



REGULAR ARTICLE

Effect of Sol-Gel Processing and Annealing on Nanocrystalline TiO₂ Thin Films for Photovoltaic Applications

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(Received 10 February 2026; revised manuscript received 19 April 2026; published online 29 April 2026)

Titanium dioxide (TiO₂) thin films were synthesized using a sol-gel spin-coating approach and evaluated for their potential use as photoanodes in dye-sensitized solar cells. A stable precursor sol was prepared by dispersing commercial P-25 TiO₂ powder in ethanol, with acetyl acetone employed as a stabilizer and p-hydroxybenzoic acid acting as a chelating agent to improve dispersion and film uniformity. Following deposition, the films were thermally treated at 400 °C to promote crystallization and enhance porous structure formation. X-ray diffraction analysis confirmed the development of monocrystalline TiO₂ with a predominant anatase phase and tetragonal crystal symmetry, with an average crystallite size of approximately 26 nm. Optical characterization carried out using UV-Visible spectroscopy revealed strong ultraviolet light absorption and high transparency in the visible region. The absorption edge observed in the range of 390-430 nm corresponds to an optical band gap of about 3.1 eV. The obtained structural and optical properties indicate improved light-harvesting capability and favorable surface characteristics. These findings demonstrate that the optimized sol-gel synthesis and annealing conditions produce TiO₂ thin films suitable for photovoltaic and photocatalytic applications.

Keywords: TiO₂, Band Gap, DSSC, Sol-Gel method, Spin coating method, XRD, UV-visible, Annealing.

DOI: [10.21272/jnep.18\(2\).02033](https://doi.org/10.21272/jnep.18(2).02033)

PACS numbers: 81.20.Fw, 81.15.Rs

1. INTRODUCTION

Globally, almost 1.3 billion people do not have access to electricity, despite the fact that it is a basic human necessity and a need for national development [1]. One of the inventive and environment friendly method of producing electricity is solar energy. A solar cell that converts electricity from solar energy, and this phenomenon is called as Photovoltaic effect. Titanium dioxide (TiO₂) has recently gained significant attention as a highly researched metal oxide in the photovoltaic process, noted for its chemical stability, transparency, high catalytic properties, affordability, and durability, TiO₂ functions as a wide band gap semiconductor, exhibiting photo-conversion properties within the UV spectrum range, which enables a variety of practical applications [2].

Grazel and O'Regan documented the initial studies on dye-sensitized solar cells (DSSC) utilizing nano-sized TiO₂ film electrodes. DSSCs are comparably inexpensive and possess a straightforward design comparing to other solar devices [3]. A DSSC is composed of four primary components: a photoanode, an electrolyte, a counter electrode, and a photo-sensitizer dye. A light energy is converted into electrical energy through the photo-sensitization of dye is referred to as a photochemical device. When DSSC is exposed to solar light, the dye's electrons become excited and move into the photoanode metal oxide's conduction band before diffusing to the counter electrode. The TiO₂ nanocrystal line meso films

used as photo anodes demonstrate a power conversion efficiency (PCE) ranging from 7% to 12% DSSCs. Under typical global simulated sunlight, a team under the direction of Michael Gratzel obtained a 15.2% power conversion efficiency, reaching a noteworthy milestone with 500 hours of operational stability. The power conversion efficiency (PCE) of DSSC under ambient light intensities is 30.2%, demonstrating high stability [4].

According to Reyes-Coronado et al., the common crystalline forms of TiO₂ are: rutile (tetragonal), anatase (tetragonal), and brookite (orthorhombic) [5]. Sun et al [6] explain how the periodic configurations of deformed TiO₆ octahedral units define the composition of various crystalline phases. Brookite is seldom reported as a photocatalyst due to challenges in synthesis, leading to rutile and anatase being the more frequently encountered Phases of crystalline TiO₂. With predicted bandgap energies E_g of 3.0 eV for rutile and 3.2 eV for anatase at ambient temperature, TiO₂ is mostly active in the ultraviolet light spectrum.

Anatase exhibits better photocatalytic activity than rutile despite having a comparatively greater bandgap, which is linked to its superior ability to adsorb hydroxyl groups. The effective mass of anatase is lower than that of rutile (m_o stands for electron mass). Photoexcited charge this characteristic allows carriers to move and transfer from the interior structure to the surface, boosting their participation in photocatalytic activities. According to Zhang et al., this might cause the photo-

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generated carriers in anatase to recombine at a slower pace [7]. Anatase's photocatalytic activity has been observed to increase for layers as thick as 5.0 nm, at 2.5 nm, the rutile variety reaches its maximum activity [8]. This indicates that charge carriers stimulated at deeper levels within the bulk exert a more significant influence on surface reactions in anatase than in rutile.

