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Comparative Study of Titanium dioxide Nanoparticles prepared using *Jasminum grandiflorum* flower extract by Green and Chemical methods

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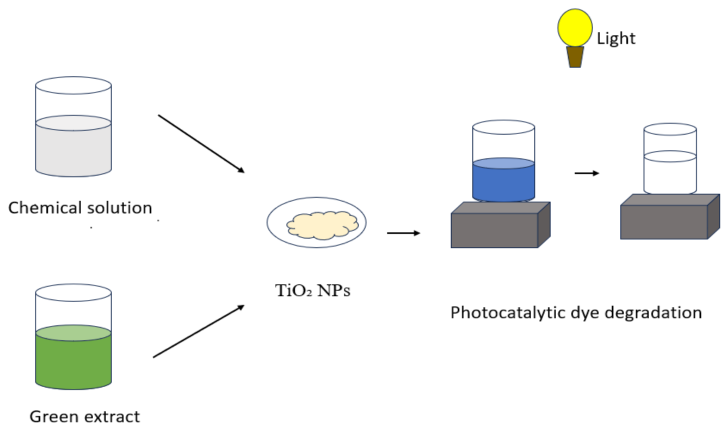
Abstract: Titanium dioxide nanoparticles were successfully synthesised using both chemical and green methods. In the green route, *Jasminum grandiflorum* flower extract acted as a natural reducing and capping agent. Spectral analysis at 328 nm shows the nanoparticles' optical responsiveness of nanoparticles in visible light. Vibrational spectroscopy confirmed the formation of TiO_2 nanoparticles by identifying Ti–O and Ti–O–Ti bonds. Both methods were evaluated for photocatalytic activity using Methylene Blue dye. Green-synthesized TiO_2 exhibited a band gap of 3.2eV, enhancing visible-light absorption and improving photocatalytic performance under artificial light compared to chemically synthesized TiO_2 with a 3.2 eV band gap.

Graphical abstract



Keywords: Titanium dioxide, *Jasminum grandiflorum*, Nanoparticles , green synthesis, Photocatalytic, Optical band gap.

1. Introduction

The introduction of dangerous materials or goods into the environment that results in negative changes is known as pollution. It may manifest as pollution of the air, water, soil, or sound. Water contamination is largely caused by industrial effluents, particularly those from the textile sector[1-2].

Colors are separated into three categories: cationic, non-ionic, and anionic. Among them, cationic dyes are employed in more extensive industrial applications such textiles, food coloring, and pharmaceuticals. By obstructing sunlight and reducing aquatic photosynthesis, these cationic dyes poisoned aquatic life when they are discharged directly into the aquatic environment. When people use this dye-contaminated water, it can lead to eye discomfort, kidney failure, cancer, and vomiting.

Industries such as textiles, leather, paper, and cosmetics make extensive use of synthetic dyes in their manufacturing processes. Many of these dyes are poisonous, carcinogenic, or mutagenic, and are resistant to light, temperature and microbial attack making them persistent in the environment[3-4].

Dye degradation refers to the breakdown of complex dye molecules into simpler, less harmful compounds, usually to reduce toxicity and color[5]. The field of nanotechnology, focused on designing and manipulating materials at the scale of 1 to 100 nanometers, has gained prominence due to its wide-ranging applications in healthcare, energy production, environmental solutions, and electronic devices. Due to their unique physicochemical properties such as increased surface area, enhanced reactivity, and quantum effects nanomaterials exhibit behaviours distinct from their bulk counterparts [6].

In medicine, nanoparticles are used in targeted drug delivery, imaging, and cancer therapy, allowing for Optimized, minimally invasive treatment options [7]. Environmental applications include the use of nano-adsorbents and photocatalysts like titanium dioxide (TiO_2) and zinc oxide (ZnO) for water purification and dye degradation, offering efficient ways to remove persistent pollutants from industrial wastewater [8]. In the energy sector, nanomaterials enhance the efficiency of solar cells, batteries, and hydrogen storage systems [9]. However, despite these advances, concerns remain about the potential toxicity and long-term environmental effects of engineered nanomaterials. Studies show that some nanoparticles can penetrate biological membranes, accumulate in tissues, and cause oxidative stress, raising questions about their biosafety [10]. As the field advances, there is growing interest in green synthesis methods and the development of regulatory frameworks to ensure the safe and sustainable use of nanotechnology [11].

