RESEARCH PAPER

Synthesis and Characterization of Titanium Dioxide Nanomaterials

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ABSTRACT
This study explores the synthesis and characterization of TiO_2 nanomaterials for cost-effective and stable electrode materials for application in solar cells. Given the significance of solar energy as a sustainable alternative to non-renewable resources, there is an urgent need to enhance the efficiency and affordability of solar cells. Conventional materials like silicon face challenges in production costs and efficiency, necessitating the exploration of alternative semiconductors. Titanium dioxide is a promising candidate due to their tunable band gaps, ease of synthesis, and environmental stability. This investigation focuses on developing simple, scalable, and environmentally friendly synthetic strategies to produce TiO_2 nanomaterials with controlled size and shape. By optimizing their structural and optical properties, the study aims to improve solar energy conversion efficiencies, thus making solar energy more competitive and viable.

Keywords: Nanomaterials, electrode materials, titanium dioxide, XRD, Field emission scanning electron microscopy.

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INTRODUCTION

Solar energy stands as a pivotal cornerstone in the realm of renewable energy sources, boasting immense potential to meet global energy demands sustainably. As the primary source of energy for all life on Earth, solar power surpasses other renewable resources in its abundance and accessibility [1]. The sun delivers an enormous amount of energy to the Earth's surface annually, estimated to be nearly twice the amount that could ever be harnessed from all of the planet's non-renewable resources combined, including coal, oil, natural gas, and mined uranium [2]. These vast potential underscores the significance of solar energy in addressing the pressing challenges of climate change and the depletion of fossil fuels. Unlike non-renewable sources, solar energy is inexhaustible, environmentally benign, and capable of being harnessed in diverse geographical locations, making it an indispensable asset in the global transition towards sustainable energy systems [3].

The efficacy of solar energy alteration deeply relies on the materials used in solar cells, mainly the electrode materials. Semiconductors play a crucial role in this process, as they are responsible for absorbing sunlight and converting it into electrical energy through the photovoltaic effect [4]. Conventional solar panels predominantly utilize group IV semiconductors like silicon, which have been instrumental in the widespread adoption of solar technology. However, these materials face several challenges, including high production costs and efficiency limitations. The electricity generated from existing solar panels is approximately five times more expensive than conventional electricity from non-renewable sources [5]. This economic disparity highlights the urgent need for the development of new, cost-effective, and stable semiconducting materials that can enhance the efficiency and affordability of solar cells [6]. The exploration of alternative semiconductors such as group III-V and II-VI compounds, including indium phosphide, gallium arsenide, cadmium telluride, and zinc oxide, has shown promise, but the search for optimal materials continues to be a significant area of research [7].

The objective of this study is to investigate the synthesis and characterization of highly specific nanomaterials titanium dioxide which is considered potential candidate for cost-effective and stable electrode materials in solar cell applications [8]. These nanomaterials offer tunable band gaps and unique properties that make it suitable for enhancing solar energy conversion efficiencies. TiO_2 has been chosen for their promising attributes, such as ease of synthesis, environmental stability, and potential for large-scale production [9]. The research aims to develop simple, scalable, and environmentally friendly synthetic strategies to produce this nanomaterial with controlled size and shape.

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By optimizing their structural and optical properties, the study seeks to improve the performance of solar cells, making solar energy a more viable and competitive alternative to traditional energy sources [10]. Through a comprehensive analysis of these materials, the study will contribute to the advancement of solar technology, ultimately promoting a more sustainable and energy-secure future.

Metal chalcogenides are compounds formed between metals and chalcogens such as sulfur, selenium, and tellurium), have gained significant attention due to their unique electrical, optical, and catalytic properties [11]. These materials have wide-ranging applications in electronics, photonics, and renewable energy sectors, particularly in the development of high-efficiency solar cells and other optoelectronic devices [12]. TiO_2 is a widely studied metal oxide known for its excellent photocatalytic properties, high stability, and non-toxicity. The use of TiO_2 in dye-sensitized solar cells (DSSCs), demonstrating its potential in solar energy conversion. Recent advancements, such as those have focused on enhancing the efficiency of TiO_2-based solar cells by manipulating the nanostructure and doping with other elements [13].

