The Physical Properties of Polyethylene/Nanotitania Composites as Thickness Function

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Abstract. *Physical properties of polyethylene (PE)/ nanotitania composite as a function of thickness have been studied. The material used is PE and nanotitania. The blending of PE with nanotitania was carried out by melting polyethylene at 95 °C for 5 minutes then added nanotitania 5 wt% and 10 wt% and heated at 124 ° C for 30 minutes. Then printed using barcoater with size 6, 12 and 25 µm with four repetitions. Variations were performed to determine the effect of composite thickness of polyethylene/nanotitania on electrical properties and functional groups. FTIR results show that as the thickness of the absorption sample increases, new functional groups appear, namely hydroxyl and carbonyl. The LCR results show the smallest conductivity value, sample 5 wt% PE / nanotitania with 6 µm size and the largest 10% PE/nanotitania sample with a size of 25 µm each at 2,302 x 10^{-5} S/m and 3,121 x 10^{-5} S/m at a frequency of 10^5 Hz. While the largest dielectric constant value is a sample of PE/nanotitania 5 wt% with a size of 6 µm and the smallest sample PE/nanotitania 10 wt% with a size of 6 µm at a frequency of 10^5 Hz. Based on these characteristics, the 5 wt% PE/nanotitania sample with a size of 6 µm is the material with the best dielectric properties so that the sample has the potential to be used as a cable insulator.*

Key words. *composite, PE, TiO_2, conductivity and dielectric constant.*

INTRODUCTION

Polyethylene (PE) is a thermoplastic which is a chemical polymer material that is widely used in daily life. Polyethylene at low temperatures is flexible and resistant to chemicals. Because it is used for various purposes including the manufacture of various containers, kitchen utensils, various small items, bottles, films, pipes, electrical cable insulators, fibers, trash bags etc. [1].

Polyethylene is a typical non-polar polymer that has good electrical properties. Especially very good in typical high frequency properties, widely used as insulation materials for radar, TV and as a communication tool. because of its unusual shape, chemical and electrical components. Furthermore, the mechanics and keelectricity of these polyethylene components such as range power, inhibition, permittivity and dielectric reduction, can be increased by adding inorganic nanoparticles due to their small size and surface effects. Inorganic nanoparticles/polyethylene/nanocomposite are used as opportunities for future industrial needs [2]–[4]. Of the various types of nanomaterials, titanium dioxide (TiO_2) or nanotitania is one of the ingredients being developed [5].

Recently, TiO_2 has been used as a filler attached to polymer matrices and electronic equipment [6]. TiO_2 is an anisotropic material used as a significant differentiator, with parallel and perpendicular dielectric permittivity [7]. The research of [8] shows the permittivity and reduction of dielectric components in nanocomposite from TiO_2/micro and TiO_2 epoxy/nano. It was reported that permittivity of TiO_2 epoxy/micro was wider than epoxy/nano TiO_2 and polymer matrix, while permittivity of epoxy/nano TiO_2 was smaller than polymer matrix and epoxy/micro TiO_2.

Nanotechnology is the understanding and control of material on the nanoscale, which is a unique phenomenon that allows for new applications [9]. Many commercially produced nanomaterials are nanosilica, nanotitania, nanoalumina, fullerenes and carbon nanotubes (CNTs). Of the various types of nanomaterials, TiO2 or nanotitania is one of the ingredients being developed [5].

Based on the research of Liu et al., (2017) TiO_2 rutile nanoparticles were chosen as fillers attached to the LDPE matrix with different mass concentrations (0.5 wt%, 1wt%, 3 wt%, and 5 wt%) and thickness of 150 µm heated in a vacuum oven at 90oC, LDPE/TiO2 has the lowest permittivity and dissolution factor due to good nanoparticle dispersion, this shows that LDPE/TiO_2 samples have sophisticated dielectric components and significant anti aging capabilities. Therefore, it is very necessary to conduct further research on the effect of the thickness of polyethylene/nanotitania on physical properties. To find out the functional groups of materials were tested by the characterization of Fourier Transform Infrared (FTIR), and LCR meter as a measure of the electrical properties of the layer of polyethylene-nanotitania.

EXPERIMENTS

This study uses basic materials of polyethylene (PE), and TiO_2 nanoparticles. The tools used in this study are digital balance sheets, measuring cups, spatulas, aluminum foil, tissue, barcoater, blender, plastic press and oven. Making polyethylene-nanotitanium is done by mixing polyethylene and nanotitania. The first step is, polyethylene is melted first using an oven at 95 °C for 5 minutes. Then melted polyethylene is added to the nanotitanium. After that, stir it using a blender. Variations in the composition of the six samples are shown in **Tabel 1.**

Tabel 1. Variation in sample composition of PE / nanotitania.