Titanium dioxide nanoparticles (TiO_2) NPs are among the most extensively studied and utilized nanomaterials due to their excellent photocatalytic activity, chemical stability, low toxicity, and affordability. At the nanoscale, TiO_2 exists primarily in three crystalline forms: anatase, rutile, and brookite, with anatase being the most photocatalytically active under UV light [12]. When exposed to UV radiation, TiO_2 NPs generate reactive oxygen species (ROS), which can degrade a wide range of organic pollutants, including dyes, pharmaceuticals, and bacteria, making them highly effective for environmental remediation and self-cleaning surfaces [13]. Additionally, TiO_2 NPs are widely used in sunscreen formulations, paints, coatings, and food products due to their UV-blocking and whitening properties [14]. However, growing concern surrounds their environmental fate and potential toxicity. Studies suggest that TiO_2 nanoparticles may induce oxidative stress in aquatic organisms and human cells, especially under prolonged UV exposure, prompting calls for more comprehensive risk assessments [15]. To mitigate these effects, research is increasingly focusing on surface modification, doping, and the development of visible-light-active TiO_2 to enhance photocatalytic efficiency while reducing adverse impacts.

Nanoparticles can be synthesized through various methods, chemical and green synthesis being two primary approaches. Chemical synthesis methods, such as sol-gel, co-precipitation, and hydrothermal techniques, offer controlled particle size and morphology but often involve toxic solvents, high energy consumption and hazardous byproducts [16]. For example, the sol-gel method enables the formation of metal oxide nanoparticles through hydrolysis and condensation reactions but frequently requires strong acids or bases and organic solvents [17]. In contrast**,** green synthesis employs biological systems such as plant extracts, microorganisms, and enzymes as reducing and stabilizing agents making it an eco-friendly and sustainable alternative [18]. Plant-mediated synthesis is particularly promising due to its simplicity, scalability and the rich phytochemical content in extracts that facilitate nanoparticle formation without toxic chemicals. This method has been widely used to produce silver, gold, and titanium dioxide nanoparticles with good stability and biocompatibility [19]. Despite its environmental benefits, green synthesis sometimes lacks precision in controlling particle size and shape compared to chemical methods. Nevertheless, as nanotechnology advances, there is increasing interest in integrating both approaches using green reducing agents in chemically robust processes to achieve scalable and environmentally responsible nanoparticle production. This present work seeks to examine the chemical and bio-synthesis of the TiO_2 nanoparticles. Alkaloids, coumarins, flavonoids, tannins, terpenoids, glycosides, steroids, essential oil and saponins are the phytochemicals found in the extracts of *Jasminum grandiflorum* flowers [20].

The novelty of this work lies in the comparative evaluation of TiO_2 nanoparticles synthesised by green and conventional chemical routes using the same precursor and reaction conditions. While TiO_2 is a well-established photocatalyst, the influence of synthesis route on its physicochemical properties and photocatalytic efficiency under identical conditions has not been systematically studied. The green route employs plant extract–mediated biomolecules that can control particle growth and enhance surface activity, providing a more eco-friendly and efficient alternative to chemical synthesis. This comparative approach offers insights into how synthesis chemistry affects the optical band gap, morphology, and photocatalytic performance of TiO_2 nanoparticles.

2. Materials and Methods

Instrumentation:

UV–Visible spectra were recorded using a Shimadzu UV-2600 spectrophotometer. FTIR analysis was performed using a PerkinElmer Spectrum Two spectrometer. Surface morphology was examined with a JEOL JSM-7610F Scanning Electron Microscope operated. Elemental analysis was carried out by Energy Dispersive X-ray Analysis (EDAX). Thermal behavior was studied using NETZCH STA instrument under nitrogen atmosphere at a heating rate of 20K/min.

2.1 Nanoparticles synthesized by chemical method

5ml of titanium tetra isopropoxide made up to 50ml using distilled water. Take 20ml of titanium tetra isopropoxide solution, to this 20ml of ethanol is added. The resulting mixture is stirred continuously for 30 minutes to ensure proper mixing and reaction. After stirring, the solution undergoes sonication for 20 minutes to aid in the dispersion of particles. The mixture is then heated in an oven for 1 hour, which helps in the formation of the desired product. Following this, the sample is cooled and then filtered to obtain the final synthesized titanium dioxide nanoparticles.

