

The 3rd International Conference on Physical Instrumentation and Advanced Materials (ICPIAM) 2021

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Preface: The 3rd International Conference on Physical Instrumentation and Advanced Materials (ICPIAM) 2021

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Preface: The 3rd International Conference on Physical Instrumentation and Advanced Materials (ICPIAM) 2021

Physics Department – University of Jember, Indonesia, 27 October 2021

The 3rd International Conference on Physical Instrumentation and Advanced Materials (ICPIAM) 2021 is a serial international seminar held as a form of collaboration bi-annual event between the Faculty of Mathematics and Natural Sciences, University of Jember and the Faculty of Science and Technology (FST) Universitas Airlangga (UNAIR).

This conference (ICPIAM 2021) was held as a means for academics, scientists and researchers to discuss, share and exchange information, experiences, methods and research findings as well as the latest innovations at the international level. Through this conference, links will also be formed between researchers and academics to establish cooperation and collaboration both in the fields of education and research internationally.

This conference was attended by 60 presenters who have submitted paper from their studies and researches. They come from Japan, Malaysia, and mostly from Indonesia such as (BPPT, LAPAN, UNAIR, UNEJ, ITB, UNAND, UNCEN, and many more). All the presenters will convey their speech in the 6 different parallel rooms as given in the book of abstract. All presented and reviewed paper will be considered to be published at the AIP Conference Proceedings (Scopus indexed proceedings). We deeply thank the authors for their enthusiastic and high-grade contribution.

The 3rd ICPIAM 2021 would not be possible running without the dedicated efforts of many people especially all organizing committee members who have made planning and organizing the programs. We are grateful to IsDB and Jember University for the funding support, also Physics Student Association (HIMAFI) and volunteers who contributed to the various processes that make up the conference and it would not be possible for me to name them all in this short message.

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Synthesis and characterization of nylon/ TiO₂ hybrid membrane for improving the electrical conductivity and activation energy

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Synthesis and Characterization of Nylon/TiO₂ Hybrid Membrane for Improving the Electrical Conductivity and Activation Energy

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Abstract. Hybrid membrane based on Nylon/with (0, 0.5, 1, 3, 5 and 7) wt.% TiO₂ were synthesized by a sol-gel method. The presence of TiO₂ in the developed membrane possesses some effect on the spectral analysis as observed via FTIR spectroscopy. As interpreted from XRD analysis, nylon/TiO₂ hybrid membrane showed a weakening of the peak intensity of the crystalline nylon and increased intensity of TiO₂ crystals gradually with the addition of TiO₂. It is indicating an increase in the amorphous phase of nylon. Hybrid membrane show enhancing the electrical conductivity by increasing TiO₂ concentration, from $(1.24 \times 10^{-5} \text{ S/cm})$ for pure nylon to $(1.94 \times 10^{-5} \text{ S/cm})$ for 5 wt.% TiO₂. Meanwhile, the activation energy decreased with increasing the TiO₂ concentration, the activation energy was (0.1103, 0.1064, 0.1055, 0.1034, 0.1025, 0.1082) eV for (pure nylon, Nylon/0.5, 1, 3, 5 and 7 wt.% TiO₂). This study points out that the optimum TiO₂ concentration into polymer blend in the hybrid membrane is 5 wt.%.

INTRODUCTION

Membrane technology is widely being utilized in separation applications. Membrane separation processes have become one of the emerging technologies in the last few decades, especially in the separation technology field. Membrane separation processes offer a number of advantages over conventional separation methods, such as separation of components at a lower temperature, ease to operation, minimized damage to the product, less energy use, reduced cost of production, and less wastage [1,2]. These advantage, encourage the development of membrane technology both in the synthesis and fabrication of membranes, gas separation, seawater desalination, membrane distillation and crystallization processes as well as the development of other membrane-based processes [3].

