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SYNTHESIS OF NB-DOPED NANOPARTICLES VIA THE SOLVOTHERMAL ROUTE AND THEIR APPLICATION IN DYE-SENSITIZED SOLAR CELLS

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Abstract:-*In this study, niobium (Nb)-doped TiO2 nanoparticles, synthesized via the solvothermal method, were used to fabricate TiO2-based dye-sensitized solar cells (DSSCs), employing an extract from the fruit of *E. conferta* as a photosensitizer and ethanol as a solvent. The synthesized semiconductor metal oxide nanoparticles were characterized using X-ray diffraction (XRD), field-emission scanning electron microscopy (FESEM), BET analysis, and UV-Visible spectroscopy. The photovoltaic performance of the cells was analyzed, including current density, voltage, and fill factor. The results demonstrate an improvement in cell performance, indicating that the solvothermal method is a suitable fabrication technique, characterized by its simplicity and a shorter processing time compared to sol-gel or solid-state methods. Furthermore, the solvothermal method helps reduce recombination phenomena, leading to superior performance compared to that achieved through other methods.*

Key Words: Solvothermal method, Nb-doped TiO2, *E. conferta*, phase analysis, photovoltaic parameters

INTRODUCTION

The constant growth of the global population makes the development of clean and affordable energy sources increasingly essential. In an effort to reduce energy consumption, researchers and technologists are striving to meet energy needs by designing efficient, low-cost devices [1]. Recently, solar energy harvesting via photovoltaic technologies has garnered significant interest due to its low cost, high performance, and durability [2-3]. Dye-Sensitized Solar Cells (DSSCs) which represent the third generation of photovoltaic cells have particularly captured the attention of researchers over the last few decades. Thanks to the use of inexpensive materials, their transparency, and their ease of fabrication, they are especially well-suited for energy development when compared to earlier generations [4]. The photoanode of a DSSC consists of a transparent conductive oxide substrate, coated with wide-bandgap materials and impregnated with a photosensitive dye [5]. Titanium dioxide (TiO_2) is the preferred semiconductor due to its excellent optical properties and high photostability. Various approaches have been explored to modify the structure of the materials that make up the photoanode in order to optimize the performance of DSSCs; one such approach is the doping of TiO_2 with metal ions. Consequently, dopants such as niobium, vanadium, iron, copper, and zinc have been incorporated into TiO_2 to develop the photoanodes for these cells [6-11].

In this study, niobium (Nb) doping is applied to TiO_2 nanoparticles, as niobium is considered one of the most promising metal dopants for enhancing optical properties. Doping the TiO_2 semiconductor in DSSCs offers three main advantages: it reduces the bandgap, which in turn increases performance; ...decreases the photocatalytic activity of the cell and minimizes charge recombination. The use of metals with a valence higher than that of Ti^{4+}, such as Nb^{5+}, provides several benefits: an increase in the number of free charge carriers, improved electrical conductivity, optimized charge-transfer kinetics, and a modification of the dye-absorption properties within the TiO_2. Previous studies on niobium (Nb)-doped TiO_2, prepared via the co-hydrolysis method, have demonstrated an improvement in electron injection and transport. This phenomenon is attributed to higher electrical conductivity, as well as a shift in the Fermi level (E_F) and the conduction band minimum (CBM) [12-13].

However, it is difficult to achieve high crystallinity and a uniform particle size using the co-hydrolysis method; furthermore, this process often introduces structural defects into the TiO_2, which impairs electron injection and transport [14]. Doping via co-hydrolysis frequently results in deep energy levels for the dopant, which are ineffective for electron injection and transport. Although the photovoltaic performance of niobium (Nb)-doped TiO_2 photoanodes has been extensively researched, their preparation via the solvothermal method has not yet been documented. Nb-doped TiO_2 synthesized via the solvothermal route offers significant advantages in terms of morphological control, particle dispersion, and increased specific surface area, compared to materials prepared using sol-gel or solid-state methods. In solvothermal synthesis, high-pressure and moderate-temperature conditions promote uniform nucleation and crystallite growth, resulting in smaller crystallites, greater homogeneity, and an increased specific surface area. Improved dye adsorption and charge transport contribute to better performance in dye-sensitized solar cells (DSSCs).