2.2 Nanoparticles synthesized by green method

50grams of *Jasminum grandiflorum* flowers were taken and 250ml of distilled water boiled about 5 min. After boiling, the mixture is allowed to cool and then filtered using Whatman filter paper No.1 to obtain the flower extract, which shows a white to yellow colouration. From this 20ml of flower extract add 50ml of titanium tetra isopropoxide solution. The resulting solution is stirred continuously for 3 hours to ensure proper reaction and mixing. After stirring, the solution is filtered using Whatman filer paper. Finally, the filtered sample is dried in an oven for 2 hours to obtain the green synthesized titanium dioxide nanoparticles.

2.3 Photodegradation of Methylene blue

For photodegradation, MB dye is employed as a model pollutant. The photodegradation process was conducted with 200 W of artificial light.100 mg of NPs were taken in a 250 ml beaker together with 100 ml of Methylene blue solution to carry out the reaction. A magnetic stirrer was used to agitate the mixture. Every 30 minutes, the dye solution's maximum absorption was examined. A quartz cuvette with a path length of 10 cm and a capacity of 200 µL was employed to measure the absorbance of Methylene blue dye. Based on the absorbance data, the dye degradation efficiency was subsequently calculated.

3. Results and Discussion

3.1. UV-Visible spectroscopy

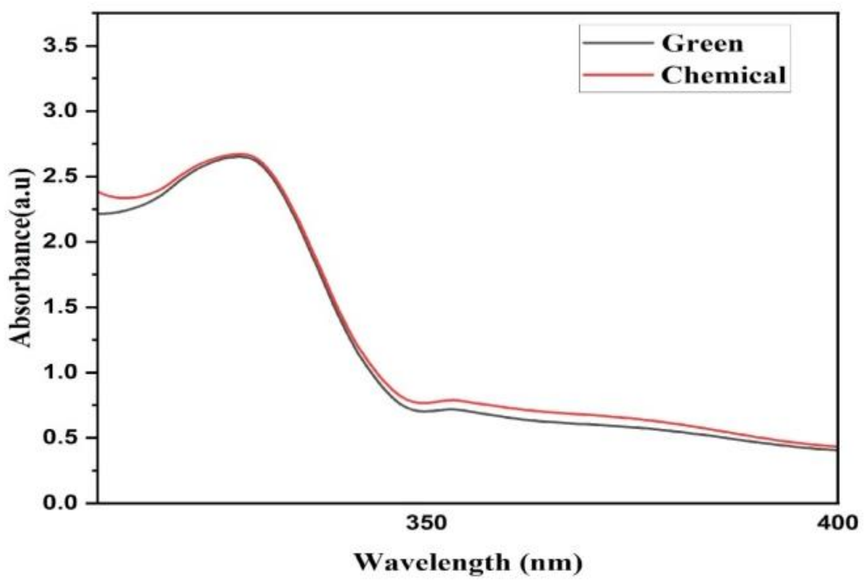


Fig.1. UV-Visible reflectance spectrum of TiO_2 NPs

The spectral properties of TiO_2 Nanoparticles were examined through UV-Visible spectroscopy. The reflectance spectrum of TiO_2 was shown in Fig.1. Over the period of green synthesis a gradual colour change was observed from white to yellow. It indicating the formation of TiO_2 NPs. The strong absorption peak around 328nm shows that the prepared nanoparticles are Titanium.

3.2 Optical band gap Determination Using Tauc Plot

The optical band gap of the synthesized Titanium dioxide nanoparticles was determined using UV-Vis absorption spectroscopy followed by Tauc plot analysis. The absorption coefficient was calculated from the absorbance data. According to the Tauc relation:

$$(\alpha h\nu)^n = A(h\nu - E_g)$$

where hν is the photon energy, A is a constant, and n depends on the nature of the electronic transition. A graph plotted against (αhν) versus hν and the linear line portion of the curve was extrapolated to intersect the energy axis, which gives band gap energy. The band gap energy for nanoparticles prepared by green and chemical method have 3.2 eV which can improve photocatalytic activity under solar or artificial illumination.

