

## Synthesis and Characterization of Cobalt-doped Titanium Dioxide Thin Films as Solar Cell Components using UV-SEM Analysis

Aris Doyan<sup>1,\*</sup>, Susilawati<sup>1</sup>, Agus Abhi Purwoko<sup>1</sup>, Muhammad Taufik<sup>2</sup>, Dedi Riyan Rizaldi<sup>3</sup>, Ziadatul Fatimah<sup>4</sup>

<sup>1</sup> Master of Science Education Program, University of Mataram, Lombok, West Nusa Tenggara, Indonesia

<sup>2</sup> Physics Education Program, FKIP, University of Mataram, Lombok, West Nusa Tenggara, Indonesia

<sup>3</sup> Madrasah Aliyah Plus Nurul Islam Sekarbela, Mataram, Lombok, West Nusa Tenggara, Indonesia

<sup>4</sup> Senior High School Nahdathul Wathan Mataram, Mataram, Lombok, West Nusa Tenggara, Indonesia

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### ABSTRACT

Research has been carried out in the form of the synthesis and characterization of thin films of titanium dioxide with the addition of cobalt doping. The purpose of this research was to synthesize and characterize thin films related to optical properties, morphological shapes, and components of thin film compounds that are good for use as basic materials for the development of nanoparticle technology in the form of solar cells. The research was carried out in two stages, namely synthesis such as preparation of glass substrates, preparation of sol-gel solutions, deposition of solutions onto glass substrates, and heating of TiO<sub>2</sub>:Co thin film samples. The next stage is the thin film characterization test, where the optical properties of the thin film, such as absorbance, transmittance, gap energy, and activation energy, are measured using a UV-vis spectrophotometer, while morphological data and components of the thin layer compounds are measured using a Scanning Electron Microscope-Energy Dispersive X-rays (SEM-EDX). Based on data analysis and discussion, it was found that the optical properties, such as the transmittance value of the thin film, experienced the lowest point at a wavelength of 250–280 nm with a maximum percentage value of 62.14 to 78.85%. The greater the doping concentration, the lower the transmittance value. This condition is inversely proportional to the absorbance value of the maximum TiO<sub>2</sub>:Co thin film, which is in the region with a wavelength of 280–300 nm and a value of 5.70–6.31. The greater the doping concentration, the greater the absorbance value. The energy bandgap values of TiO<sub>2</sub>:Co thin films for doping concentrations (0, 5, 10, 15, 20)% were 3.44; 3.39; 3.38; 3.32; 3.22 eV for the direct energy bandgap and 3.86; 3.85; 3.84; 3.85; 3.82 eV for the indirect energy gap. The activation energy of the TiO<sub>2</sub>:Co thin film decreased from 2.86 to 2.26 eV. As for the morphological data of the TiO<sub>2</sub>:Co thin film, it was found that the greater the concentration of doping, the layer was deposited evenly, finely, and homogeneously. Regarding the data on the components of the thin layer compounds, it was found that the percentage of TiO<sub>2</sub> content was in the range of 64.48–95.94%, while for Co, it was in the range of 0.00–19.21%.

\* Corresponding author.

E-mail address: [aris\\_doyan@unram.ac.id](mailto:aris_doyan@unram.ac.id)

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## 1. Introduction

Solar cells are a nanoparticle-based technology that continues to experience a process of development and improvement both in terms of basic materials and the resulting performance in supporting various human activities [1]. This is, of course, because solar cell technology utilizes unlimited energy sources, namely sunlight, and is environmentally friendly, so it is a good alternative to replacing limited energy sources such as coal, gasoline, and others [2,3]. To be able to develop solar cells, materials that are semiconductors and have good absorption levels are needed in order to maximize solar radiation as the main energy source [4,5]. One of the compounds that can be used as a base material or precursor in the development of thin film-based solar cells is titanium dioxide, also known as  $\text{TiO}_2$  [6,7].

$\text{TiO}_2$  compounds are often used in the development of solar cells due to several advantages, such as having high stability against photo corrosion, a high level of photocatalyst, a relatively cheap price compared to other compounds with the same use, and not causing side effects that are harmful to health. However, based on the various advantages previously possessed, there is one drawback of the  $\text{TiO}_2$  material, namely that it still has a relatively large energy gap that has an impact on the level of absorption of light, which is still limited [8,9]. The absorption limitation is only in the ultraviolet light spectrum with low wavelengths. In general, the good properties of photocatalysts in solar cell technology are that the process of absorbing energy from sunlight can also occur in the visible light wavelength range [10]. A photocatalyst is a combined process between chemical synthesis processes involving light as a triggering factor and a catalyst as a factor that accelerates the reaction process [11,12].

Efforts that can be used to increase photocatalyst activity on  $\text{TiO}_2$  materials are doped with either metal or non-metal elements. The purpose of the doping process is to optimize the electrical and optical properties of the thin films to be developed [13,14]. One of the dopings that can be used to change the characteristics of thin films is cobalt (Co) because the atoms of this metal can enhance the process of photocatalytic reduction and degradation. This is supported by several previous studies using cobalt doping, which was found to be able to reduce the band gap so that it can work in visible light (up to 550 nm) [15]. This condition is certainly expected to form on a thin layer of  $\text{TiO}_2$  with Co doping using the sol-gel spin coating method as a material for developing solar cells.