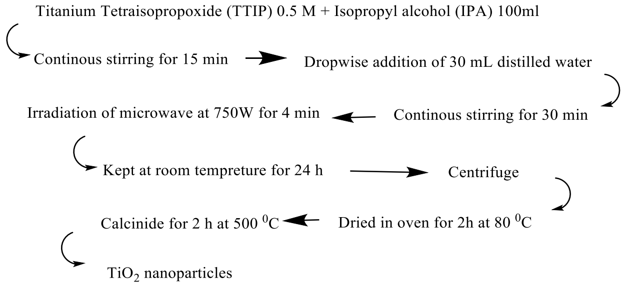
X-ray Diffraction is a powerful tool for determining the crystalline structure and phase composition of materials. It has been extensively used to characterize TiO_2 thin films and nanoparticles. For instance, the X-ray Diffraction studies to confirm the successful synthesis of highly crystalline TiO_2 nanorods [14].

Field emission scanning electron microscopy provides detailed images of the surface morphology and microstructure of materials. This study analyzes the morphology of TiO_2 thin films, revealing the uniformity and nanoscale features of the deposited layers [15]. UV-Visible Spectroscopy technique is used to study the optical properties and band gap of semiconductor materials. providing insights into their suitability for photovoltaic applications [16].

Methodology

The synthesis of nanomaterials involves a variety of techniques tailored to produce materials with specific properties suitable for solar energy applications. Sometime chemical bath deposition is employed to produce TiO_2 thin films. In this method, a substrate is immersed in a solution containing titanium precursors, where a controlled chemical reaction precipitates TiO_2 onto the substrate, forming a thin film. Both methods are effective in producing high-quality TiO_2 with desirable properties for solar cell applications. However electrochemical codeposition technique involves the simultaneous reduction of metal ions from an electrolyte solution onto a conductive substrate [17]. The electrochemical codeposition of TiO_2 demonstrating improved film quality and uniformity. Microwave-Assisted Synthesis method utilizes microwave radiation to heat the reaction mixture, leading to rapid and uniform synthesis of nanomaterials [18]. The microwave-assisted synthesis of TiO_2 nanoparticles, highlighting the advantages of this technique in terms of speed and energy efficiency [19]. The successful synthesis of TiO_2 nanoparticles using microwave irradiation, showcasing its applicability to a range of metal chalcogenides [20].

First of all, TiO_2 was synthesized by sol-gel method. This process involves the hydrolysis and polymerization of titanium alkoxides in a solution to form a gel-like network. Subsequent drying and calcination yield TiO_2 nanoparticles with high purity and controlled morphology. In the experimental work titanium isopropoxide (TTIP, 0.5 M) in isopropyl alcohol (IPA, 100 mL) was stirred with a magnetic stirrer for 15 min, then distilled water (30 mL) was added dropwise and the mixture was continuously stirred for 30 min. The mixture was exposed to microwave irradiation in 750 W for 4 min. Next, the mixture was left at room temperature for 24 h, then centrifuged. The mixture was dried in an oven at 80 °C for 12 h, and then calcined at 500 °C for 2 h. The flow chart of synthesis by sol-gel method is presented below:



Then we had tried hydrothermal method. In this method TiO_2 powder was used. The powder of TiO_2 micro particles was mixed with 10M NaOH. It is then treated hydrothermally at 150ºC in an autoclave for 24 hours. After hydrothermal reaction the sample was washed by 0.1M HCl solution and made the sample neutral at PH = 7. The white solid was then dried at 80ºC for 24 hours in vacuum. Finally, the powder was heated at 500ºC in a hot plate in static air for 2 hours to produce the TiO_2 nano particles.

TiO_2 thin films were also obtained by chemical bath deposition method. In this method peroxo-titanium precursor was first prepared in the aqueous acidic medium. This is done by mixing $TiOSO_4$ powder and H_2O_2 with constant stirring for 25 min to get a red clear solution. The obtained solution was kept in the bath at room temperature with constant stirring. The glass substrate was cleaned in an ultrasonic bath with acetone and ethanol solution, and then rinsed with by distilled water and finally purged with nitrogen gas. The glass substrates were immersed vertically in the solution. 12 h deposition time was used for getting better and uniform films. Then the substrate was withdrawn from the solution. The substrate with deposited film was rinsed with the double-distilled water and purged with nitrogen gas at room temperature.

The films were then dried at 100 °C for 1 h. Finally, the films are annealed at 500°C, 600°C and 700°C in air for 1h. It was found that annealing at 600°C produces best result.