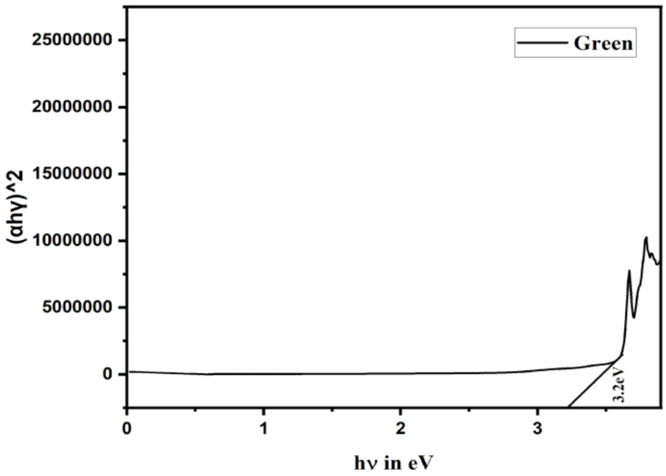


Fig.2 Tauc plot for TiO_2 NPs using Green method

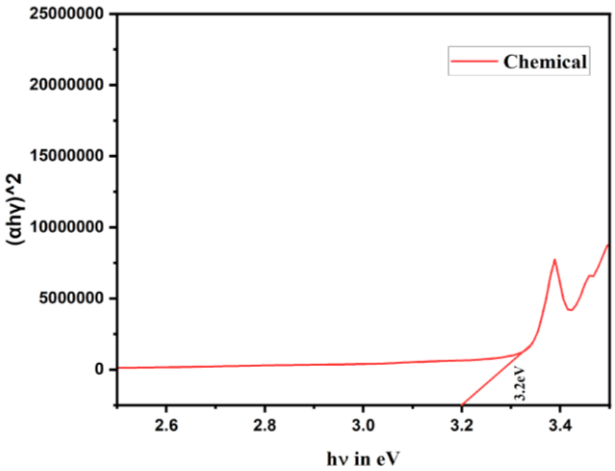


Fig.3 Tauc plot for TiO_2 NPs using chemical method

3.3 FT-IR-Spectroscopy

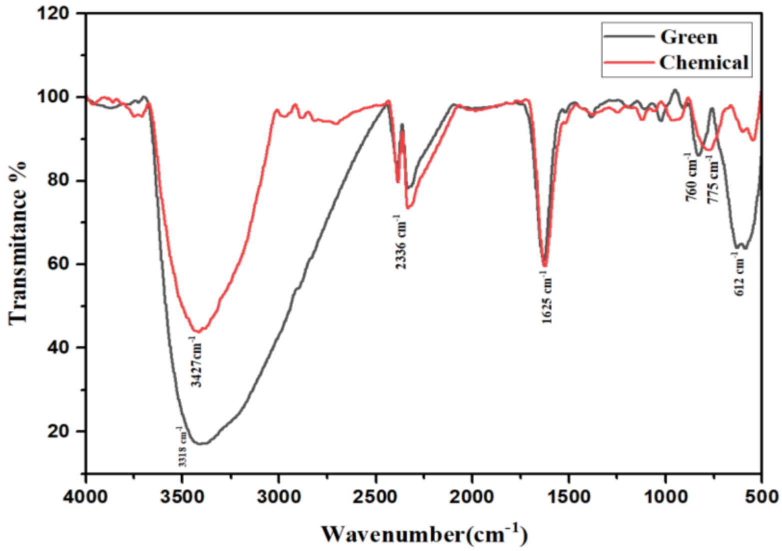


Fig.4. FT-IR spectrum of TiO_2 NPs

FT-IR spectrum of the synthesized TiO_2 nanoparticle was shown above the Fig.4. The peak at 3413-3429 shows that O-H stretching and peaks at 2332-2381 also shows O-H stretching. The peaks observed in the range of 777-820 cm^{-1} corresponds to Ti–O–Ti bending vibrations, while those in the range of 540-637 cm^{-1} are attributed to Ti–O bending vibrations. These metal-oxide bonds, Ti–O–Ti and Ti–O, confirm the presence of TiO_2 in the synthesized nanoparticles.