## 2. Methodology

This research is pure experimental research. The variables of this research consist of the independent variable, namely variations in cobalt doping, while the dependent variable is the synthesis and characterization of  $\text{TiO}_2$  thin layers as materials for developing solar cell components. This research was carried out through two stages of the process, namely synthesis and characterization. The synthesis process aims to produce  $\text{TiO}_2$ :Co thin film samples with concentration criteria of 0, 5, 10, 15, and 20%, consisting of four layers, and heated at 200 °C. Stages of synthesis start with: a) preparation of a glass substrate with a size of 15 x 15 x 3 mm; b) preparation of sol-gel solutions utilizing titanium dioxide precursor materials, cobalt doping materials, ethanol, and hydrochloric acid solvents with the help of a magnetic stirrer; c) deposition of sol-gel solutions on a glass substrate using a spin-coating method with the aid of a modified centrifuge; and d) heating the sample using an oven. In the next stage, after the sample has been successfully synthesized, the characterization process is carried out with the aim of determining the optical properties of the thin layer using a UV-Vis Spectrofotometer and the morphology and constituent components of the thin layer using a Scanning Electron Microscope-Energy Dispersive X-ray (SEM-EDX).

Analysis technique for measuring transmittance and absorption of UV light in the ultraviolet wavelength range (starting from 150 nm) to cover all wavelengths of visible light (up to 800 nm) using a UV-Vis Spectrophotometer. From the characterization process, data was obtained on the comparative value of the quality of pure TiO<sub>2</sub> thin films with TiO<sub>2</sub>:Co thin films in terms of transmittance and absorbance levels. Then the absorbance values that have been obtained are analyzed using Microsoft Excel to find the energy band gap and activation energy values.

The optical energy band gap value of a material is obtained by the following equation

$$\alpha h\nu = C(h\nu - E_g)^n \quad (1)$$

Information:

$\alpha$  = absorbance coefficient

$h\nu$  = photon energy (eV)

$E_g$  = energy band gap (eV)

$C$  = constant

The step to obtain the thin layer absorbance coefficient value is obtained by using the absorbance spectrum value in the equation.

$$\alpha(\nu) = \frac{2.303A}{d} \quad (2)$$

Information:

$A$ =absorbance (obtained directly through the reading process with a UV-Vis Spectrophotometer)  
 $d$ =thin layer sample thickness (mm)

Then the final step to determine the energy band gap value of the pure TiO<sub>2</sub> and TiO<sub>2</sub>:Co thin films is obtained from the slope of the graph  $(\alpha h\nu)^n$  against the photon energy  $h\nu$ , with the electron transition constant  $n = \frac{1}{2}$  for direct allowed and  $n=2$  for indirect allowed. Meanwhile, to calculate the activation energy using the Urbach-edge method, it is mathematically expressed through the equation

$$\alpha(\omega) = \alpha_0 \exp\left(\frac{\hbar\omega}{\Delta E}\right) \quad (3)$$

$$\ln \alpha(\omega) - \alpha_0 = \frac{\hbar\omega}{\Delta E} \quad (4)$$

$$\ln \alpha(\omega) = \frac{\hbar\omega}{\Delta E} + \alpha_0 \quad (5)$$

Because  $\hbar = \frac{h}{2\pi}$  and  $\omega = 2\pi\nu$ , so

$$\ln \alpha(\omega) = \frac{h}{2\pi} \frac{2\pi\nu}{\Delta E} + \alpha_0 \quad (6)$$

$$\ln \alpha(\omega) = \frac{h\nu}{\Delta E} + \alpha_0 \quad (7)$$

Because  $E_f = h\nu$ , so

$$\ln \alpha(\omega) = \frac{1}{\Delta E} E_f + \alpha_0 \quad (8)$$

Eq. (8) is a linear equation that is proportional to the equation  $y = mx + C$ , with  $m = \frac{1}{\Delta E}$ , so the activation energy ( $E_a$ ) =  $\Delta E$ . So that

$$E_a = \frac{1}{m} \quad (9)$$

Information:

$\alpha(\omega)$  = absorbance coefficient

$\alpha_0$  = constant

$\omega = 2\pi\nu$

$E_a$  = Activation energy (eV)

The research flow procedure can be seen in the following Figure 1.