The development of other membrane-based technologies that have gained considerable interest in recent years is organic/inorganic hybrid membranes consisting of a polymer matrix and inorganic nanoparticles, which can be widely applied in various fields including ultrafiltration, pervaporation and direct methanol fuel cells (DMFC) [4,5]. Polymeric membranes are made from either glassy or rubbery materials including polysulfone, polyamide, polyethersulfone, polyvinyl acetate etc. Polymeric membranes possess the inability to withstand chemical degradation and thermal instability, thus limiting their applications when faced with the separation process [6]. Meanwhile, inorganic materials play an important role in improving membrane performance, because of thermal and chemical stability, high flux, resistance to high pressure, good mechanical strength, and longer lifetime [7]. Many types of inorganic nanoparticles have been added to the polymer matrix, such as SiO₂, Al₂O₃, Fe₃O₄, ZnO, ZrO₂, CdS, and TiO₂ [8-14]. Among these inorganic materials, titanium dioxide (TiO₂) has attracted much attention in this field because of its high chemical and thermal stability, resisting and decomposing bacteria, UV resistance, and super hydrophilicity. Moreover, TiO₂ can also create preferential permeation pathways for selective permeation while posing a barrier for undesired permeation in order to improve separation performance [15]. Meanwhile, poly(amide-imide) such as nylon chosen as the polymer matrix material because of its superior mechanical properties and the high thermal stability, solvent resistance, and high permeability [16].

To promote the fine dispersion of the inorganic component in polymers, a simple method is to grow the inorganic phase by a sol-gel process in the polymer solution [17]. Sol-gel is a very flexible route for the synthesis of inorganic, organic-inorganic networks such as glasses, ceramics, films or powders. The combination of inorganic material with a polymer gives the resultant hybrid properties from both materials – the flexibility of the polymer with the strength of the inorganic. Depending on the types of interactions that occur between the materials, better properties than either of the components could emerge. Creating these materials can be as easy as mixing them together in a sol-gel process to produce a homogeneous hybrid [18]. In this paper, inorganic-polymer hybrid membranes from nylon and TiO₂ were prepared by a sol-gel method. This article will focus on the effect of TiO₂ content on the physical properties of nylon and study the electrical conductivity and activation energy of different wt.% TiO₂ at a temperature ranging between 25°C and 95°C.

MATERIAL AND METHODS

Hybrid Membrane Preparation

The nylon/TiO₂ hybrid membrane was prepared by a sol-gel method. TiO₂ was obtained from Merck KGaA, Darmstadt, Germany. The nylon/TiO₂ hybrid membrane was synthesized via an aqueous route. Nylon (6 g) was fully dissolved with 20 mL concentrated HCl (Mallinckrodt Baker, Inc, Paris, Kentucky) and 2 mL of acetone. Then, (0, 0.5, 1, 3, 5 and 7) wt.% TiO₂ was added to the nylon solution and stirred until fully dissolved using a magnetic stirrer. The mixed solutions were stirred for sufficient time to increase homogeneity. The resulting homogeneous mixture was cast on a glass plate and immersed for 10 minutes in distilled water. Finally, the obtained film sample was dried at room temperature overnight.

Based on the addition of the concentration of TiO₂ into the nylon polymer, the following samples were obtained in this study:

- Membrane A: pure nylon membrane
- Membrane B: nylon/0.5 wt.% TiO₂ hybrid membrane
- Membrane C: nylon/1 wt.% TiO₂ hybrid membrane
- Membrane D: nylon/3 wt.% TiO₂ hybrid membrane
- Membrane E: nylon/5 wt.% TiO₂ hybrid membrane
- Membrane F: nylon/7 wt.% TiO₂ hybrid membrane

Furthermore, the sample was characterized using Fourier Transform Infrared (FTIR) and X-Ray Diffraction (XRD). In addition, the electrical conductivity and activation energy were calculated to evaluate the membrane performance.

