

# Production and Characterization of Nylon Fiber Nanomembrane from Fishing Line using Electrospinning

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## Abstract

Nylon is an alternative material for making membranes considering that nylon is a material that is easy to obtain, cheap and strong. The electrospinning method is capable of producing nanoscale membranes or filters, compared to other methods. In this research, we prepared nanomembrane from fishing line material, using the electrospinning method. The fishing line is made of nylon 6 after being identified using FTIR and TGA. Small pieces of fishing line were dissolved in glacial acetic acid and formic acid in a 1:1 ratio, then the solution was processed on an electrospinning machine with various voltage variations. Next, the membrane was characterized using FTIR, TGA, SEM and UTM. The membrane characterization results show that the higher the tension, the thicker the membrane and the higher the tensile strength and elongation of the membrane. Fiber sizes were produced at tensions of 33.3% (150-200 nm), 30% (200-250 nm), 20% (100-150 nm) and 16.67% (250-300 nm). The greater the flow rate of the nylon solution, the smaller the percentage of nanofibers. The addition of hydroxyapatite additives increases the tensile strength of the membrane and increases the percentage of TGA residue.

**Key words:** nylon; fishing line; membrane; electro spinning; hydroxyapatite

## Introduction

Studies on membranes have continued to progress so far. There are many interesting things that can be learned from membranes, so many ideas are emerging in terms of making synthetic membranes from various materials, which can then be used in various industries. Membranes can act as very specific filters, such as their use as membrane filters in medical masks to filter viruses and bacteria with a size of 0.1 microns (100 nm). Only molecules of a certain size can pass through the membrane, while the rest are stuck on the membrane surface.[1] The membrane can function as a thin, highly selective barrier between two phases and can only pass certain components and pass other components of a fluid flow that is passed through the membrane. [2] These phases have different characteristics, namely: concentration, pressure, temperature, solution composition and viscosity.

Membrane technology continues to develop over time, because this technology has advantages that other separation technologies do not have. The separation technique using a membrane has several advantages, including a simple operational

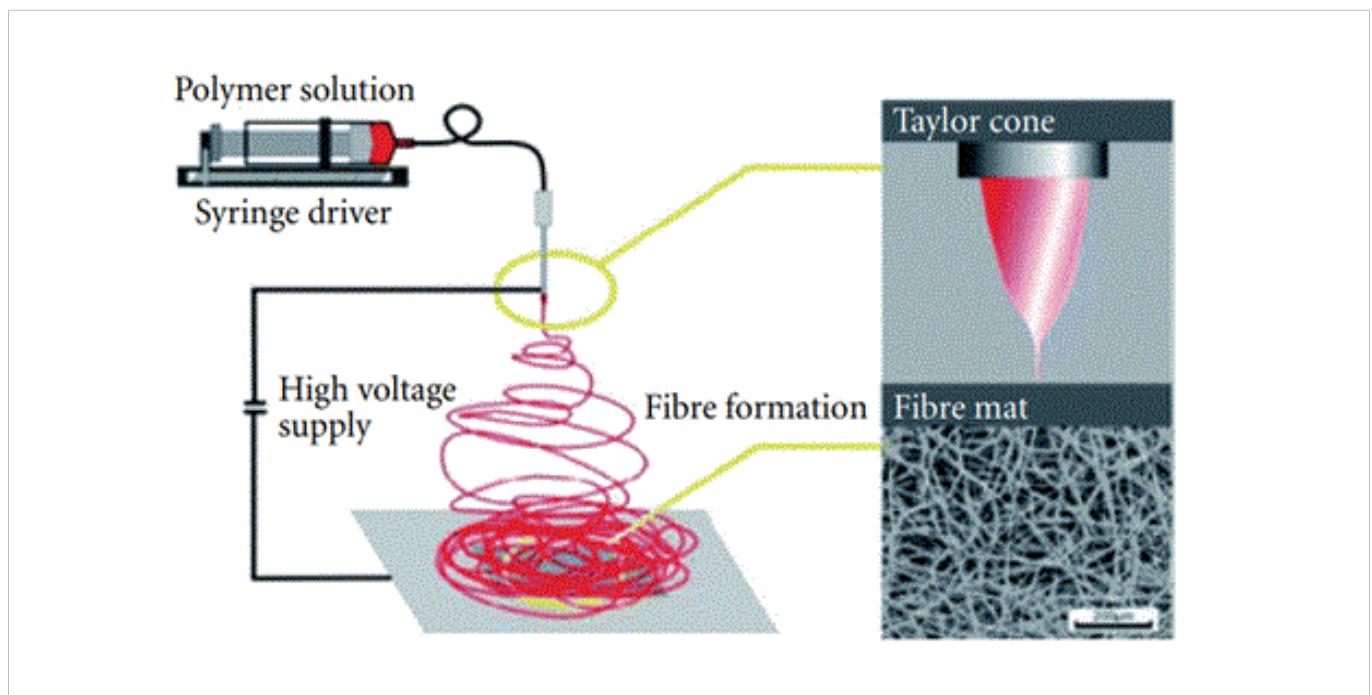
process, can take place at room temperature, is not destructive, so it does not result in changes (degradation) of the substances being separated either physically or chemically, and the separation can take place continuously. Therefore, membranes are included in clean technology.[3]

The development of nanotechnology is currently growing rapidly, one of which is nanofiber technology. Several methods for producing nanofibers are drawing, template synthesis, phase separation [4], self-assembly [5-7], template synthesis [8,9], and mechanical drawing [10], and electrospinning [11-14]. The Electrospinning method is one of several methods for making nanometer to micrometer sized polymer fibers [15,16]. To produce nanofibers, physical principles are used in the electrospinning method, namely by utilizing Coulomb forces due to the presence of free charges or ions on the surface of the polymer solution, so that when subjected to a very high potential difference or voltage, this charged polymer solution can be attracted to the collector and form polymer fibers. [12,15].

Electrospinning is a simple and versatile technique that utilizes electrostatic forces to produce very fine polymer fibers

ranging in size from submicron to nanometer. This technique can be applied to produce fibers from various types of polymers, be they synthetic polymers [17], natural polymers [18], biodegradable polymers [19,20], nondegradable polymers [21], or mixtures thereof [19,22]. Electrospinning has gained much importance and is a preferred technique because it is relatively easy, cost-effective, requires simple tooling, [23-26] and can be applied to produce ultrafine fibers with simple step-up production that is not easily achieved with other conventional fiber forming techniques.

In the electrospinning process, a high voltage electric field, applied to a liquid polymer (solution or melt) results in the release of a continuous jet from the elution nozzle that accelerates towards the oppositely charged ground collector. In the absence of an electric field, polymer droplets are held at the tip of the capillary by the surface tension of the liquid [17,27]. Upon application of an electric field, as the surface tension is balanced by electrostatic forces, the droplet elongates and develops into a cone known as a "Taylor Cone". When the electric field strength is sufficient to overcome the surface tension of the liquid, a jet of fine fibers is ejected from the tip of the Taylor Cone [28,29]. As the fiber jet moves through the atmosphere, the solvent evaporates and the solid polymer fibers are deposited on a grounded collector as a mesh or scaffold. Figure 1 provides a schematic representation of a typical electrospinning process [30].



**Figure 1:** Schematic of a typical electrospinning setup showing Taylor cone and SEM images of an electrospun mat. Adapted from Wallace et al [30].

