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Fabrication of PVA/TiO_2 Nanofibers by Electrospinning Method as Photocatalytic Degradation

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ABSTRACT

The nanofiber fabrication process was optimized to determine the effect of stress variations on the morphology and diameter of the nanofibers produced for the absorption of methylene blue dye in water. PVA/TiO_2 nanofibers were made using the electrospinning method. The nanofiber fabrication process begins by dissolving 10% PVA with the addition of 0.05 g of TiO_2 nanoparticles, the collector tip distance is 15 cm with varying voltages of 12, 15, and 18 kV. PVA/TiO_2 nanofiber characterization was carried out using FTIR, SEM, EDS, and UV-Vis Spectrophotometer. The results showed success in forming PVA/TiO_2 nanofibers. The nanofibers show a fibrous structures with a diameter of 252 nm for PVA and 237 nm for PVA/TiO_2 with a uniform surface and contained TiO_2 nanoparticles scattered within the nanofibers. The photocatalytic testing within 5 hours shows that PVA nanofibers can degrade up to 39.29% of the methylene blue dye, while TiO_2 powder is up to 55.48%. Hence, the PVA/TiO_2 nanofibers can degrade the dye up to 97.02%. The results of good transparency on the UV-Vis Spectrophotometer test are shown in the visible light region caused by the uniform distribution of TiO_2 nanoparticles in the nanofiber.

Introduction

Living conditions have improved significantly in recent years, which has also led to an increase in the demand for water for human and industrial consumption. Sufficient water is water that meets health requirements, i.e., water that is clear, colorless, and odorless. With the growing activity in various industries, the number and diversity of chemical contaminants in various waters are increasing every year [1]. With minimal impact on the environment, wastewater is one of the solutions to restore it to viable water. Wastewater use and recycling are proposed for sustainable water supply to cities [2]. Many ways have been developed to recycle wastewater, for example, physically, namely sedimentation and adsorption, chemically, using coagulants but producing sludge. Previous researchers have also carried out conventional methods, but of the existing techniques, the easiest method to apply is photocatalytic.

Photocatalytic is an effective method in separating pollutant compounds, decomposing water, and air as well as decomposition of organic pollutants because photocatalytic has advantages such as having strong oxidation-reduction properties, stability to light and chemical bonds that are not soluble in water [3]. The photocatalytic process occurs when semiconductor particles in a liquid or gas medium are exposed to UV light either from the sun or from a UV lamp, it will produce a pair of electrons and holes. This electron and hole pair will diffuse to the surface of the semiconductor particle and cause the oxidation and reduction of pollutants in the medium.

The most common semiconductor material used as a photocatalyst is TiO_2. The advantages of TiO_2 over other semiconductor materials are non-toxicity, relatively low cost, excellent chemical stability, high thermal stability, and high photocatalytic activity [4]. Various forms of TiO_2 nanomaterials have been prepared including nanoparticles, nanotubes, nanofibers, and nanosheets [5]. Not only in UV light but in visible light and fluorescent light, the photocatalytic activity of nanofibers containing TiO nanoparticles can occur [6]. The presence of a catalyst can purify water and eliminate odors. In addition, photocatalyst can reduce the TDS value in organic wastewater by 44.08%, BOD by 73.44%, and COD by 71.21% [7]. After the photocatalytic, cleaning process can cause new problems, one of which is the deposition of TiO_2 particles that cannot be separated from the waste after the degradation process. So that by making nanofibers synthesized by electrospinning method for photocatalytic processes is a solution to this problem.

Electrospinning is a method of using a power source to form nanometer or micrometer-sized thin lines (nanofibers) from a liquid [8]. Electrospinning is a very flexible technique because variations in electrospinning parameters can produce different morphologies and diameters [9]. Among the parameters that affect the process of forming nanofiber membranes are the properties of the solution which include molecular weight, concentration, viscosity, elasticity, electrical conductivity, and surface tension. Then, the process parameters include the high voltage, the distance between the syringe to the collector, and the pressure of the syringe pump. Then control parameters which include temperature and humidity [10]. Therefore in this study, optimizing the electrospinning parameter, namely the voltage variation to produce the smallest fiber that is suitable for good and maximum absorption applications.