Characterization of Hybrid Membrane

FTIR Analysis: Fourier Transform Infrared Spectroscopy (FTIR) spectra of the hybrid membranes were recorded between 4000 and 500 cm⁻¹ on Routine Spectrometers. Samples for FTIR were prepared in the form of KBr pellets. The FTIR instrument was used to assess the functional structure of hybrid membrane samples.

XRD: The X-Ray Diffraction (XRD) patterns of hybrid membrane samples were characterized by PANalytical type E'xpert Pro using Cu K α ($\lambda = 1.54060 \text{ \AA}$) radiation. The obtained XRD patterns were analyzed using High Score Plus software and then matched with the International Center Diffraction Database (ICDD) in Powder Diffraction File (PDF). Crystallite size (L_c) measurements included in this study were obtained by X-ray crystallography data and calculated with Scherrer's equation [19]:

$$L_c = \frac{k\lambda}{\beta \cos \theta} \quad (1)$$

where k is the shape coefficient (value between 0.9 and 1.0), λ is the wavelength (1.54 Å), β is full width at half maximum (FWHM) of each phase, and θ is the diffraction angle. Meanwhile, crystallinity is calculated by comparing the area of the crystal curve with the total area of amorphous and crystal [20].

$$\text{crystallinity} = \frac{\text{Broad crystal fraction}}{\text{Broad crystal fraction} + \text{amorphous outer fraction}} \quad (2)$$

Electrical conductivity and Activation Energy: Measurement of the electrical properties of the hybrid membrane using the dual chamber system method. The nylon/TiO₂ hybrid membranes were cut into small discs and put in a chamber. A chamber was filled with a different solution, the first compartment was filled with a (NaCl) salt aqueous solution with a concentration of 4000 ppm, and the second compartment was filled with distilled water in the same volume. The electrical resistance of the nylon/TiO₂ hybrid membranes has been measured using a Lutron 9183 LCR meter as a function of temperature in the range (25 – 95)°C at 10°C interval with the frequency used is 1 kHz. The electrical conductivity of the nylon/TiO₂ hybrid membranes were calculated using the equation below:

$$\sigma = \frac{1}{\rho} = \frac{L}{AR} \quad (3)$$

where σ is electrical conductivity (S/cm), ρ is an electrical resistivity (Ohm.cm), R is a nylon/TiO₂ hybrid membranes resistance (Ohm), L is a thickness of a nylon/TiO₂ hybrid membranes, and A is the cross-sectional area of the electrode (cm²) [21].

The activation energy is obtained by plotting the graph of the relationship between $\ln\sigma$ to $1/T$ on hybrid membrane samples. The slope of the graph is then entered into equation (4) to get the value of the activation energy (E_a). The activation energy values of the nylon/TiO₂ hybrid membranes according to Arrhenius equation:

$$\sigma = \sigma_0 \exp(E_a/k_B T) \quad (4)$$

Where E_a is activation energy (eV), T is the absolute temperature (K), k_B is Boltzmann constant (1.38 × 10⁻²³ J/K) and dividing by the electron charge, k_B = 8.62 × 10⁻⁵ eV, σ_0 is the minimum electrical conductivity at 0 K [22].

RESULTS

FTIR RESULTS

Figure 1 shows the FTIR spectra of nylon/TiO₂ hybrid membranes. FTIR spectra confirmed the formation of nylon/TiO₂ hybrid with network crosslinking. The pure nylon sample (Membrane A) shows the typical primary amide C = O stretching band (1636 cm⁻¹), secondary amide N – H stretching band (3297 cm⁻¹), and secondary amide N – H bending band (1542 cm⁻¹). The peak at 2868 – 2940 cm⁻¹ was assigned to the CH₂ stretching band. Furthermore, the peak at around 1200 cm⁻¹ was assigned to Amide III crystalline band. The presence of these functional groups indicates the presence of an amide group (–CO–NH–), which proves that the nylon membrane is a polyamide. For the hybrid nylon/TiO₂ membrane (Membrane B, Membrane C, Membrane D, Membrane E, and Membrane F), it was noticed there was a functional group of the TiO₂ molecule that appear include O – H stretching band (3361 - 3373 cm⁻¹), O – H bending band (1635 - 1636 cm⁻¹), Ti – O – Ti stretching band (1416 - 1417 cm⁻¹) and Ti – O stretching (503 - 507 cm⁻¹). The appearance of these functional groups confirms that the inorganic material (TiO₂) has been successfully added to the nylon/TiO₂ hybrid membrane [23].