Thus, electrospun nanofibers can have a tremendous impact in HEPA (high efficiency particulate air) and ULPA (ultralow penetration air) filtration [31-34]. In the textile sector, nanofibers in woven and nonwoven form can be used for smart textiles, sports, medical textiles, etc. [35]. For nonwovens, nanofibers can be produced as membranes with nano sizes up to <100 nm, which cannot be produced by other membrane technologies.

Electrospun nanofibers from polyamide (PA) have been used in a number of applications [36-43]. PA has superior fiber forming ability. It is a biodegradable and biocompatible synthetic polymer with good mechanical properties, which are further enhanced by hydrogen bonding. Unlike other polymers, such as polyvinyl alcohol, PA is resistant to water and humidity. PA can be dissolved in formic acid or formic/acetic acid solutions; this ensures an environmentally friendly process compared to other solvents such as dimethylformamide (DMF), which is toxic, according to EU directive 67/548/EEC. PA 6 and PA 6/6 are the most studied polyamide species, but discussion of their application for

filtration purposes is rather limited [32,44,45]. Recent reports on the properties of electrospun nylon-6 nanofibers indicate PA as a very attractive material for filtration applications [43].

The morphology and size of the resulting fibers can be adjusted by changing device parameters, such as electrical voltage, needle tip distance to collector, needle shape, polymer solution flow rate, and collector geometry, or by changing polymer solution parameters, including conductivity, viscosity, and surface tension. [46,47]. The electrospinning method is also a simple, easy and cheap technique for producing nanofibers [48]. Nanofiber membranes can be applied to drug carrier systems [47], wound dressings [49,50], cosmetics [51,52], and capacitors [53,54].

Many polymers can be used as carrier matrices in electrospinning techniques. In this study, we used nylon to produce nanofibers. Nylon was chosen because it is easy to spin (electrospinning), and can dissolve in polar and nonpolar solvents

[55,56]. Nylon-6 nanofiber membranes have been widely used as aerosol filtration media and filters because they can produce uniform fibers, have good mechanical properties, low density and high porosity [57]. Some research related to nylon-6 nanofibers is nylon-6/graphene to eliminate Cr(VI) adsorption [44]) and preparation of multilayer nylon-6 nanofibers [58]. However, Nylon-6 nanofibers produced using the electrospinning method with different concentrations and analysis of the characteristics of the nanofibers (morphology, X-Ray Diffraction (XRD) and Fourier transform of infrared spectrophotometer (FTIR)) are rarely found.

In this study, nylon fiber from fishing line was produced using electrospinning because the method is efficient, continuous fiber production, little or no solvent waste is generated. The research is limited only to the influence of voltage and flow rate on electrospinning, because voltage and flow rate are very influential on making nanomembranes, although there are several parameters that also have an influence, such as: the distance between the injector and collector, solvent and others. Apart from that, we tried adding the additive hydroxyapatite to the formation of nanomembranes, then analyzed its effect on the membrane properties, although this is only additional research that needs to be investigated further.

Membranes or filters that are used for several purposes, for example for medical purposes, require certain properties such as anti-bacterial, burn-resistant, melt-resistant and others that are useful for the user. These properties can be achieved by including certain chemicals which are expected to react with the nylon fiber or trap in the fiber which results in affecting the properties of the fiber. For this reason, one of the efforts used is to include hydroxyapatite in the fiber formation process. It is hoped that the phosphorus content in hydroxyapatite can increase the membrane's resistance to fire and can also prevent melting. As is known, melting during a fire will speed up the spread of the flame and if it comes into contact with the human body it will cause serious fire injuries.

## Materials And Methods

### Material

The main ingredients are fishing line, formic acid, glacial acetic acid and methylformamide, hydroxyapatite is obtained commercially on the market. The laboratory equipment used is; magnetic stirrer, petri dish, glass bottle, beaker, spatula, and the main equipment, namely an Electrospinning machine.

### Method

The fishing line material is dissolved first using formic acid and glacial acetic acid solvents. The formation of nanofibers and membranes in this activity uses electrospinning tools in the PTM laboratory and their characterization uses FTIR, TGA, SEM and UTM tools. Morphological characterization using SEM Phenom Pro X and tensile testing using UTM Gotech 2 kN.

To produce nylon fibernanomembrane, it is carried out in 2 stages, namely dissolving the nylon fiber (fishing line) and

making the membrane using an electrospinning machine. In this research, electrospinning voltage variations were carried out (10 kV, 15 kV and 20 kV). To determine the effect of additives on membrane properties, an experiment was carried out by adding the additive hydroxyapatite (5% by weight) to the solution, while the electrospinning voltage was 20 kV.

### Preparation Nylon Solution

Nylon solution is the main formula used to make the desired nanomembrane. For this reason, what needs to be done is to look for a suitable solvent to obtain a suitable nylon solution. From several references it has been found that good solvents for nylon are acetic acid and formic acid. [59]

First, prepare the nylon solution as follows: cut the nylon fishing line into small pieces then weigh 1 gram, see Figure 2. Put the pieces of nylon line in 5 mL of solvent made from acetic acid and formic acid (1:1) then stir for 90 minutes. After that, let it sit for a while to remove the foam. This nylon solution is used as a material for making nanomembranes using electrospinning.

### Manufacturing of Nylon Fiber Nanomembrane

The experimental stages that have been carried out can be seen in the flow diagram in Figure 3. Stage 1 is the preparation of the nylon solution and stage 2 is making the nanomembrane. Making the nylon solution begins with small pieces of fishing line measuring 1 gram being put into a mixture of 2.5 ml of glacial acetic acid and 2.5 ml of formic acid (1: 1), stirring using a magnetic stirrer for 90 minutes. The prepared nylon solution is then added + 3 ml as needed into the syringe.

In this case, the experiment used a composition of 1 gram of nylon and 3 mL of acetic acid: formic acid (1:1) solvent. Electrospinning was set with a flow rate of 1 mL/hour and a distance from the needle tip to the collector of 10 cm [60]. Meanwhile, the voltage used varies from 10kV, 15kV [60] and 20kV, to find the optimal voltage. The experiments carried out in this research used information from several references that had been carried out previously, and optimal conditions were taken from what they had carried out, such as the distance between the injector and collector and the voltage used. On the other hand, we see that the potential for optimization of this research is to find the appropriate voltage to obtain certain results.

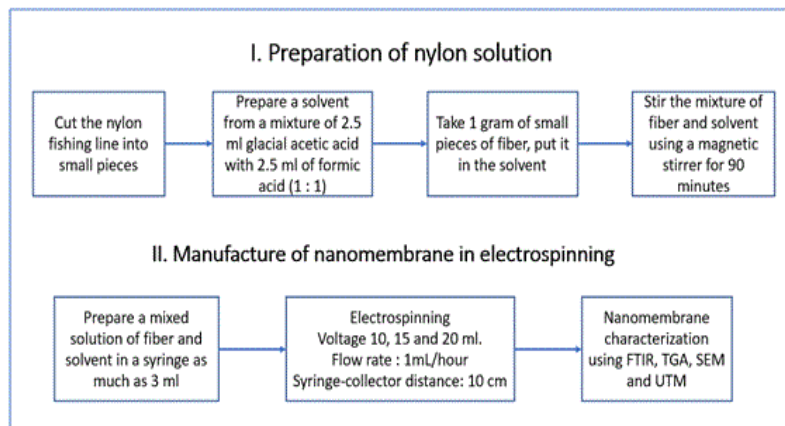
In this research, identification was limited to 3 samples, namely fishing line samples (N), nylon fibernanomembrane with an electrospinning voltage of 20V (N20) and nylon fibernanomembrane with 5% hydroxyapatite (NH5). Besides that, the effect of voltage on fiber diameter was also observed at voltages of 10kV, 15 kV and 20kV. The effect of flow rate was also observed by comparing 2 flow rate parameters, namely 0.5 ml/hour and 1.5 ml/hour, at a voltage of 15kV.