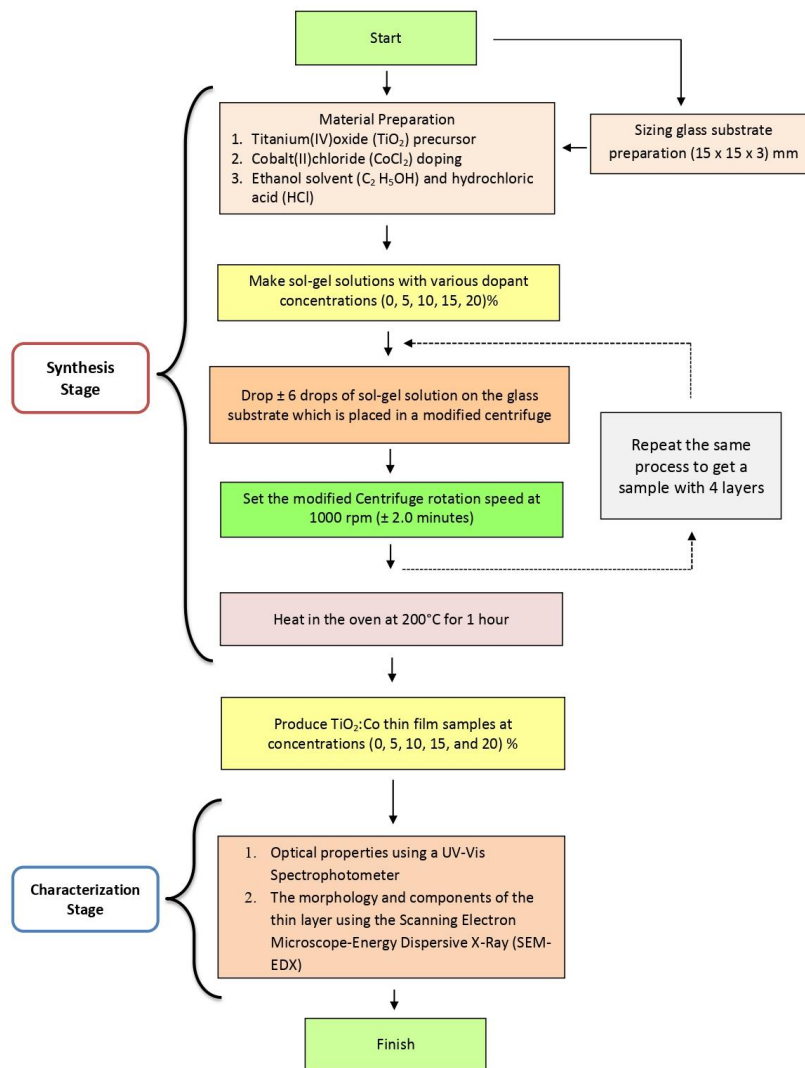


Fig. 1. Thin film research flow procedure TiO<sub>2</sub>:Co

### 3. Results

#### 3.1 Synthesis of Thin Film $TiO_2:Co$

Based on the thin film synthesis process that has been carried out at the Basic Chemistry Laboratory, Faculty of Mathematics and Natural Sciences, University of Mataram, it can be seen that the sample of  $TiO_2$  thin layer with cobalt doping has a white surface appearance, with the condition that the greater the concentration of doping given, the more average the deposition results of the solution on the surface of the substrate glass, as shown in Figure 2 below.

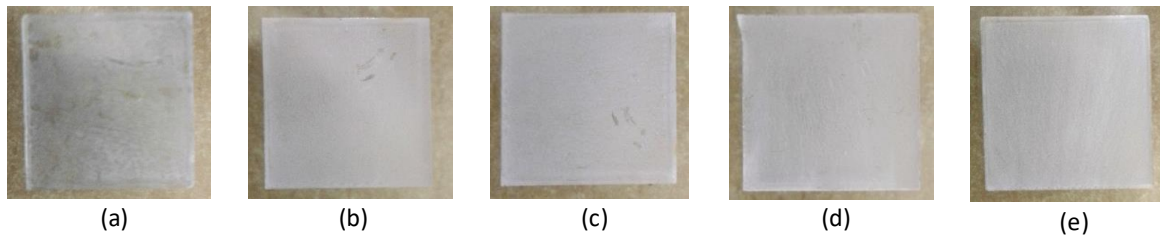
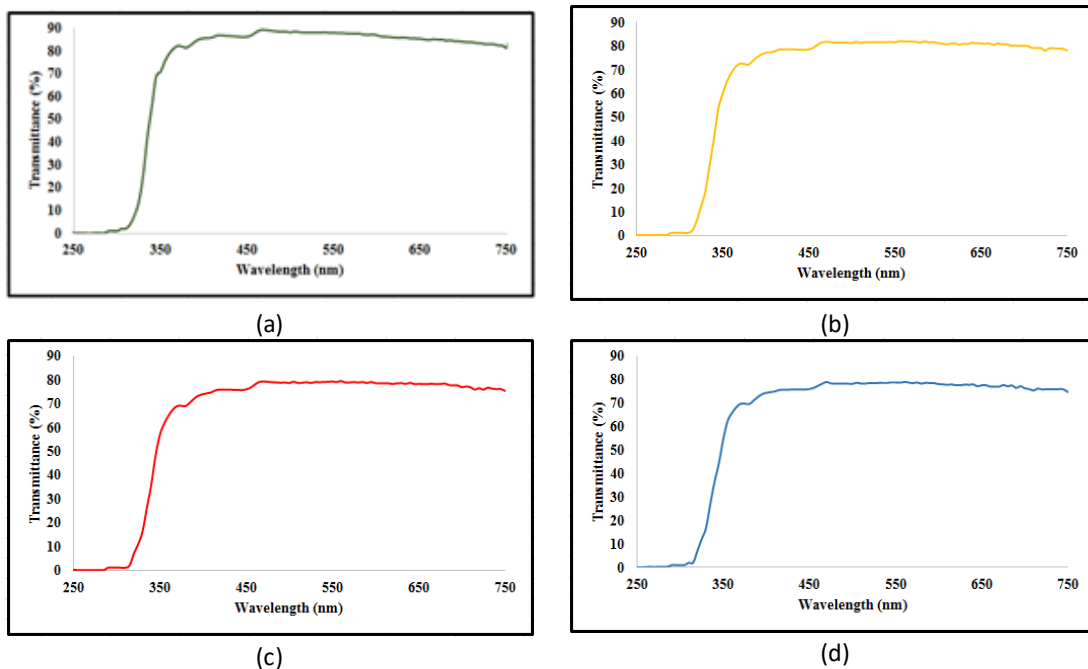
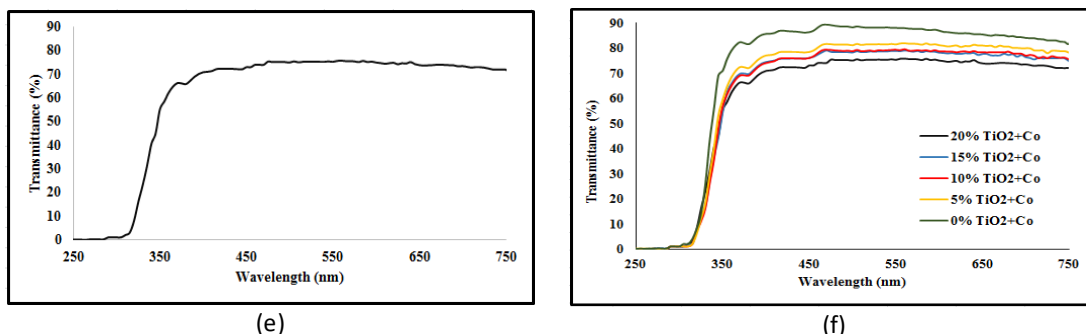


Fig. 2. Thin film samples  $TiO_2:Co$  at various concentrations (a) 0%, (b) 5%, (c) 10%, (d) 15%, (e) 20%

#### 3.2 Characterization of Thin Film Optical Properties $TiO_2:Co$

The process of testing the characterization of the optical properties of  $TiO_2:Co$  thin films using UV-Vis spectrophotometers was carried out at the Integrated Science Laboratory, Mataram State Islamic University. The optical properties of the thin films tested and observed include the transmittance and absorbance properties of the thin film samples. Graphs of the transmittance and absorbance values of the  $TiO_2:Co$  thin film can be seen in Figures 3 and 4.



