



Exploring the Variation of Urbach Energies Between Anatase and Rutile Phases of TiO₂ Nanoparticles in Polymer-Based Hybrid Composites

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Abstract

Titanium dioxide (TiO₂) nanoparticles were combined with a mixture of polymers: polyvinyl alcohol (PVA), polyethylene glycol (PEG), and polyvinylpyrrolidone (PVP). TiO₂ nanoparticles were formed by the sol-gel process, and nanocomposites were prepared with concentrations 1, 5, 10, 15, 20, and 25 wt% of TiO₂ nanoparticles after subjecting them to a calcination process at temperatures of about 400 °C and 700 °C, and with polymer blends of different concentrations (PVA various wt%, PEG constant wt%, and PVP constant wt%). A UV-Vis spectrometer was used to determine the optical constants of the prepared samples, namely, the absorption coefficients and Urbach energies. It was observed that the Urbach energies were highest for the samples with calcination temperature 400 °C and 20 wt% TiO₂ nanoparticles blended with PVA 65 wt%, PEG 10 wt%, and PVP 5 wt%. The Urbach energy for the PVA-PEG-PVP polymer blend was 0.32 eV. For the anatase phase (calcination at 400 °C), the Urbach energy was in the range of 0.41–5.55 eV for PVA-PEG-PVP-TiO₂ nanocomposites, and for the rutile phase (calcination at 700 °C) it was in the range of 0.31–1.94 eV. The findings have important ramifications for applications of these nanocomposites as reusable photocatalysts, by providing a means of extending their useful life.

Keywords: Titanium dioxide nanoparticle, PVA, PEG, PVP, Urbach energy, polymer blend.

1. Introduction

Nanocomposites have emerged as a prominent field of study, holding significant potential in a wide variety of application domains. The use of polymers in nanomaterial synthesis has been extensively explored owing to their remarkable properties. Titanium dioxide, also known as titania (TiO₂), is a white material that is widely used in environmental photocatalysis, self-



cleaning and anti-fogging surfaces, and photoelectrochemical conversion of solar energy (1,2). Titanium dioxide nanoparticles are among the most widely used nanomaterials due to their distinctive optical and chemical characteristics. Previous studies have demonstrated that the addition of TiO₂ nanoparticles to polyvinyl alcohol (PVA) enhances its photocatalytic properties for water purification.

The Urbach energy of nanocomposite films comprising a polyvinylpyrrolidone (PVP) matrix and TiO₂ nanoparticles increases with the TiO₂ concentration, indicating a rise in structural defects and interstitial gaps in the polymeric network. These defects are crucial in determining the optical and electrical properties of the films, affecting their practical applications(3) . The Urbach energy of TiO₂ nanocrystalline films is significantly influenced by the annealing rate. The obtained Urbach energy values ranged from 432 meV to 505 meV, with the highest value observed at an annealing rate of 1 °C/min. A higher Urbach energy indicates an increase in structural defects and interstitial gaps in the films, reflecting a greater degree of disorder in the crystalline structure of the films. It was observed that the optical band energy decreased as the Urbach energy increased, due to improved crystallinity and reduced structural defects in the films (4) .

Nanostructured TiO₂ has a larger surface area, improving its photocatalytic efficiency. When exposed to UV light, TiO₂ generates electron–hole pairs, which can react with water molecules or organic contaminants. This leads to the creation of reactive oxygen species (ROS), like hydroxyl radicals. These ROS can decompose organic compounds in water (2–5) . PVA serves as a stable support for TiO₂ nanoparticles, preventing aggregation and ensuring uniform dispersion. It also provides mechanical strength and flexibility to the composite material, making it durable and suitable for practical use (6) . Furthermore, PVA enhances the absorption of visible light, expanding the photocatalytic activity of TiO₂ to the visible region of the spectrum (7,8) . In summary, the combination of nanostructured TiO₂ and PVA in a composite material offers improved photocatalytic properties(9), enabling the degradation of organic pollutants and the inactivation of microorganisms in water, making it a promising solution for water purification (10).

However, these nanoparticles constitute a threat to the environment because they are often only used once to treat wastewater and disposed into bodies of water, from where they are challenging to recover. The nanoparticles can be incorporated into or supported by a polymeric matrix, like electrospun fibers, to reduce this risk (11). Immobilizing the nanoparticles on these supports enables the catalysts to be reused, extending their useful lives.