### Fishing Line Material Characterization

Characterization of fishing line materials is carried out to determine the type of nylon used. The fishing line used is commercial fishing line obtained in the market. Characterization was carried out using the Nicolet iS50 FTIR and Thermo Scientific



**Figure 2:** Commercial Fishing Line (a), Small Piece of Fishing Line (b)



**Figure 3:** Flow Diagram for Making Nylon Solution (I) and Making Nanomembranes (II)

Setaram TGA at the PTM-BPPT Laboratory. FTIR is a tool or instrument that can be used to detect functional groups, identify compounds and analyze mixtures of the sample being analyzed without damaging the sample, meanwhile TGA is an analytical technique for determining the thermal stability of a material and the volatile component fraction by calculating the associated weight change. with changes in temperature.

Nylon is a synthetic fiber which is often called polyamide fiber which is derived from diamine and dicarboxylic acid. The best characteristic of nylon fiber for the textile industry is its flexibility which contributes to strength and abrasion resistance. Nylon is a semi-crystalline polymer that is widely used in various fields due to its good physical and mechanical properties. Nylon has many varieties, but the most common are nylon 6 and nylon 66. These two types of nylon are produced for more than 80% of total nylon

production. The differences between nylon 6 and nylon 66 are presented in Table 1.

Nylon 6.6 has four repeating hydrogen bonds per unit, so its melting range is greater than nylon 6 (215-225oC). Nylon 6 has only one hydrogen bond per repeating unit, so less energy is required to break intermolecular forces than nylon 6.6. Nylon 6.6 is a polymer consisting of two monomers: adipoyl chloride and hexamethylenediamine. Due to the strong chemical bond between the two styles, Nylon 6.6 has a more crystalline structure, making it slightly stiffer and more heat resistant than Nylon 6.



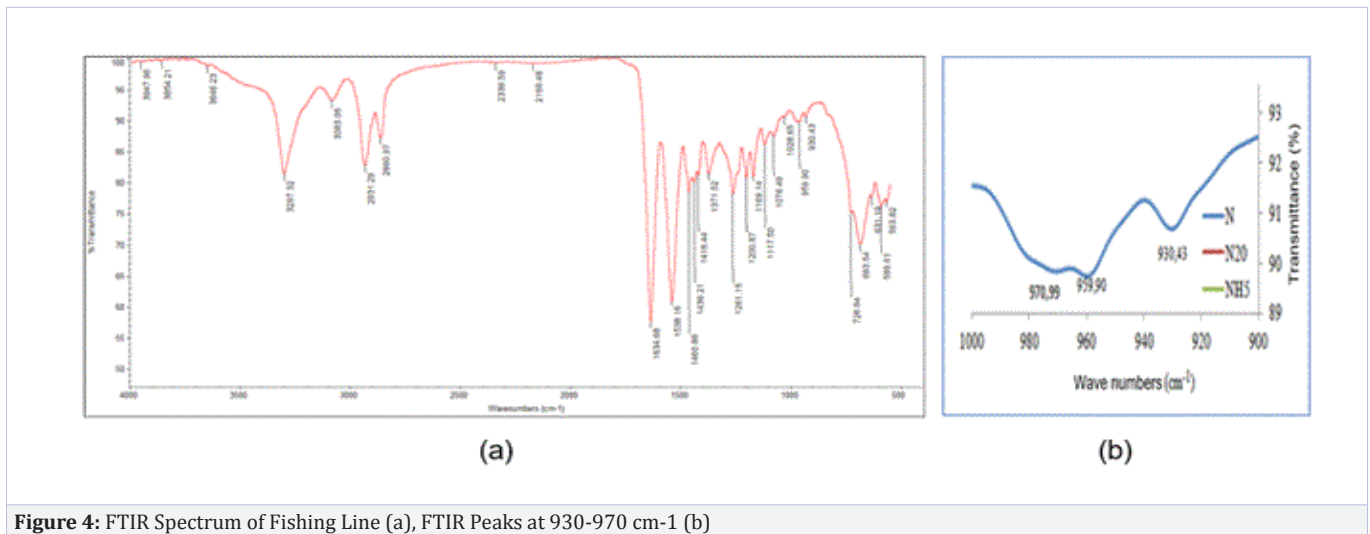
Properties	Nylon 6.6	Nylon 6
Utility	Fiber, thermoplastic	Fiber, thermoplastic
Monomer	Adipic acid and hexamethylenediamine	Caprolactam
Polymerization	Condensation polymerization with acid catalyst	Ring opening polymerization
Morphology	Crystalline	Crystalline
Melting point	260°C	215°C
Glass transition point	50°C	40°C

**Research Results And Discussion**

**Fishing Line Material Analysis**

Initial characterization was carried out using FTIR to determine the type of polyamide from the fishing line. After being cleaned with aqua, small pieces of fishing line were then dried in an oven at 60oC for 1 hour, then characterized using an FTIR tool. The results can be seen in Figure 4.

The results of FTIR characterization of commercial fishing line material show that the spectrum from this test tends to indicate that the fishing line used to form the membrane prototype is of the nylon 6 type, Figure 4. To facilitate analysis, functional group determination was carried out by dividing the FTIR spectrum into five observation areas , namely 3500 – 3000, 3000 – 2500, 1800 – 1490, 1490 – 1000, 1000 – 900 cm-1. This analysis was carried out in the 1000 – 900 cm-1 area, which is based on research by Ma Y et al. (2016)[61] who used this area to determine the difference between nylon 6 and nylon 6.6. They reported that nylon 6.6 had no peak at wave number 960 cm-1. In the N sample, there are three main peaks, namely 930.43, 959.90, and 970.99 cm-1 (Figure 4b). These peaks indicate respectively CONH bending ( $\alpha$  phase), CONH bending ( $\alpha$  phase), and CONH bending ( $\gamma$  phase). From this spectrum, the fishing line sample is most likely nylon 6.



**Figure 4:** FTIR Spectrum of Fishing Line (a), FTIR Peaks at 930-970 cm-1 (b)

Next, to strengthen the FTIR characterization results, the same fishing line was tested against the TGA test. The fishing line is prepared and then tested on a TGA tool with a heating rate setting of 10°C/minute with oxygen gas from room temperature to 800°C. Figure 5 shows the TGA graph showing that the dTG curve tends to have one decomposition peak which indicates the TGA curve of nylon 6.

Table 2 shows a recapitulation of the TGA curve from Figure 6 which has several parameters taken to be used as analysis material for the fishing line material used, including the temperature when the material begins to decompose at 350oC (IDT) and reaches a

maximum decomposition of 442oC and the residue at 800oC still remains 6 .23%.