The electrospinning method produces a nanofiber membrane derived from TiO_2 nanoparticles that have been bonded into a nanofiber composite using Polyvinyl Alcohol or PVA as a fiber template. PVA is soluble in water and has excellent chemical properties (easy to process, non-toxic and biodegradable) PVA can be used for various purposes [11]. PVA is a biocompatible, inexpensive, and non-toxic polymer that is widely used in the biomedical field [12]. In industry, PVA is used in adhesives and coatings, filtration applications, and gas retention applications [13]. The main purpose why TiO_2 is bound into nanofibers is to prevent TiO_2 particles from becoming new pollutants in the water. PVA/TiO_2 composites could degrade dyes in solution by 70% in 5 hours [14], and TiO_2/PVA nanofibers succeeded in degrading Methyl Orange dye by 89.3% for 4 hours [15]. The explanation above is the basis for this research, namely the fabrication of PVA/TiO_2 nanofibers using the electrospinning method with variations in voltage to observe its effect on the diameter size and uniformity of the fibers formed as an application for the absorption of methylene blue dye in water.

Experimental Method

This study refers to previous research [16]. The composition of the solution was prepared with a concentration of 10% PVA and TiO_2 of 0.05 g. There is a difference in this study, namely the electrospinning parameter. By manipulating the voltage of 12, 15, and 18 kV. Meanwhile, other parameters were controlled such as the distance of the syringe to the collector of 15 cm and the flow rate of 1 ml/hour.

Preparation of PVA/TiO_2 Solution

The fabrication of nanofibers begins with making a solution of PVA/TiO_2 by dissolving 1 g of PVA into 10 ml of distilled water, stirring using a magnetic stirrer at a temperature of 100°C. Then 0.05g of TiO_2 particles were added and mixed until a homogeneous solution was formed.

Preparation of PVA/TiO_2 Nanofiber

The electrospinning begins with coating the collector drum with aluminum foil which is covered with glass preparations. PVA/TiO_2 solution was inserted into the syringe. After that setting up the electrospinning parameters. The polymer solution passes through the spinneret attached to the syringe. The polymer leaving the spinnerate always forms nanofibers due to the electrostatic forces generated by the charged particles due to the high voltage between the spinnerate and the collector as shown in Figure 1. The results in the form of nanofibers that were then characterized by using Fourier Transform Infrared (FT-IR), Scanning Electron Microscope (SEM) EDS, and UV-Vis Spectrometer.

Photocatalytic activity in degrading methylene blue by PVA/TiO_2 nanofibers was observed under UV light (365 nm) as a light source shown in Figure 2. The reaction was carried out with PVA/TiO_2 nanofibers placed in a beaker containing 10 ppm methylene blue solution (10 mL). The photocatalytic activity of methylene blue was observed every 1 hour, 3 hours, and 5 hours to show that the PVA/TiO_2 nanofibers succeeded in degrading the methylene blue solution.