While the sol-gel technique allows for a more uniform nanostructure, the calcination step can reduce the specific surface area, promote grain growth, and alter the properties of the cell [15]. Crystallite sizes obtained via the sol-gel method typically range from 15 to 30 nm, while the specific surface area tends to decrease due to particle agglomeration [16]. The solid-state method, which involves dry mixing at high temperatures, results in larger crystallites and particle agglomeration; this leads to a lower specific surface area and a less homogeneous dispersion of Nb[17]. The solvothermal method allows for the control of light absorption through the effective dispersion of Nb within the TiO_2 crystal lattice. The advantages of this approach include more efficient charge transport, reduced recombination, and an enhanced capacity for light harvesting.

In this study, we synthesized Nb-doped TiO_2 photoanodes using the solvothermal method, employing *E. conferta* as a source of natural dye (sensitizer). *E. conferta* was selected due to its high content of anthocyanin pigments, which facilitate the conversion of photons into electrical energy [18]. The use of the solvothermal method eliminates the need for steps such as pH adjustment, rigorous temperature control, and the preparation of complex chemical mixtures during the fabrication process, while simultaneously reducing the time required for the synthesis of Nb-doped TiO_2. These advantages promote more efficient electron injection and transport, resulting in improved photovoltaic performance.

EXPERIMENTAL METHODS

Nb Doped TiO2 Nanoparticles Synthesis

For the synthesis of undoped TiO2 via the solvothermal method, 6 mL of titanium isopropoxide was mixed with 12 mL of acetic acid. Subsequently, 12 mL of ethanol was added to the mixture, which was then stirred for 2 hours. The solution was placed in a Teflon-lined autoclave and heated at 200 °C for 90 minutes. After cooling to room temperature, the product was filtered, washed with ethanol, and then dried at 80 °C for several hours. Separately, niobium-doped TiO2 was prepared following the same protocol, with the addition of NbCl5 and continuous stirring for 30 minutes. Different niobium doping levels 0, 0.5, 1.0, and 1.5 wt% were tested using this same procedure.

Natural Dye Preparation

In this study, the fruits of *E. conferta* were used as a natural colorant. The pulp and seeds were separated and subsequently dried at room temperature. The dried pulp was ground in a mortar until a fine powder was obtained. Fifty grams (50 g) of this powder were placed in a beaker, mixed with 500 ml of ethanol (at a 1:10 ratio), and stirred thoroughly; the mixture was then left to stand for 24 hours, protected from light. Afterward, the mixture was filtered to remove residues, yielding a natural colorant solution that was purified in a single step. Further details regarding this natural colorant can be found in our previous research [19].

DSSC Assembly

An FTO glass substrate (7 Ω/cm^2) was cleaned with ethanol before applying a niobium (Nb)-doped TiO_2 paste. The paste was prepared by mixing 0.3 g of TiO_2 with 0.5 mL of acetic acid and a 1:1 mixture of deionized water and ethanol; the resulting mixture was stirred for 20 minutes. During this stirring process, 0.5 mL of Triton X was added to improve the cohesion of the paste intended for the photoanode. The TiO_2 paste was spread onto the FTO glass using the "doctor blade" technique, and the resulting films were subsequently sintered at 450 °C for 30 minutes. Following this same protocol, Nb-doped TiO_2 films with doping concentrations of 0.5%, 1.0%, and 1.5% by mass were prepared and left immersed for 24 hours. After the sensitization stage, the electrodes were rinsed with ethanol to remove any unbound dye. The counter electrode was fabricated according to procedures described in previous studies [20]. A redox electrolyte was introduced between the photoanode and the counter electrode, ensuring that it adequately covered the active areas of both electrodes. Finally, the cell was sealed using a specific adhesive tape; the active surface area of the cell is 7 cm^2.