**Characterization of Nanofibre Membranes**

The nylon material (fishing line) is dissolved first using a solvent from a mixture of formic acid and glacial acetic acid. The solution (solvent and nylon) is divided into 2 parts. The first part is processed without additives and the second part is added with hydroxyapatite additives. Both are processed on an electrospinning machine in the PTM BPPT laboratory. The results of the nanofiber membrane were characterized morphologically

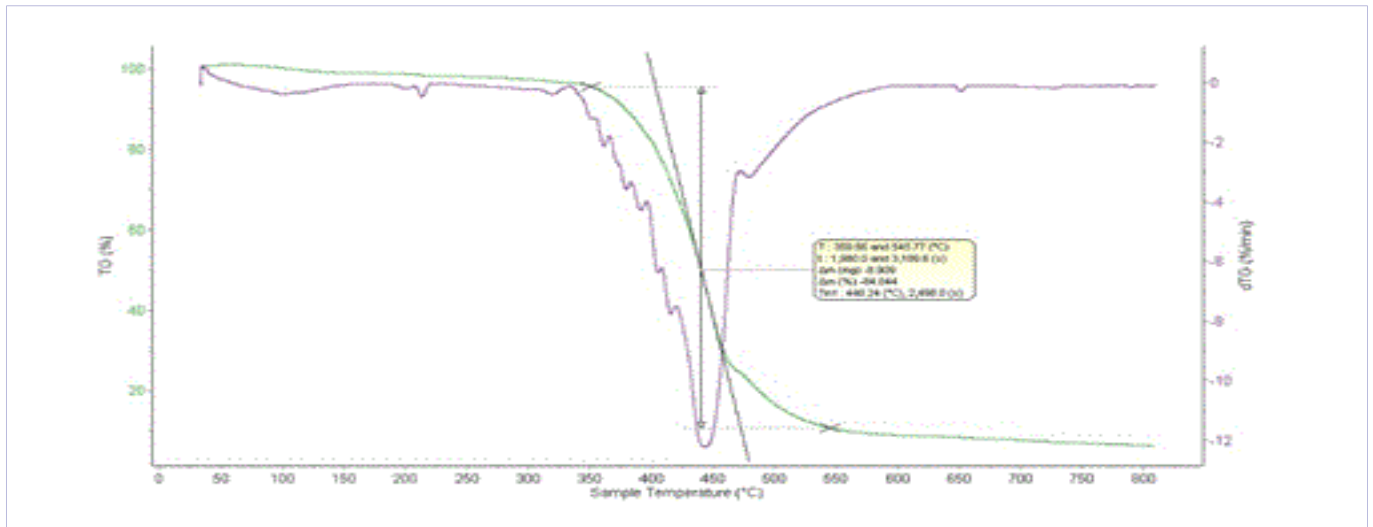


Figure 5: TGA Curve of Fishing Line

Table 2. Recapitulation of TGA Curve of Fishing Line (N)

Test results	Sample N
IDT (°C)	350,66
T PeakdTG (°C)	442,7
MRDT (°C)	442,7
FR (%) (T =797,2)	6,23

using SEM Phenom Pro X and tensile testing using UTM Gotech 2 kN.

The nylon material (fishing line) is dissolved first using a solvent from a mixture of formic acid and glacial acetic acid. The solution (solvent and nylon) is divided into 2 parts. The first part is processed without additives and the second part is added with hydroxyapatite additives. Both are processed on an electrospinning machine in the PTM BPPT laboratory. The results of the nanofiber membrane were characterized morphologically using SEM Phenom Pro X and tensile testing using UTM Gotech 2 kN. FTIR is used to determine the functional groups in materials and TGA is used to determine the temperature at which material degradation occurs.

#### Fourier Transform Infrared (FTIR) Spectroscopic

The nanomembranes resulting from electrospinning have been characterized for their functional groups using FTIR in the wave number range of 400 – 5000 cm<sup>-1</sup> with a resolution of 4 cm<sup>-1</sup>. To facilitate analysis, functional group determination was carried out by dividing the FTIR spectrum into five observation areas, namely: 3500 – 3000, 3000 – 2500, 1800 – 1490, 1490 – 1000, 1000 – 900 cm<sup>-1</sup>.

##### 1. Region I 3500 – 3000 cm-1

Region I is the NH group area. In the N sample there is a weak peak at a wavelength of 3446.23 cm<sup>-1</sup>. Ma Y et al indicated this peak as N-H stretching of the free NH group [61]. This free NH

group is the NH group which is located at the very end of the nylon chain. In samples N20 and NH5 this peak shifted to 3441.41 cm<sup>-1</sup> (Figure 7). For sample N, in region I there is also a peak at the wave number 3297.32 cm<sup>-1</sup> which indicates NH stretching from the hydrogen bonded NH group [61] as well as a peak at the wave number 3083.06 cm<sup>-1</sup> which indicates NH angular deformation in plane [62]. The electrospinning process apparently makes the peaks shift towards higher wave numbers (the smaller the wave number). The NH stretching of the hydrogen bonded NH group in the N20 sample shifted to 3296.43 cm<sup>-1</sup> and 3295.63 cm<sup>-1</sup> for the NH5 sample. This also occurs in NH angular deformation in plan, N20 shifts to 3080.79 cm<sup>-1</sup> and NH5 shifts to 3088.61 cm<sup>-1</sup>.

##### 2). Region II 3000 – 2800 cm-1

Region II is the CH<sub>2</sub> group area. In sample N there are peaks at wavelengths of 2931.29 cm<sup>-1</sup> and 2860.97 cm<sup>-1</sup>. Guerrini et al indicated that these peaks are CH<sub>2</sub> α – NH axial deformation and CH<sub>2</sub> β – NH & γ – NH axial deformation [63]. In samples N20 and NH5, these peaks shifted to 2932.16 cm<sup>-1</sup> and 2859.57 cm<sup>-1</sup> for sample N20, and 2933.44 and 2862.96 cm<sup>-1</sup> for sample NH5 (Figure 8). α, β, γ are the positions of CH<sub>2</sub> towards the NH group.

##### 3). Region III 1700 – 1490 cm-1

Region III is the amide group area. In sample N there are peaks at wavelengths of 1634.68 cm<sup>-1</sup> and 1538.16 cm<sup>-1</sup>. Ma Y et al indicated this peak as CO stretching of the hydrogen-bonded CO group (CO axial deformation, amide I) and NH bending of the hydrogen-bonded NH group (CN axial deformation and CONH angular deformation, amide II) [61]. In samples N20 and NH5 these peaks shifted to 1638.62 cm<sup>-1</sup> and 1540.37 cm<sup>-1</sup> for sample N20 and 1640.28 and 1541.91 cm<sup>-1</sup> for sample NH5 .