Sample	Comparison of PE (gr) with nanotitania	Thickness (µm)
A6	1 : 5	6
A12	1 : 5	12
A25	1 : 5	25
B6	1 : 10	6
B12	1 : 10	12
B25	1 : 10	25

The results of the mixture of polyethylene-nanotitanium were printed using a barcoater to produce layers with a size of 6 µm, 12 µm, and 25 µm. The coating process is repeated four times at 124 °C for 30 minutes at each size of the barcoater.

After that the polyethylene/nanotitania samples were characterized using FTIR in the frequency range 400-4000 cm^{-1} and LCR Hi Tester type Hioki 3520-52. Characterization was carried out in order to be able to analyze the functional groups and electrical properties of polyethylene/nanotitania composites. To calculate the electrical conductivity and real dielectric permittivity or dielectric constant can use the equation:

$$\sigma = \frac{GL}{A} \qquad (1)$$

$$\varepsilon_r = \frac{Cd}{\varepsilon_0 \mathrm{A}} \qquad (2)$$

With σ are electrical conductivity (S/m), G conductance (S), L sample thickness (m), A sample surface area (m^2), ε_r permittivity dielectric, C sample capacitance (F), ε_o vacuum permittivity (8,854 x 10^{-12} F / m), d sample thickness (m) and A sample surface area (m^2).

DISCUSSIONS

The results of making polyethylene / nanotitania

layers with a filler concentration of 5 wt% and 10 wt% using barcoater measuring 6, 12 and 25 µm were repeated four times in each size. The results obtained can be seen in **Table 2** and **Table 3**.

Table 2. Results of sample thickness of titania 5 wt%

Sample	Repetition (times)	Thickness (µm)
A6	4	180
A12	4	210
A25	4	240

Table 3. Results of sample thickness of titania 10 wt%

Sample	Repetition (times)	Thickness (µm)
B6	4	190
B12	4	230
B25	4	250

Based on Table 2, it can be seen that the fourth repetition produces the thinnest size. For PE / nanotitania with 5 wt% nanotitania content for barcoater 6, 12 and 25 µm, the thickness of each of 180, 210 and 240 µm was obtained. In Table 3 shows the results of thickness of PE/nanotitania with a level of 10 wt% nanotitania with barcoater sizes 6, 12 and 25 µm obtained by the thickness of 190, 230 and 250 µm respectively. The results of the coating are not in accordance with the size of the bar coater used. Layer mismatch because the coating process still uses manual barcoater.

FTIR results are used to detect functional groups present in polyethylene and TiO_2 composites. Samples are coded in presentation based on their thickness. The results of the PE/nanotitani sample FTIR spectrum are shown in **Figure 1** with the absorption peaks shown in **Table 4.**