RESULTS AND DISCUSSION

In this section, properties related to the crystal structure specifically lattice strain and crystallite size have been examined. Visible light absorption, which allows for the assessment of photon absorption efficiency, was studied using UV-Vis spectroscopy. Additionally, scanning electron microscopy (SEM) was employed to characterize the morphology of the samples, revealing their polycrystalline nature. Electrochemical impedance spectroscopy (EIS) was used to analyze recombination phenomena; finally, the photovoltaic performance of niobium-doped TiO2, synthesized via a solvothermal method, was evaluated.

XRD Analysis

Figure 1 presents the results obtained for pure TiO_2 nanoparticles and niobium (Nb)-doped TiO_2 nanoparticles. The observed diffraction peaks correspond to reflections of the anatase phase and are consistent with JCPDS card no. 98-004-5316. As

illustrated in Figure 4.10(a), distortions in the crystal lattice induced by defects such as vacancies, interstitial atoms, or substitutions can cause a shift in the peaks, reflecting the presence of stresses (whether tensile or otherwise) within the material. To evaluate variations in the crystal lattice parameters, the (101) peak, located at 25.3° for the Nb-doped TiO_2 (see Figure 1(b)), was analyzed. The magnified view in Figure 1(b) reveals shifts in peak positions, as well as changes in peak broadening. At a concentration of 0.5 wt%, the peaks shift toward the right, indicating that niobium has been incorporated into the TiO_2 lattice, thereby altering its structure. Despite this shift, the anatase structure remains intact, and X-ray diffraction (XRD) results show no evidence of any separate phase associated with the dopant. Since the ionic radius of Nb^{5+} (0.78 Å) is significantly larger than that of Ti^{4+} (0.75 Å), the crystal lattice becomes distorted; furthermore, an increase in crystallite size is observed, as shown in Table 1 [21]. Nb^{5+} preferentially occupies octahedral interstitial sites, a configuration that is energetically favorable. The incorporation of Nb^{5+} is advantageous, as it allows for the supply of electrons to TiO_2, thereby reinforcing its n-type semiconductor characteristics. Consequently, the use of a substituent element that possesses a greater number of valence electrons than Ti^{4+} such as Nb^{5+} serves as an effective strategy for modifying the electronic properties of TiO_2.

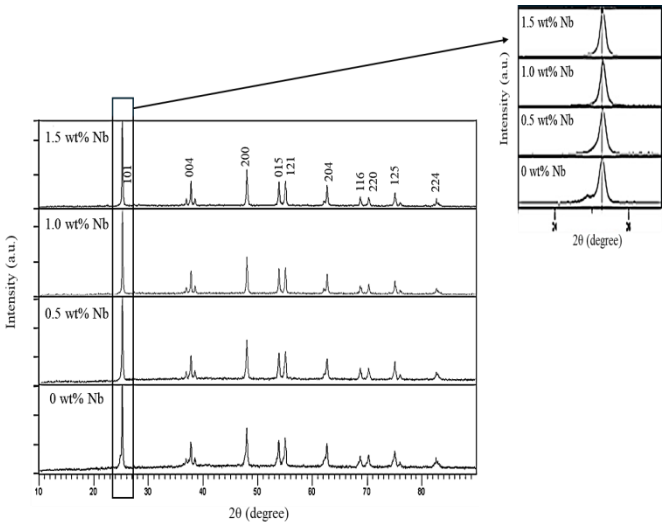

Figure 1. (a) Pure oiO2 and different amounts of Nb (b) an enlarged view of the (101) peaks for undoped TiO_2 and Nb-doped TiO_2.