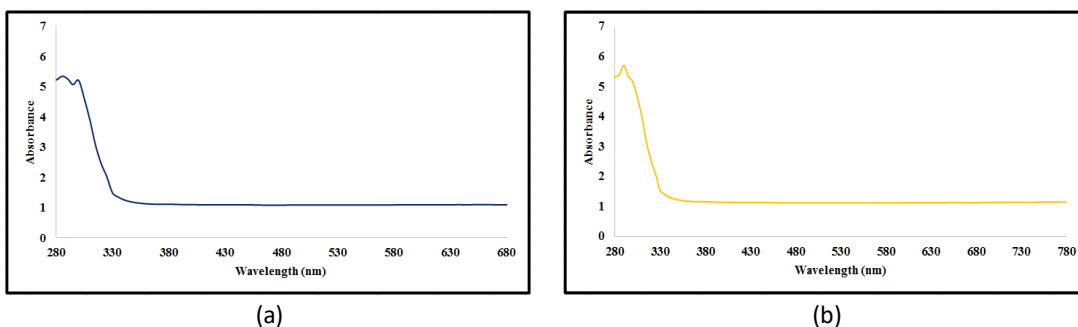


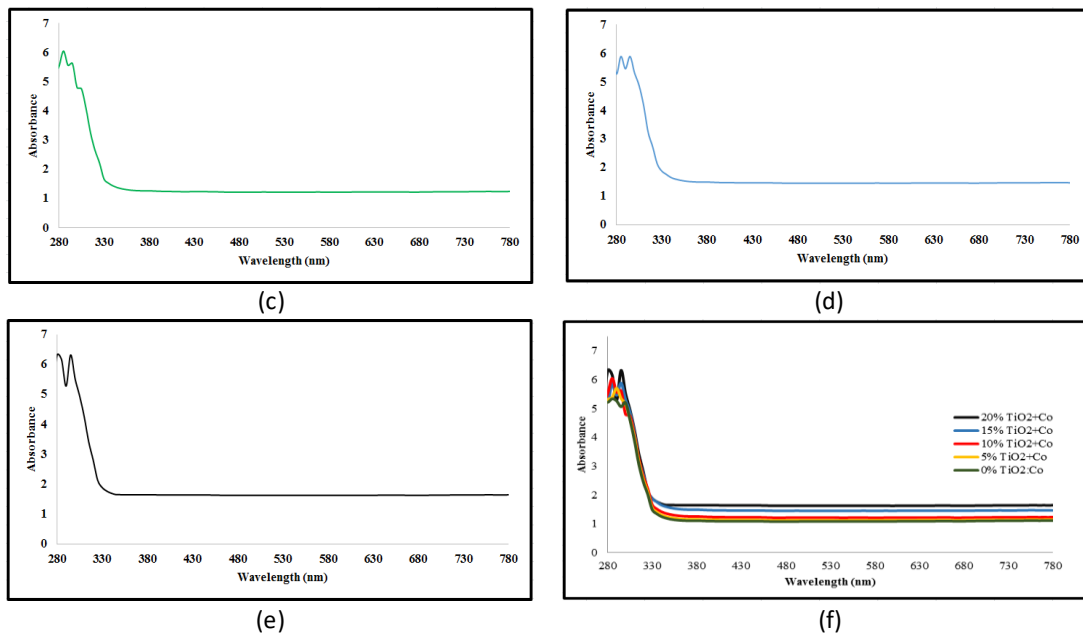
**Fig. 3.** Graph of  $\text{TiO}_2\text{:Co}$  thin film transmittance at various concentrations (a) 0%, (b) 5%, (c) 10%, (d) 15%, (e) 20%, and (f) all concentrations

The transmittance value describes the ability of a thin layer to transmit or receive light emitted from a light source, in this case, a UV-Vis spectrophotometer [16]. Based on Figure 3, it can be seen that the lowest transmittance spectrum is in the wavelength range 250–280 nm (ultraviolet light region), while the highest intensity is in the wavelength range 350–750 nm (visible light region), with the highest value found in the  $\text{TiO}_2\text{:Co}$  thin layer sample at a concentration of 0% and a value of 89%. The low percentage of transmittance of the  $\text{TiO}_2\text{:Co}$  thin films is thought to be due to the addition of cobalt doping, which results in a thicker surface on the thin layer samples when compared to the thin layer samples without additional doping treatment. This is in line with the data that the higher the doping concentration is given, the percentage of transmittance of the thin film will decrease [17], [18]. Meanwhile, the increase in the transmittance spectrum shown in the graph shows that the  $\text{TiO}_2\text{:Co}$  thin layer samples are composed of materials with relatively homogeneous chemical stoichiometry on the surface of the glass substrate [19].