Nanocomposites have gained significant attention in various fields due to their versatile applications and unique properties. In this work, we focus on preparing nanocomposites using a polymer blend of PVA, polyethylene glycol (PEG), PVP, and TiO₂ nanoparticles. The Urbach energies of these nanocomposites are investigated, as they play a crucial role in potential applications such as optoelectronics and sensors (12).

This study addresses the gap in our understanding of how different calcination temperatures affect the anatase and rutile phases of TiO₂ nanoparticles in a polymer blend, specifically their Urbach energies. This enhances our understanding of the optical and electrical properties of these

composites, enabling improvements in photocatalytic efficiency for environmental applications and guiding future research in developing tailored nanomaterials for advanced technological uses.

2. Materials and methods

The synthesis of titanium dioxide nanoparticles was carried out using the sol–gel method. The procedure of hydrolysis and condensation of titanium alkoxides in water was used to synthesize nanoscale TiO₂ colloids. Alkoxides were hydrolyzed and then polymerized to form a three-dimensional oxide network in the water. These interactions can be described as follows:

Hydrolysis:



Condensation:



In the above, R represents a group such as ethyl, *i*-propyl, or *n*-butyl. Nucleation of the stable hydroxide Ti(OH)₄ cannot occur. This is attributable to the powerful acidity of tetravalent cations. Water molecules resulting from reaction (2) always have a fractional positive charge. Accordingly, sporulation and axis can proceed at the same time during the progression of nucleation and growth. This will result in an amorphous type of TiO₂·*n*H₂O oxide, where *n* is the number of water molecules, which depends on the experimental conditions. The formation of rutile or anatase phases by TiO₂ deposition is observed on the experimental route. By adapting the initial water concentration and pH, the deoxidation stage before decomposition can be controlled. Precipitation of TiO₂ anatase nanoparticles will occur as a result of controlling the experimental work steps.

Precursor solution A was made from a mixture of 5 ml of titanium tetra-isopropoxide (TTIP, 98%, Aldrich) and 15 ml of ethanol (99%, Merck). The hydrolysis catalyst used was solution B of distilled water, with a volume of 250 ml. HNO₃ and NH₄OH (99%, Merck) were used to control the pH of the solution. Solution B was then added dropwise to solution A to obtain a clear and transparent solution. The solution became milky during the peptization procedure, in which it was heated to a temperature of 60–70 °C for 16 hours. After peptization, a suspension was produced. The synthesized precipitate was washed three times with deionized water and then three times with ethanol. The product was left to dry for 12 hours at 100 °C.

Subsequently, the synthesized TiO₂ nanoparticles were calcined at two different temperatures, 400 °C and 700 °C, to optimize their crystalline structure and remove any residual organic species (13). The choice of calcination temperature (400 °C for anatase and 700 °C for rutile) was based on their well-documented effects on the crystalline structure of TiO₂. Calcination at 400 °C favors the formation of the anatase phase, which is known for its higher photocatalytic activity due to its larger surface area and higher reactivity. In contrast, calcination at 700 °C induces the formation of the rutile phase, which possesses different structural and electronic properties that can significantly influence the optical characteristics and stability of the nanocomposites. Calcination is a crucial step that influences the morphology, size, and crystallinity of the nanoparticles, affecting the overall properties of the nanocomposite. The prepared TiO₂ nanoparticles of concentrations 1, 5, 10, 15, 20, and 25 wt% TiO₂ were then mixed with the polymer blend in

specific weight ratios (PVA various wt%, PEG 10 wt%, and PVP 5 wt%). The detailed composition of each sample is presented in **Table 1**. The selection of PVA, PEG, and PVP as the polymer components was based on their compatibility with the nanoparticles and their ability to form a stable composite system (12,14). The weight ratios of the components were carefully chosen to ensure a homogeneous dispersion of nanoparticles within the polymer matrix (15). The solvent casting method was employed to fabricate the nanocomposite (16).

The polymer blend and TiO₂ nanoparticle mixture were dissolved in a suitable solvent, followed by the casting of the solution onto a flat substrate. The solvent was evaporated under controlled conditions to obtain uniform nanocomposite with a desired thickness.

Table 1. Mixing ratios of the prepared samples for the PVA–PEG–PVP–TiO₂ nanocomposite.