The influence of dopants on lattice parameters is presented in Table 1. As indicated in the table, Nb-doping leads to an increase in the length of Ti–O bonds; this is explained by an increase in the unit cell volume, given that the Nb–O bond is longer than the Ti–O bond. The result is an expansion of the unit cell volume in the doped TiO_2, accompanied by the emergence of a local stress field at the sites occupied by the dopant, which modifies atomic interactions and induces distortion in the crystal lattice [22-23]. Consequently, significant variations in crystal parameters are observed as the Nb content increases, as confirmed by analyses of the lattice parameters and crystallite size. Furthermore, a decrease in the full width at half maximum (FWHM) of the peaks in the X-ray diffraction (XRD) patterns is observed as the Nb concentration increases, which is attributed to an increase in crystallite size. This phenomenon stems from the expansion of the Nb-doped TiO_2 samples, caused by the insertion of Nb^{5+} ions into interstitial positions within the TiO_2 crystal lattice [24]. The lattice parameters (a and c), as well as the unit cell volume, increase with Nb content. Consequently, a higher Nb content leads to notable modifications in the crystal parameters as evidenced by the analysis of lattice parameters and crystallite size which, in turn, influences the performance of dye-sensitized solar cells (DSSCs).

Table 1 Analysis based on peak (101) for pure and doped TiO2

Sample	Crystallitesize(nm)	(Å3)	a (Å)	c (Å)
PureTiO2	9.00	140	3.80	10.50
0.5NbdopedTiO2	9.16	146	3.81	10.51
1.0NbdopedTiO2	12.00	146.3	3.81	10.51
1.5NbdopedTiO2	12.10	146.4	3.85	10.52

Compared to other synthesis methods such as sol-gel, hydrothermal, or solid-state reaction processes the solvothermal method allows for the production of small-sized crystallites (between 8 and 11 nm), in contrast to the other methods (20–25 nm for sol-gel and solid-state reaction processes, or even greater than 30 nm) [25–26]. Thus, precise control over crystallite size facilitates better electron diffusion.

UV-VIS Analysis

To study the behavior and understand the characteristics of the photoanode, an optical analysis was conducted, as illustrated in Figure 2. It is observed that the range between 350 and 420 nm exhibits the highest photon absorbance for the given Nb concentrations. The absorption wavelength increases with the concentration of the Nb dopant up to 1.0 wt%; this phenomenon is attributed to the formation of new conduction bands or the creation of new energy levels within the TiO_2 structure [27]. Conversely, an increase in the Nb concentration to 1.5 wt% leads to a decrease in the optical absorption wavelength. Higher energy

values indicate that Nb has been incorporated via substitution into the TiO_2 crystal lattice, causing a shift in the band gap. This demonstrates that the introduced dopants can modify the optical band gap energy. The improvement in absorption can be attributed to the emergence of a new energy level between the conduction and valence bands, which leads to a slight reduction in the band gap and, consequently, a "redshift" in light absorption. The decrease in absorbance at anNb concentration of 1.5 wt% is attributed to a deterioration in crystalline quality, which increases photon scattering a finding confirmed by X-ray diffraction (XRD) results [28]. This phenomenon is visible in Figure 1, where the broadening of the XRD peaks indicates increased strain within the crystal lattice. Degraded crystallinity reduces the mean free paths of both photons and electrons, which can affect both electrical and optical properties. With crystallinity degraded at 1.5 wt% Nb, optical absorbance decreases due to an increase in the number of recombination centers, which reduces the quantity of photogenerated charge carriers.

$$\alpha hv = A(hv - E_{gg})^n \qquad (1)$$

According to Equation 1, the optical band gap of Nb-doped TiO2 can be determined using a Tauc plot.