The addition of TiO₂ into nylon polymer on the nylon/TiO₂ hybrid membrane showed a shift in the wavenumber value and the appearance of TiO₂ functional groups. The shift in wavenumber value along with the addition of the TiO₂ occurs because nylon molecules will absorb different wavelengths of electromagnetic radiation along with the addition of TiO₂ and undergo a transition as a result of the energy transfer process between the radiation field and atoms/molecules [24].

The addition of TiO₂ into nylon polymer on the nylon/TiO₂ hybrid membrane also causes changes in the absorbance value in the FTIR spectra. The highest absorbance value is in the functional group of Ti – O stretching band on the nylon/TiO₂ hybrid membrane, presumably due to the stronger intermolecular interactions with the addition of TiO₂ concentration. While in the primary amide C = O stretching band, there is an overlap with the O – H bending band so that interactions occur between molecules and cause high absorbance values in the membrane. In addition, the increase in the absorbance value of the primary amide C = O stretching band is thought to be caused by the dipole bond in the monomer unit, which affects the intermolecular forces in the polymer. Where in the N – H stretching band, the hydrogen atom which has a positive charge is strongly attracted by the oxygen atom, which has a negative charge in the primary amide C = O stretching band, resulting in a strong hydrogen bond. This will be affected in increasing the tensile strength, melting point and flexibility of the nylon/TiO₂ hybrid membrane [25].

From the FTIR spectra of the nylon/TiO₂ hybrid membrane that has been carried out, it shows the appearance of functional groups from the polyamide and TiO₂, which indicates that TiO₂ has been successfully added into the nylon polymer. The addition of TiO₂ into the nylon polymer on the hybrid membrane did not remove the functional groups from each material, but only a shift in the wavenumber along with the addition of the TiO₂ concentrations. The intermolecular interactions indicated by the shift in wavenumber and the absorbance increased with the addition of the TiO₂ concentrations into nylon polymer on the hybrid membrane, which showed an increase in the flexibility of the nylon/TiO₂ hybrid membrane.

