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# Synthesis of PVA/TiO<sub>2</sub> Composite Layer over Conductive Textile Sheet Using Electrospinning Method for Enhancing Self-Cleaning Properties

Lisa Samura<sup>1</sup>, Daffa Rizky Febrian<sup>2</sup>, Cahaya Rosyidan<sup>1</sup>, Mustamina Maulani<sup>1</sup>, Mentari Gracia, Soekardy<sup>1</sup>, Valentinus Galih Vidia Putra<sup>2</sup>, Sigit Dwi Yudanto<sup>3</sup>, Ferry Budhi Susetyo<sup>4</sup>

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**ABSTRACT:** The present research used the electrospinning method to apply a polyvinyl alcohol/titanium dioxide (PVA/TiO2) layer over a conductive textile (70 % polyester and 30 % cotton) sheet. PVA with 10, 12.5, and 15 g concentrations was mixed into 100 ml distilled water. Then, each PVA solution was mixed with 1.5 wt.% of TiO2. Afterward, the electrospinning method applied a PVA/TiO2 composite onto a conductive textile sheet. Various characterizations were conducted, such as resistivity, scanning electron microscopy (SEM), Fourier transforming infrared (FTIR), and photocatalytic activity. The resistivity result is 9.5, 10, and  $10 \Box$  for A, B, and C samples. According to SEM investigation, higher PVA concentration leads to higher fiber sizes around 0.65  $\mu$ m. An increase in PVA content does not affect the bands that were formed. The size of the fiber diameter contributed to the photocatalytic activity of MB. A smaller fiber diameter could enhance photocatalytic activity.

KEYWORDS: Conductive textile, Electrospinning, FTIR, PVA/TiO2 composite, Photocatalytic, SEM.

#### INTRODUCTION

The textile industry has a global market of around USD 1 trillion. Polyester and cotton dominated the textile industries due to relatively low cost and outstanding performance [1]. According to Islam et al., textiles comprising 70 % polyester and 30 % cotton have a tensile strength of 74.34 kg [2]. Unfortunately, textiles with characteristic self-cleaning are limited. Therefore, textiles or materials with characteristic self-cleaning have received more attention from researchers. That material could be embedded into a textile sheet; hence, textile properties could be enhanced to be self-cleaning characteristics.

Superhydrophobic criteria could enhance the self-cleaning characteristic. Chen et al. modified silk to have superhydrophobic properties using the enzim-etching method [3]. Thorvaldsson et al. have modified the nanofiber-coated microfiber to have superhydrophobic properties using a plasma technique [4]. Moreover, the polyvinyl alcohol/titanium dioxide (PVA/TiO2) composite is also a promising candidate for self-cleaning characteristics, as shown by dye degradation. Liu et al. fabricated transparent PVA/TiO2 composite with TiO2 composition 5, 10, 20, 30 and 40 wt.% and found increasing TiO2 content in the composite led to a higher degradation of methyl orange (MO) [5].

PVA/TiO2 composite could fabricated using casting [6], sol-gel [7], and electrospinning [8]. The electrospinning method is more precise for embedding onto a textile sheet due to several advantages, such as a high surface ratio to volume, adapted porosity, and various resulting shapes and sizes [9]. Nasikhudin et al. fabricated PVA/TiO2 using electrospinning and followed crosslinked in 2.5% Glutaraldehyde solution, resulting in PVA/TiO2 being more degraded of methylene blue (MB) than TiO2 [10]. Khan et al. Fabricating PVA/TiO2 using electrospinning and crosslinked in the HCL for 60, resulting in degraded MB around 83% at 3 h [11]. Varied PVA in the PVA/TiO2 composite needs further investigation. Therefore, the present research would vary the composition of PVA and then mixed with TiO2. Afterward, PVA/TiO2 composite coating was fabricated using the electrospinning method over the conductive textile sheet. PVA/TiO2 composite is not crosslinked to reduce the time and cost of fabrication. Due to its cost-effectiveness, the conductive textile sheet was prepared using manual screen printing [12]. Various characterizations of the PVA/TiO2 composite were conducted, such as scanning electron microscopy (SEM), Fourier transforming infrared (FTIR), and photocatalytic activity. Moreover, the conductive textile sheet resistivity properties were also investigated.