Referring to these conditions, the low transmittance factor in the ultraviolet region indicates that the  $\text{TiO}_2\text{:Co}$  thin layer sample developed in this study has a relatively high level of light absorption. This is in line with the relationship between transmittance properties, which is inversely proportional to the absorbance properties of a layer, namely that the higher the percentage transmittance of a surface, the lower the absorbance value [20-22]. This condition can be compared with the graph shown in Figure 4, where the highest absorbance of the  $\text{TiO}_2\text{:Co}$  thin film is in the wavelength range of 280–300 nm, with the highest value in the  $\text{TiO}_2\text{:Co}$  thin layer sample with a concentration of 20% and a value of 6.31, while the lowest is in the wavelength range of 330–680 nm.  $\text{TiO}_2$  has the highest absorption rate in the ultraviolet wavelength range, which is characterized by its maximum absorbance value [23,24].

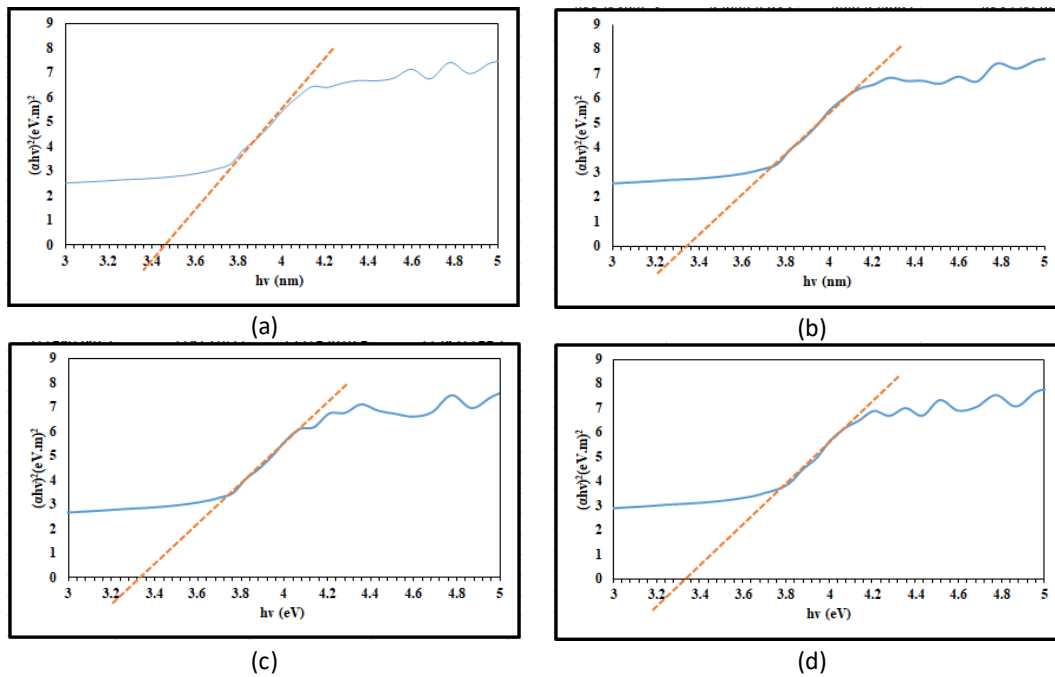
In addition to increasing the intensity of the absorbance value in the ultraviolet light wavelength range, the addition of cobalt doping is also able to increase the absorbance value of the thin film along the visible light wavelength range. Of course, this provides an opportunity to increase the chances of the absorption of light energy in various wavelength ranges.

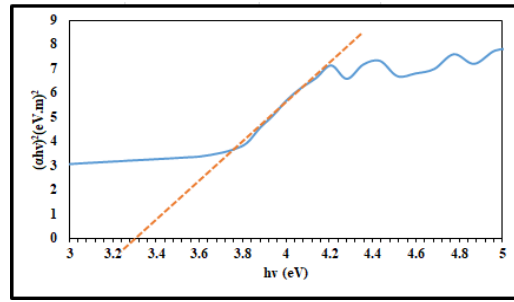




**Fig. 4.** Graph of absorbance of  $\text{TiO}_2\text{:Co}$  thin films at various concentrations (a) 0%, (b) 5%, (c) 10%, (d) 15%, (e) 20%, and (f) all concentrations