It is also reported that electrochemical co-deposition is a powerful technique for creating thin films of various materials onto a conductive substrate. It leverages the magic of electricity to precisely control the deposition process. Let us delve deeper into the fundamental principles that govern electrochemical co-deposition process. We also tried this technique in our system. The experimental setup used for this purpose is shown schematically in Figure 1. This voltammetry arrangement consists of three electrodes. Titanium plates (M/S Titanium Equipment and Anode Manufacturing Co. Ltd. Madras) were used as working and counter electrodes. Saturated calomel electrode was used as reference electrode. The working and counter electrodes were cleaned and polished properly. The electrodes were finally washed with acetone and deionized water. The working, counter and reference electrodes were then dipped into electroplating solution, and a predefined fixed potential was applied across the working and reference electrode.

The potential used for the deposition of good quality of films is between −0.8 to −1.0 V in the electrolyte solution of 0.1 M $TiCl_4$. The pH was maintained at 2.0 by adding a few drops of 2 M Na_2CO_3. After the deposition, the electrodes were rinsed with double distilled water then dried in air at room temperature and finally the samples were heated at 500°C for 2 h under N_2 atmosphere.

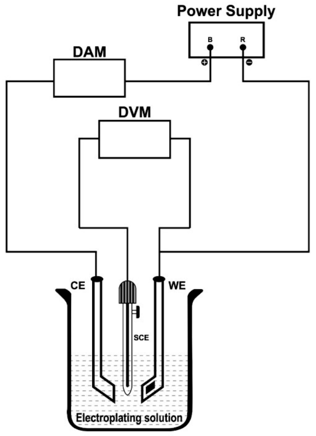


Figure 1: Experimental set up for electrochemical co-deposition of TiO_2 films.

W.E - Working Electrode; C.E - Counter Electrode; DVM - Digital Voltmeter; DAM - Digital Ammeter; SCE - Saturated Calomel Electrode.

Results and Discussion:

Although the synthesis of nanomaterials TiO_2 can be accomplished using multiple methods, each presenting distinct advantages and limitations but the sol-gel method is simpler. The sol-gel method produced nanoparticles with high purity and well-defined morphology, while chemical bath deposition method resulted in uniform thin films, although with slightly lower purity [21]. The hydrothermal method yielded highly crystalline nanoparticles, whereas electrochemical co-deposition provided excellent control over film thickness and composition. extracts were explored. Electrochemical codeposition method produced high-purity films with strong adhesion to substrates, and green synthesis offered an eco-friendly alternative with commendable purity and size control [22]. The yield and purity of the synthesized materials were critically assessed. The sol-gel method for TiO_2 achieved a yield of over 80-85% with high phase purity, while chemical bath deposition method offered slightly lower yields around 70-75% due to precursor losses. TiO_2 synthesized via the hydrothermal method showed a yield of 85-90% with excellent crystallinity, while electrochemical codeposition resulted in an 75-80% yield with high compositional accuracy. attributed to the variability in plant extract composition. Overall, these methods provided high-quality materials, though optimization is needed to balance yield and purity.

Table -1: Percentage yield of TiO_2 synthesized by various methods.

S.No.	Synthetic method	Percentage Yield
1	Chemical bath deposition method	70-75%
2	Sol-gel method	80-85%
3	Electrochemical codeposition method	75-80%
4	Hydrothermal method	85-90%

Although it is clear from Table-1 that TiO_2 synthesized via the hydrothermal method showed maximum yield, high purity and well-defined morphology is not obtained from this method. Sol-gel method also gives comparable yield. Further it leads to high purity and well-defined morphology. Therefore, we restrict our investigation on synthesis of TiO_2 by sol-gel method. We synthesized a number of samples of TiO_2 the sol-gel method. The synthesized TiO_2 nanomaterials were then subjected to comprehensive characterization to determine their structural, optical, and electrochemical properties, ensuring their suitability for solar energy conversion applications [23].

To study the photovoltaic performance of TiO_2 nanoparticle synthesized by sol–gel method photoelectric conversion efficiency, fill factor ratio etc were investigated. The photoelectric conversion efficiency was calculated by the following formula.

$$\eta\% = \frac{Jsc \times Voc \times FF}{Pin \times 100} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ (1)$$

Where FF is the fill factor ratio between the maximum output power density available (JmVm) and the maximum power combining short-circuit and open-circuit situations. It describes the extent of square of the J–V curve according to the equation

$$FF(\%) = \frac{Jm \times Vm}{Jsc \times Voc \times 100} \quad \ldots\ldots\ldots\ldots\ldots (2)$$

Where JSC, VOC, FF and Pin are the short-circuit current density, open-circuit voltage, fill factor and incident light power density, respectively. To calculate the energy conversion efficiency of the as-fabricated solar cells using the deposited film of TiO2 aggregates of different sizes, the measurement of the current versus voltage under AM 1.5 simulated sunlight conditions of 100 mW/cm^2 power density. The results are shown in Figure 8.

