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GREEN SYNTHESIS OF TiO_2 NANOPARTICLES WITH *PSIDIUM GUAJAVA* (GUAVA) LEAVES EXTRACT

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Abstract:
The green synthesis of titanium dioxide (TiO_2) nanoparticles using *Psidium guajava* (guava) leaves has gained significant attention due to its eco-friendly approach and cost-effectiveness. In this study, TiO_2 nanoparticles were synthesized by reducing titanium precursor (titanium tetrachloride) in the presence of aqueous extracts of *Psidium guajava* leaves. The phytochemical components of the leaf extract, such as polyphenols, flavonoids, and organic acids, act as reducing agents and stabilizers during the synthesis process. The resulting TiO_2 nanoparticles were characterized using various techniques, including UV-Visible spectroscopy, X-ray diffraction (XRD), Field Emission Scanning Electron Microscopy (FESEM), and Fourier-Transform Infrared (FTIR) spectroscopy. This green synthesis approach provides a sustainable alternative to traditional methods, offering a low-cost and environmentally benign route for the production of TiO_2 nanoparticles.

Keywords : Titanium dioxide, *Psidium guajava*, Green synthesis, XRD, FTIR, and FESEM.

1. Introduction

Nanotechnology has emerged as a significant field of scientific innovation in the 21st century, offering advancements in various sectors such as medicine, environmental protection, and material sciences (Jadoun,S., *et al*., 2021). Nanoparticles, which range in size from 1 to 100 nanometers, exhibit unique physical, chemical, and mechanical properties that differentiate them from their bulk counterparts (Horikoshi.S.A.T.O.S.H.I., and Serpone.N.I.C.K., 2013). Among different nanoparticles, titanium dioxide (TiO_2) nanoparticles have gained considerable attention due to their strong photocatalytic activity, antibacterial properties, and environmental applications (Miller.R.J., *et al*., 2012).Traditionally, TiO_2 nanoparticles have been synthesized using chemical and physical methods that often involve toxic reagents, high energy consumption, and adverse environmental impacts. These drawbacks have led researchers to explore alternative, sustainable synthesis methods that are eco-friendly and cost-effective.

Green synthesis of nanoparticles is an emerging approach that utilizes biological resources such as plants, microorganisms, and natural polymers as reducing and stabilizing agents. This method aligns with the principles of green chemistry, reducing hazardous waste and making the process safer for both human health and the environment (Jadoun,S., *et al*., 2021). Among plant-based approaches, *Psidium guajava* (guava) leaves have been widely studied for their rich composition of bioactive compounds, including flavonoids, tannins, and phenolics, which facilitate the reduction and stabilization of nanoparticles. The use of *Psidium guajava* leaf extract for TiO_2 nanoparticle synthesis not only enhances the biocompatibility of the nanoparticles but also minimizes the need for harmful chemicals (Patil.S.P., and Rane.P.M., 2020).

This study focuses on the green synthesis of TiO_2 nanoparticles using *Psidium guajava* leaf extract, highlighting its advantages over conventional synthesis methods. The synthesized nanoparticles were characterized using various analytical techniques such as UV-Visible spectroscopy, FTIR, XRD, and FESEM to understand their structural and functional properties. By adopting a green synthesis approach, this research contributes to the development of sustainable nanotechnology, paving the way for environmentally friendly innovations in material science.

2. Materials and methods

2.1. Materials: The fresh leaves of *Psidium guajava* were gathered from the Chidambaram area, Tamilnadu. A Titanium tetrachloride ($TiCl_4$) solution with 99.9% purity was obtained from Sigma Aldrich Company, Bangalore . Sodium hydroxide (NaOH) pellets and double distilled water were also used in the nanoparticles synthesis process. All the reagents used were of analytical grade and were utilized without any additional purification.

2.2. Preparation of Guava leaves extract: Fresh *Psidium guajava* leaves were collected from the Annamalai University area in Chidambaram. The leaves were thoroughly washed with water, finely chopped, and crushed. Then, 60 g of crushed leaves was added to

350 ml of deionized water in a conical flask and boiled continuously for 3 hours at a specific temperature until the volume was reduce from 425 ml to 400 ml. The obtained extract was then filtered using Whatman filter paper.The collected samples were stored in a refrigerator for further analysis (Santhoshkumar. T., *et al*., 2014).