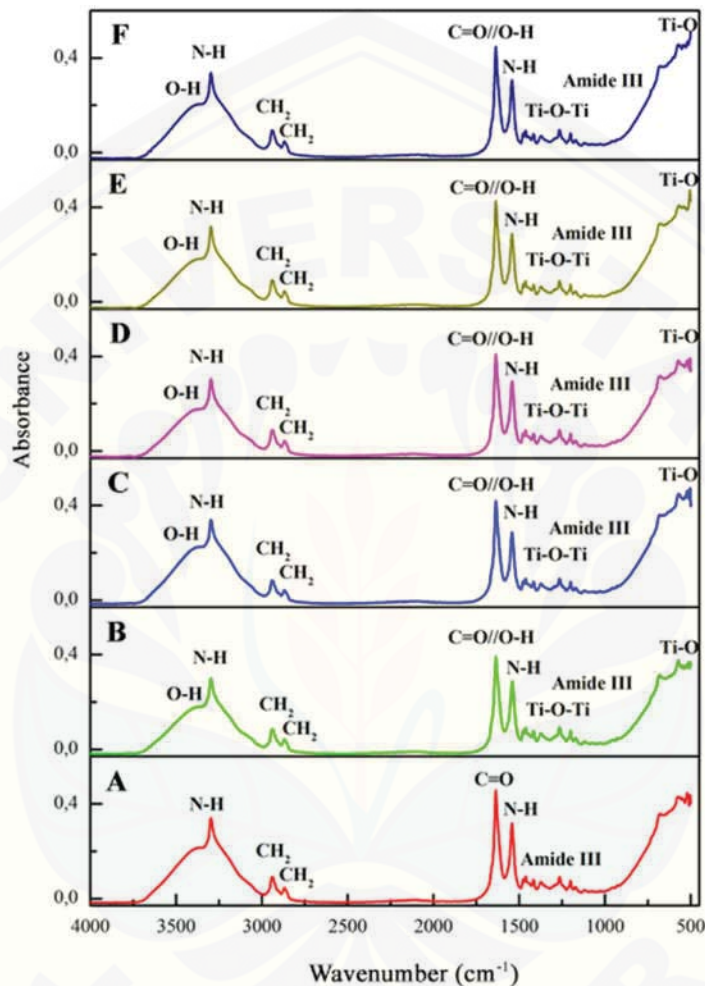


FIGURE 1. FTIR spectra of the nylon/TiO₂ hybrid membrane

XRD RESULTS

The crystallinity of nylon and nylon/TiO₂ hybrid membrane was studied by XRD analysis and the results are shown in Figure 2. For nylon membrane, the characteristic peak of nylon appeared at approximately $2\theta = 20^\circ$ and $2\theta = 24^\circ$. This is in agreement with results obtained by Millot et al. [26] working with pure nylon-6. XRD pattern of the nylon/TiO₂ hybrid membrane exhibited new reflection peaks at $2\theta = 38.26^\circ, 39.15^\circ, 25.29^\circ, 25.33^\circ, 25.16^\circ$ (for membrane B, membrane C, membrane D, membrane E, membrane F, respectively), which were attributed to TiO₂ phase with tetragonal anatase structure. The presence of anatase structure in the nylon/TiO₂ hybrid membrane indicates high chemical and thermal stability, antifouling, and super hydrophilicity, which are important characteristics of separation membranes [27]. With the addition of TiO₂, diffraction patterns of the nylon/TiO₂ hybrid membrane showed that the intensity of the typical peak of nylon became smaller and the peak of TiO₂

became larger. This indicates a decrease of the crystallinity of the nylon/TiO₂ hybrid membrane and an increase of amorphous character due to the crosslinking among nylon and TiO₂.

The results showed that the crystallinity of the nylon/TiO₂ hybrid membranes obtained were 85.65%, 79.20%, 82.68%, 77.78%, 70.60%, and 71.45% (for membrane A, membrane B, membrane C, membrane D, membrane E, membrane F, respectively). The highest crystallinity was in sample membrane A (pure nylon), while the lowest was obtained in sample membrane E. Based on these results, it can be seen that the crystallinity of the nylon/TiO₂ hybrid membrane decreased when the TiO₂ concentration was added. This is due to the presence of TiO₂ which hinders the generation of the crystal lattice and leads to the decrease of nylon crystallinity. The addition of TiO₂ into the nylon polymer can decrease the crystallinity of the hybrid membrane and ultimately increase the flexibility of the polymer chains [25].