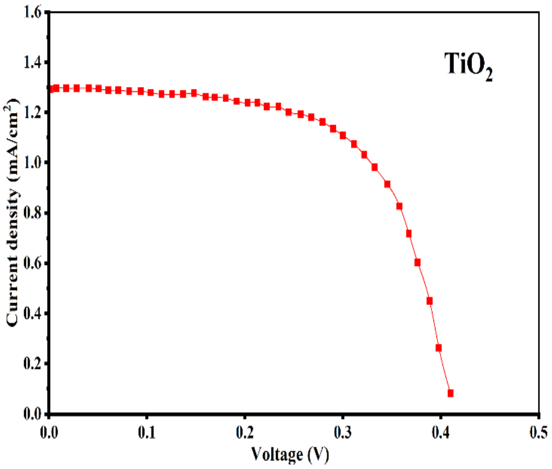


Figure 2: Photovoltaic performance of TiO$_2$ nanoparticles.

The short-circuit current density, open-circuit voltage, fill factor and incident light power density calculated from the curves are presented in Table-2. the VOC was controlled by the immersion time to the highest value and then decrease. When the film was made of TiO2 nanoparticle synthesized by sol-gel method, the efficiency was the highest at 64 % for VOC = 0.465 V, JSC = 1.361 mA/cm^2 and FF = 0.647.

Table- 2: Photovoltaic characteristics of TiO$_2$ nanoparticle synthesized by sol–gel method.

Sample	Open-circuit voltage (V)	Short-circuit current density (mA/cm^2)	Fill Factor	Efficiency (%)
Sol-gel method	0.465	1.361	0.647	64

UV-Visible spectroscopy is utilized to determine the optical properties of the synthesized nanomaterials, particularly the band gap. By measuring the absorption spectra, the optical band gap can be calculated, providing insights into the material's light-harvesting capabilities. Photoluminescence studies are conducted to investigate the emission properties and defect states within the nanomaterials. Photoluminescence spectra help understand the recombination mechanisms of charge carriers, which are critical for optimizing the efficiency of solar cells.

Photoluminescence measurements were performed with an excitation wavelength of 340 nm. Because the photoluminescence signal is contributed through recombination of the free charge carriers, we have utilized photoluminescence studies to understand the fate of the photoinduced charge carriers. The result is presented in Figure 2. An instant suppression of the emission of TiO$_2$ is noticed. Further Figure 3. shows that the photoluminescence peaks of the TiO$_2$ nanorods at 396 nm. From this analysis band gap is measured.

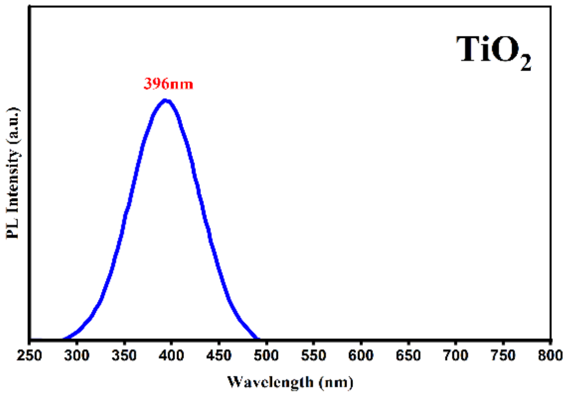


Figure 3: Photoluminescence spectra of TiO$_2$ nanoparticles.

The optical properties of the synthesized nanomaterials were evaluated through UV-Visible spectroscopy and photoluminescence studies. The band gap energies for TiO$_2$ were determined from the absorption spectra. TiO$_2$ showed a band gap of approximately 3.31 eV, consistent with literature values for anatase TiO$_2$. The band gap is obtained from Tauc's plot. The graph is presented in Figure 4.