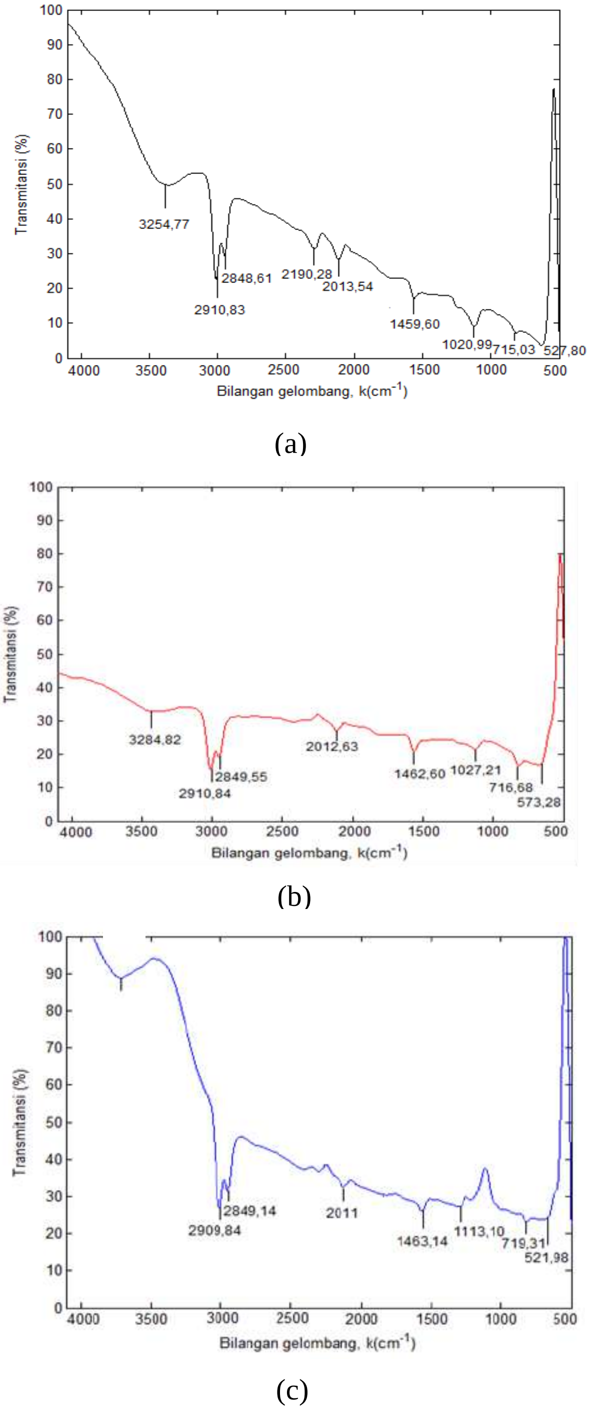
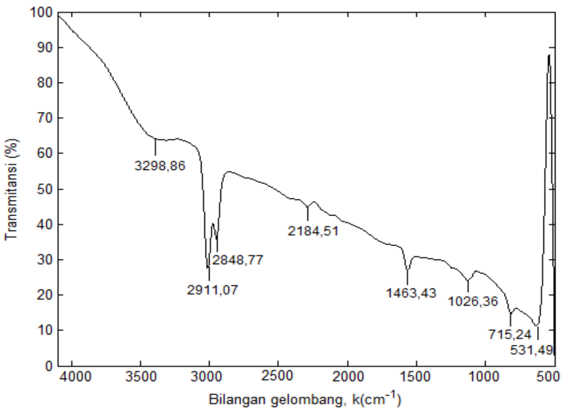


Figure 1. Results of composite FTIR spectra of PE/nanotitania with a mass percentage of 5 wt%. (a) sample A6 (6 µm), (b) sample A12 (12 µm) and (c) sample A25 (25 µm).

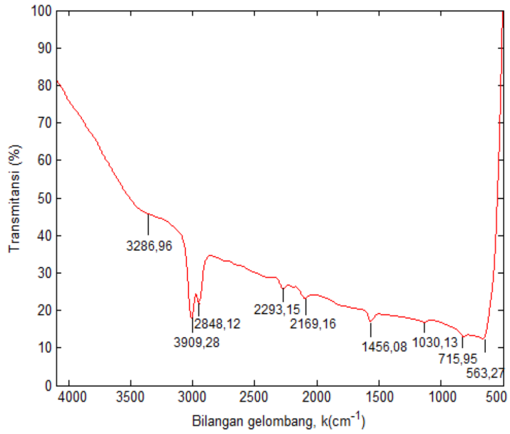
Table 4. Absorption of detected wave numbers

Wavenumber / cm^{-1}	Functional groups
3498-3254	O-H
2910-2800	C-H
2190-2010	C-O
1463-715	CH_2
719-521	TiO_2

Based on **Table 4.** All samples show the same results where each sample produces a functional group C-H at wavenumbers 2910 - 2800 cm^{-1} and CH_2 at wave numbers 1463 - 715 cm^{-1}. These absorption bands are typical polyethylene absorption bands [1] and wavenumber 719-521 cm^{-1} is a vibration of TiO_2. The functional group determination is used to find out the existence of inter-atomic bonds that are expected to be present in the sample. Function group testing is the process of matching wave number absorption values from FTIR Spectroscopy devices with the value of atomic bond vibrations. The absorption value of wave numbers on FTIR is obtained from the amount of energy absorbed by the sample because it has the same energy as the vibrations of the atoms in the sample.The FTIR results in the 10 wt% polyethylene/nanotitania sample are shown in **Figure 2**.



(a)



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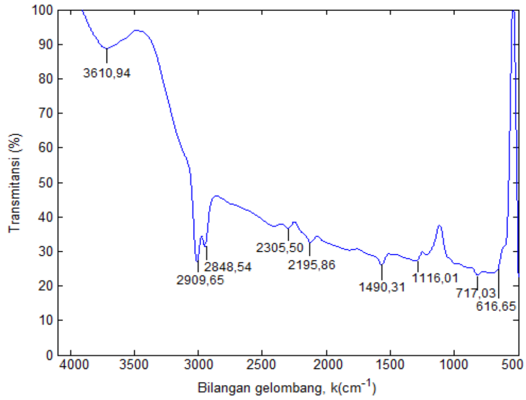


