alternanthera philoxeroides* indicates that it is a good food source[23].This study deals with titanium Dioxide for nanoparticle synthesis using *Alternanthera philoxeroides* and the characterization of nanoparticles with UV-vis spectrophotometer, FTIR, and XRD.

MATERIALS AND METHODS

Titanium dioxide (no purification is needed) of analytical grade was purchased from RANKEM Laboratory Reagent (Avantor), ethanol, sterile distilled water, What's man filter paper No. 1, and all the other chemicals used in the proposed study were of analytical grade.

1. **Collection of plant material:** *Alternanthera philoxeroides*' whole plants were collected from across the sides of the Chambal River. It was identified by the Department of Botany, Government College, Kota. It is rinsed with tap water followed by distilled water to remove dirt and dried in an oven at 45^{0}C separately.
2. **Preparation of Extracts:** These were further ground to a fine powder using a blender. Approximately 5g of powder was boiled in 200 ml sterile distilled water at 50^{0}C for one hour. After cooling, the solutions were filtrated through Whatman's No. 1 paper and stored in a refrigerator for further use within a week.
3. **Preparation of solution TiO_2:** A 5mM solution of TiO_2 (MW 79.87 g) was prepared by dissolving it in 100ml of Sterile distilled water (without any filtration process) and kept on a Magnetic stirrer for two hours.
4. **Synthesis of APTiO_2NP:** Two Erlenmeyer flasks (250 ml) were taken and marked as CON. (control) and APTiO_2NP. 80 mL of 5 mM solution of TiO_2 was taken except for the control (100 ml) and given two hours for incubation on a magnetic stirrer. 20 ml of aliquot extracts was added into the flask slowly and carefully in a ratio of 4:1 (v:v) under stirring conditions for 4 h. After 4 h, the color of TiO_2 changed with plant extract. In contrast, the same time control did not change its color. Nanoparticle formation was confirmed by the color change of the reaction solution. For further processing, the nanoparticle was centrifuged at 5000 rpm for 15 minutes and washed 2-3 times. Pellets were collected and dried in an oven at 45-50ºC. Dried powders were ready for further characterization.

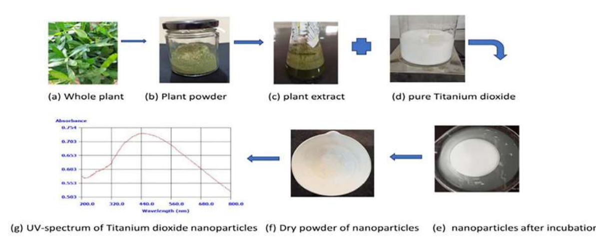


Figure 1: Green synthesis methods of titanium Dioxide nanoparticles using ***Alternanthera Philoxeroides***

Characterization of titanium dioxide nanoparticles:

UV–visible spectroscopy is a frequently used technique in the interpretation of nanoparticles. To characterize different metal nanoparticles with sizes ranging from 1 to 100 nm and beyond, UV-vis spectrophotometers are typically utilized in the 200–800 nm range.

The optical property of TiO_2 nanoparticles was analyzed via ultraviolet and visible absorption spectroscopy (UV-vis Double Beam Spectrophotometer 2202TS) in the range of 200 -800 nm. The energy bandgap of TiO_2 NPs was calculated using the equation:

$$Eg = hc/\lambda$$

Eg depicts the energy bandgap, h is Planck's constant (4.135×10^{-15} eV Hz^{-1}), c is the speed of light (3×10^{-8} m s^{-1}), and λ is the wavelength of the radiation (in nm).