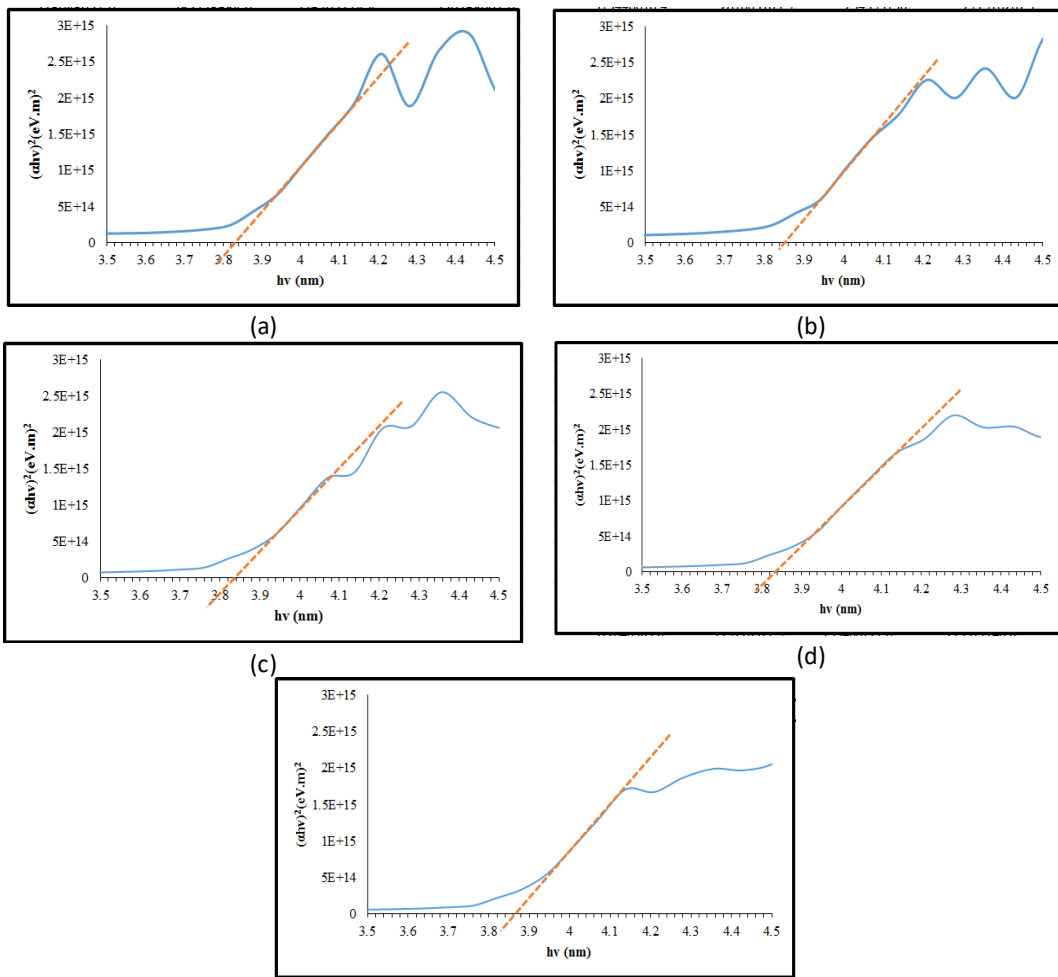
The absorbance value of the measurement results from the UV-Vis spectrophotometer was analyzed using Microsoft Excel to obtain the energy band gap value of the  $\text{TiO}_2\text{:Co}$  thin film sample. Analysis of the value of the energy band gap with reference to the Tauc Plot graphical method, which describes the relationship between  $h\nu$  and  $(ah\nu)^n$  [25-27]. The value of the energy band gap is seen from the slope gradient formed at the intersection of  $(ah\nu)^n$  with the  $h\nu$  axis. The energy band gap values obtained consist of direct and indirect energy band gaps, which can be seen in Figures 5 and 6.





(e)

**Fig. 5.** Graph of the direct energy band gap of TiO<sub>2</sub>:Co thin films at concentrations of (a) 0%, (b) 5%, (c) 10%, (d) 15%, and (e) 20%



(e)

**Fig. 6.** Indirect energy band gap graph of TiO<sub>2</sub>:Co thin films at concentrations of (a) 0%, (b) 5%, (c) 10%, (d) 15%, and (e) 20%

In general, TiO<sub>2</sub> shows the highest absorbance value in the wavelength range of 280–300 nm, followed by a constant intensity at a wavelength of 300–600 nm with a value of 1.08. However, after the process of adding cobalt doping, there was an increase in absorbance at a wavelength of 300–600 nm, with values ranging at concentrations of 5, 10, 15, and 20%, respectively: 1.12, 1.21, 1.45, and 1.62.

The energy band gap describes the intrinsic properties of semiconductor materials, which can be estimated through optical absorbance measurements [28,29]. Based on Figure 5, it can be seen that



the addition of cobalt doping reduces the energy band gap in the TiO<sub>2</sub> compound, which is the basic material (precursor) in the development of thin films based on solar cells. Decreasing the energy band gap is an important advance in this research because decreasing it reduces the minimum energy required for electrons to undergo an excitation process or move from the valence band to the conduction band. This is because a decrease in the energy band gap in a thin film causes more energy ranges that can be used as a source of electron and hole pairs [30-32]. The decrease in the energy band gap causes the absorption spectrum or the absorbance ability of the thin film to shift to the right towards a wider wavelength so that more visible radiation intensity can be absorbed by the TiO<sub>2</sub>:Co thin layer [33,34]. The importance of a shift in absorption in the visible light region is due to the presence of these rays on Earth more than ultraviolet rays. This is in line with research conducted by Doyan and Munadar which proves that the addition of doping can reduce the gap energy in a thin layer precursor with direct gap energy starting from 3.58 eV to 3.54 eV, while indirect gap energy starts from 3.90 eV towards 3.87 eV [17]. In addition, by reducing the band gap energy of thin layers, the performance of photocatalysts in these layers as semiconductor materials can be improved [15].

A comparison of the band gap values of direct and indirect TiO<sub>2</sub>:Co thin films can be seen in the following table 1.

**Table 1**

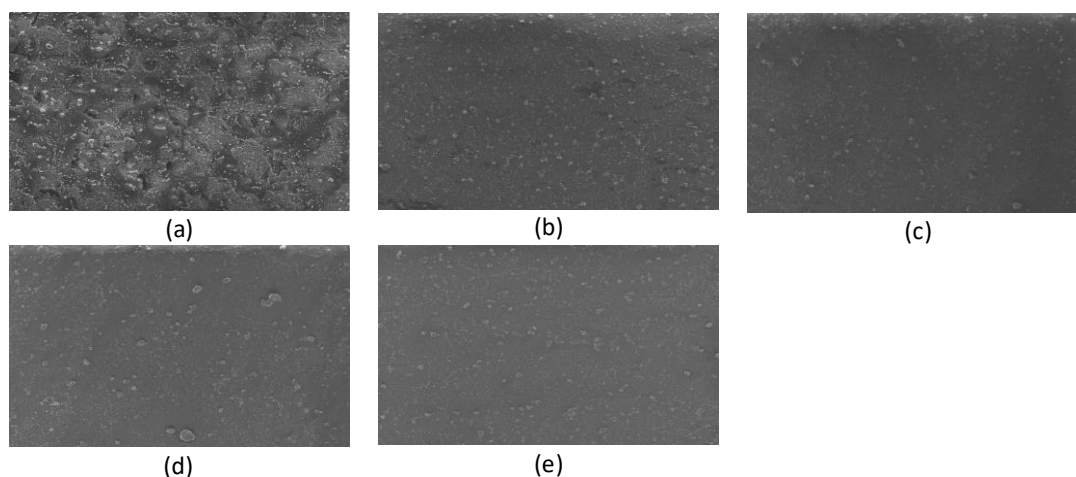
Comparison of the direct, indirect, and activation energy band gaps of TiO<sub>2</sub>:Co