Various techniques and substances may be employed to deposit solar cells. Nonetheless, TiO₂ stands out as a widely utilized material, and spin coating presents significant potential. A variety of techniques have been developed and effectively utilized for thin film deposition. These encompass techniques like Spray pyrolysis, Sputtering, Screen printing, electroplating, Dip-coating, Chemical vapor deposition, thermal evaporation, pulsed laser deposition, atomic layer deposition and chemical bath deposition [9] various methods exist for thin-film fabrication, yet the spin coating technique is particularly notable for its simplicity, easily controllable, and cost-effective procedure that is widely utilized [10]. This method is currently widely utilized in the microelectronics sector to apply a consistent layer of photosensitive and photolithographic film onto a silicon substrate [11]. A key aspect of spin coating technology that enhances its appeal is its reproducibility. Significant differences in the coated film can result from small changes to the parameters controlling the spin process [12]. In this report, a cost effective and efficient spin coating techniques is employed to synthesized TiO₂ thin film for photovoltaic application.

2. METHODOLOGY

2.1 Substrate Cleaning

A Glass substrate used for deposition. Fig. 1 illustrates the process used for cleaning the glass substrate (of dimension 20 mm × 25 mm × 2 mm). The surface of the substrate was meticulously wiped to eliminate any residues and impurities, followed by a thorough wash with detergent. The samples undergo additional treatment with deionized water, acetone, and ethanol for 15 minutes each, followed by ultrasonication for 40 minutes at a temperature of 95 °C and dry under IR light for 15 minutes [13]. Effective substrate cleaning is essential to enhance material-to-substrate adhesion and to prevent contamination.



Fig. 1 – The procedure employed for substrate cleaning

2.2 Materials

The Commercial TiO₂ powder P25 (Degussa AG, Germany), consisting of mixed phases of 80 % anatase and 20% rutile, was obtained from Degussa, Germany, acetyl acetone (Sigma Aldrich), p-hydroxybenzoic acid, and ethanol absolute. Every chemical utilized was of analytical reagent (AR) quality and didn't require any additional purification.

2.3 Preparation of the TiO₂ Precursor Solution

TiO₂ thin coatings were produced on glass substrates using sol-gel spin coating. In this P-25 synthesis, TiO₂ sourced from Degussa, Germany, serves as the precursor, while ethanol and acetyl acetone function as solvents, with p-hydroxybenzoic acid acting as the catalyst. The TiO₂ layer was deposited using the spin coating process, which necessitated the preparation of a TiO₂ gel. 0.5 gm of P-25 (Degussa P-25), 4.5 ml of ethanol, 0.8 ml of acetyl acetone, and 1 gm of p-hydroxybenzoic acid were combined to create this gel. The resulting mixture was continuously stirred for 1 hour at 1000 revolution per minute (r.p.m) at room temperature and subsequently subjected to ultra-sonication at a frequency of 20 kHz for 1 hour while maintained in an ice bath. Later 1 ml of the prepared TiO₂ gel was applied once to the center of the glass substrate. The substrate underwent acceleration at 500-1000 rpm for a duration of 1 minute, leading to the development of a consistent film. The synthesized films were subjected to annealing at 400 °C for one hour, resulting in the development of a TiO₂ thin film. Fig. 2 illustrates the comprehensive process for fabricating TiO₂ thin films.

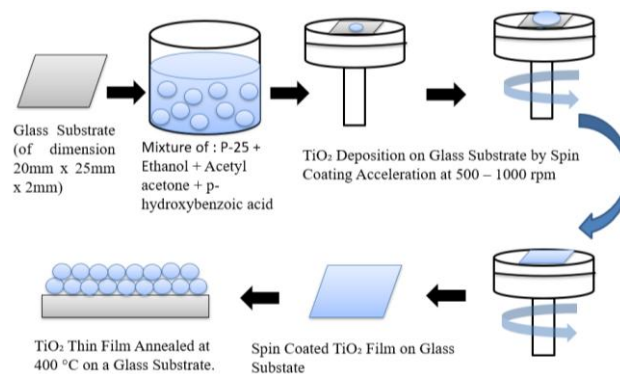


Fig. 2 – A Schematic of the steps used to make a TiO₂ thin film

3. CHARACTERIZATION OF TiO₂

The as-synthesized sample underwent characterization through a range of techniques. The phase composition and structure of the coatings were examined by X-ray diffraction (XRD) using an automated Bruker D8 Advance X-ray diffractometer, employing CuK α radiation ($\theta = 1.541 \text{ \AA}$) over a 2θ range of 10° to 80° . S-4800 Type-II (HITACHI HIGH TECHNOLOGY CORPORATION, Tokyo, Japan). A UV-VIS spectrophotometer (V-630) was used to acquire absorbance spectra in the 370-900 nm range to examine the optical characteristics of the film.