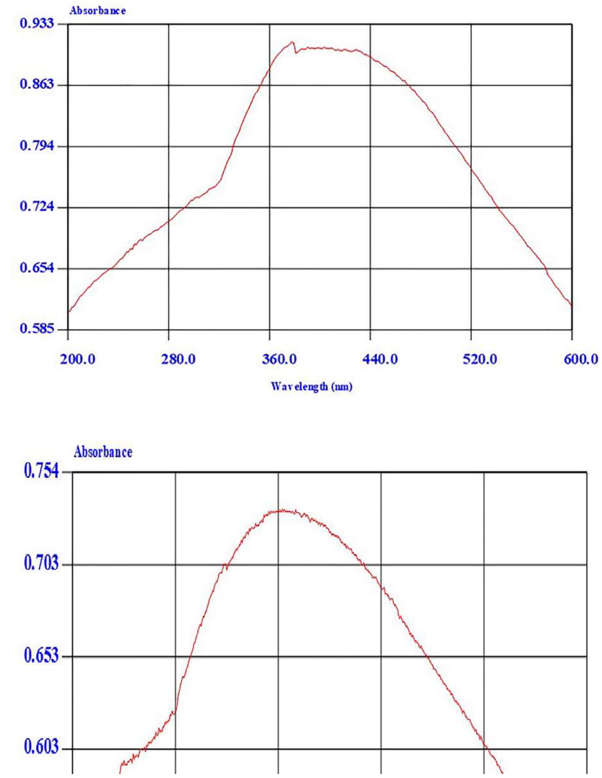
For FT-IR spectroscopy analysis, standard KBr pellets were prepared using dry powder of the $APTiO_2$ NP and *Alternanthera philoxeroides*' whole plant extract, which was dried at 40 °C and powdered, and the transmittance was measured using FTIR in the range from 500 to 4000 cm^{-1} to study the functional groups. X-ray diffraction (XRD) is used to determine the atomic and molecular structure of samples. This method was performed for irradiating the material with incident X-rays and to calculate the intensities and scattering angles of the X-rays which was scattered by the nanomaterial. The XRD was performed using an X-ray diffractometer–Cu, K*a* radiation λ 1.54 nm in the 2*θ* range.

RESULTS AND DISCUSSION

In the current study, *Alternanthera philoxeroides* was used as a solvent medium in place of organic solvents to synthesize the TiO_2NPs**.** The plant extract may serve as a capping and reducing agent in the green synthesis of metal nanoparticles.

The production of the $APTiO_2$NPs was verified by turning the white suspension into a soiled light-yellow hue. Figure 2 (a) displays the UV absorption spectrum of untreated TiO_2, revealing a prominent absorption band at 377.6 nm. The UV-Vis spectral pattern recognized the colloidal $APTiO_2$NPs. The surface plasmon resonance (SPR)of $APTiO_2$NP revealed the highest absorption at 372.8 nm (Figure 2 b).The surface plasmon resonance (SPR) of the TiO_2NPs is excited by the collective oscillation of conduction electrons, which interact with electromagnetic radiation to produce the color. It is commonly recognized that the surface plasmon resonance capping agents found in the leaf extract dominate the optical absorption spectra of metal nanoparticles.

TiO_2NPs synthesis with the leaf extract of *Trigonella foenum* (11), *Aloe vera*(12), and *Syzygium cumini* (23) showed absorbance at 400 nm, 356 nm, and 393 nm wavelength which arein excellent agreement with a band gap of the anatase phase.



(b)

Figure 2: UV-spectrum of titanium dioxide **(a)** Nanoparticles of Titanium dioxide

1. Using Alternanthera, 2. Alternanthera philoxeroides

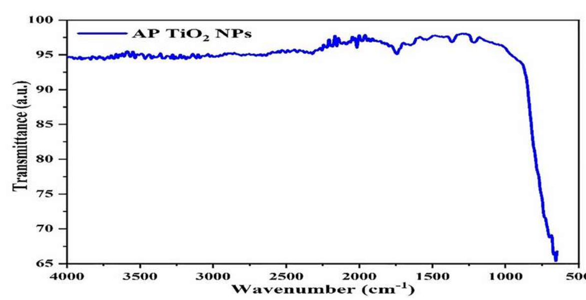


Figure 3: FTIR result of titanium dioxide nanoparticles using ***Alternanthera Philoxeroides***

FTIR spectrum was recorded using the wave number of 400–4000 cm^{-1} at room temperature for the synthesized TiO_2 nanoparticles using *Alternanthera philoxeroides*. The FTIR spectra of the synthesized titanium dioxide nanoparticles are given in Figure 3. This analysis revealed the presence of various functional groups at different wavelengths. The functional groups such as, -CH, –CN, -OH, -CC-, -CO, and -OH might have been derived from Protein, Tannins, Saponins, Phenols, flavonoids, Glycosides, and Terpenoids which are present in *Alternanthera philoxeroides* that would act as capping agents for TiO_2 NPs.

Different IR bands were observed at 2259 cm^{-1}, 1748 cm^{-1}, 1372 cm^{-1}, and 676 $cm^{-1,}$ respectively, for TiO_2NPs. The band at 2259 cm^{-1} was assigned to the nitrile group of C.N stretching the band at 1748 cm^{-1} was the characteristic band to C=O stretching in esters or -lactones, which indicates the presence of alcohol and phenol group. The band at 1372 cm^{-1} indicated the -OH bending of phenol. The band at 676$cm^{-1}$was mainly assigned to the Ti-O vibrational mode, approving the formation of the TiO_2 NPs[24].