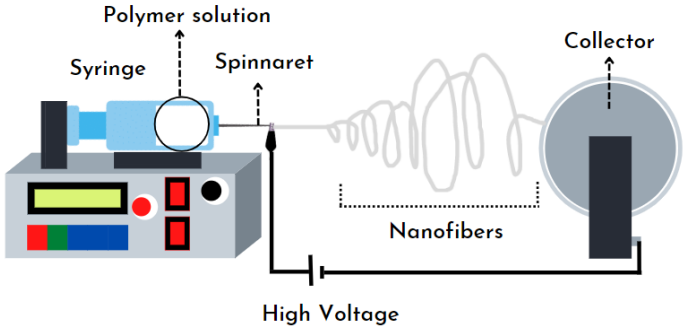

Figure 1. Research Design

Result and Discussion

The results of the fabrication of PVA/TiO_2 nanofibers with voltage variations of 12 kV, 15 kV, and 18 kV using an Optical Microscope are shown in Figure 3 where it is clear that the formation of fibers, beads that appear indicate the presence of TiO_2 compounds dispersed in solution. Nanofiber fabrication is strongly influenced by several parameters, one of which is voltage. The voltage used in electrospinning is 7-32 kV [17]. Fiber formation is affected by the output voltage which is above the threshold, because high voltage can cause rotational instability from taylor cone shape to random shape of several nozzles. Thus, surface tension is the main cause of droplet formation after an unsuccessful electrospinning process, this is related to spinning conditions that are too dilute, or the concentration of the solution [13]. There is a decrease in the average fiber diameter from 60 to 350 nm and 70 to 230 nm with an increasing applied electric field (0.8 and 1.0 $kV.cm^{-1}$) [18], the spinneret distance parameter to the collector can also decrease the fiber diameter between 70-150 nm with a distance (of 10 cm and 12 cm) [19].

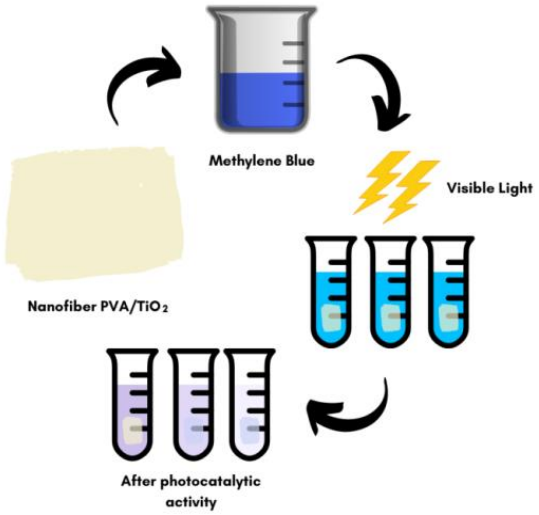

Figure 2. Photocatalytic Mechanism

The greater the voltage used, the smaller the fiber diameter, and if the applied voltage is small, the resulting fiber diameter becomes larger as shown in Table 1. The fiber diameters are 0.67, 0.59, and 0.53µm, respectively. The decrease in nanofiber diameter caused by the increase in high voltage can shorten the solution time from the tip of the syringe to the collector [17].

The electrostatic force is not strong enough to attract the droplets which are offset by the surface tension. So that the drip deformation is not completely attenuated. The elongation of the jet causes the jet to become thinner, resulting in a reduction in the diameter of the fiber. The viscosity of the solution can optimally balance the electrostatic force and electric field. So that the resulting fiber is continuous. The application of a high voltage that is too high with a collector nozzle distance that is too close causes a corona discharge. Corona discharge is an electrical phenomenon caused by an excessive electric field between two electrodes [20].

Table 1. Voltage Variations Used in the Synthesis of PVA/TiO_2 Nanofibers

Voltage (kV)	Syringe to collector distance (cm)	Diameter of nanofiber (µm)

12	15	0.67
15	15	0.59
18	15	0.53

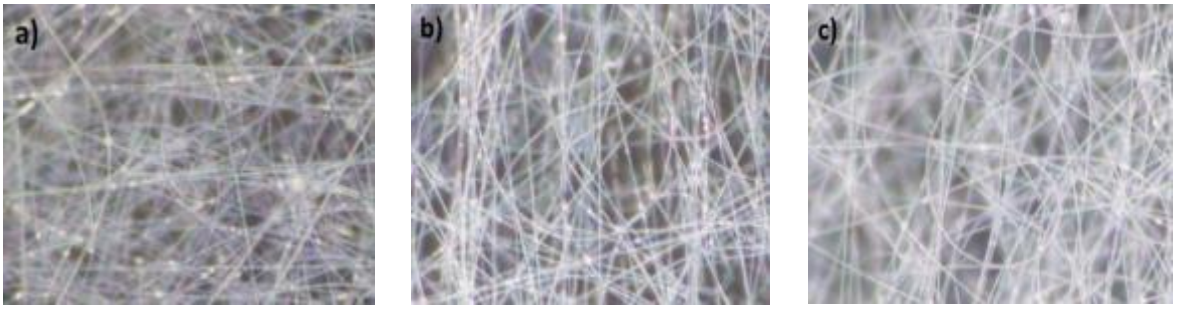

Figure 3. Optical Microscopy Results from Nanofiber (a) 12 kV PVA/TiO_2, (b) 15 kV PVA/TiO_2 and (c) 18 kV PVA/TiO_2