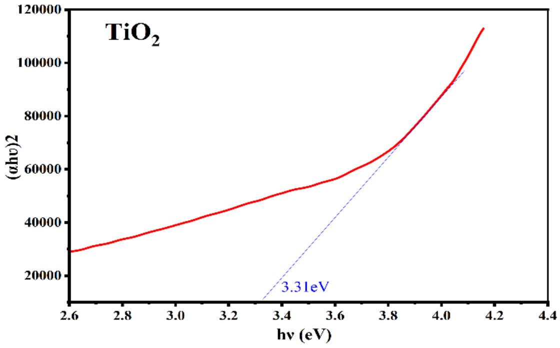


Figure 4: Band gap measurement by Tauc's plot.

The morphology of the synthesized TiO_2 was examined using field emission scanning electron microscopy. Such an image is presented in Figure 5. This figure shows that TiO_2 particles are uniform in size. The size of the particles was calculated and found to be about 45 nm. The field emission scanning electron microscopy image also shows that the grains are sufficiently larger and well connected uniformly distributed and is thick enough. However, the image exhibits porosity to some extent. The particles also seem to have agglomerated.

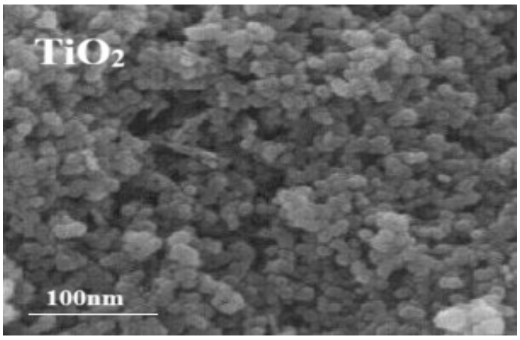
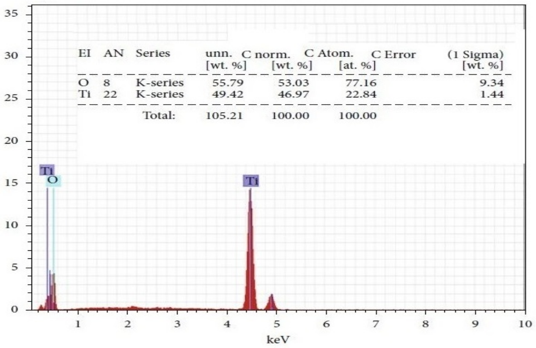


Figure 5: Field emission scanning electron microscopic study of TiO_2.



El	AN	Series	unn. C [wt. %]	norm. C [wt. %]	Atom. C [at. %]	Error (1 Sigma) [wt. %]
O	8	K-series	55.79	53.03	77.16	9.34
Ti	22	K-series	49.42	46.97	22.84	1.44
		Total:	105.21	100.00	100.00	

Figure 6: EDAX analysis of TiO_2 nanoparticles synthesized by sol–gel method.

The TiO_2 nanoparticles were analysed using EDAX analysis to identify their composition. The EDAX image of TiO_2 nanoparticle synthesized by sol–gel method is presented in Figure 6. Only Ti and oxygen (O) were shown to have peak values in Figure, with no additional peak values for any other metals. This confirms the formation of TiO_2 particles.

Structural analysis X-ray diffraction studies were investigated. The result is presented in Figure 7. This study revealed that the synthesized nanomaterials possessed the expected crystal structures and phases. X-ray diffraction patterns confirmed the formation of the anatase phase, which is known for its photocatalytic efficiency.

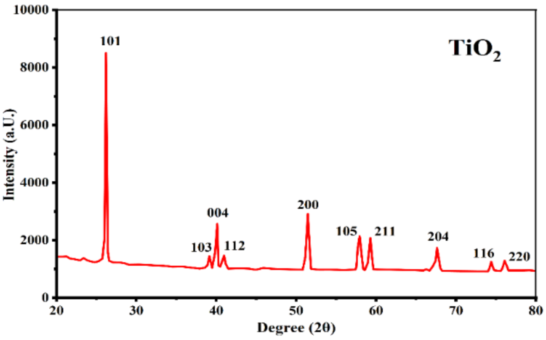


Figure 7: X-ray diffraction studies of TiO_2.