Figure 4 depicts the X-ray diffraction graph of APTiO_2NPs produced from the aqueous extract of *Alternanthera philoxeroides'* whole plant. The presence of sharp diffraction peaks in the XRD result confirmed that the products were highly crystalline. The peaks' positions and intensities correspond to tetragonal, anatase polymorph of TiO_2NP. Main peak was exhibited at 25.58° corresponding to (101) plane and other peak positions at 37.16° (103), 37.98° (004), 38.83° (112), 48.15° (200), 54.15° (105), 55.33° (211), 62.83° (213), 69.00° (116), 70.58° (220), 75.23° (107), 76.25° (215). A strong peak at 25.58° with high intensity was observed.

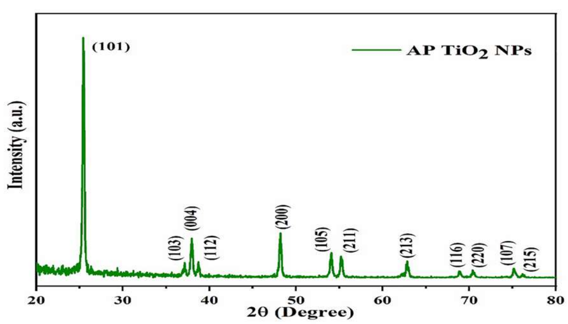


Figure 4: XRD pattern of titanium dioxide nanoparticles (APTiO_2NPs) produced from aqueous extract of ***Alternanthera philoxeroides*** whole plant

That is a peak which characterizes the anatase phase of TiO_2NPs at (101) plane. The XRD pattern displayed here is consistent with earlier reports[30].

These 2θ Values could be well indexed to anatase with the tetragonal crystal structure. These values matched the standard data file (JCPDS no. 75-1537). The products are pure in phases with the calculated lattice constants, a = b = 3.73 Å and c = 9.37 Å. The presence of sharp diffraction peaks in the XRD confirms that the products are highly crystalline. The expansion of the peaks was attributed to the nanoscale size of the nanocrystals. The average size of crystallite was estimated as 35.31 nm by Scherer's formula as given in the equation:

$$D = \frac{K\lambda}{\beta \cos\theta}$$

Where D is the average crystallite size, K is the Scherrer's constant (0.9), λ is the wavelength of X-ray (1.54 Å/ 0.154 nm), β is full width at half maxima (FWHM) for the peak, and θ is Bragg's angle.

The current study established that the manufacture of TiO_2 nanoparticles by utilizing an aqueous extract of *Alternanthera philoxeroides* whole plants is a novel method using inexpensive components. Furthermore, this process can be extended for the synthesis of other metal nanoparticles.

CONCLUSION

In this paper, we offered a simple and reproducible way for the synthesis of titanium dioxide nanoparticles. The use of natural plant extracts, distilled water, and practically nontoxic reagents allows the synthesis pathways presented to be measured as 'green' and so permits the synthesized TiO_2NPs to be used in sensitive areas such as wastewater treatment. The methodology employed here is very simple, easy to perform, inexpensive, and eco-friendly. The green synthesis of TiO_2 nanoparticles utilizing an aqueous extract of *Alternanthera philoxeroides* whole plants is an easy, eco-friendly, and very effective approach. The results of UV-visible spectroscopy showed the absorbance maxima of approximately 372.8 nm of green synthesized nanoparticles. XRD results confirmed that the products were highly crystalline. A strong peak at 25.58° with high intensity and some other peaks were observed, which characterize the anatase phase of APTiO_2NPs.The peaks' positions and intensities correspond to tetragonal, anatase polymorph of TiO_2NPs. The bands at 3119 cm^{-1} and 3569 cm^{-1} represented -OH stretching of alcohols. The band at 2259 cm^{-1} was assigned to the nitrile group of CN stretching. The band at 1748 cm^{-1} was the characteristic band for C=O stretching in esters or lactones. The band at 1372 cm^{-1} indicated the -OH bending of phenol.

Most importantly, these $APTiO_2NPs$ showed a significantly longer life expectancy with sustained reactivity in water pollution abatement, which means inwastewater treatment for reducing BOD, COD, turbidity, pH, heavy metal, dye, and diseases-causing microbesin the laboratory under controlled conditions. Through $APTiO_2NPs$, dyes have degraded by 80-90 percent of those present in textile waste and laboratory wastes.

Conflict of Interest: The authors declare no competing interest in this work

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