Characterization using FT-IR was used to determine the functional groups formed from TiO_2 particles bound to PVA to form nanofibers as shown in Figure 4. The functional groups that appear include O-H, C-H, CH_2, C-O, C-C, and Ti-O-Ti. The stretching of the O-H group occurred in the range of 3000-3800 cm^{-1} [14]. The results of the study occurred at a wavenumber of 3275 cm^{-1}. O-H symmetry strain vibration occurs at a wavenumber of 3336 cm^{-1} [16]. The vibration of the C-H group occurs at 2910 cm^{-1}, while the asymmetrical vibration of the C-H occurs at a wavenumber of 2900 cm^{-1} [16]. The vibration of the CH_2 group occurs at a wavenumber of 1430 cm^{-1} [21]. The results of the study occurred at 1420 cm^{-1}.

The peak at wavenumber 1331 cm^{-1} indicates the C-H group. C-H group vibrations occur at 1330 and 1377 cm^{-1} [14] and occur at 1375 cm^{-1} [21]. The absorption bandwidth at 1078 cm^{-1} indicates the presence of the C-O strain. Vibration at a wavenumber of 849 cm^{-1} indicates a C-C PVA strain [21], while in the study it occurred at 857 cm^{-1}. For the new wideband around 419-731 cm^{-1} is an indication of the Ti-O-Ti group. Ti-O-Ti vibrations occur at wavenumbers 400-600 cm^{-1} [21] and occur at 438 cm^{-1} [16].

Table 2. Comparison of FTIR Results with References.

Peak (cm^{-1})		Functional Groups	Reference
Experiment	**Data Base**		
3275	3200-3550	O-H stretching	Nasikhudin et al, 2017
	3336	Symmetric stretching vibration O-H	Kurian et al, 2019
2910	2910	C-H Streching	Nasikhudin et al, 2017
	2900	C-H Asymmetric	Kurian et al, 2019
1420	1430	CH_2 Vibration	Lou et al, 2020
1331	1330 dan 1377	C-H Group	Nasikhudin et al, 2017
	1375	C-H Bending	Lou et al, 2020
1078	1095	C-O Streching	Nasikhudin et al, 2017
857	849	C-C Streching of PVA	Lou et al, 2020
731	550-800	Ti-O-Ti Band	Nasikhudin et al, 2017
419	400-600	Ti-O-Ti Group	Lou et al, 2020
	438	Ti-O-Ti Bonding	Kurian et al, 2019