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#### **EXPERIMENTAL METHODS**

#### Material

Consist of 70 % polyester and 30 % cotton ( $10 \square 10$  cm) were used as a conductive sheet substrate. Polyvinyl alcohol (PVA), TiO2, Methylene blue (MB), Polyacrylic binder, Alco printing, and copper-silver powder (70Cu30Ag) were used in the present research and are industrial grade.

#### Fabricated and characterization conductive sheet

Mix 6 g of alco printing with 94 g of distilled water and stir it properly. Prepare 15 g of mixed alco printing, 10 g of polyacrylic binder, 10 g of copper-silver powder, then mix and stir properly. Afterward, print onto 70 % polyester and 30 % cotton sheets using manual screen printing. Dry the formed conductive sheet at  $100 \square C$  for 2 min and cure at  $160-180 \square C$  for 2 min. Afterwards, the resistivity of the conductive sheet was measured using a Sanwa CD 800 A multimeter.

#### Synthesis and characterization of PVA/TiO2

PVA with 10, 12.5, and 15 g concentrations was mixed into 100 ml of distilled water separately, then heated at 40 □C and stirred for around 2 h. Afterward, each PVA solution was mixed with 1.5 wt.% of TiO2, heated at 40 □C, and stirred for around 2 h. Composited sample with composition 10PVA/1.5TiO2, 12.5PVA/1.5TiO2 and 15PVA/1.5TiO2 wt. % were designated as A, B, and C. Electrospinning was conducted in various PVA/TiO2 compositions over a conductive sheet for 3 min using 2.25 kV. The distance between the needle tip and the conductive sheet was around 5.5 cm. Afterward, the coating sample was investigated using SEM JEOL JSM 6360 LA and FTIR Prestige 21 Shimadzu. According to the SEM result, origin software was used to examine the average diameter of the fiber.

Degradation of the MB was investigated using single GINGA UV light (GA-7W-UV) with specifications of 7 watts and 220240V 50Hz/60Hz for photocatalytic activity. The distance between the sample and UV light was fixed to around 10 cm. MB degradation was conducted in 1, 2, and 3 h of irradiation times.

#### RESULT AND DISCUSSION

#### SEM Resistivity Test

Figure 1 shows the resistivity test result of various conductive textile sheets. The conductive textile sheet was prepared using manual screen printing. The resistivity measurement result is 9.5, 10, and  $10 \square$  for A, B, and C samples, respectively. Besides the material conductivity, the alloy's composition is one factor that results in various resistivity. Hsieh and Hung et al. found Increasing Cu content in the sputtered CuAg alloy (as deposited) from 40 to 80 at. % led to an increase in resistivity [12]. Moreover, Liu et al have found that Cu-24 wt. % Ag has les conductivity than Cu-6 wt. % Ag and Cu-12 wt. % Ag [13].





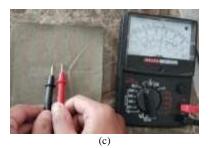


Figure 1. Resistivity test (a) A sample, (b) B sample, and (c) C sample.

#### **SEM**

An SEM investigation determined the fiber size according to increasing PVA content. Figure 2 shows SEM and frequency distribution results of various layers.

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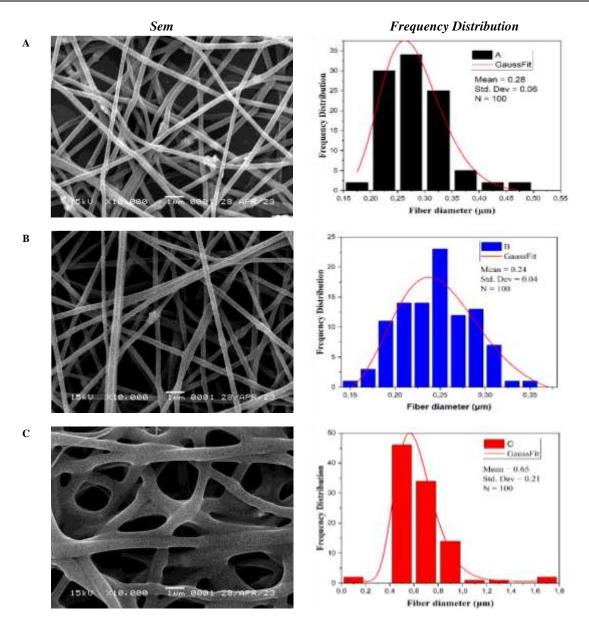


Figure 2. SEM and frequency distribution of various sample layers.