2.3.Preparation of TiO_2 nanoparticles: To synthesize TiO_2 nanoparticles, 1.0 N titanium tetrachloride ($TiCl_4$) was dissolved in 80 ml of water. Then 80 ml of leaf extract was slowly added while stirring until the pH reached 7. To adjust the pH, 0.8 g of 2N NaOH dissolved in 40 ml of water was gradually added. The mixture was stirred for 4 hours, changing color from dark brown to light brown, indicating nanoparticle formation. It was then cooled for 12 hours. Then the mixture was filtered, washed, dried at 100°C for 4 hours, and calcinated at 500°C for another 4 hours. The dried sample was kept in a desiccator for subsequent characterization(Rao. K. G., *et al*., 2015).

2.4. Analysis of TiO_2 nanoparticles: The optical characteristics of the nanoparticles were evaluated using the SHIMADZU-UV 1800 UV-VIS spectrometer. The surface morphology and elemental composition were examined using FESEM and EDAX (SIGMA 300) analysis. The crystalline structure and particle size were obtained using an X-ray diffractometer operating at 40 kV and 30 mA with a copper Kα radiation source. FTIR (PERKIN ELMER) is employed to detect and characterize the chemical bonds and functional groups present in the nanoparticles.

3. Results and discussion

3.1.UV-VIS Analysis : UV-Vis absorption spectroscopy was used to confirm the formation of TiO_2 nanoparticles, which showed a characteristic absorption peak at 265 nm, indicating their presence in the UV region and providing insights into their optical properties. These findings are consistent with previous studies (Hong.S.M., *et al*., 2013). The energy gap of TiO_2 nanoparticles was calculated using the Eqn 3.1.

$$Eg = \frac{hc}{\lambda} \quad (3.1)$$

Where h is Planck's constant (4.135×10^{-15} eV.s), c is velocity of light ($3\times10^{8}ms^{-1}$) and λ is wavelength (in nm) (Patidar.Vivek and Jain.P., 2017). From eqn 3.1, The band gap energy to be calculated as 4.7 eV at wavelength 265 nm corresponds to absorption peak. Fig 3.1. depicts the UV-Visible absorption peak of synthesized TiO_2.

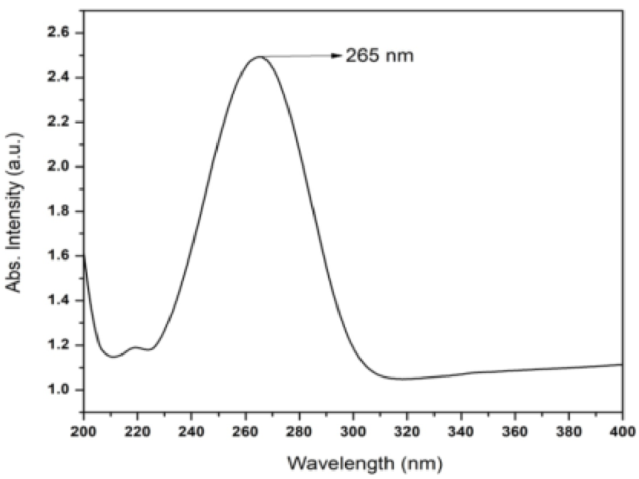


Fig 3.1. UV-Visible absorption peak of synthesized TiO_2

3.2. XRD Analysis : X-ray diffraction (XRD) was used to analyze the crystal structure of TiO_2 nanoparticles synthesized using *Psidium guajava* leaf extract. The diffraction pattern confirmed a well-defined and highly crystalline structure, with prominent peaks at 2θ values of 27.56°, 45.62°, and 56.61°, corresponding to the crystallographic planes (110), (210), and (211), respectively. These peaks align with the standard reference data (JCPDS card no: 21-1276), confirming the presence of the tetragonal Rutile phase of TiO_2 nanoparticles. The most intense peak at 31.88˚, corresponding to (121) crystallographic plane indicates the presence of Brookite phase of TiO_2 nanoparticles (JCPDS card no: 29-1360). The peak 24.66˚ indicates the presence of anatase phase in traces, corresponding to crystallographic plane (101)(JCPDS 21-1272). Based on the diffraction pattern, the Rutile and Brookite phase of TiO_2 was identified predominately (Rathore.C., *et al*., 2024). The calculated lattice parameters for the tetragonal Rutile phase were A = 4.5467 Å, B = 4.4452 Å, and C = 2.8284 Å, consistent with previously reported values (Kang.C.S., and Evans.E., 2021) .The average size of TiO2 NPs was calculated using the Debye–Scherrer equation.(Eqn.3.2)