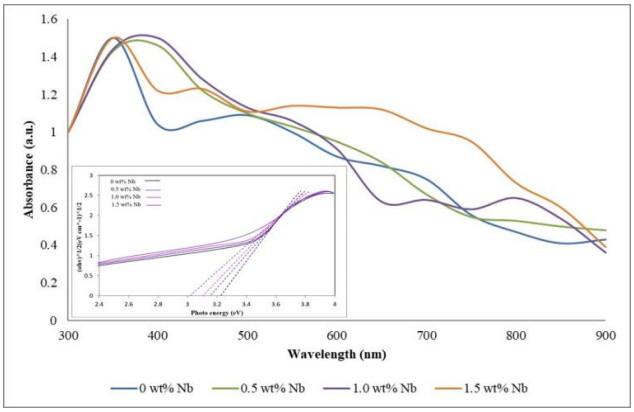

Figure2. Analysis of UV vis photo absorbance for pure and doped TiO2

The band gap width decreases as the dopant concentration increases. In particular, it is possible to precisely tune this band gap to a desired value by selecting an appropriate concentration of transition metal ions within the TiO_2 matrix; this allows the optical absorption threshold to be shifted from the ultraviolet toward the visible region [29]. The reduction of the band gap leads to the creation of new electronic states within the material. This decrease facilitates electron transport and improves the performance of niobium (Nb)-doped TiO_2 in dye-sensitized solar cells (DSSCs).

The solvothermal method enables better incorporation of niobium and greater optical absorption in Nb-doped TiO_2 compared to sol-gel or solid-state methods [30–31]. Niobium doping via the solvothermal route promotes light absorption by narrowing the band gap in contrast to sol-gel and solid-state methods, which tend to widen the band gap, thereby reducing the efficiency of electron transport.

FESEM Analysis

Figure 3 shows the surface morphology of the samples obtained via solvothermal synthesis. In samples containing 0 wt% and 0.5 wt% Nb, slight agglomerates are observed. In contrast, samples with 1.0 wt% and 1.5 wt% Nb exhibit dense, continuous particles with low porosity. At 1.0 wt% and 1.5 wt% Nb, the surface tends to become denser and smoother than in the samples with 0 wt% and 0.5 wt% Nb. The presence of pores facilitates better dye penetration into the photoanode, as well as more efficient electron injection [32]. Penetration into the pores decreases when particles are smaller, due to the reduced spacing between them. This phenomenon can hinder electron transport from the electrolyte to the photoanode, promote the formation of recombination centers, and reduce cell efficiency.

Particle size and porosity were measured using ImageJ software. As shown in Table 2, the grain size decreases from 131.3 nm to 126 nm as the Nb content increases from 0 wt% to 1.0 wt%. Subsequently, it increases again to 128.5 nm at anNb content of 1.5 wt%. A similar trend is observed regarding porosity: an increase in particle size leads to a decrease in porosity.

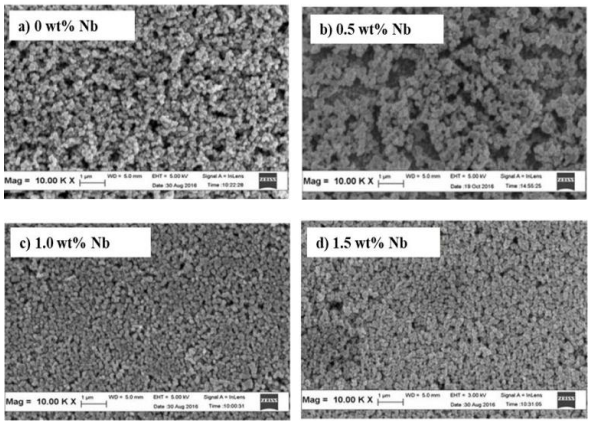

Figure 3. FESEM images of pure and doped TiO_2 at varying Nb concentrations.