3.4 XRD-Analysis

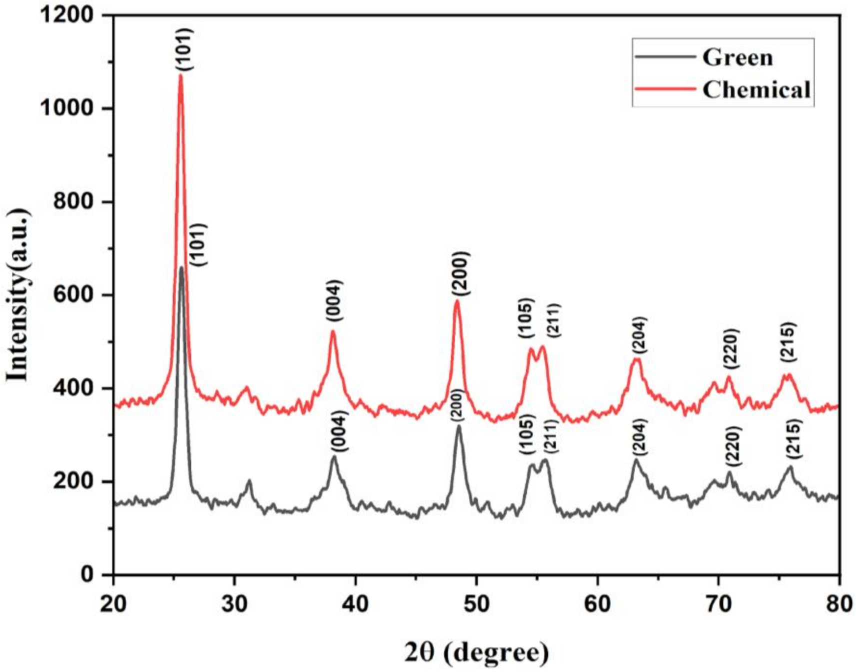


Fig. 5. XRD pattern of TiO_2 nanoparticles

Figure.5. shows the X-ray diffraction pattern for the TiO_2 nanoparticles produced by Green and Chemical methods. The synthesized TiO_2 nanoparticles are Anatase phase. The peaks obtained at 2θ values of (25.5°), (37.9°), (48.3°), (54.4°), (55.3°), (62.9°), (70.8°), (75.8°) can be attributed to the planes (101), (004), (200), (105), (211), (204), (220), (215) for both green and chemical method. The XRD pattern of TiO_2 nanoparticles shows good agreement with the JCPDS card number 21-1272.The Debye Scherer formula was utilized to compute the size of the nanoparticles.

$$Dhkl = k\lambda/\beta\cos\theta$$

In this case, μ represents the wavelength, β the line's full width at half maximum (FWHM), and θ the diffraction angle. The crystalline size was determined to be 5nm for green and 11nm for chemically prepared nanoparticles.

3.5 SEM and EDAX-Analysis

Energy Dispersive X-ray Analysis is referred to as EDAX analysis. This method is employed to determine the specimen's elemental composition. It's an investigation technique that's frequently used to characterize an object's elements or chemical makeup. EDAX experiments are used to determine the photocatalyst's elemental composition and purity.

Table 1: Chemical and Green method of EDAX-analysis

Element	At.No	Series name	Wt [%]	
			Green	Chemical
O	8	K-Series	33.14	45.79
Ti	22	K-Series	66.86	54.21