$$D = \frac{K\lambda,}{\beta \cos\theta} \quad (3.2)$$

where D is the crystalline size under UV radiation, K is the shape factor (0.9), λ is the wavelength of X-rays (1.5406 Å), β is the full width at half maximum and θ is the Bragg angle (Selvi.J.M., et al., 2022). The average crystalline size was found to be 56.96 nm, with individual sizes of 69.30 nm, 43.91 nm, and 57.68 nm corresponding to the diffraction peaks at 24.66, 27.56°, 45.62°, and 56.60°, respectively. These values closely align with previous studies, indicating the successful synthesis of TiO_2 nanoparticles with nanoscale dimensions (Siah.W.R., *et al*., 2014). The results suggest that *Psidium guajava* extract effectively acts as a stabilizing and reducing agent in the green synthesis of TiO_2 nanoparticles, influencing their crystalline structure and size. Fig.3.2. shows the recorded XRD pattern of the synthesis TiO_2 nanoparticles. The XRD data of green synthesized TiO_2 NPs were given in the Table 3.1 along with the standard values.

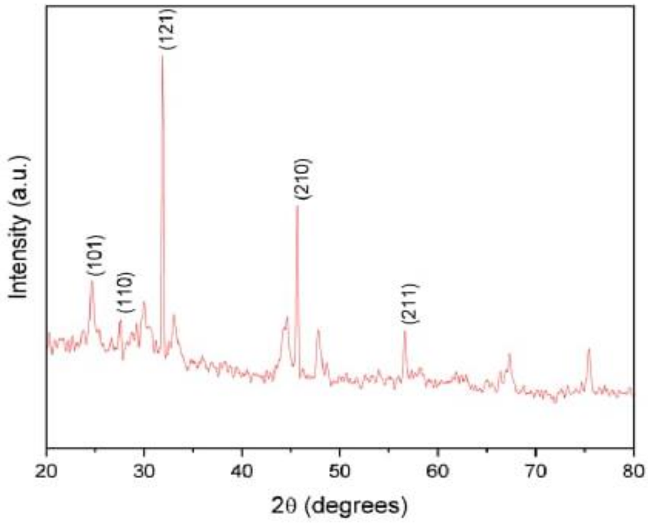


Fig.3.2. XRD pattern of green synthesized TiO2 NPs

TABLE 3.1. XRD data of green synthesized TiO_2 NPs

2θ		Full width Half Maximum (FWHM)	h k l	Crystalline size (nm)
Observed values	Standard values			
27.56	27.49	0.1181	110	69.30
45.62	44.10	0.1968	210	43.91
56.61	56.64	0.1574	211	57.68
Average crystalline size				56.96 nm

3.3 FTIR Analysis :FTIR spectroscopy was used to identify functional groups involved in the reduction and stabilization of TiO_2 nanoparticles synthesized using *Psidium guajava* leaf extract. The FTIR spectrum (Fig. 3.3) was analyzed in the 400–4000 cm^{-1} range, with peak assignments listed in Table 3.2. A broad peak at 3433 cm^{-1} corresponds to O-H stretching, indicating hydroxyl groups from water and biomolecules (Perumal.S., *et al*., 2014). Peaks at 2924 cm^{-1} and 2854 cm^{-1} correspond to C-H and CH_2 stretching, respectively, suggesting the presence of organic compounds. The 1638 cm^{-1} peak is attributed to H-Ti-O bending, confirming TiO_2 formation (Babu.N., *et al*., 2019). The peak at 1421 cm^{-1} corresponds to C=O stretching, likely from carboxyl groups aiding in nanoparticle stabilization (Goodarzi.M., *et al*., 2020). Peaks at 712, 536, and 482 cm^{-1} confirm Ti-O and Ti-O-Ti stretching, verifying the presence of TiO_2 nanoparticles (Soni.P., *et al*., 2024),(Jalaukhan. A.H., 2020). These results indicate that biomolecules from *Psidium guajava* extract played a key role in nanoparticle formation stabilization. Fig 3.3. shows the FTIR spectrum of green synthesized TiO_2 NPs.