##### 4). Region IV 1490 – 1000 cm-1

Region IV has many peaks. The peaks and their analysis are presented in Table 3. In this area, there is a peak of nylon or polyamide in the α form (phase α). The α phase is a form of polyamide crystal which has a monoclinic structure with the full

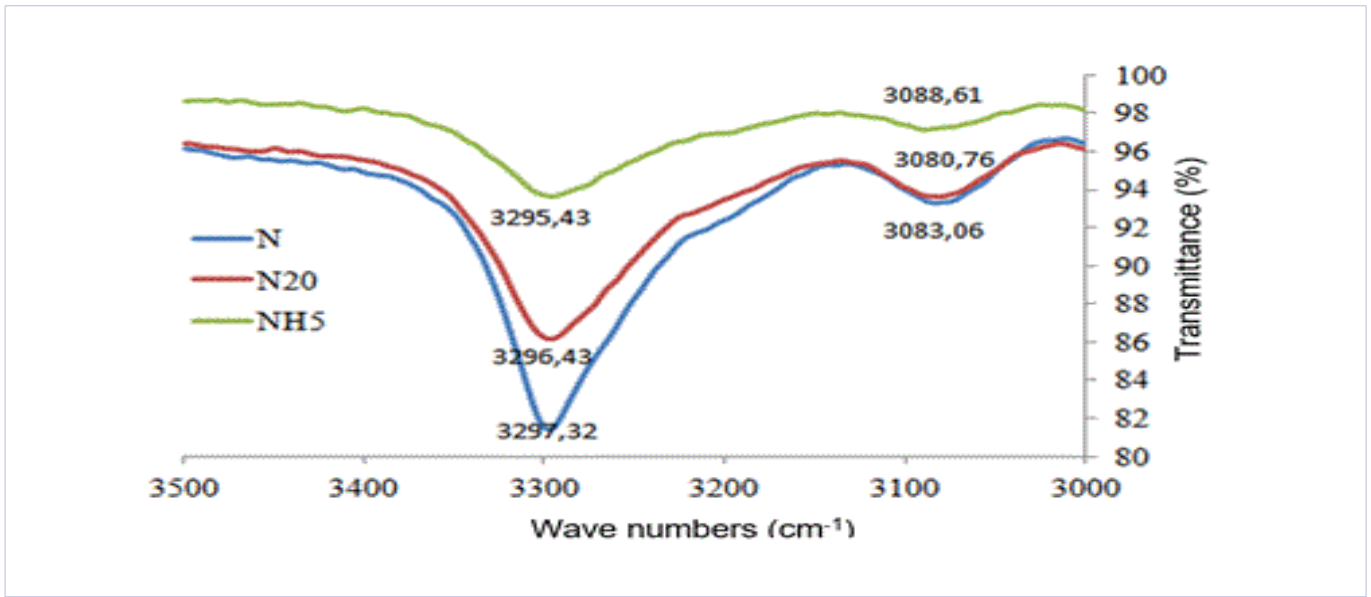


Figure 6: FTIR spectrum of region I on samples of N, N20, and NH5

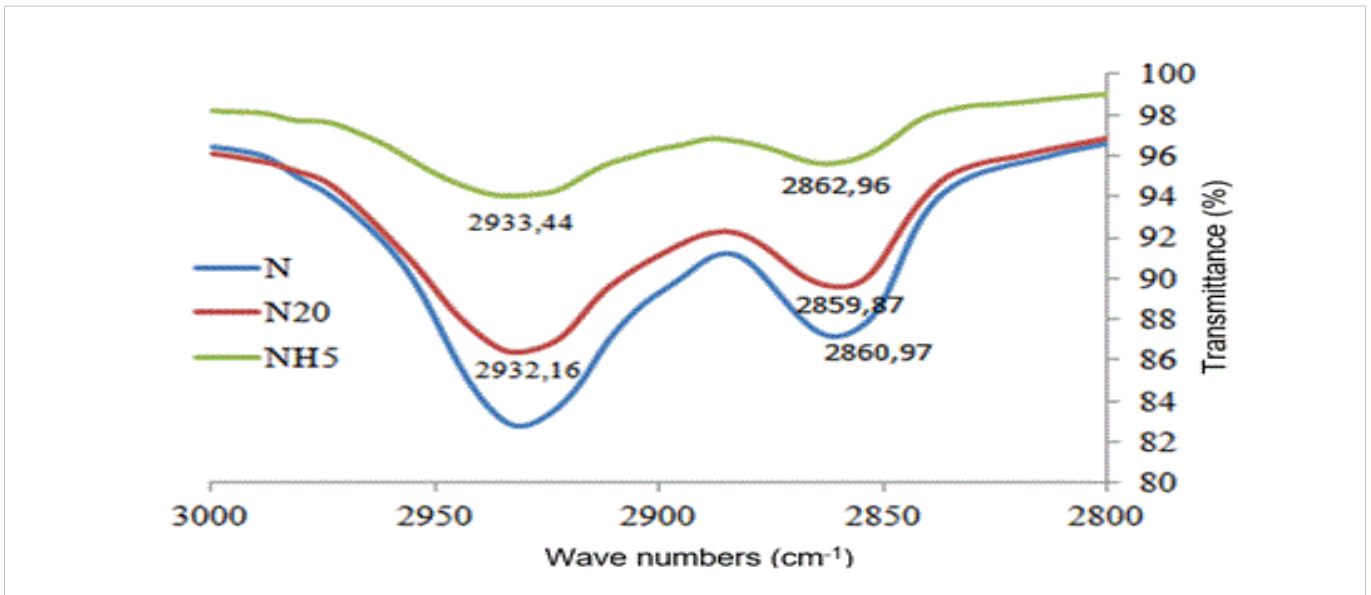


Figure 7: FTIR Spectrum of Region II on Samples of N, N20, and NH5

Table 3. Analysis of functional groups in the FTIR spectrum of samples of N, N20, and NH5 in the IV region

Characteristics	Wave number (cm <sup>-1</sup> )			Reference
	N	N20	NH5	
NH deformation / CH2 scissoring	1460,88	1461,70	1461,57	[65]
CH2 bending (γ-phase)	1436,21	1437,57	1437,22	[63]
CH2 bending (α)	1428,44	1420,34	1421,30	
CN axial deformation	1371,52	1371,44	1370,57	[63]
Amida III stretching	1261,98	1261,18	1261,15	[65]
Amida III	1200,87	1201,36	1201,77	[66]
CO-NH	1169,14	1170,44	1169,73	[67]
PO <sub>4</sub> <sup>3-</sup>	-	-	1046,19	[68]
α-phase	1076,49	1076,49	-	[67]