Doping concentration TiO <sub>2</sub> :Co	Energy band gap (eV)		Activation energy (eV)
	Direct	Indirect	
0%	3.44	3.86	2.86
5%	3.39	3.85	2.69
10%	3.38	3.84	2.42
15%	3.32	3.85	2.28
20%	3.22	3.82	2.26

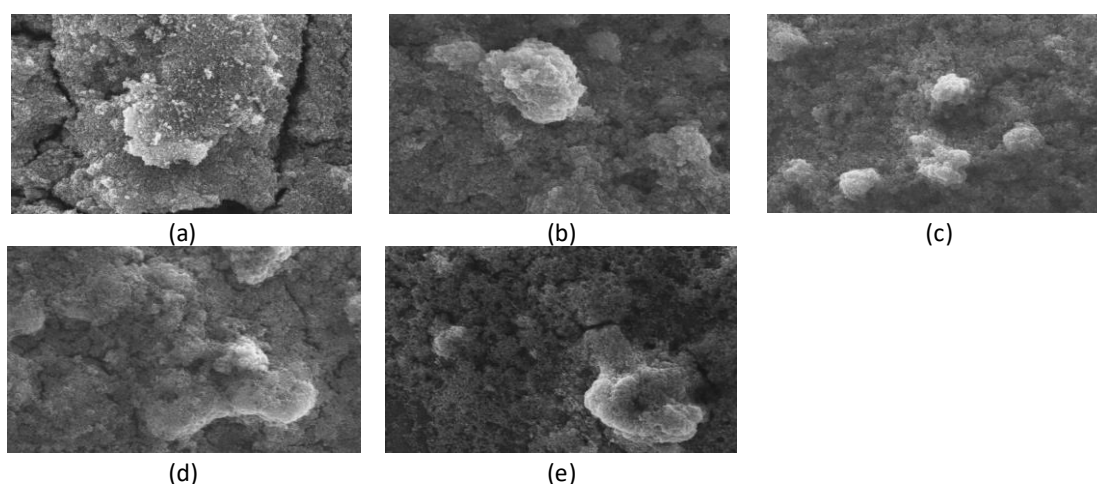
In addition to obtaining the energy band gap value, the next indicator obtained from the data analysis process of the absorbance value of the thin film is the activation energy value of the TiO<sub>2</sub>:Co thin film. Activation energy is the energy required for a chemical reaction to occur in a thin film sample [35,36]. The smaller the activation energy, the easier it will be for a compound to react with other compounds. It can be seen in Table 1 that there is an effect of the concentration of the addition of cobalt doping, where the higher the percentage of doping used, the lower the activation energy of the thin layer sample.

### 3.3 Morphological Characterization and Thin Film Composition Components TiO<sub>2</sub>:Co

The appearance of the morphology of TiO<sub>2</sub>:Co thin films can be done by a surface scanning process using a Scanning Electron Microscope (SEM). SEM analysis provides a two-dimensional picture of the material by utilizing high resolution. The morphology of the TiO<sub>2</sub>:Co thin film from the measurements can be seen in Figures 7 and 8 as follows.



**Fig. 7.** Surface morphology of  $\text{TiO}_2\text{:Co}$  thin film with 100x magnification at concentrations of (a) 0%, (b) 5%, (c) 10%, (d) 15%, and (e) 20%

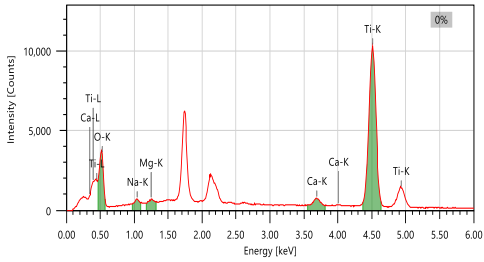
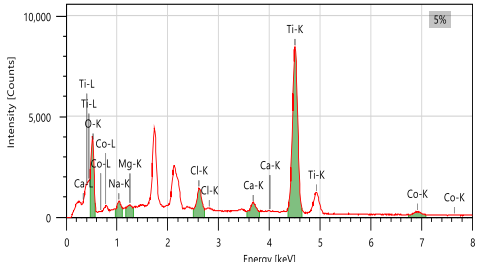
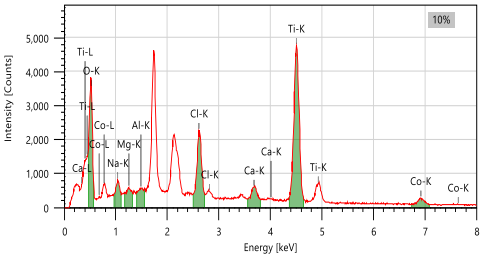
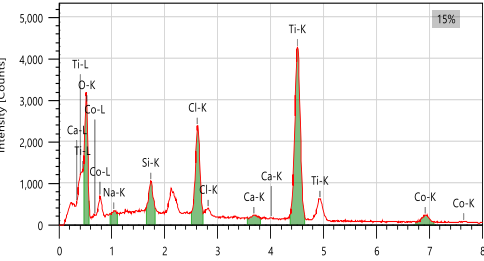
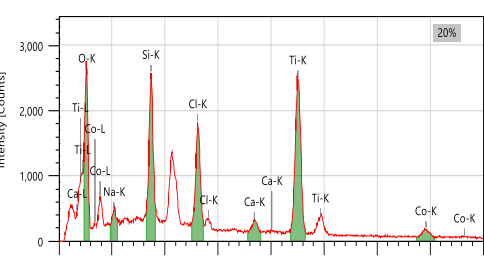