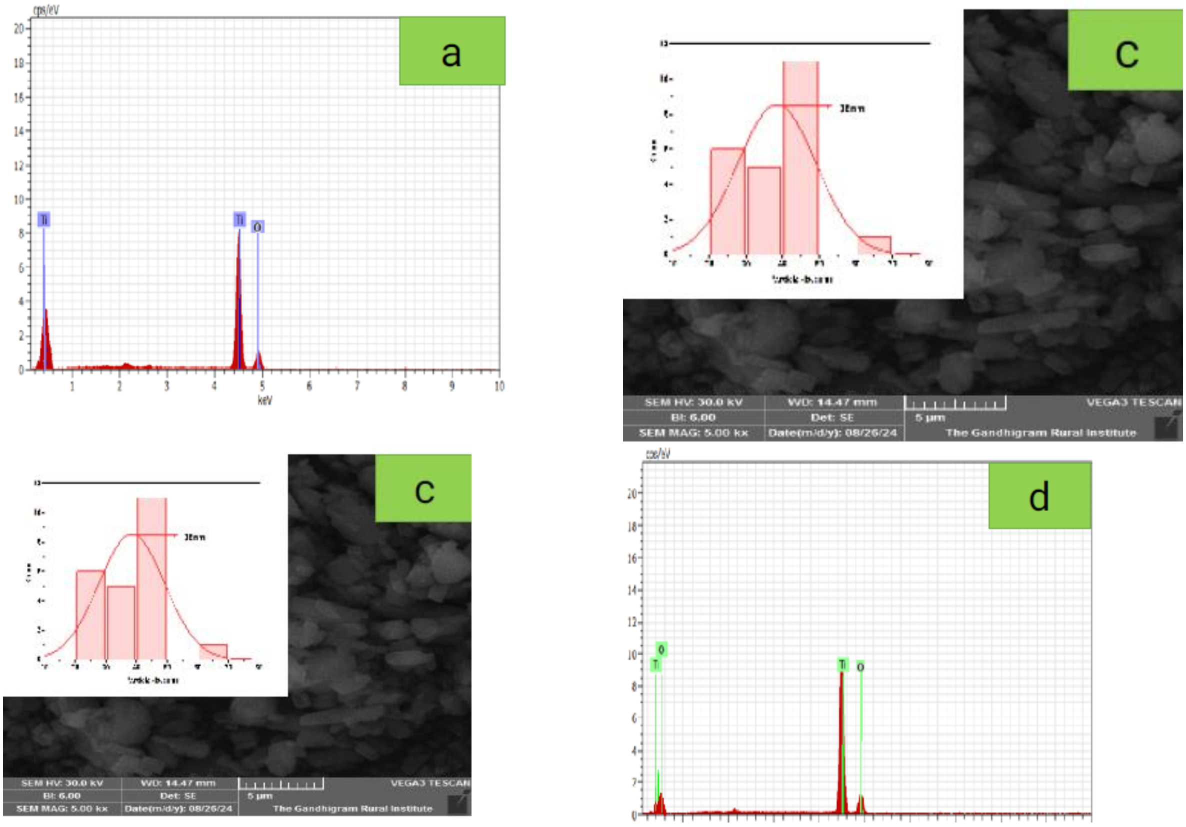


Fig. 6. (a-d) SEM and EDAX of TiO_2 NPs using Green and Chemical method

The SEM image of the nanoparticles are shown in the above figure.6 (a,b) green method and (c,d) chemical method The morphology analysis revealed that the particles exhibited a spherical shape. Furthermore, the EDAX spectrum confirms the presence of Titanium and Oxygen in the sample. Particle size of the nanoparticles were estimated using imageJ software. The size of the nanoparticles prepared by green synthesis method have 1.3 nm for chemical synthesis method have 38 nm respectively, the smallest size have the highest surface area. Therefore nanoparticles prepared by green synthesis method have higher surface area. Although SEM analysis provided valuable morphological information, the accurate determination of particle size and crystallinity requires TEM analysis. This will be considered in future work to obtain precise particle size distribution and confirm nanostructure.

3.6. Photodegradation Study of Methylene Blue Dye

The UV-visible irradiation method was employed to investigate the photodegradation of Methylene blue dye. The photodegradation efficiency of TiO_2 nanoparticles was calculated using the following formula:

$$\text{Dye removal (\%)} = C0 - Ct / C0 \times 100$$

Where, Ct is the temporal concentration of MB at time t and C0 is the starting concentration of MB, Methylene blue was used to test the photocatalytic activity of TiO_2 NPs produced by chemically and green method. Since Methylene blue dye is more toxic to humans and is frequently used for colouring purposes in the textile sector, it is included as a pollutant in this study. Therefore, eliminating Methylene blue from wastewater is a difficult task. Methylene blue's UV absorption spectrum at 665 nm is consistent with the π–π* transition. Results of absorption peak intensity reduction point to Methylene blue's deterioration. The degradation efficiency of TiO_2 NPs generated by green method is higher than that of TiO_2 NPs synthesized chemically. Under 180 minutes of radiation, the highest degradation of bio-mediated TiO_2 NPs is 68%. Both the chemically studied TiO_2 NPs and the bio-mediated TiO_2 NPs have a degradation efficiency of 68.18% and 50%, respectively.