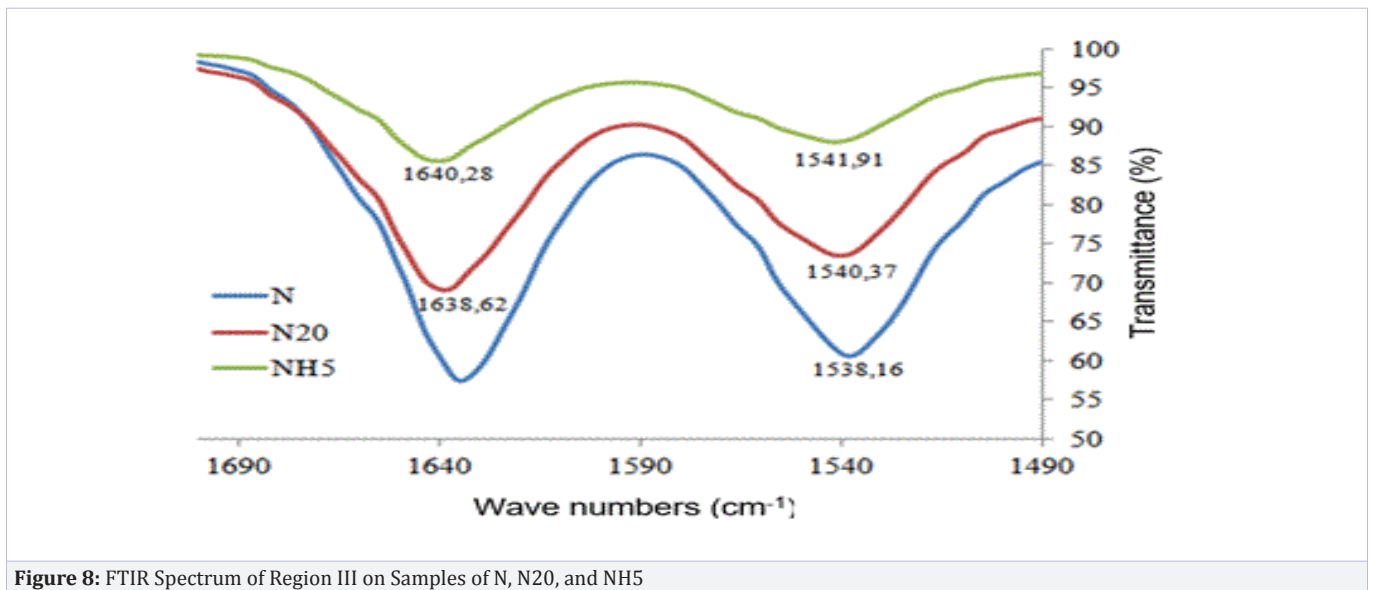


Figure 8: FTIR Spectrum of Region III on Samples of N, N20, and NH5

extension of the nylon 6 chain, and hydrogen bonds are formed between the antiparallel chains with the ethylene part and the amide group in the same plane, this produces a sheet-like shape. Apart from the  $\alpha$  phase, polyamide can also be in the  $\gamma$  phase. The  $\gamma$  phase is composed of pleated chains, where hydrogen bonds are formed between parallel chains, and the direction of the hydrogen bonds between molecules is perpendicular to the plane of the carbon framework [64]. Apart from that, in the NH5 sample, the peak at wave number 1076  $\text{cm}^{-1}$  disappeared and was replaced by a peak of 1046  $\text{cm}^{-1}$  which is the hydroxyapatite peak.

In Table 3, the results of the recapitulation of functional groups on each membrane are visible, although there is a shift in the wavelengths of the three types of samples, the shift is not very significant. This is in accordance with the prediction that in the NH5 sample there is a PO4-3 functional group, this shows that the hydroxyapatite additive added in the electrospinning process has the burn resistance and drip properties of the membrane

### 5). Region V 1000 – 900 $\text{cm}^{-1}$

Ma Y et al used this area to determine the difference between nylon 6 and nylon 66. They reported that nylon 66 did not have a peak at a wave number of 960  $\text{cm}^{-1}$ . In the N sample, there are three main peaks, namely 930.43, 959.90, and 970.99  $\text{cm}^{-1}$  (Figure 5). These peaks indicate sequentially CONH bending ( $\alpha$  phase), CONH bending ( $\alpha$  phase), and CONH bending ( $\gamma$  phase) [61,67]. In the N20 sample, the peak at 960  $\text{cm}^{-1}$  seems to disappear and peak 973  $\text{cm}^{-1}$  becomes more dominant. This possibly indicates a phase change from the  $\alpha$  phase to the  $\gamma$  phase. From this spectrum, sample N is most likely nylon 6, see Figure 9.

### Thermogravimetry Analysis (TGA)

The TGA test was also carried out on nylon fibernanomembrane samples (N20) and nanomembrane samples with the addition of hydroxyapatite (NH5), see Figure 11. In Figure 11, where the curves for N, N20 and NH5 samples are overlaid, there are 2 important results, namely deviations in the curves at

temperature. The TG is between 450-500oC in the N20 and NH5 samples compared to the initial Nylon sample. Another result is that the TG residue at a temperature of 800oC decreased from 6.23% (sample N) to 4.72% (sample N20), but increased to 16.05% (sample NH5), see Table 4

The NH5 sample (Figure 11.) uses the same conditions as the process for the N20 sample. This is related to the FTIR test results where before the electrospinning process, the peaks were identical to nylon 6 but after the electrospinning process, there was a missing nylon 6 peak which was probably due to the formation of nylon 6.6. This is shown by the results of the dTg curve in Figure 12. The TGA curve shows that before electrospinning, the dTG curve tends to have one decomposition peak Figure 12 (sample N), but after electrospinning the dTG curve has two decomposition peaks, sample N20. This also applies to the NH5 sample.

An interesting result is that at a temperature of +797oC, the residue in the N sample was 6.23%, decreased in the nanomembrane sample (N20) by 4.72%, but increased again after adding hydroxyapatite (NH5 sample) reaching 16.05% or almost 3 (three) times the residue in the nylon N sample. This indicates that hydroxyapatite containing phosphorus has an effect on the burning of nylon, in other words the phosphorus in hydroxyapatite has a flame retardant effect on nylon [69, 70]. However, special research for hydroxyapatite needs to be carried out separately regarding its effect on nylon melting, because charcoal formation occurs.

Thermal decomposition of nylon has been shown to begin with a primary cleavage reaction of the  $-\text{NH}^*\text{CH}_2$  bond followed by a series of complex secondary reactions. The subsequent intermediate mechanism of decomposition depends on the specific structure of the nylon, but the combustion and products do not differ much between types of nylon [71, 72]. Strauss and Wall [73] studied various nylons and concluded that decomposition is caused by radical formation and random cutting, and the main



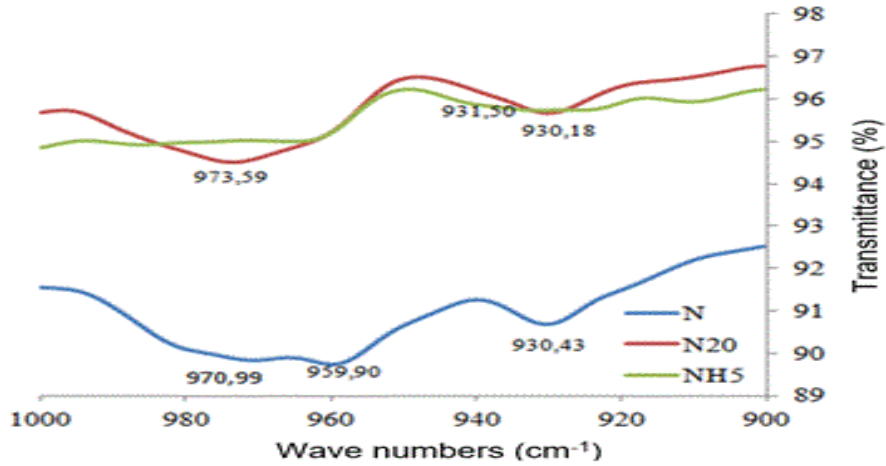


Figure 9: FTIR spectra of region IV on samples of N (fishing string), N20 (membrane), and NH5 (membrane mixed with hydroxyapatite)