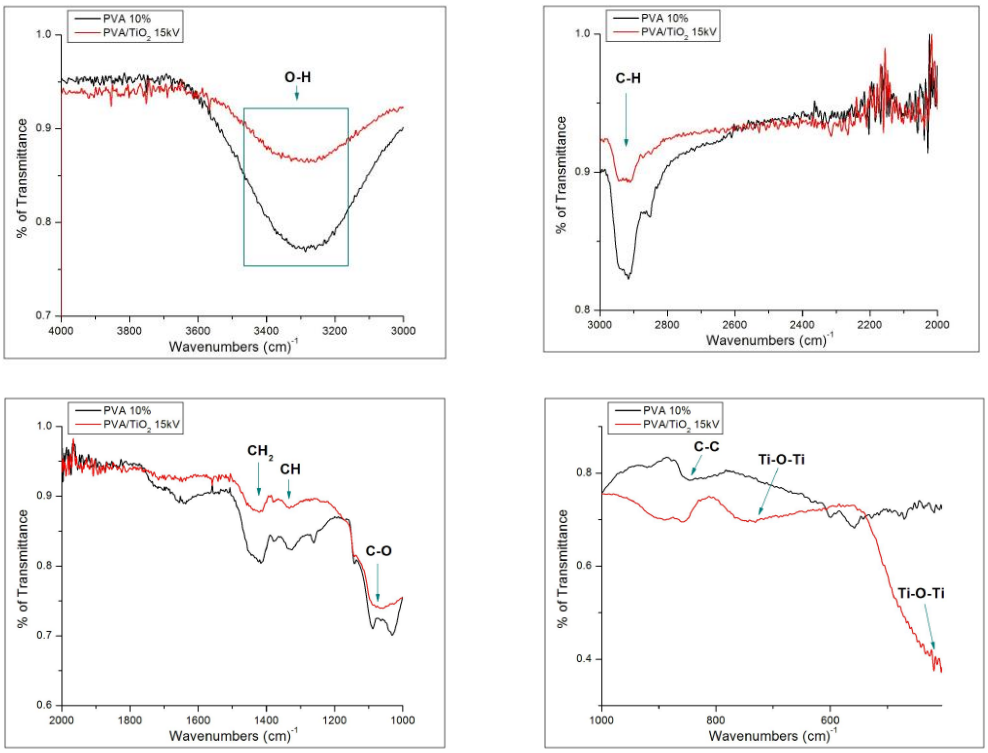

Figure 4. FTIR spectrum of PVA/TiO_2 nanofibers with wavelengths from 4000-400 cm-1

The arrangement of PVA and PVA/TiO_2 nanofibers is shown in Figure 5 using SEM wherein the nanofibers look uniform, clear, and smooth. There is an aggregate of TiO_2 nanoparticles in certain parts of the fiber which shows a good distribution. The dot is uneven on the fiber surface but jumps. The effect of the repulsive force between TiO_2 particles caused by the electric field separates the particles from one another. The presence of a PVA polymer that binds TiO_2 particles causes the particles to do not separate but instead form a collection of TiO_2 particles at certain points along with the fiber [22].

The average diameter of nanofibers from PVA is 252 nm, while that of PVA/TiO_2 is 237 nm as shown in the histogram of Figure 6. The diameter of the fibers decreases as the TiO_2 composition increases [23]. Thus, the addition of TiO_2 is known to reduce fiber diameter. The viscosity of the solution decreases while the conductivity of the solution increases due to the presence of TiO_2 particles. Reduction of fiber diameter is very useful in increasing the active surface area to increase photocatalytic activity [14].