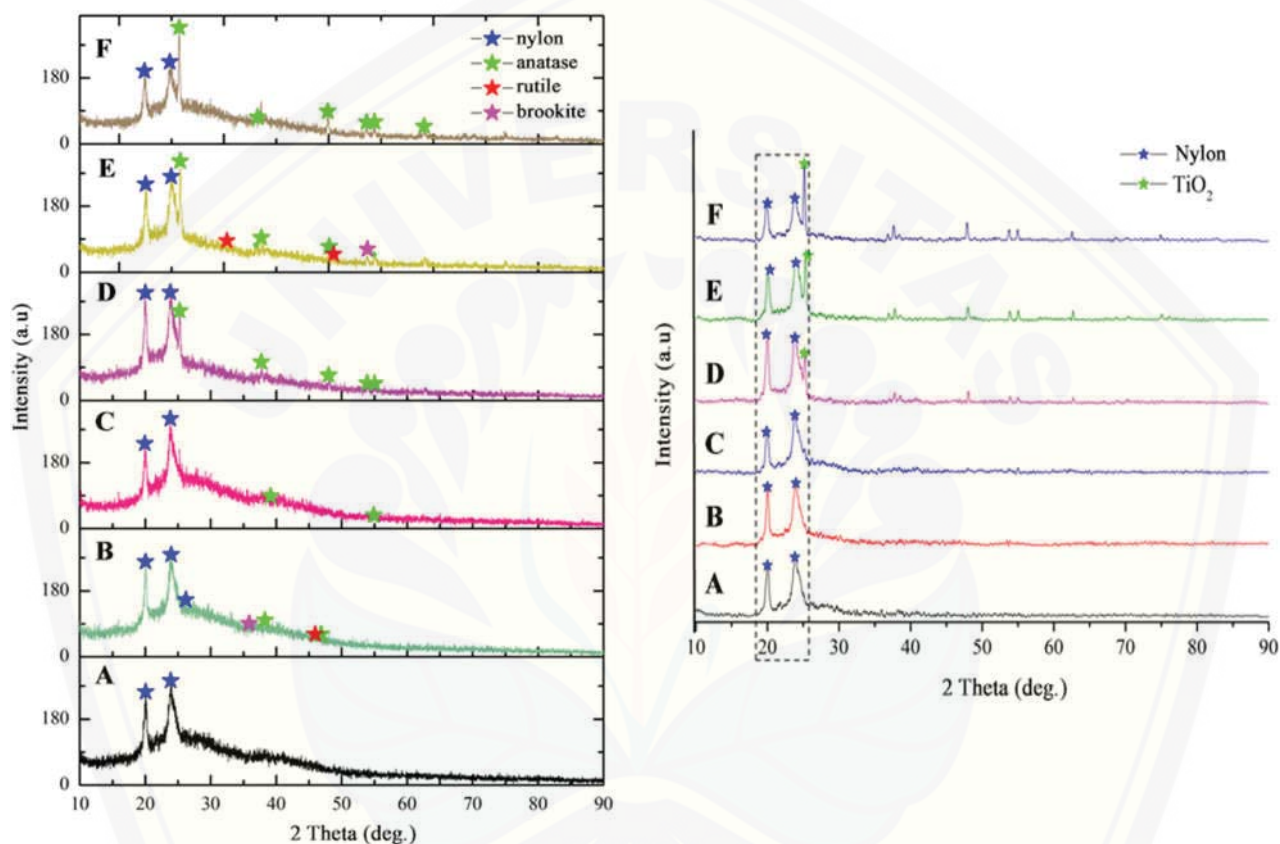


FIGURE 2. XRD Spectrum of the nylon/TiO₂ hybrid membranes with various TiO₂ contents: 0 wt.% (membrane A), 0.5 wt.% (membrane B), 1 wt.% (membrane C), 3 wt.% (membrane D), 5 wt.% (membrane E) and 7 wt.% (membrane F)

Results of Electrical Conductivity and Activation Energy Measurements

Electrical conductivity is the ability of a material to conduct the electrical current which is shown by the magnitude [28]. The result of this test show increasing the electrical conductivity with increasing the temperature from 25°C and 95°C on the nylon/TiO₂ hybrid membranes. The pure nylon membrane has electrical conductivity equal to 1.24×10^{-5} S/cm. The electrical conductivity of (0.5, 1, 3, 5, and 7) wt.% TiO₂ on the hybrid membrane are (1.50×10^{-5} , 1.54×10^{-5} , 1.63×10^{-5} , 1.94×10^{-5} , 1.59×10^{-5}) S/cm respectively. The electrical conductivity increased due to increasing the mobility of charge carriers when the temperature was increased. Thus, the more charge carriers, the more current was carried so that the current flow through the nylon/TiO₂ hybrid membranes would increase, followed by an increase in the measured electrical conductivity [29]. The maximum electrical conductivity resulted with 5 wt.% TiO₂, which represented the maximum addition of concentration of TiO₂ on the nylon/TiO₂ hybrid membranes.