The average diameter of the fiber sample based on 100 spot measurements is 0.28, 0.24, and 0.65  $\mu m$  for the A, B, and C samples, respectively. Shift to more PVA content leads to higher fiber diameter. Montallana et al. found an increase in PVA content from 8 to 12 wt. % in PVA/TiO<sub>2</sub>, resulting in a wider fiber diameter due to the viscosity of the PVA/TiO<sub>2</sub> [8]. Those results are in line with the present study.

#### **FTIR**

FTIR investigations were conducted in the various samples to examine the bond of the coatings. Figure 3 shows the FTIR result of different composite.

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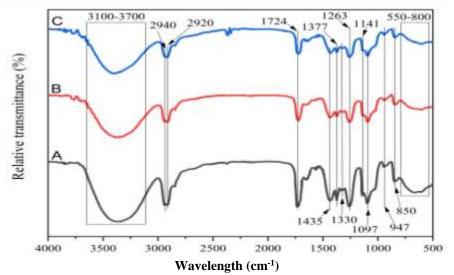
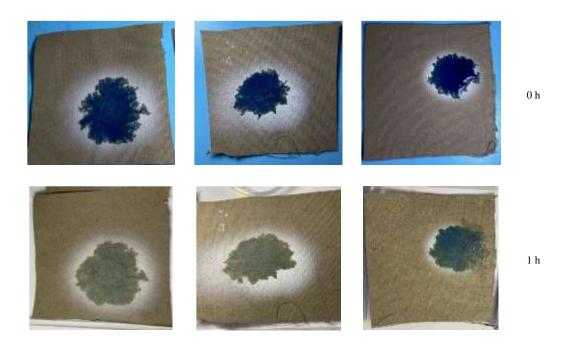


Figure 3. FTIR result.

According to Figure 3, bands were formed could be following explained. 3100-3700 cm<sup>-1</sup> band is associated with OH stretching [14]. 2940 cm<sup>-1</sup>band is related to the methylene group [15]. 2920 cm<sup>-1</sup> band associated with C-H stretching vibrating [16]. 1720 cm<sup>-1</sup> band assignment to C=O bond [17]. 1435 cm<sup>-1</sup> band related to the C=O bond [18]. 1330 and 1377 cm<sup>-1</sup> bands are related to the C-H group [10]. 1263 cm<sup>-1</sup> band corresponding to C-N stretching [19]. 1141 cm<sup>-1</sup> band is associated with the relative crystallinity of PVA [20]. 1097 cm<sup>-1</sup> band is associated with the acetal group [15]. 947 cm<sup>-1</sup> band related to the COH group [21]. 850 cm<sup>-1</sup> is the skeletal vibration of PVA [6]. 550-800 cm<sup>-1</sup> band attributed to Ti-O-Ti band [10]. According to FTIR, it can be concluded that an increase in PVA content does not affect the bands that were formed.

#### Photocatalytic activity

Figure 4 shows the visual observation of MB degradation results using UV light for various composites.



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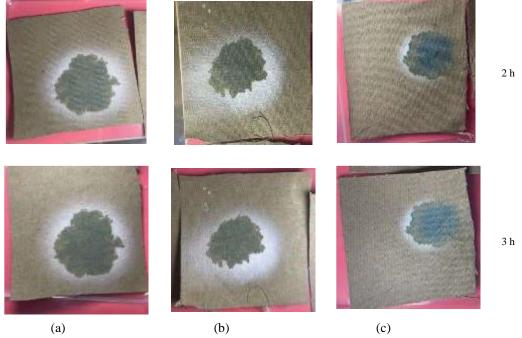


Figure 4. Photocatalytic activity for 1, 2, and 3 h for (a) A sample, (b) B sample, and (c) C sample.