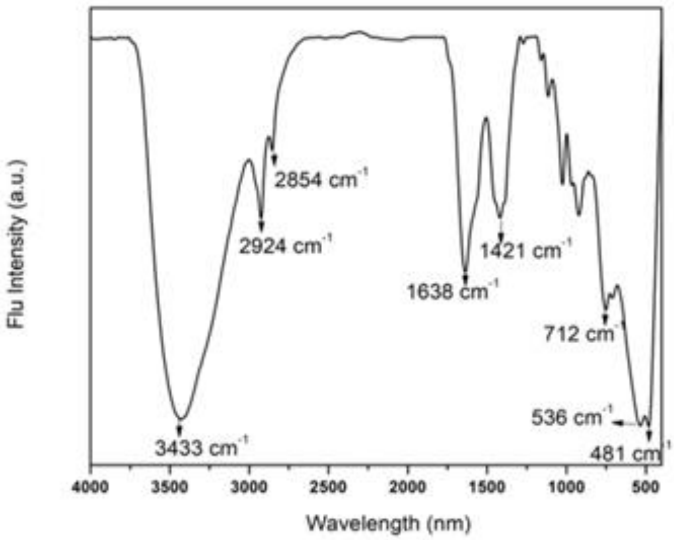
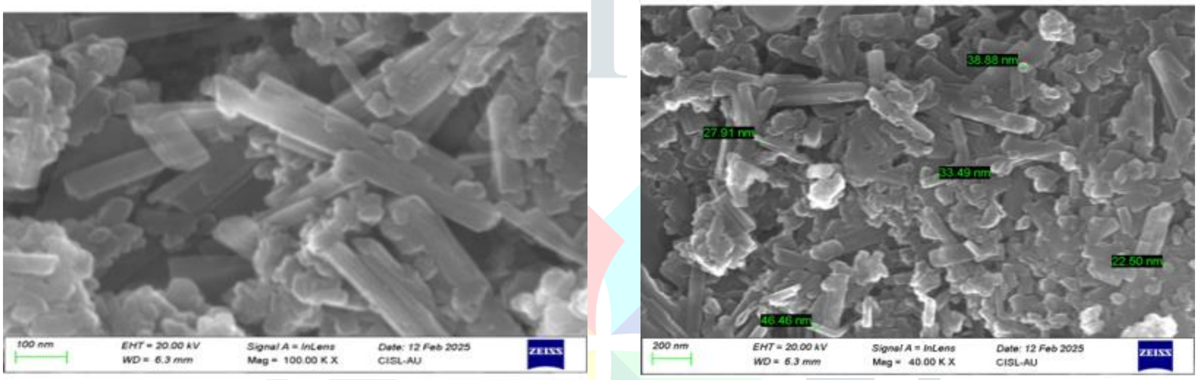


Fig 3.3. FTIR spectrum of green synthesized TiO_2 NPs

Table 3.2. Tentative peak assignments for synthesized TiO_2 NP

Wavenumber (cm^{-1})	Tentative peak assignments
3433	O-H stretching
2924	C-H stretching
2854	H-C-H stretching
1638	H-Ti-O bending
1421	C=O stretching
712	Ti-O stretching
536	Ti-O-Ti stretching
482	Ti-O-Ti stretching

3.4. FIELD EMISSION SCANNING ELECTRON MICROSCOPY (FESEM) ANALYSIS : FESEM was used to analyze the morphology of green-synthesized TiO_2 nanoparticles (TiO_2 NPs) using *Psidium guajava* leaf extract. The images confirmed uniform rod-like structures with sizes ranging from 23 nm to 46 nm, showing some agglomeration. The morphology closely matches findings from previous findings (Alosfur.F.K.M.,*et al.*, 2019). FESEM images at 100kx, and 40.00kx magnifications further validated the uniformity and structural integrity of the nanoparticles have been shown in Figure 3.4 (a), and Figure 3.4 (b) respectively. EDAX confirmed the elemental composition of the synthesized TiO_2 NPs, showing the presence of titanium (Ti) and oxygen (O) without impurities (Figure 3.5). The elemental analysis (Table 3.3) indicated a 1:1 atomic ratio of Ti to O, confirming the successful formation of TiO_2. FESEM and EDAX analyses confirm the successful green synthesis of TiO_2 nanoparticles, with well-defined morphology and high purity. The results highlight the efficiency of *Psidium guajava* leaf extract in nanoparticle synthesis, supporting its eco-friendly and sustainable application.