Cyclic voltammetry is performed to evaluate the electrochemical behavior of the nanomaterials. This measurement provides information on the redox activity, charge storage capacity, and reaction kinetics of the materials. Tafel plots are used to study the corrosion properties of the nanomaterials. By analyzing the Tafel curves, the corrosion rate and stability of the materials in various environments can be determined. Understanding the electrochemical properties is essential for assessing the long-term stability and performance of the nanomaterials in solar energy conversion devices**.** Through these synthesis and characterization techniques, TiO_2 nanomaterial is optimized for solar energy conversion, contributing to the development of cost-effective and efficient solar cells.

The integration of advanced synthesis methods and comprehensive characterization ensures that these materials meet the necessary performance criteria for sustainable energy applications. Stability and corrosion resistance were evaluated using cyclic voltammetry and Tafel plots. TiO_2 exhibited excellent stability, with minimal photocurrent degradation over multiple cycles and high resistance to corrosion. These results highlight the importance of stability in ensuring the longevity and reliability of solar materials**.**

The characterization technique Fourier transform infrared analysis was used to identify the functional groups in TiO_2 nanoparticles. This analysis is shown in figure 8. It can be observed the strong bond length between 580 to 660 cm $^{-1}$ to Ti-O stretching bands. This analysis concludes absorption spectra of TiO_2 nanoparticles is almost around 3426 cm^{-1} which specify the presence of -OH group with stretched bonds. There is not any peak at 2900 cm^{-1} which is indicated that all organic compounds are removed from the sample after calcification. The 1630 cm^{-1} absorption peak may be similarly to -OH (bending) it is indicated that the water as moisture is present in the sample. It is reported that the similar absorption peaks observed in the synthesis of TiO_2 nanoparticles**.**

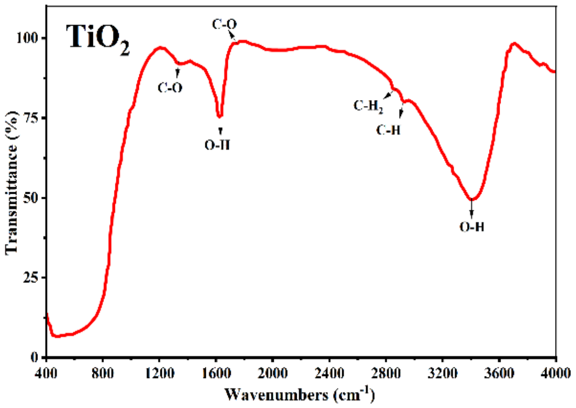


Figure 8: Fourier transform infrared spectrum of TiO₂ nanoparticles.

Raman spectroscopy ware employed to further characterize the synthesised TiO_2 nanoparticle. The spectra clearly confirmed the phase purity of anatase and rutile TiO_2, as shown in Figure 9. Both phases exhibit well-defined and distinct Raman fingerprints. Anatase possesses a tetragonal crystal structure, with its conventional unit cell consisting of two primitive cells, each containing two TiO_2 units. The Raman-active modes of anatase include $A_{1}g$ (516 cm^{-1}), two $B_{1}g$ modes (395 and 519 cm^{-1}), and three Eg modes (143, 196, and 653 cm^{-1}). In the present study, samples synthesized by sol–gel method displayed prominent Raman peaks at 146, 395, 517, and 649 cm^{-1}, confirming the presence of the TiO_2 nanoparticle**.**

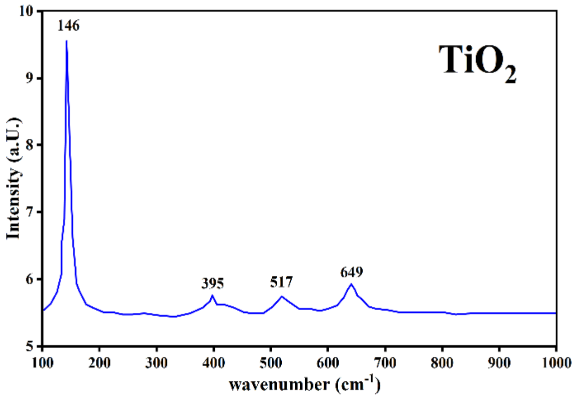


Figure 9: Raman spectra of TiO₂ nanoparticles.

Conclusion:

The TiO_2, synthesized via sol-gel method demonstrated sufficient yield, high purity and suitable crystal structures, leading to effective charge transport and stability in solar cells. The band gap energy was found to be 3.31 eV. The X-ray diffraction patterns in conjunction with Fourier transform infrared analysis and Raman spectral analysis confirmed the formation of the anatase phase of TiO_2. The grains were observed to be uniform in size.

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