<i>TiO₂</i> wt%	PVA wt%	PVP wt%	PEG wt%
0	85	5	10
1	84	5	10
5	80	5	10
10	75	5	10
15	70	5	10
20	65	5	10
25	60	5	10

2. Results and Discussio

The absorption coefficient of a material is strongly correlated with both photon energy and band gap energy. It depends on the incident photon energy and the kind of electronic transitions that take place between the energy bands, and can be defined as the attenuation in the flood of radiation energy or the intensity per unit of area in the direction of the wave in the medium (15,16). The attenuation in incident photon energy is caused by the absorption processes. The following equation connects the absorbance with the absorption coefficient (13,18–20):

$$\alpha = 2.303 \left(\frac{A}{d} \right) \quad (3)$$

where d is the thickness and α is the absorption coefficient (19,21).

When electrons move from the valence band to the conduction band, they come across a density of states given by $\rho(h\nu)$, with $h\nu$ representing the photon energy. This interaction commonly results in disturbances within the energy gap caused by irregularities. This occurrence is identified by the Urbach energy E_u and absorption tails at the band edge. The extension of $\rho(h\nu)$ into the energy band gap is commonly known as the Urbach tail. Consequently, the energy associated with this tail is termed the Urbach energy. This phenomenon arises from the exponential decay of the absorption coefficient $\alpha(h\nu)$. The calculation of the Urbach energy is described by the following (17,22):

$$\alpha = \alpha_0 + \exp\left(\frac{h\nu}{E_u}\right) \quad (4)$$

where α_0 is a constant [17,23]. In practice, the Urbach energy is obtained by plotting $\ln \alpha$ against $h\nu$, which is shown in **Figure 1- 3** for prepared samples. The Urbach energy is calculated by finding the inverse of the slope of the regression below the optical band gap area. These E_u values are presented in **Table 2**.

Table 2. Calculated Urbach energy values of the prepared samples.

	Mixture Percentage (wt%)	Urbach Energy (eV)
Pure polymer	0	0.3222
Anatase phase (400 °C)	1	0.4100
	5	1.1550
	10	1.1605
	15	0.5426
	20	5.5506
	25	5.2910
Rutile phase (700 °C)	1	0.3159
	5	0.3115
	10	0.3879
	15	0.6117
	20	1.9493
	25	1.9146

From **Table 2.**, it can be seen that with an increase in the TiO₂ nanoparticles, the resulting increase in Urbach energy is linked to the rise in oxygen defect centers within the system (24). **Figure 1.** shows the Urbach energy for the PVA–PEG–PVP polymer blend for the anatase phase (with calcination at 400 °C).

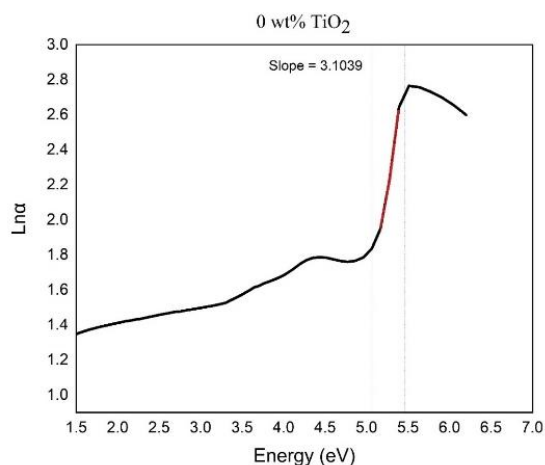


Figure 1. Plot of Urbach energy for the PVA–PEG–PVP polymer blend for the anatase phase (400 °C).

Based on **Figure 1**, the Urbach energies of the PVA–PEG–PVP polymer blend, with concentrations of PVA 85 wt%, PEG 10 wt%, and PVP 5 wt%, are observed to be 0.322 eV.