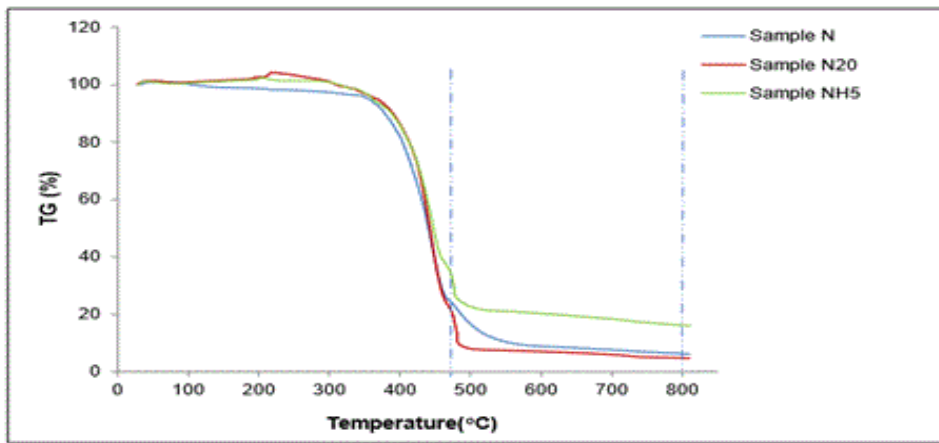


Figure 10: TGA Curve of Sample Fishing Line (N), Nanomembrane (N20) and Nanomembrane + HA (NH5)

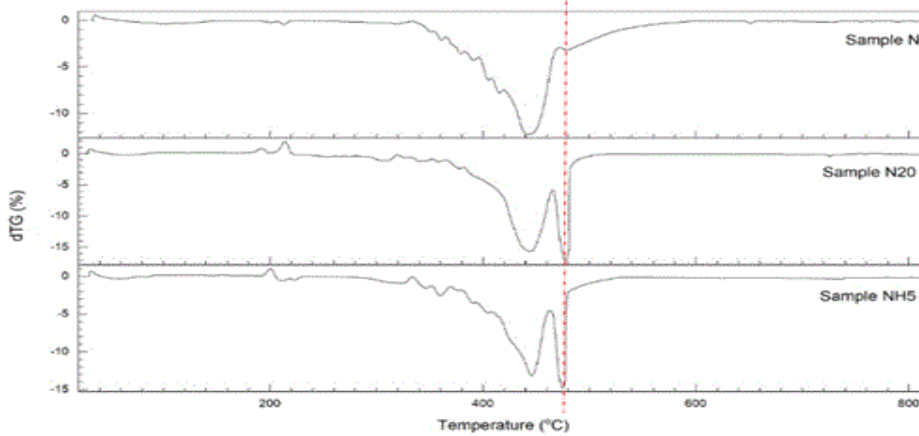


Figure 11: dTG Curves of Samples N, N 20 and NH5



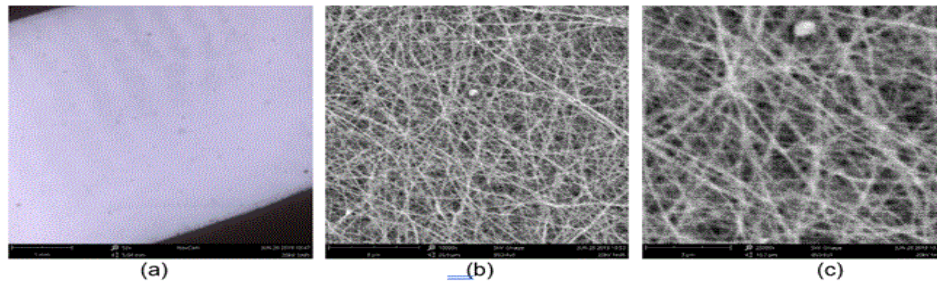


Figure 13: Results of SEM characterization of Nanomembrane with 52x magnification (a), 10000x magnification (b), 25000x magnification (c)

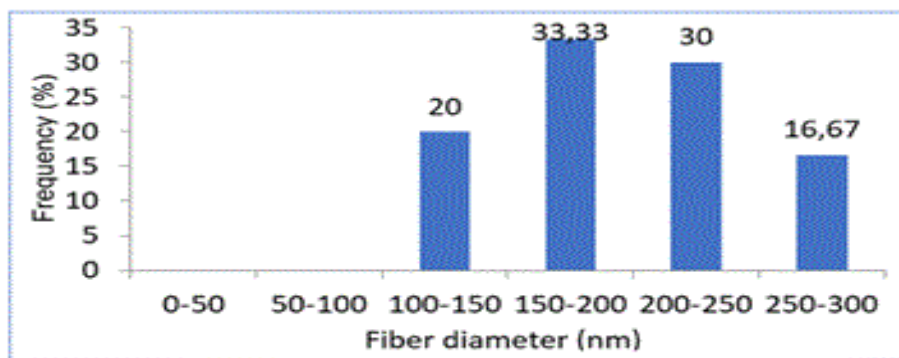


Figure 14: Results of SEM characterization of Nanomembrane with 52x magnification (a), 10000x magnification (b), 25000x magnification (c)

diameter of  $196.33 \pm 43.17$  nm with a diameter range of 117 nm to 268 nm, Figure 14.

In Figure 15, it is shown that the nylon fiber diameter achieved respectively, fiber diameter between 150-200 nm was  $33.3\%+3.15\%$ , fiber diameter between 200-250 nm was  $30\%+2.91\%$ , fiber diameter between 100-150 nm by  $20\%+2.23\%$  and fiber diameter between 250-300 nm by  $16.67\%+2.41\%$ . This shows that the fiber diameter achieved is still between 0.1 and 0.3 microns, while the diameter of 0.1 microns or 100nm is still relatively small. Optimization of the electrospinning process needs to be studied further by further examining the influence of voltage, flow rate, tailor distance to collector, viscosity of the nylon solution.

#### Tensile Strength Test of the Membrane

The samples for this tensile test are in the form of membranes with different thicknesses due to differences in the process of using voltage in the electrospinning tool used. In the formation of this membrane, it can be seen that the greater the voltage value used, the thicker the membrane will be with a smaller nanofiber diameter as seen in Figure 16. The results of the thickness analysis can be seen in Table 5, where the average thickness of the membrane for the N10 sample (voltage 10 kV) of 0.057 mm, sample N15 (voltage 15 kV) of 0.061 mm and sample N20 (voltage 20 kV) of 0.068. Thus, the greater the voltage, the thicker the resulting membrane.

Morphological testing using SEM is carried out by randomly selecting the membrane that forms. This random sampling was carried out because the frequency of testing using SEM was high, so that out of 3 membranes only 1 sample was tested. In the SEM testing process, membranes were taken using a voltage of 20 kV. Fibers produced using an electrospinning voltage of 20 kV produced an average fiber diameter of  $196.33 \pm 43.17$  nm with a diameter range of 117 nm to 268 nm.

The membrane tensile strength test was carried out using a Gotech 2kN UTM tool. Tensile tests were carried out on membranes formed using voltage variations on an electrospinning tool.

Tensile strength testing uses a speed of 10 mm per minute. The tensile strength test results of each membrane produced can be seen in the graph in Figure 17, and the summary is in Table 5. It can be seen that the voltage in electrospinning significantly influences the tensile strength and elongation of the membrane.

Figure 16 shows the tensile strength of several membranes with varying stresses, namely samples N10, N15 and N20. A recapitulation of the nylon membrane test results using the UTM tensile strength test tool can be seen in Table 5, where each sample is looked at for tensile strength and elongation (Figure 17) and thickness (Figure 16).