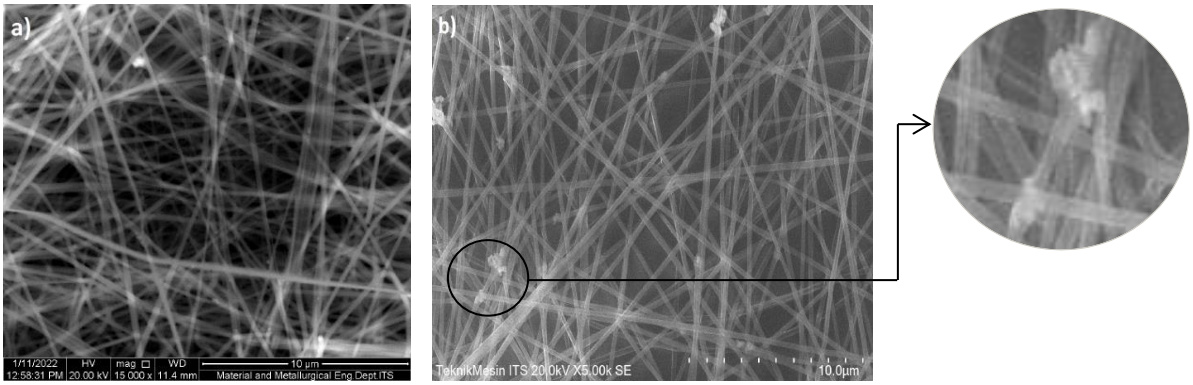

Figure 5. SEM images of (a) PVA Nanofibers and (b) PVA/TiO_2 Nanofibers

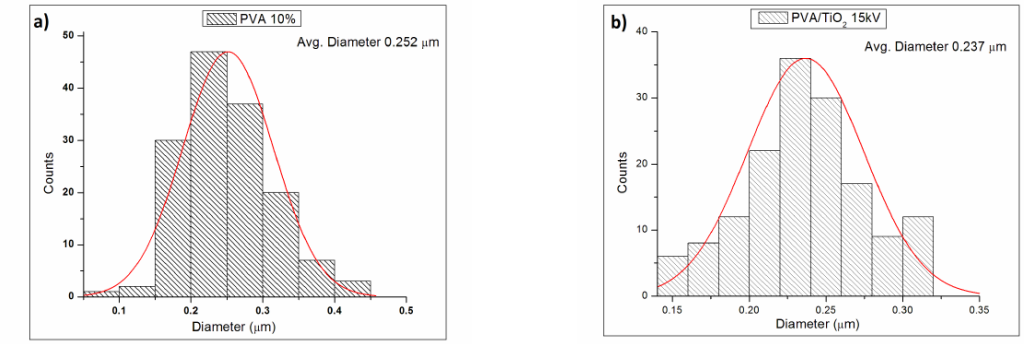

Figure 6. Average Fiber Diameter of (a) PVA Nanofibers and (b) PVA/TiO_2 Nanofibers

EDS was used to investigate the elemental composition of PVA/TiO_2 nanofibers as shown in Figure 7. The results showed that the TiO_2 nanoparticles were charged on the PVA surface and spread in the direction of the fiber direction generated due to polarization and orientation through a high electric field in the electrospinning method [21]. Elemental analysis showed that TiO_2 nanoparticles was distributed in the fiber, the elemental ratios of C, O, and Ti were 13.51, 84.91, and 1.58%, respectively, in the entire area of PVA/TiO_2 nanofibers as shown in Table 3. This proves that the nanofibers contain TiO_2.