This phenomenon is attributed to the formation of new nucleation centers, which reinforce the process of heterogeneous nucleation at preferential sites, such as

phase interfaces or impurities. Dopant atoms hinder the growth of TiO_2 crystals through their interaction with Ti^{4+} ions, thereby slowing down crystal growth [33]. Higher niobium (Nb) doping leads to an increase in particle size due to agglomeration as well as a decrease in porosity. The doping of TiO_2 with large cations, such as niobium, can be explained by the fact that if these bulky dopants replace Ti ions, the segregated TiO_2 may form a liquid phase. This liquid phase, in turn, can promote grain growth during the subsequent addition of dopants. The agglomeration of Nb-doped TiO_2 tends to become more pronounced as the Nb concentration increases, resulting in larger particles and reduced porosity. Furthermore, Nb acts as a defect, preventing the dye from attaching to the Nb-doped TiO_2 photoanode. The presence of Nb, whether on the surface or in its immediate vicinity, modifies the surface chemistry of TiO_2: Nb can occupy active sites or create competing binding sites, thereby hindering the efficient attachment of the dye. Consequently, Nb doping can also modify the surface energy levels of TiO_2, which may alter the nature of the electronic interaction between the dye and the surface, thereby affecting dye anchoring and electron injection.

Table2FESEM Analysis

Sample	Grainsize(nm)	Porosity (%)	Surfacearea(m^2/g)
0wt%Nb	131.3	75.06	6.1407
0.5wt%Nb	126.5	75.13	-
1.0wt%Nb	126.0	75.16	8.8314
1.5wt%Nb	128.5	75.12	7.3451

DSSC Photovoltaic Performance

Figure 4 presents the photovoltaic performance in terms of short-circuit current density (J_{SC}) and open-circuit voltage (V_{OC}), while Table 3 summarizes the analysis derived from this figure. The addition of niobium improved the properties of the photovoltaic cells; however, beyond a certain concentration, these properties degrade due to an excess of dopants. The best result was obtained with a content of 2.50%. The increase in short-circuit current density (J_{SC}) indicates an improvement in electron injection and transport, attributed to higher cell conductivity and the introduction of a new energy level that facilitates more efficient electron transfer. This phenomenon can be linked to the reduction in the band gap mentioned in Section 3.2. The creation of this new energy level allows for the emergence of additional free electrons in the valence band, which can be excited to this new level without energy loss. Consequently, it is possible to achieve a higher J_{SC} and design higher-performance cells using these solvothermal synthesis techniques, as the number of charge carriers increases at the new energy level created near the conduction band. A correlation can also be observed in Table 2: a smaller grain size increases the specific surface area of the nanoparticles, which promotes better dye absorption by the photoanodes. Greater absorption leads to a higher J_{SC}, thereby improving the performance of dye-sensitized solar cells (DSSCs). Conversely, the decrease in J_{SC} is attributed to the oxidation of niobium, which generates new recombination centers (electron recombination reactions at the photoanode/dye/electrolyte interface). This process reduces the number of charge carriers, which leads to a decrease in J_{SC}, as observed for a niobium content of 1.5 wt% [34].

Furthermore, the increase in open-circuit voltage (V_{OC}) can be attributed to enhanced optical absorption. Niobium (Nb) can act as an inhibiting agent that reduces charge recombination while simultaneously increasing the V_{OC} value. The introduction of a new energy level between the conduction and valence bands helps eliminate recombination centers located between these bands, thereby improving electron mobility by preventing electrons from becoming trapped at recombination sites. Consequently, Nb-doping creates an energy level capable of enhancing photovoltaic properties, even when natural dyes are used as is the case in this study. Nb-doping strengthens the photoresponse by introducing additional energy levels within the band gap of TiO_2. This leads to a reduction in internal resistance, resulting from the modification of the TiO_2 electronic structure. The short-circuit current density (J_{SC}) and open-circuit voltage (V_{OC}) values in dye-sensitized solar cells (DSSCs) depend significantly on the synthesis method used for the Nb-doped TiO_2. Among common fabrication techniques solvothermal, sol-gel, and solid-state synthesis the solvothermal method typically yields better photovoltaic performance. This is primarily due to its ability to produce well-crystallized, smaller, and more uniform nanoparticles (typically 8–11 nm in size) that exhibit a larger specific surface area and higher porosity [25–26]. These characteristics promote dye adsorption, facilitate electrolyte infiltration, and improve electron transport, leading to higher J_{SC} values, generally ranging between 5.20 and 6.00 mA/cm^2. A higher V_{OC}, ranging between 0.4 and 0.5 V, is also observed, thanks to improved crystallinity and a reduction in defects, which helps limit charge recombination at the photoanode. Compared to sol-gel and solid-state methods, the solvothermal method is more effective for achieving a high V_{OC}, due to uniform particle size and a larger specific surface area [35]. Although the solid-state method is simpler and more economical, it leads to significant particle