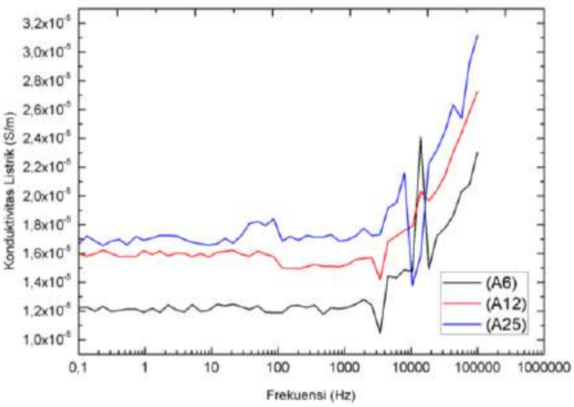
(c)

Figure 2. Results of composite FTIR spectra of PE / nanotitania with a mass percentage of 10 wt%. (a) samples B6 (6 µm), (b) samples B12 (12 µm) and (c) samples B25 (25 µm).

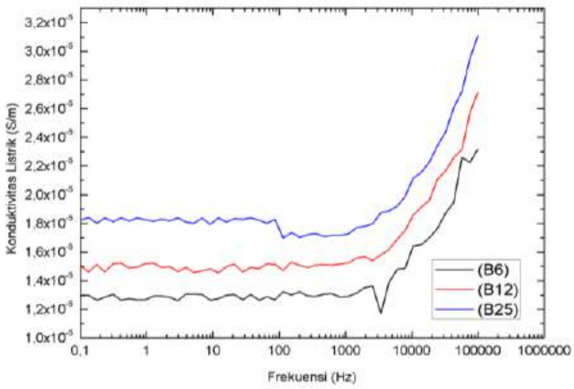
Based on **Figure 2**, the same absorption peaks show that the absorption results show the functional groups C-H and CH_2. Based on the FTIR results in samples with a fi lter of 5 wt% and 10 wt% showed that the absorbance produced was less. The results of FTIR spectrum showed that the emergence of OH functional groups derived from the use of titanium butoxide precursors on TiO_2 and CO which showed that tere had been an interaction between polyethylene and nanotitania due to the oxidation of polyethylene molecule chain chains and branches, so that oxygen gas reacted with carbon atoms or hydrogen atoms attached to the main carbon chain that produces hydroxyl and carbonyl bonds [10].

The LCR results show measurements of electrical conductivity in the frequency range 0.1-105 Hz shown in **Figure 3**. The measurement results show that the electrical conductivity increases with increasing frequency. The electrical conductivity values of samples A25 and B25 increase more sharply as the frequency increases compared to the other samples. Increasing the value of electrical conductivity can be due to the influence of the elements of combining, impurity or imperfections in the crystal, which greatly affects the conductivity of an conductor [11]. Meanwhile, based on the results of FTIR characterization, it shows that the sample contains an O-H functional group which is a water molecule that causes dielectric properties (polarized insulators). Water molecules can affect electrical conductivity. However, as the thickness of the sample increases O-H bond absorption decreases which is confirmed based on the results of the FTIR spectrum in **Figure 1** and **Figure 2**. This is what causes the value of electrical conductivity to increase. In accordance with the research conducted by

[12] that the higher the content of water molecules in the sample the stronger the dielectric properties.



(a)



(b)

Figure 3. Value of electrical conductivity of PE/nanotitania samples, (a) titania filler 5 wt%, (b) titania filler 10 wt%.

In general, the value of the electrical conductivity of all samples obtained in this study reaches a high value that is reaching the level of 10^{-5} S/m at a frequency of 1 x 105 Hz. The highest conductivity value for PE / nanotitania 5 wt% which is equal to 3.11 x 10^{-5} S / m with a thickness of 25 µm seen in **Table 5** and for the largest electrical conductivity value in the sample 10 wt% PE/nanotitania which is 3.12 x 10^{-5} S / m with a thickness of 25 µm as shown in **Table 6**.