(a) (b)

Fig 3.4. FESEM images of green synthesized TiO2 NPs using *Psidium guajava* leaves extract at (a) 100kx, and (c) 40.00kx magnifications.

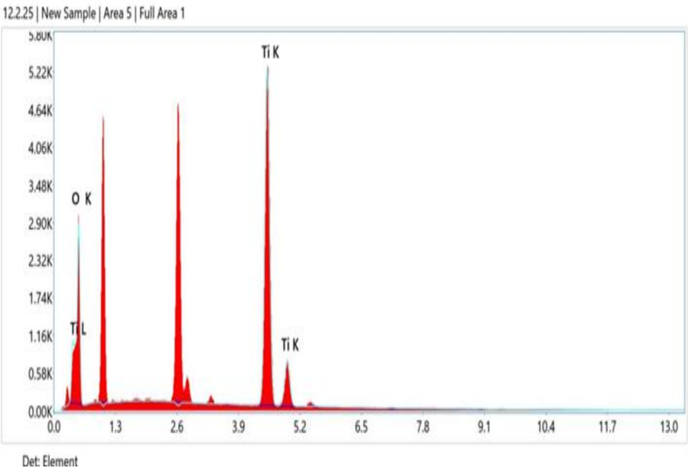


Fig.3.5 EDAX analysis of TiO_2 NPs

Table 3.3. Percentage of atomic weight ratio of TiO_2 NPs

Element	Weight (%)	Atomic (%)
Ti K	43.43	20.41
O K	56.57	79.59
Total	100.00	100.00

4. Conclusion

This study successfully demonstrated the green synthesis of TiO_2 nanoparticles using *Psidium guajava* leaf extract, providing a sustainable and eco-friendly alternative to conventional chemical methods. The phytochemicals in the guava extract acted as natural reducing and stabilizing agents, eliminating the need for toxic chemicals and high-energy processes. This approach aligns with green chemistry principles, offering a cost-effective and environmentally safe method for nanoparticle synthesis. Characterization techniques confirmed the successful formation and properties of the synthesized TiO_2 nanoparticles. UV-Vis spectroscopy revealed an absorption peak at 265 nm, indicating their optical properties. XRD analysis confirmed the rutile phase with an average crystalline size of 56.96 nm. FTIR analysis identified functional groups responsible for stabilization, while FESEM imaging showed rod-like morphology with particle sizes ranging from 23 nm to 46 nm. EDAX analysis further validated the elemental composition, confirming the presence of titanium and oxygen in the expected ratio. The synthesized TiO_2 nanoparticles will hold great potential for various applications, including photocatalysis, antibacterial treatments, and environmental remediation. Their eco-friendly production method makes them highly suitable for biomedical and industrial applications while minimizing environmental impact. This study provides a strong foundation for future research, focusing on optimizing synthesis parameters, enhancing stability, and expanding their applications in nanotechnology.