The electrical conductivity and the activation energy were measured for nylon/TiO₂ hybrid membranes with different concentrations of TiO₂ at different temperatures (298 – 368) K. Figure 3 represented $\ln \sigma$ versus $1/T$ for

nylon/TiO₂ hybrid membranes. From resulted values, the electrical conductivity and activation energy were listed in Table 1. As shown from the results, the electrical conductivity increases with the addition of concentration of TiO₂ wt.%. The electrical conductivity increased due to increasing the density and mobility of charge carriers. The activation energy decreases with increasing the TiO₂ wt.% in nylon polymer while the electrical conductivity increases with increasing concentration of TiO₂ wt.%.

Activation energy can be used as a parameter to determine the minimum energy needed for a reaction to proceed. Activation energy stays the same with increasing temperature. However, the average increase in particle kinetic energy caused by the absorbed heat means that a greater proportion of the reactant molecules now have the minimum energy necessary to collide and react. During the reaction, it can be indicated by the presence of a frequency factor constant, which in these results shown the value of the initial electrical conductivity (σ_0). An increase in temperature causes a rise in the energy levels of the molecules involved in the reaction, so the rate of the reaction increases. Similarly, the rate of reaction will decrease with a decrease in temperature [30].

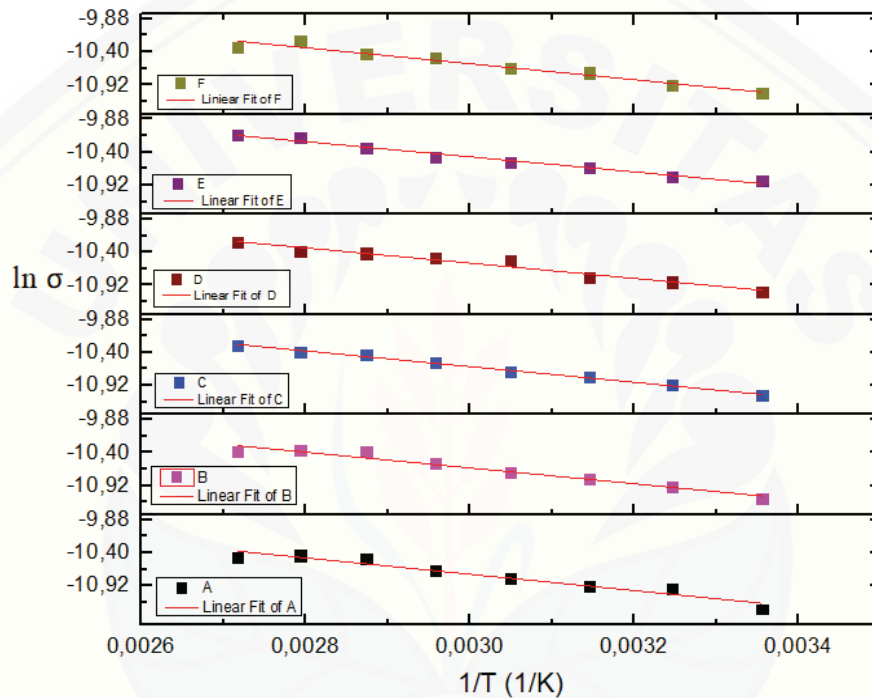


FIGURE 3. Plots of $\ln \sigma$ versus $1/T$ of the nylon/TiO₂ hybrid membranes at different temperature (298 – 368) K

TABLE 1. Activation energy and the electrical conductivity of different compositions of the nylon/TiO₂ hybrid membranes