4. RESULTS AND DISCUSSION

4.1 X-ray Diffraction

The XRD pattern of the as-synthesized sample generated on a glass substrate is shown in Fig. 3, clearly revealing its polycrystalline features. The most pronounced peak observed at $2\theta \approx 25.26^\circ$, attributed to the (101) plane, which is the distinctive peak of the anatase phase exhibiting a tetragonal TiO₂ crystal structure [JCPDS card no. 84-1286]. In addition, several low-intensity peaks at $2\theta \approx 37.87^\circ$, 48.15° , 54.54° , and 55.06° are identified, corresponding to reflections from the

(004), (200), (105), and (211) planes of TiO₂, respectively.

The application of Scherer's formula has facilitated the determination of crystallite size (D) [14] for TiO₂ at a 25.26° angle in the films.

$$D = \frac{k\lambda}{\beta \cos \theta} \quad (1)$$

In this context, k is a constant valued at 0.94 [15], λ represents the wavelength of the X-ray, β denotes the full width at half maximum (FWHM) of the peak measured in radians, and θ signifies Bragg's angle. The derived equation suggests that the crystallite size of TiO₂ is roughly 26 nm.

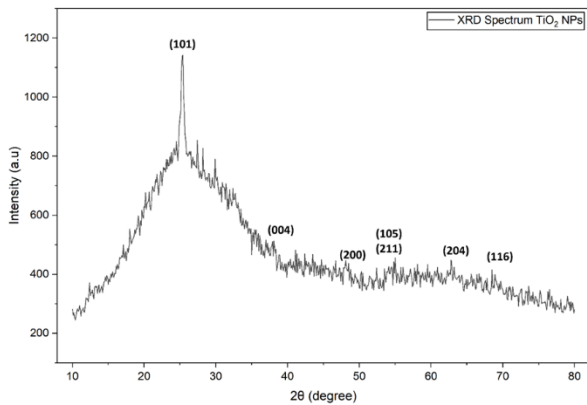


Fig. 3 – XRD diffraction pattern of TiO₂ placed on a glass substrate

4.2 UV-Vis Spectroscopy

Figs. 4(a) and (b) presents a detailed examination of the optical characteristics exhibited by the TiO₂ thin films, which underwent a spin-coating process followed by annealing at a temperature of 400 °C. The Tauc plot of $(\alpha h\nu)^2$ versus incident photon energy ($h\nu$) and its absorption and spectrum, Tauc and David Mott's relation was used to illustrate the optical bandgap of the produced TiO₂ thin films that were annealed at 400 °C [16] as shown in Eq. (2).

$$(\alpha h\nu)^n = K \cdot (h\nu - E_g) \quad (2)$$

In this context, the absorption coefficient as defined by Beer-Lambert's law is represented by α , while the incident photon's energy is denoted as $h\nu$. K is a constant that doesn't depend on energy, while E_g denotes the optical bandgap value of the TiO₂ thin films. However, for direct permitted change, a value n depends on the type of transition ($n = 2$). The analysis of Tauc's wavelength in relation to absorbance demonstrated that the TiO₂ thin films show considerable absorption within the visible light spectrum. As seen in Fig. 4(b), the produced thin layer absorbed UV radiation at wavelengths of 400 nm and less. Other authors have published such absorption spectrums. High absorbance is a defining feature of anatase TiO₂, enhancing energy conversion efficiency as increased energy results in greater photon excitation.

The bandgap was determined from the linear relationship observed in the graph of $(\alpha h\nu)^2$ against photon energy ($h\nu$). The evaluation required extending a

straight line from the linear part of the curve to meet the x-axis, as seen in Fig. 4(b). The coated TiO₂ thin film's direct bandgap was measured and found to be 3.1 eV. The value is in closer alignment with the reported optimal range of 3.0 to 3.2 eV, which is characteristic of the anatase and rutile phases [17, 18]. The proximity of the acquired bandgap value to the ideal range is advantageous, as optimal values facilitate the swift functionalization of materials upon exposure to UV irradiation. Elevated calcination temperatures, specifically those reaching 400 °C or beyond, have demonstrated a capacity to augment the bandgap of TiO₂ [19]. Annealing enhances the thin film's crystalline structures and helps eliminate internal tensions that develop within the coatings during the thin film deposition process. The enhancement of the crystal structure will lead to further optimization of the material's bandgap.