According to Figure 4, it can be seen that the C sample until 3 hours has a more difficult MB to degrade, probably due to the 0.65  $\mu m$  diameter according to SEM investigation. On the contrary, the A and B samples have behavior that is easy to MB degradation at one first hour. This behavior is probably due to the lower fiber diameter, around 0.28 and 0.24  $\mu m$ , for the A and B samples. According to Montallana's study, a smaller fiber diameter leads to a higher surface area-to-volume ratio. This characteristic is desirable for photocatalytic activity [8]. Therefore, this statement is that corroborated samples A and B have higher photocatalytic activity to MB degradation.

Nashikhudin et al. have stated that increasing the time duration of PVA/TiO<sub>2</sub> composited nanofiber led to more MB dye removal. At 3 hours of exposure time using  $4 \Box 10$  W UV light source, MB was removed around 45 % [10]. Khan et al. stated that PVA/TiO<sub>2</sub> could remove MB dye at 3 hours for 83% using light intensity 1000 W/m<sup>2</sup> [11].

#### CONCLUSION

 $PVA/TiO_2$  coating over conductive textile sheets has been wholly synthesized. Higher PVA content resulted in higher fiber size due to viscosity. An increase in PVA content does not affect the bands that were formed. A  $10PVA/1.5TiO_2$  and  $12.5PVA/1.5TiO_2$  have better MB degradation or self cleaning than  $15PVA/1.5TiO_2$  due to the smaller diameter of the fiber.

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# Synthesis of PVA/TiO2 Composite Layer Over Conductive Textile Sheet Using Electrospinning Method

by Lisa Samura FTKE

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#### ABSTRACT

The present research used the electrospinning method to apply a polyvinyl alcohol/titanium dioxide (PVA/TiO2) layer over a conductive textile (70 % polyester and 30 % cotton) sheet. PVA with 10, 12.5, and 15 g concentrations was mixed into 100 ml distilled water. Then, each PVA solution was mixed with 1.5 wt.% of TiO2. Afterward, the electrospinning method applied a PVA/TiO2 composite onto a conductive textile sheet. Various characterizations were conducted, such as resistivity, scanning electron microscopy (SEM), Fourier transforming infrared (FTIR), and photocatalytic activity. The resistivity result is 9.5, 10, and 10  $\Omega$  for A, B, and C samples. According to SEM investigation, higher PVA concentration leads to higher fiber sizes around 0.65  $\mu$ m. An increase in PVA content does not affect the bands that were formed. The size of the fiber diameter contributed to the photocatalytic activity of MB. A smaller fiber diameter could enhance photocatalytic activity.

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Varied PVA in the PVA/TiO2 composite needs further investigation. Therefore, the present research would vary the composition of PVA and then mixed with TiO2. Afterward, PVA/TiO2 composite coating was fabricated using the electrospinning method over the conductive textile sheet. PVA/TiO2 composite is not crosslinked to reduce the time and cost of fabrication. Due to its cost-effectiveness, the conductive textile sheet was prepared using manual screen printing [12]. Various characterizations of the PVA/TiO2 composite were conducted, such as scanning electron microscopy (SEM), Fourier

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#### EXPERIMENTAL METHODS

Material

Consist of 70 % polyester and 30 % cotton ( $10 \times 10$  cm) were used as a conductive sheet substrate. Polyvinyl alcohol (PVA), TiO2, Methylene blue (MB), Polyacrylic binder, Alco printing, and copper-silver powder (70Cu30Ag) were used in the present research and are industrial grade.

Fabricated and characterization conductive sheet

Mix 6 g of alco printing with 94 g of distilled water and stir it properly. Prepare 15 g of mixed alco printing, 10 g of polyacrylic binder, 10 g of copper-silver powder, then mix and stir properly. Afterward, print onto 70 % polyester and 30 % cotton sheets using manual screen printing. Dry the formed conductive sheet at 100°C for 2 min and cure at 160-180 °C for 2 min. Afterwards, the resistivity of the conductive sheet was measured using a Sanwa CD 800 A multimeter.