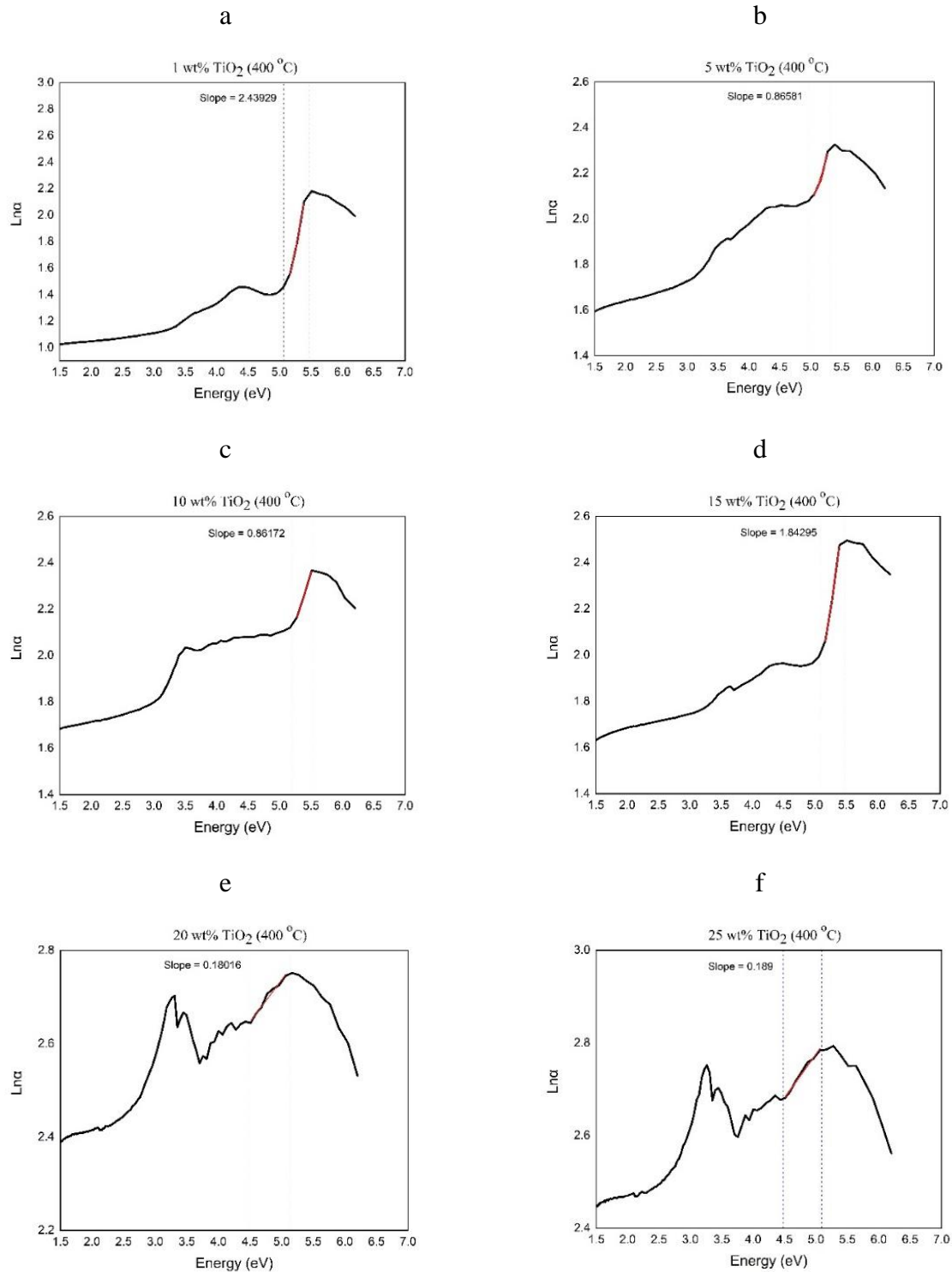
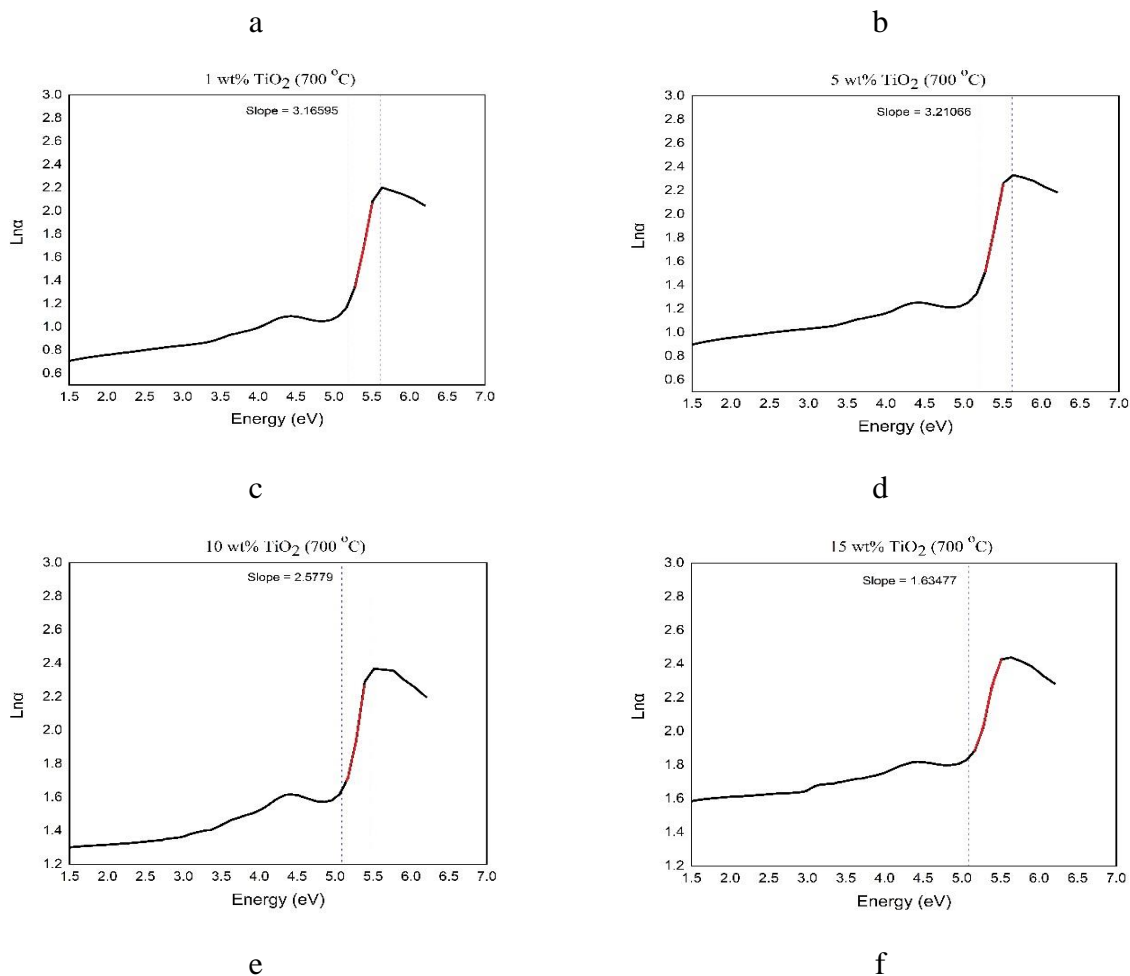