Hybrid Membrane	The activation energy (eV)	Electrical conductivity (S/cm) of hybrid membrane at 25°C
Pure Nylon (Membrane A)	0.1103	1.24×10^{-5}
Nylon/0.5 wt.% TiO ₂ (Membrane B)	0.1064	1.50×10^{-5}
Nylon/1 wt.% TiO ₂ (Membrane C)	0.1055	1.54×10^{-5}
Nylon/3 wt.% TiO ₂ (Membrane D)	0.1034	1.63×10^{-5}
Nylon/5 wt.% TiO ₂ (Membrane E)	0.1025	1.94×10^{-5}
Nylon/7 wt.% TiO ₂ (Membrane F)	0.1082	1.59×10^{-5}

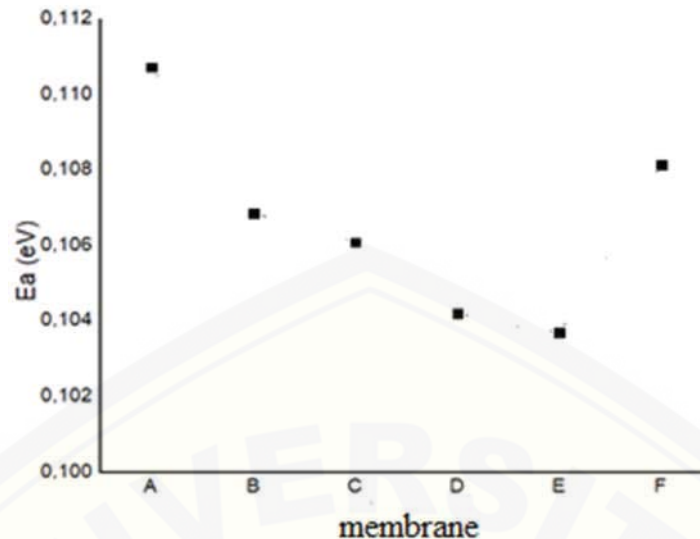


FIGURE 4. The activation energy affected by adding different concentration of TiO_2 into nylon polymer on the hybrid membrane

The parameter that has the lowest activation energy was the key. So, in this study, the lowest activation energy indicated the best hybrid membrane. The activation energy (E_a) can be used to see the characteristics of the mechanism for the rate of stability of ions that pass through the membrane under the influence of temperature. The greater activation energy means the rate of stability of the ions that pass through the membrane will be slower with changes in temperature (indicated by the steeper slope of the line). On the other hand, the smaller activation energy indicates the rate of stability of the ion passing through the membrane will be faster with changes in temperature (indicated by the slope of the line getting gentler). The large activation energy indicates that the hybrid membrane has a stiffer and denser structure [31]. According to Kujawa et al. [32], the value of activation energy can be slightly different depending on the type of membrane, its character (hydrophobicity), and thickness.

Based on Figure 4, for nylon/7 wt.% TiO_2 (Membrane F) the activation energy increased more than the activation energy for hybrid membrane else. Since a good dispersion is very important to establish the conductive pathway in the hybrid membrane, increasing the TiO_2 concentration led to a lack of dispersion, causing dispersion and interfacial interactions. It is suggesting that the addition of a small amount of TiO_2 probably induces heterogeneous nucleation. Nevertheless, the addition of more TiO_2 into nylon polymer reduced the transportation ability of polymer chains during the crystallization process and thus increased the activation energy [22].

CONCLUSIONS

Hybrid membranes, based on a nylon polymer matrix and a TiO_2 component, have been successfully synthesized by a sol-gel method. The characterization of studies confirmed the existence of TiO_2 in the nylon membrane matrix based on FTIR spectra. From XRD analysis, the addition of TiO_2 into the nylon polymer can decrease the crystallinity of the hybrid membrane. Finally, the electrical conductivity of nylon/ TiO_2 hybrid membranes increases with increasing temperature. The addition of TiO_2 most likely increased the charge carrier concentration and mobility, leading to an increase the electrical conductivity while the activation energy decreased with increasing wt.% of TiO_2 in the polymeric matrix.

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