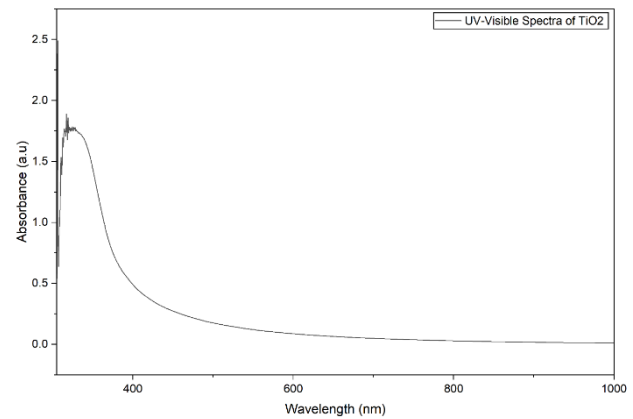


Fig. 4 (a) – Absorbance with respect to wavelength for TiO₂ thin film

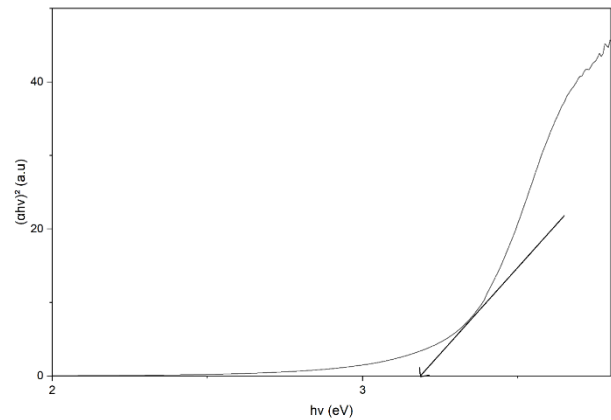


Fig. 4 (b) – Energy band gap calculation by Tauc and baseline approach. Direct Tauc Plot $(\alpha h\nu)^2$ vs. Photon Energy

5. CONCLUSION

The present investigation establishes a reproducible and cost-effective sol-gel spin-coating method for fabricating nanostructured TiO₂ thin films suitable for DSSC photoanode applications. The synthesized films exhibited a polycrystalline anatase structure with nanoscale crystallites that enhance electron transport and suppress charge recombination. The optical studies confirmed a direct allowed transition with a band gap of approximately 3.1 eV, consistent with high photocatalytic activity under UV illumination. The synergy

between nanoscale morphology and crystalline phase purity is critical for optimizing light absorption and electron mobility in photovoltaic devices. Overall, the developed TiO₂ films demonstrate significant potential for integration into efficient, stable, optoelectronic systems and low-cost DSSCs.

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ACKNOWLEDGEMENTS

The authors of this work declared that they have no conflict of interest.

Вплив золь-гель обробки та відпаду на нанокристалічні тонкі плівки TiO₂ для фотоелектричних застосувань

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Тонкі плівки діоксиду титану (TiO₂) були синтезовані з використанням методу золь-гель спінового покриття та оцінені для їх потенційного використання як фотоанодів у сонячних елементах, сенсibiliзованих барвником. Стабільний золь-попередник був отриманий шляхом диспергування комерційного порошку P-25 TiO₂ в етанолі, з використанням ацетилацетону як стабілізатора та п-гідроксибензойної кислоти як хелатного агента для покращення дисперсії та однорідності плівки. Після осадження плівки були термічно оброблені при 400 °C для стимулювання кристалізації та посилення формування пористої структури. Рентгенівський дифракційний аналіз підтвердив розвиток монокристалічного TiO₂ з переважною фазою анатазу та тетрагональною кристалічною симетрією, із середнім розміром кристалітів приблизно 26 нм. Оптична характеристика, проведена за допомогою УФ-видимої спектроскопії, виявила сильне поглинання ультрафіолетового світла та високу прозорість у видимій області. Край поглинання, що спостерігається в діапазоні 390-430 нм, відповідає оптичній забороненій зоні близько 3,1 еВ. Отримані структурні та оптичні властивості вказують на покращену здатність до збирання світла та сприятливі характеристики поверхні. Ці результати демонструють, що оптимізовані умови золь-гель синтезу та відпаду дозволяють отримати тонкі плівки TiO₂, придатні для фотоелектричних та фотокаталітичних застосувань.

Ключові слова: TiO₂, Ширина забороненої зони, DSSC, Золь-гель метод, Метод спінового покриття, Рентгенівська дифракція, УФ-видиме випромінювання, Відпал.