Figure 2. Plots of the Urbach energy for PVA-PEG-PVP-TiO₂ nanocomposite for the anatase phase (400 °C), with TiO₂ weight percentages of a) 1%, b) 5%, c) 10%, d) 15%, e) 20%, and f) 25%.

A rise in the concentration of TiO₂ nanoparticles leads to a higher Urbach energy. This indicates the presence of oxygen vacancies when introducing TiO₂ nanoparticles.

The increase in oxygen vacancy defects can be explained by the concept of charge neutrality (25,26). This is crucial, as the concentration of TiO_2 rises from 1% to 25% in the polymer blend lattice (27,28). The creation of oxygen vacancies leads to localized defect sites significantly impacting the valence and conduction band edges (29,30). This could also be a factor contributing to the decrease in the band gap in the polymer blend doped with TiO_2 . More clearly, the presence of oxygen vacancies leads to the creation of imperfections within the material's band structure, causing an increase in Urbach energy. From **Figure 2**, the Urbach energies of PVA-PEG-PVP- TiO_2 nanocomposite, for the anatase phase (400 °C) and with TiO_2 nanoparticle weight percentages of 1, 5, 10, 15, 20, and 25, were 0.410, 1.15, 1.16, 0.542, 5.55, and 5.29 eV, respectively. The Urbach energies were higher for a calcination temperature of 400 °C and 20 wt% TiO_2 nanoparticles blended with PVA 65 wt%, PEG 10 wt%, and PVP 5 wt% than those of the other samples.