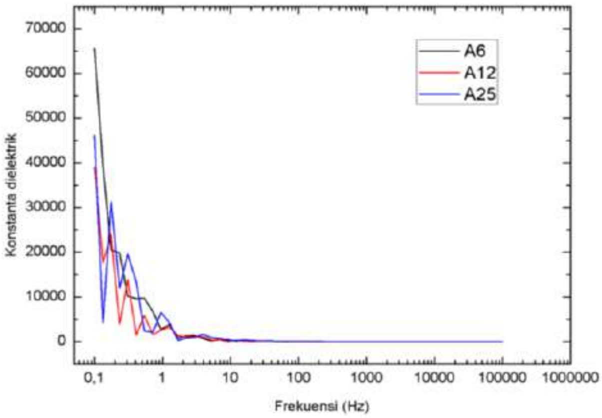
Table 5. Value of electrical conductivity of PE / nanotitania 5 wt%

Sample	Thickness (µm)	Electrical conductivity (S/m)
A6	6	2,30 x 10^{-5}
A12	12	2,72 x 10^{-5}
A25	25	3,11 x 10^{-5}

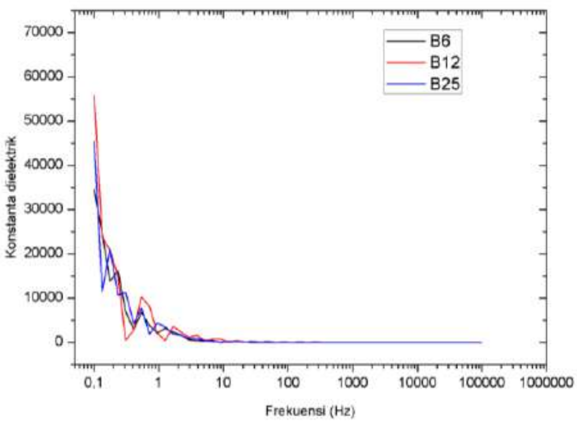
Table 6. Value of electrical conductivity of PE / nanotitania 10 wt%

Sample	Thickness (µm)	Electrical conductivity (S/m)
B6	6	2,31 x 10^{-5}
B12	12	2,71 x 10^{-5}
B25	25	3,12 x 10^{-5}

The measurement results of dielectric constants in the frequency range 0.1-105 Hz are shown in **Figure 4**. Based on the measurement results in both PE/nanotitania samples (5 wt% and 10 wt%) seen at low frequencies, various types of polarization (dipolar polarization, electric charge , ions, and surface polarization) are very easily generated by electric fields. However, at high frequencies only the polarization of electric charge is easily generated by an electric field. Therefore, the dielectric constant value decreases at high frequencies [13], [14]. The characteristics of the dielectric constant obtained in **Figure 4** show the suitability of the electrical conductivity value in **Figure 3**. In accordance with the Hanjitsuwan et al (2011) study, the smaller the dielectric constant value, the greater the electrical conductivity value because polarization decreases.



(a)



(b)

Table 7. Value of PE / nanotitania composite dielectric constant 5 wt%

Sample	Thickness (µm)	Dielectric constant
A6	6	6.563 – 1,57
A12	12	3.911 – 2,89
A25	25	4.632 – 2,62

Table 8. Dielectric constant value of PE / nanotitania 10 wt%

Sample	Thickness (µm)	Dielectric constant
B6	6	3.457 – 1,57
B12	12	5.586 – 2,85
B25	25	4552 – 2,81

The following results from the dielectric constant of PE/nanotitania 5 wt% composites in **Table 7** and the dielectric constant of PE/nanotitania 10 wt% shown in **Table 8**.

Based on **Table 7** and **Table 8**, the thicker the sample the more the dielectric constant decreases. The biggest dielectric constant value is 5 wt% PE/nanotitania with 6 µm size and the smallest 10 wt% PE/nanotitania with 6 µm at 10^5 Hz. According to Liu et al (2017) research, the high dileketik constant values in the sample can show that the sample is a sample that has good anti-aging or anti-aging ability to heat. Based on these characteristics, the 5 wt% PE/nanotitania sample with a size of 6 µm is the material with the best dielectric properties so that the sample has the potential to be used as a cable insulator.

CONCLUSION

Based on the results of FTIR shows that the thicker the sample, the absorption bands are getting lower. In the sample PE/nanotitania with a mass percentage of 5% and 10%, it shows that there are OH and CO bonds which indicate that there has been an interaction betweenpolyethylene and nanotitania due to the oxidation of polyethylene molecule chain chains and branches, so that oxygen gas acts with carbon atoms or a hydrogen atom attached to the main carbon chain that produces hydroxyl and carbonyl bonds. The LCR Results show that the thicker the sample, the higher the conductivity value along with the reduction of O-H group absorption. The smallest conductivity value occurs in the 5 wt% PE/nanotitania sample with a size of 6 µm and the largest in the 10% PE/nanotitania sample with a size of 25 µm each of 2,302 x 10^{-5} S/m and 3,121 x 10^{-5} S/m at a frequency of 10^5 Hz. While the largest dielectric constant value is a sample of PE/nanotitania 5 wt% with a size of 6 µm and the smallest sample PE/nanotitania 10 wt% with a size of 6 µm at a frequency of 10^5 Hz.

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