5.References

1. Alosfur.F.K.M., Ouda.A.A., Ridha.N.J., and Abud.S.H., **(2019).** Structure and Optical properties of TiO_2 nanorods prepared using polyol solvothermal method. In *AIP conference proceedings* (Vol.2144, No.1). AIP Publishing.
2. Babu.N., Pathak.V.M., Singh.A., and Navneet.A., **(2019).** Sonchus asper leaves aqueous extract mediated synthesis of titanium dioxide nanoparticles. *Pharma Innov. J*, (8), (817-822). *Chemical Society*, 34(1), 279-282.
3. Goodarzi.M., Naderi.F., and Ghanbari.D., **(2020).** Preparation of hollow TiO_2 nano-spheres, ZnO and Fe_2O_3 in sunscreens to absorb harmful ultraviolet rays. *Journal of Solar Energy Research*, 5(4), 594-602.
4. Hong.S.M., Lee.S., Jung.H.J., Yu.Y., Shin.J.H., Kwon.K.Y., and Choi.M.Y., **(2013).** Simple preparation of anatase TiO2 nanoparticles via pulsed laser ablation in liquid. *Bulletin of the Korean Chemical Society*, 34(1), 279-282.
5. Horikoshi.S.A.T.O.S.H.I., and Serpone.N.I.C.K., **(2013).** Introduction to nanoparticles. Microwaves in nanoparticle synthesis*: fundamentals and applications*, 1-24.
6. Jadoun,S., Arif.R., Jangid.N.K., and Meena.R.K., **(2021).** Green synthesis of nanoparticles using plant extracts: A review, *Environmental Chemistry Letters*, 19(1), 355-374.
7. Jalaukhan. A.H., **(2020**). Optical investigation of TiO_2/ graphene oxide thinflim prepared by spin coating technique. *Materials Science and Engineering*, (Vol. 871, no. 1, p. 012087).
8. Kang.C.S., and Evans.E., **(2021).** Synthesis and Transformation of Hollow Rutile Titania Wires by Single Spinneret Electrospinning with Sol-Gel Chemistry. *Fibers* 2021,9,18.
9. Miller.R.J., Bennett.S., Keller. A. A., Pease.S., and Lenihan.H.S., **(2012).** TiO2 nanoparticles are phototoxic to marine phytoplankton. *PloS one*, 7(1), e30321.
10. Patidar.V. and Jain.P., **(2017).** Green synthesis of TiO_2 nanoparticles using Moringa oleifera leaf extract. *Int. Res. J. Eng. Technol*, 4(3), 1-4
11. Patil.S.P., and Rane.P.M., **(2020).** Psidium guajava leaves assisted green synthesis of metallic nanoparticles: a review, *Beni-Suef University Journal of Basic and Applied Sciences*, 9, 1-7.
12. Perumal.S., Sambandam.C.G., and Mohamed.A.P., **(2014).** Synthesis and characterization studies of solvothermally synthesized undoped and Ag-doped TiO_2 nanoparticles using toluene as a solvent. *Journal of Engineering Research and Applications*, 4(7), 184-187.
13. Rao. K. G., Ashok. C. H., Rao. K.V., Chakra. C.S., and Tambur. P., **(2015)**. Green synthesis of TiO_2 nanoparticles using Aloe vera extract. *Int.journal Adv Res. Phyics. Science*, 2(1A), 28-34.
14. Rathore.C., Yadav.V.K., Amari.A., Meena.A., Chinedu Egbosiuba.T., Verma.R.K., and Patel.A., **(2024).** Synthesis and characterization of titanium dioxide nanoparticles from Bacillus subtilis MTCC 8322 and its application for the removal of methylene blue and orange G dyes under UV light and visible light. *Frontiers in Bioengineering and Biotechnology*, 11, 1323249.
15. Santhoshkumar. T., Rahuman, A.A., Jayaseelan. C., Rajkumar. G., Marimuthu. S., Kirthi, A.V., and Kim, S.K., **(2014).** Green synthesis of titanium dioxide nanoparticles using psidium guajava extract and its antibacterial and antioxidant properties. *Asian pacific journal of tropical medicine*, 7(12), 968-976.
16. Selvi. J.M., Murugalakshmi. M., Sami. P., Gnanaprakash. M., and Thanalakshmi. R.J.C.S., **(2022).** Green synthesis, characterization and applications of TiO_2 nanoparticles using aqueous extract of Erythrina variegate leaves. *Current science*, 123(1),(00113891).
17. Siah.W.R., Lintang.H.O., Shamsuddin.M., and Yuliati.L., **(2016).** High photocatalytic activity of mixed anatase-rutile phases on commercial TiO2 nanoparticles. In IOP Conference Series: *Materials Science and Engineering* (Vol.107,No.1,p.012005). IOP Publishing.
18. Soni.P., Pal.B., and Das.R.K., **(2024).** Influence of β-CD and Ag deposition over TiO_2 towards photocatalytic oxidation of urea under solar irradiation. *Journal of Environmental Chemical Engineering*, 12(2), 112150.