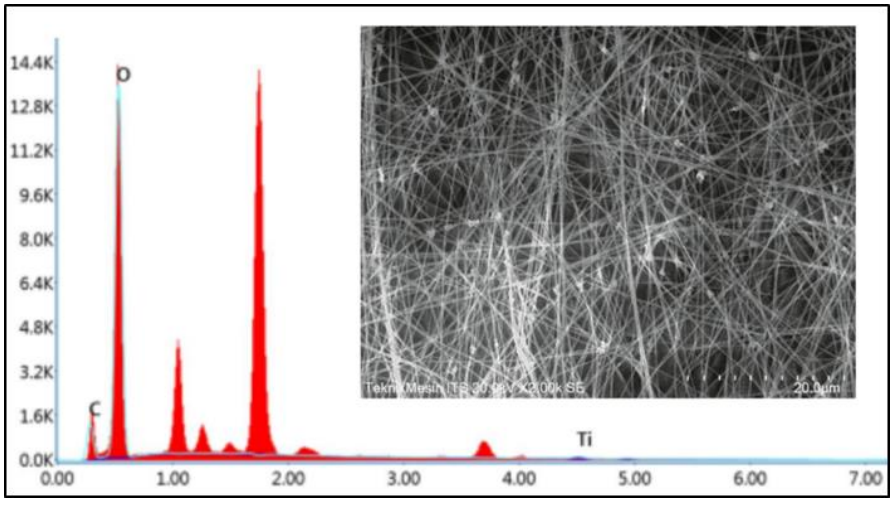

Figure 7. SEM-EDS Image of PVA/TiO_2 Nanofibers

Table 3. EDS Profile of PVA/TiO_2 Nanofibers

Element	Weight (%)	Atomic (%)
C	13.51	17.40
O	84.91	82.09
Ti	1.58	0.51

Characterization UV-vis spectrometer was used to determine the absorbance value of PVA/TiO_2 nanofibers after photocatalytic activity by using UV light (365 nm). In the visible light region, which is in the wavelength range of 600-700 nm, it shows good transparency results shown in Figure 8. This is due to the presence of TiO_2 nanoparticles that are evenly distributed in the matrix. Even distribution is often hindered by Van der Waals forces between nanoparticles, which causes aggregation [24]. PVA nanofibers in photocatalytic activity succeeded in removing methylene blue dye by 39.29%, and TiO_2 powder up to 55.48%, within 5 hours. Meanwhile, PVA/TiO_2 nanofiber was able to degrade methylene blue in 1 hour by 39.29%, 3 hours by 92.17%, and 5 hours by 97.02%. The photocatalytic activity of PVA/TiO_2 nanofibers with sonication treatment increased by 60%, and if surfactant was added it increased by 80% within 5 hours [25]. The photocatalytic activity of nanofibers increases with the length of irradiation time because more electrons are excited and h^+ is formed. The more h^+, the more hydroxyl radicals play a role in oxidizing the dye [26]. The longer the irradiation time also causes more photon energy to be absorbed by the photocatalyst, so the damage to the dye increases because the catalyst in the reactor experiences higher radiation and is more activated. As in this study, within 5 hours PVA/TiO_2 nanofibers can absorb almost all dyes. This can also be caused by the addition of TiO_2 to the nanofibers causes the diameter to become smaller and denser so that the nanofibers can absorb optimally.

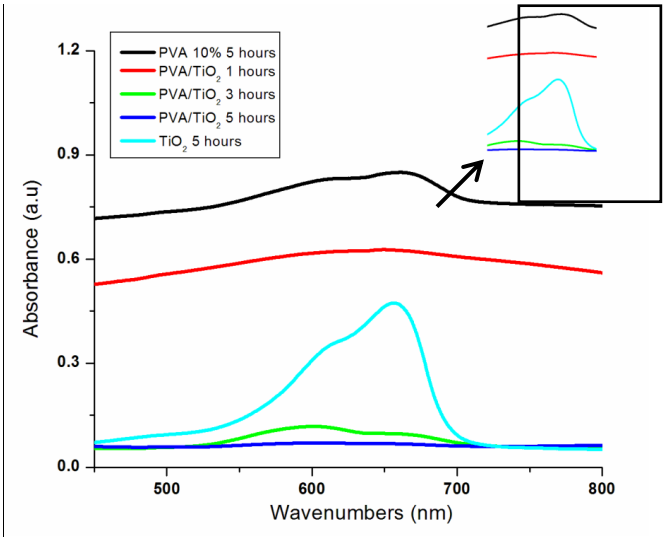

Figure 8. UV-vis Spectra After Photocatalytic Activity for 1, 3 and 5 Hours by PVA, PVA/TiO_2 Nanofibers and TiO_2 Powder

The bandgap energy is obtained from the function of the Kubelka-Munk equation with the photon energy as shown in Figure 9. The bandgap calculation for PVA/TiO_2 nanofibers is listed in Table 4. The TiO_2 particle powder has an energy band gap of 3.22 eV, which means

TiO_2 particles is in the anatase phase. TiO_2 particles for the anatase phase generally have bandgap energy of 3.2 eV which is equivalent to light having a wavelength of 290 nm, namely middle ultraviolet (UV) light [27]. TiO_2 particles have several phases including anatase, rutile, and brookite. TiO_2 anatase has been widely used as an effective photocatalyst [17]. TiO_2 for anatase phase is more photoactive than rutile phase [28]. Meanwhile, PVA/TiO_2 nanofibers that have been treated with photocatalytic activity for 1 hour, 3 hours, and 5 hours have bandgap energies of 3.87, 3.92, and 3.96 eV, respectively. It can be seen that the bandgap increases with the length of time variation of the photocatalytic activity. There is a slight shift in bandgap energy indicating that the shape of the fiber requires slightly more energy to excite the electrons, where the energy gap for pure PVA is 4.98 eV [29],6.27 eV [30] this is because the energy gap facilitates the transition of electrons from the band valence to the conduction band. In addition, the shift in bandgap energy can also be caused by the effect of PVA polymer coating that affects the density of charge carriers, the presence of power losses due to diffusion, and changes in the structure of TiO_2 particles [31].