agglomeration and a low specific surface area, which reduces the amount of adsorbed dye, limits electron transport, and lowers photovoltaic performance [36]. Consequently, DSSCs fabricated via the solid-state method generally exhibit the lowest J_{SC} (0.2–2.29 mA/cm^2) and V_{OC} (0.28–0.5 V) values among the three methods analyzed [36]. Overall, the solvothermal method provides a more favorable nanostructure and electronic environment, making it the most effective approach for enhancing the performance in terms of J_{SC} and V_{OC} of dye-sensitized solar cells (DSSCs) based on Nb-doped TiO_2.

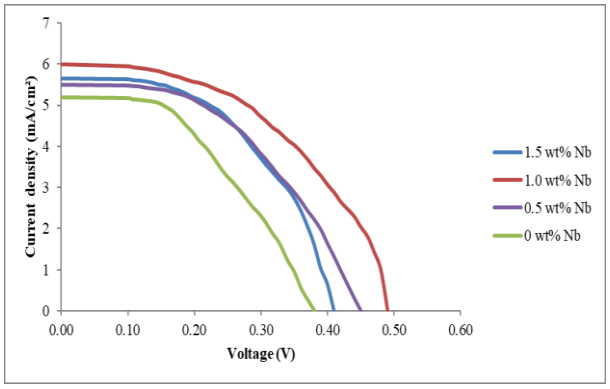

Figure 4. Performance of photovoltaic based on short circuit current density and open circuit current voltage for all samples using solvothermal method.

Table 3 Analysis of photovoltaic performance for all samples using solvothermal method

Sample	JSC(mA/cm2)	VOC(V)	FF	η(%)
0wt%Nb	5.20	0.38	0.78	1.54
0.5wt%Nb	5.65	0.45	0.80	2.03
1.0wt%Nb	6.00	0.49	0.85	2.50
1.5wt%Nb	5.71	0.41	0.80	1.87

CONCLUSION

The successful synthesis of niobium (Nb)-doped TiO_2 nanoparticles via the solvothermal method, combined with their integration into dye-sensitized solar cells (DSSCs) that utilize an extract from *E. conferta* fruit as a natural sensitizer, highlights a promising approach for the development of low-cost, eco-friendly photovoltaic devices. Structural analysis via X-ray diffraction (XRD) confirms the formation of a hexagonal TiO_2 phase with nanoscale crystallinity; meanwhile, UV-Vis and Tauc analyses reveal a systematic decrease in the bandgap as the Nb content increases, indicating enhanced visible-light absorption. Notably, the DSSC fabricated using TiO_2 doped with 1.0 wt% Nb achieved a maximum power conversion efficiency (PCE) of 2.50%, with an open-circuit voltage (Voc) of 0.49 V and a short-circuit current density (Jsc) of 6 mA/cm^2, outperforming results obtained with other doping levels. Doping with Nb allows for the tuning of the electronic structure and electron transport, thereby improving the performance of DSSCs. The combination with natural dyes can further enhance this performance facilitated by more effective molecular binding to the Nb-doped TiO_2 photoanode while simultaneously promoting environmental sustainability. In the context of this work, understanding the modification of metal oxides not only enables the tuning of DSSC properties but also contributes to the development of more environmentally friendly approaches for the next generation of solar cells.

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BIOGRAPHIES



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