Synthesis and characterization of PVA/TiO2

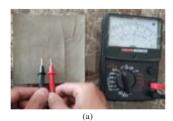
PVA with 10, 12.5, and 15 g concentrations was mixed into 100 ml of distilled water separately, then heated at 40 °C and stirred for around 2 h. Afterward, each PVA solution was mixed with 1.5 wt.% of TiO2, heated at 40 °C, and stirred for around 2 h. Composited sample with composition 10PVA/1.5TiO2, 12.5PVA/1.5TiO2 and 15PVA/1.5TiO2 wt. % were designated as A, B, and C. Electrospinning was conducted in various PVA/TiO2 compositions over a conductive sheet for 3 min using 2.25 kV. The distance between the needle tip and the conductive sheet was around 5.5 cm. Afterward, the coating sample was investigated using SEM JEOL JSM 6360 LA and FTIR Prestige 21 Shimadzu. According to the SEM result, origin software was used to examine the average diameter of the fiber.

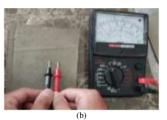
Degradation of the MB was investigated using single GINGA UV light (GA-7W-UV) with specifications of 7 watts and 220-240V 50Hz/60Hz for photocatalytic activity. The distance between the sample and UV light was fixed to around 10 cm. MB degradation was conducted in 1, 2, and 3 h of irradiation times.

#### RESULT AND DISCUSSION

SEM Resistivity Test

Figure 1 shows the resistivity test result of various conductive textile sheets. The conductive textile sheet was prepared using manual screen printing. The resistivity measurement result is 9.5, 10, and 10  $\Omega$  for A, B, and C samples, respectively. Besides the material conductivity, the alloy's composition is one factor that results in various resistivity. Hsieh and Hung et al. found Increasing Cu content in the sputtered CuAg alloy (as deposited) from 40 to 80 at. % led to an increase in resistivity [12]. Moreover, Liu et al have found that Cu-24 wt. % Ag has les conductivity than Cu-6 wt. % Ag and Cu-12 wt. % Ag [13].





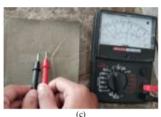


Figure 1. Resistivity test (a) A sample, (b) B sample, and (c) C sample.

SEM

An SEM investigation determined the fiber size according to increasing PVA content. Figure 2 shows SEM and frequency distribution results of various layers.

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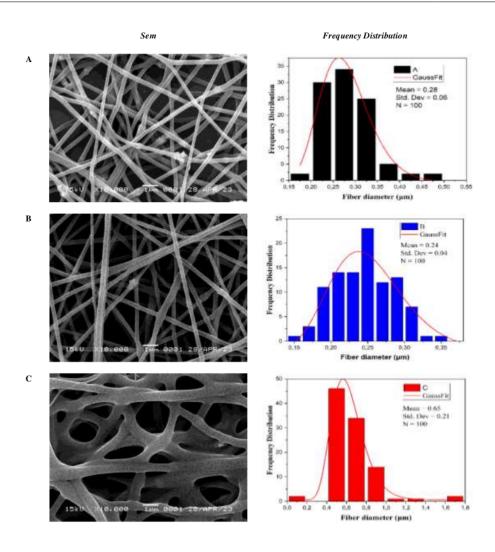


Figure 2. SEM and frequency distribution of various sample layers.

The average diameter of the fiber sample based on 100 spot measurements is 0.28, 0.24, and 0.65  $\mu$ m for the A, B, and C samples, respectively. Shift to more PVA content leads to higher fiber diameter. Montallana et al. found an increase in PVA content from 8 to 12 wt. % in PVA/TiO<sub>2</sub>, resulting in a wider fiber diameter due to the viscosity of the PVA/TiO<sub>2</sub> [8]. Those results are in line with the present study.

#### FTIR

FTIR investigations were conducted in the various samples to examine the bond of the coatings. Figure 3 shows the FTIR result of different composite.