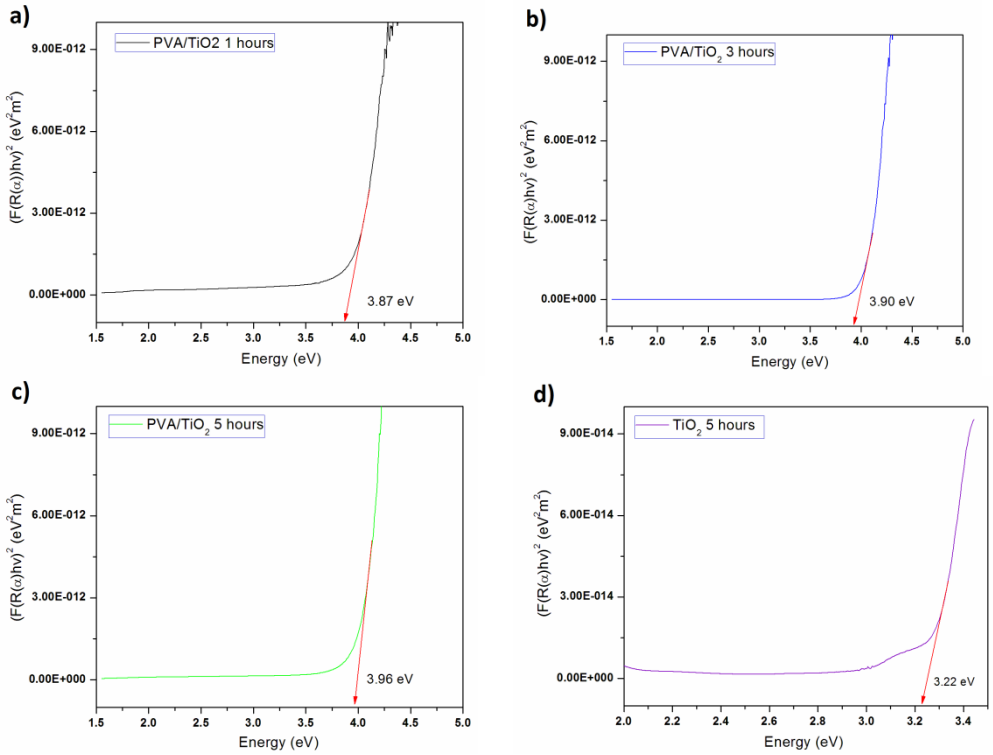

Figure 9. (a-c) Bandgap Energy of PVA/TiO_2 Nanofibers and (d) Bandgap Energy of TiO_2 Powder After Photocatalytic Activity

Table 4. Bandgap Energy of PVA/TiO_2 Nanofibers

Sample	% of Transmission	Absorbance	Bandgap value (eV)
PVA/TiO_2 1 hours	83.16	0.6300	3.87
PVA/TiO_2 3 hours	32.53	0.1203	3.92
PVA/TiO_2 5 hours	20.33	0.0732	3.96
PVA 5 hours	89.10	0.8515	-
TiO_2	87.15	0.4740	3.22

Conclusion

PVA/TiO_2 nanofibers were made by the electrospinning method which was used to decompose methylene blue dye. Nanofibers formed from variations in voltage indicate that the higher the voltage used, the smaller the fiber diameter. The nanofibers show a fibrous structures with a diameter of 252 nm for PVA nanofibers and 237 nm for PVA/TiO_2 nanofibers. The photocatalytic testing within 5 hours shows that PVA nanofibers can degrade up to 39.29% of the methylene blue dye, while TiO_2 powder is up to 55.48%. Hence, the PVA/TiO_2 nanofibers can degrade the dye up to 97.02%.

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