**Fig. 8.** Surface morphology of  $\text{TiO}_2\text{:Co}$  thin film with 5000x magnification at concentrations of (a) 0%, (b) 5%, (c) 10%, (d) 15%, (e) 20%

Based on Figure 7, it can be seen that the morphology of the  $\text{TiO}_2\text{:Co}$  thin film at 100x magnification changes towards a denser and more homogeneous direction as the concentration of cobalt doping is increased. It can be seen in Figure 7(a) that the morphology of the  $\text{TiO}_2$  thin layer sample without doping was obtained and that the sol-gel solution was not evenly distributed on the surface of the glass substrate. In contrast to the morphological appearance of the  $\text{TiO}_2$  thin layer after being given cobalt doping, the sol-gel solution was deposited evenly on almost all surfaces of the glass substrate. This can happen because cobalt as a dopant material functions to dissolve  $\text{TiO}_2$  compound precursors so that they are homogeneous during the deposition process by utilizing centrifugal force in a modified centrifuge device.

Meanwhile, based on Figure 8(a) for 5000x magnification, the  $\text{TiO}_2$  thin layer without cobalt doping has an irregular shape, a broken texture, an inhomogeneous size, and a brighter color. In contrast to Figure 8(e), the thin layer of cobalt-doped  $\text{TiO}_2$  with the highest concentration shows that the arrangement is almost uniform, there is no significant difference in size, and it looks darker. This indicates that the  $\text{TiO}_2\text{:Co}$  thin layer sample has a high absorption rate, which is supported by the high absorbance value in the previous discussion.

**Table 2**  
 EDX analysis results of TiO<sub>2</sub>:Co

Concentration TiO <sub>2</sub> :Co	Mass (%)				Compiler composition chart
	TiO <sub>2</sub>	CoO	SiO <sub>2</sub>	Cl	
0%	95.94±0.86	-	2.15±0.56	1.70±0.12	
5%	88.33±0.87	4.12±0.28	5.07±0.21	2.70±0.08	
10%	76.89±1.03	7.48±0.47	5.42±0.19	7.60±0.17	
15%	73.14±1.07	8.11±0.51	4.36±0.21	9.16±0.20	
20%	64.48±1.11	19.21±0.56	6.85±0.48	9.18±0.23	

EDX characterization was carried out to determine the various elemental contents contained in the TiO<sub>2</sub>:Co thin film sample. Based on the test results, there were at least four elements or compounds that were found to have the highest percentage level in the TiO<sub>2</sub>:Co thin layer samples, including TiO<sub>2</sub> (precursor base material), CoO (doping agent), SiO<sub>2</sub> (glass substrate content), and Cl (solvent), as shown in Table 2.

The two elements or compounds that appeared, namely  $\text{TiO}_2$  and  $\text{CoO}$ , indicated that the thin film samples already contained precursor and doping, as expected by the researchers, which were obtained through a deposition process using a modified centrifuge. Meanwhile, two other elements or compounds with a high percentage that appeared, namely  $\text{SiO}_2$ , indicated that there was still a surface of the glass substrate that had not been deposited perfectly or evenly, while the Cl element was the residual result of the heating process, which was sourced from the solvent used, namely ethanol ( $\text{C}_2\text{H}_5\text{OH}$ ) and hydrochloric acid (HCl). The appearance of the percentage of  $\text{SiO}_2$  and Cl in the thin film synthesis process is a natural thing because it is closely related to the substrate used and the dissolution process between the precursor and doping materials [37-40]. So it is very likely that these two elements/compounds were found through the SEM-EDX test process.

If seen in Table 2, it can be seen that the greater the percentage of doping concentration used, the higher the percentage of  $\text{CoO}$  elements, which causes a decrease in the percentage of  $\text{TiO}_2$ . This is in line with the treatment carried out by researchers, where the greater the amount of dopants used, the smaller the amount of precursor material. In addition, the high percentage of  $\text{TiO}_2$  and  $\text{CoO}$  indicates that the deposition process on the substrate using the spin-coating method provides [41-44] a great opportunity for the sol-gel solution to adhere to the surface of the glass substrate. The bonding process between the solution and the substrate is influenced by the adhesion force, which is strengthened by utilizing the centrifugal force, which will spread the sol-gel solution along the surface of the glass substrate [45].

#### 4. Conclusions

The process of synthesis and characterization of  $\text{TiO}_2$  thin films with cobalt doping has been carried out. The results of the UV-Vis spectrophotometer test showed that the energy bandgap values of the  $\text{TiO}_2\text{:Co}$  thin films for doping concentrations (0, 5, 10, 15, 20)% were 3.44; 3.39; 3.38; 3.32; 3.22 eV for the direct energy bandgap and 3.86; 3.85; 3.84; 3.85; 3.82 eV for the indirect energy gap. The activation energy of the  $\text{TiO}_2\text{:Co}$  thin film decreased from 2.86 to 2.26 eV. As for the morphological data of the  $\text{TiO}_2\text{:Co}$  thin film, the results of the SEM-EDX test showed that the greater the doping concentration, the layer was deposited evenly, finely, and homogeneously. Regarding the data on the components of the thin layer compounds, it was found that the percentage of  $\text{TiO}_2$  content was in the range of 64.48–95.94%, while for Co, it was in the range of 0.00–19.21%.

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