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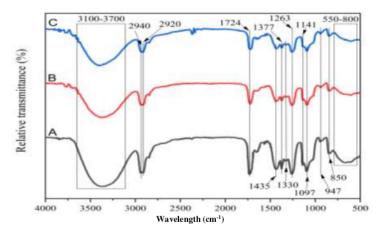
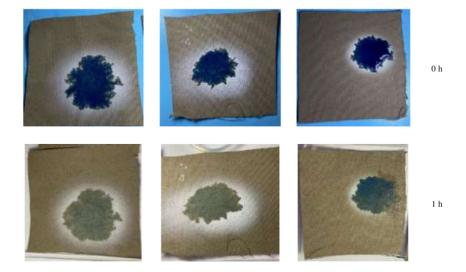


Figure 3. FTIR result.

According to Figure 3, bands were formed could be following explained. 3100-3700 cm<sup>-1</sup> band is associated with OH stretching [14]. 2940 cm<sup>-1</sup>band is related to the methylene group [15]. 2920 cm<sup>-1</sup> band associated with C-H stretching vibrating [16]. 1720 cm<sup>-1</sup> band assignment to C=O bond [17]. 1435 cm<sup>-1</sup> band related to the C=O bond [18]. 1330 and 1377 cm<sup>-1</sup> bands are related to the C-H group [10]. 1263 cm<sup>-1</sup> band corresponding to C-N stretching [19]. 1141 cm<sup>-1</sup> band is associated with the relative crystallinity of PVA [20]. 1097 cm<sup>-1</sup> band is associated with the acetal group [15]. 947 cm<sup>-1</sup> band related to the C-OH group [21]. 850 cm<sup>-1</sup> is the skeletal vibration of PVA [6]. 550-800 cm<sup>-1</sup> band attributed to Ti-O-Ti band [10]. According to FTIR, it can be concluded that an increase in PVA content does not affect the bands that were formed.

#### Photocatalytic activity

Figure 4 shows the visual observation of MB degradation results using UV light for various composites.



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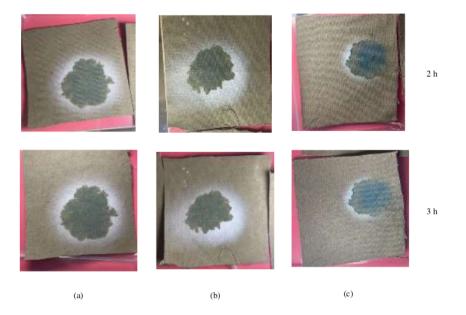


Figure 4. Photocatalytic activity for 1, 2, and 3 h for (a) A sample, (b) B sample, and (c) C sample.

According to Figure 4, it can be seen that the C sample until 3 hours has a more difficult MB to degrade, probably due to the 0.65  $\mu$ m diameter according to SEM investigation. On the contrary, the A and B samples have behavior that is easy to MB degradation at one first hour. This behavior is probably due to the lower fiber diameter, around 0.28 and 0.24  $\mu$ m, for the A and B samples. According to Montallana's study, a smaller fiber diameter leads to a higher surface area-to-volume ratio. This characteristic is desirable for photocatalytic activity [8]. Therefore, this statement is that corroborated samples A and B have higher photocatalytic activity to MB degradation.

Nashikhudin et al. have stated that increasing the time duration of  $PVA/TiO_2$  composited nanofiber led to more MB dye removal. At 3 hours of exposure time using  $4 \times 10$  W UV light source, MB was removed around 45 % [10]. Khan et al. stated that  $PVA/TiO_2$  could remove MB dye at 3 hours for 83% using light intensity 1000 W/m² [11].

#### CONCLUSION

PVA/TiO<sub>2</sub> coating over conductive textile sheets has been wholly synthesized. Higher PVA content resulted in higher fiber size due to viscosity. An increase in PVA content does not affect the bands that were formed. A 10PVA/1.5TiO<sub>2</sub> and 12.5PVA/1.5TiO<sub>2</sub> have better MB degradation or self cleaning than 15PVA/1.5TiO<sub>2</sub> due to the smaller diameter of the fiber.

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