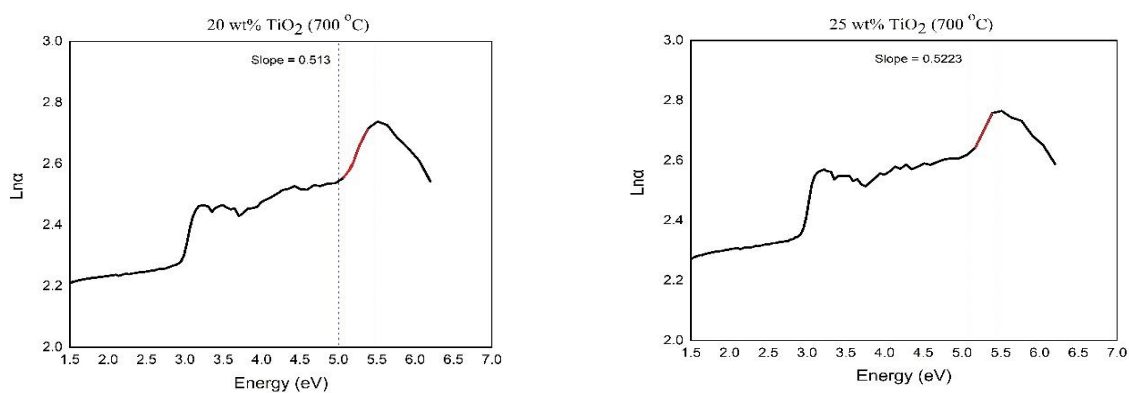


Figure 3. Plots of the Urbach energy for PVA–PEG–PVP–TiO₂ nanocomposite for Rutile phase (700 °C) and TiO₂ weight percentages of a) 1%, b) 5%, c) 10%, d) 15%, e) 20%, and f) 25%.

The decrease in E_u from 0.315 to 0.311 eV can be accounted for by a change in the structure of the films from amorphous to microcrystalline. The anatase phase, formed at 400 °C, represents a transition from an amorphous state to a microcrystalline structure, enhancing the photocatalytic properties. Similarly, the rutile phase, formed at 700 °C, indicates further crystallization and the formation of oxygen vacancies induced by lattice vibrations during absorption leads to a rise in Urbach energy from 0.387 to 1.949 eV. From **Figure 3**, it can be seen that the Urbach energies of PVA–PEG–PVP–TiO₂ nanocomposite for the rutile phase (700 °C) with TiO₂ nanoparticle weight percentages of 1, 5, 10, 15, 20, and 25 were 0.315, 3.11, 0.387, 0.611, 1.949, 1.914 eV, respectively. The Urbach energies for a calcination temperature of 700 °C and 20 wt% TiO₂ nanoparticles blended with PVA 65 wt%, PEG 10 wt%, and PVP 5 wt% were higher than those of the other samples. High Urbach energy values indicate a significant number of structural defects and interstitial gaps in the polymeric network. At a 5% TiO₂ concentration, the highest Urbach energy was observed, suggesting an imbalanced and unstable structure, making this ratio potentially unsuitable for practical applications. In contrast, at 10% and 15% TiO₂ concentrations, the Urbach energy values were lower, indicating that these ratios may be ideal for achieving greater structural stability. Therefore, it can be concluded that low-to-moderate TiO₂ ratios (10% and 15%) are preferable to achieve a balance between structural stability and reducing defects in the polymeric network.

4. Conclusion

Titanium dioxide (TiO₂) nanoparticles were successfully formed via the sol–gel procedure. The anatase and rutile phases were obtained by subjecting the prepared powder to a calcination process at temperatures of about 400 °C and 700 °C, respectively. Nanocomposites were prepared from polymer mixtures (variable weight PVA, constant weight PEG, and constant weight PVP) and different concentrations of TiO₂ nanoparticles. Studies on the Urbach energy of PVA–PEG–PVP blends with different weight percentages of TiO₂ nanoparticles yielded valuable findings. The results showed that the incorporation of TiO₂ nanoparticles into the PVA–PEG–PVP matrix has a significant influence on the Urbach energies of the composites. Based on the research findings, the Urbach energy values for anatase and rutile TiO₂ nanocomposite were obtained. For anatase TiO₂, the Urbach energy was determined to be about 5.5 eV, and for rutile, 1.94 eV. These values indicate

the characteristic width of the exponential absorption tail within the Urbach area of the substances' absorption spectra. The differences in Urbach energy between anatase and rutile TiO₂ can be attributed to variations in crystalline structure, defects, and surface characteristics. These findings offer useful insights into the optical properties and electronic structure of anatase and rutile TiO₂, which might be important for diverse applications such as photocatalysis.

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Conflict of Interest

The authors declare that they have no conflicts of interest.

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Ethical Clearance

The local ethical committee at the University of Baghdad approved the project.

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