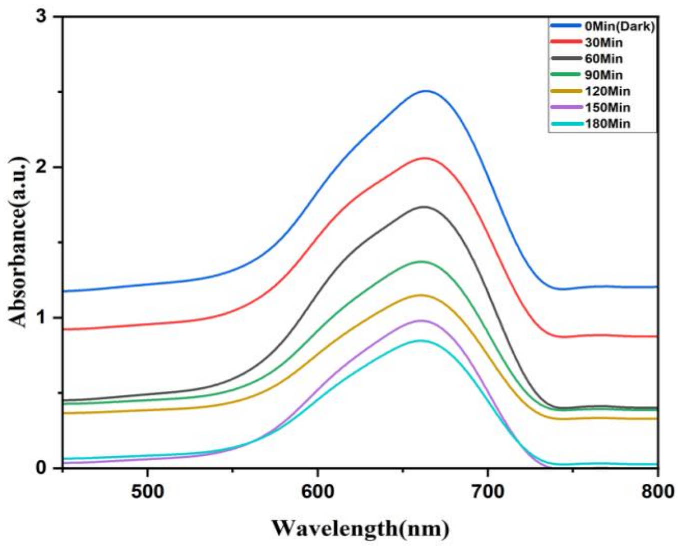


Fig.7 Photocatalytic performance of TiO_2 NPs Green synthesis method

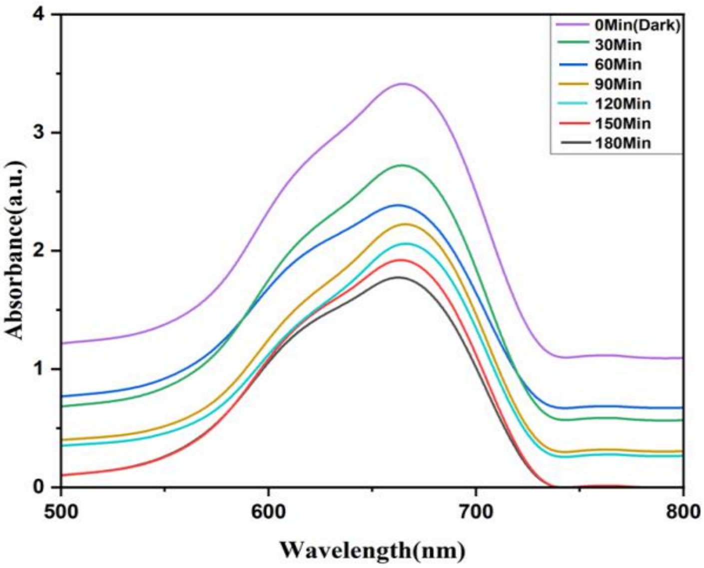


Fig.8 Photocatalytic performance of TiO_2 NPs Chemical synthesis method

Table 2:

Slope of the linear plot and rate constant value of Green and Chemical synthesis method

Synthesis Method	**Slope of the -ln(C/C0)**	**Rate Constant (k)**
Green synthesis	0.006	0.006 min^{-1}
Chemical synthesis	0.003	0.003 min^{-1}

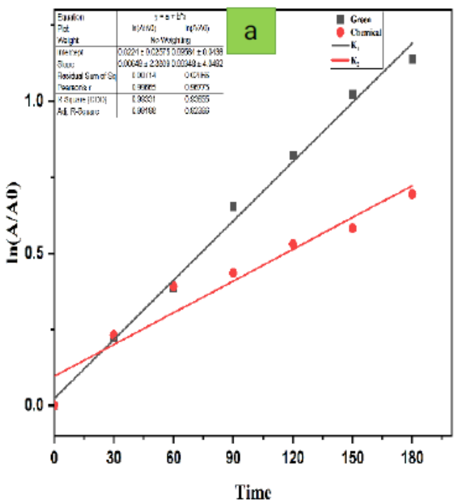


Fig.9.(a)
Kinetic curves of photocatalyst as a function of time for Methylene blue

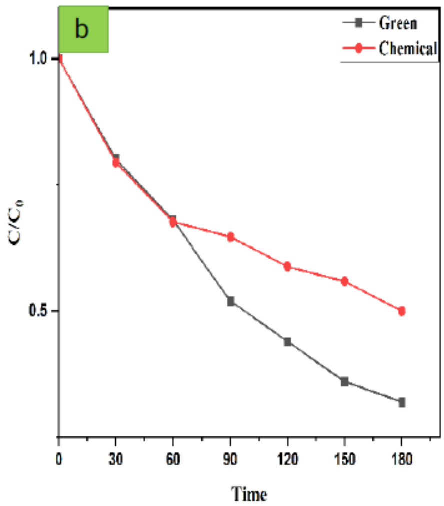


Fig.9. (b)
Time-dependent efficiency curves of prepared photocatalyst for the Methylene blue degradation

The photodegradation of the Methylene blue dye using TiO_2 NPs followed pseudo-first order kinetics**.** Kinetic curves of photocatalyst as a function of time for Methylene blue and Time – dependent efficiency curves of prepared photocatalyst for the Methylene blue degradation was shown in the fig.9.The rate constants calculated from the slope of the linear plot and rate constant value is higher for green synthesis when comparing chemical synthesis method rate constant K_1= 0.006 min^{-1} and K_2= 0.003 min^{-1} here K1 is green and K2 is chemical method rate constants. The photodegradation effectiveness of the chemical and green methods was computed every 30 minutes.

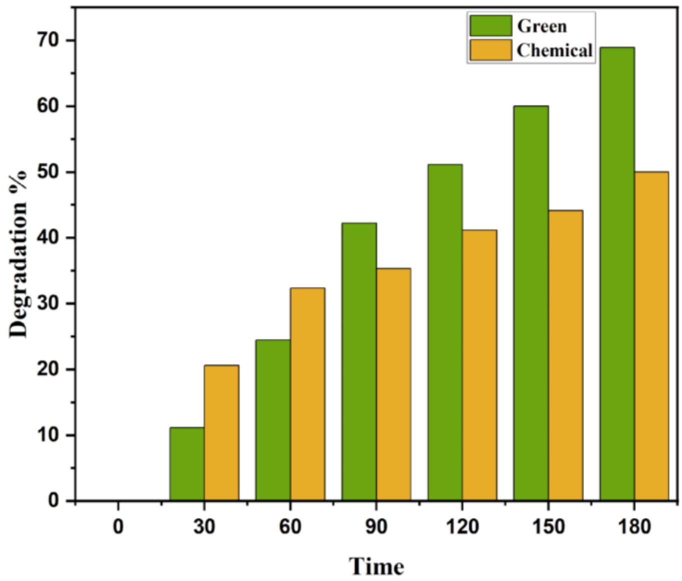


Fig.10. shows the photodegradation efficiency of green and chemical synthesis method over time

The degradation efficiency of Methylene blue using green and chemically prepared nanoparticles against time were shown in the above fig.10. The degradation efficiency from 0 min -180 min of difference shows that the green method nanoparticles have high degradation efficiency compared to chemically prepared nanoparticles. At 30 min and 60 min of evaluation chemical method have high efficiency after that from 90 min to 180 min green method nanoparticles efficiency was increased gradually it shown in the below table.3.

Table 3: Degradation efficiency for Green and Chemical method

Time	Degradation efficiency %	
	Green	Chemical
0 Min	0	0
30 Min	11.11111	20.58824
60 Min	24.44444	32.35294
90 Min	42.22222	35.29412
120 Min	51.11111	41.17647
150 Min	60	44.11765
180 Min	68.88889	50

The green-synthesised TiO_2 nanoparticles showed better photocatalytic performance due to several factors:

- The smaller particle size increases the surface area and number of active sites.
- The presence of plant-derived organic functional groups enhances charge separation and reduces electron–hole recombination.
- The slightly lower band gap energy allows better light absorption.

These synergistic effects lead to higher degradation efficiency of methylene blue compared to chemically synthesised TiO_2 nanoparticles.

4. Conclusion

TiO_2 NPs were successfully synthesized in this work using both chemical and green methods. In the presence of a 200W of artificial induced light. Bio-mediated TiO_2 exhibits a maximum breakdown efficiency of 68.18% after 180 minutes of radiation. Green and chemical approaches have been successfully used to generate TiO_2 nanoparticles. The XRD pattern indicates that the generated nanoparticles are anatase and spherical in shape. According to the XRD pattern, the nanoparticles' sizes were 11nm for the chemical approach and 5 nm for the green method. The green and chemical approach method's sharp absorbance peak at around 328 nm confirms the formation of TiO_2 NPs, according to UV-visible spectroscopy. From tauc plot we know that the optical band gap for anatase TiO_2 have 3.2eV. The EDAX spectra verified the presence of oxygen and titanium. Photocatalytic efficiency for green method is higher when comparing chemical method because of the phytochemicals present in the flower and shape, size and surface area of the nanoparticles. In the green synthesis route, phytochemicals such as flavonoids, phenols, and terpenoids present in the plant extract act as reducing and stabilizing agents. Titanium tetraisopropoxide (TTIP) undergoes hydrolysis and condensation, where the bioactive compounds cap the growing TiO_2 nuclei, preventing agglomeration and resulting in smaller, more uniform nanoparticles.

In contrast, in the chemical method, TTIP reacts with ethanol and water without any natural capping agents, leading to rapid nucleation and growth. The absence of stabilizing biomolecules allows aggregation, which results in larger particle sizes. Thus, the controlled release of ions and surface binding by biomolecules in the green method effectively limits particle growth and yields smaller nanoparticles.

Credit authorship contribution statement

L.R. Lalin Mary: Writing – original draft, Investigation, Data curation, Formal analysis.
A. Jeena Pearl: Supervision.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data Availability Statements

The authors declare that the data that support the findings of this study are available from the corresponding author upon reasonable request.

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