In Table 5, it can be seen that the voltage used in electrospinning greatly influences the thickness of the membrane produced in



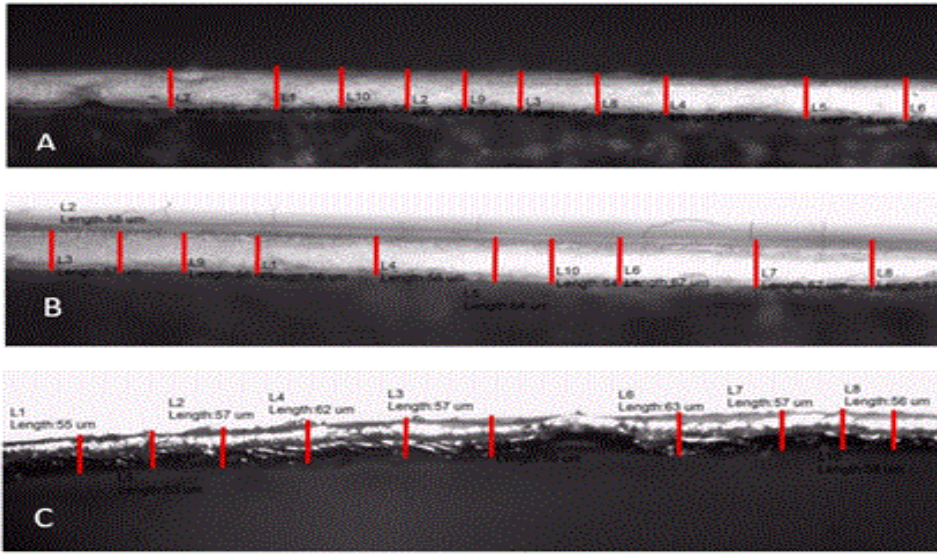
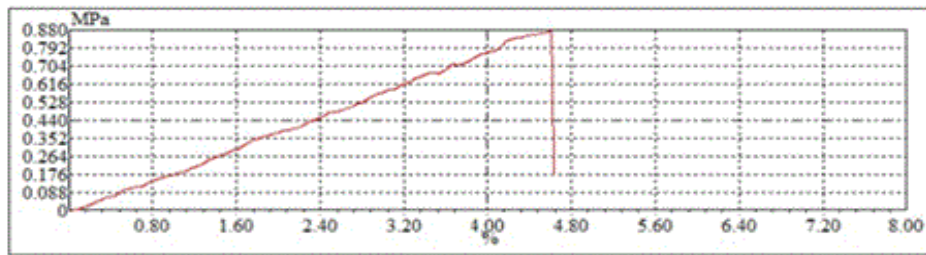
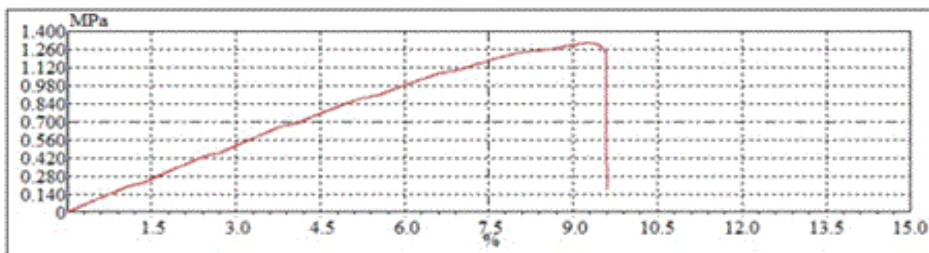


Figure 15: Thickness of the Membrane Resulting from the Variation of Voltage 10 kV (A), 15 kV (B), 20 kV (C)



(a)



(b)

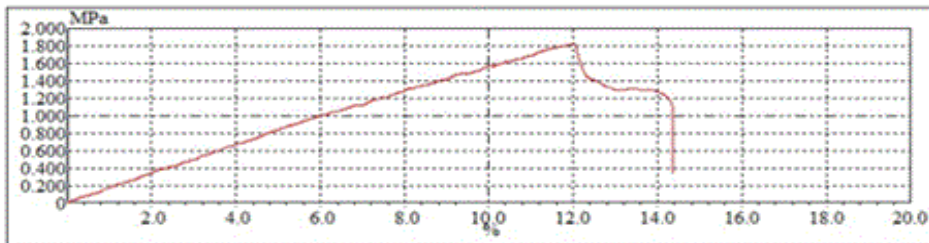


Figure 16: Graph of membrane tensile strength produced by electro spinning of samples N10 (a), N15 (b) and N20 (c)



**Table 5. The results of the tensile strength test of the membrane**

Test results	N10	N15	N20
Tensile strength (MPa)	0,87	1,31	1,82
Elongation (%)	4,61	9,25	12,01
Thickness (mm), rata-rata	0,057	0,061	0,068

the same time span. This also affects the elongation and tensile strength. The thicker the membrane, the higher the elongation and tensile strength. From this table it can also be seen that the tensile strength of the membrane at a voltage of 20kV (N20) is 2 times greater than the tensile strength of the membrane at a voltage of 10kV (N10), even the elongation of the membrane at a voltage of 20kV is almost 3 times compared to the elongation of the membrane at a voltage of 10kV.

**Effect of Voltage and Flow Rate on Fiber Size**

**Effect of Tension on Fiber Size**

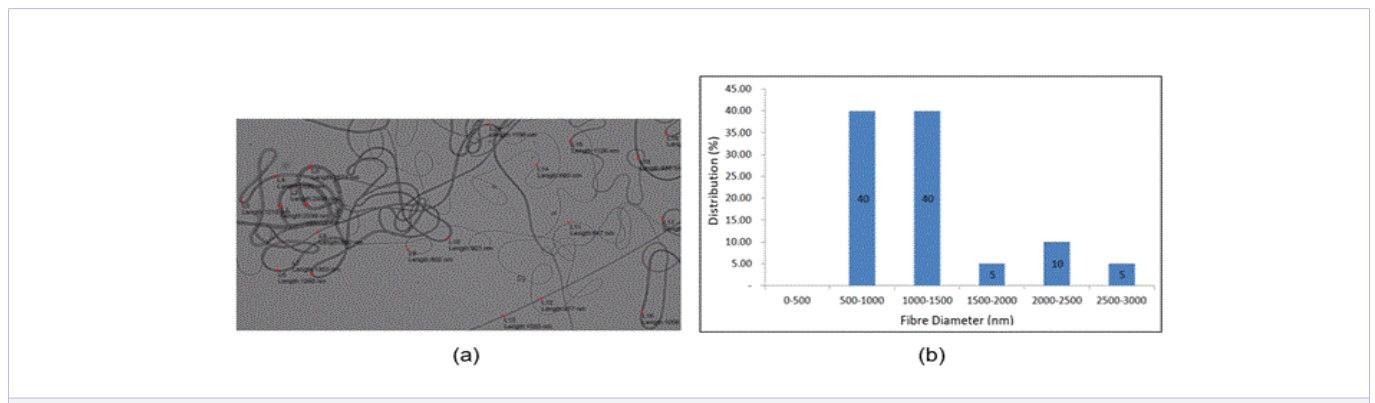
In the previous chapter, indications were obtained that tension greatly influences fiber size. To confirm this, observations were carried out using a polarizing microscope owned by the Advanced Materials Research Center, BRIN. The results are as follows:

a). At a voltage of 10 kV, the fibers formed tend to be free from beads, it's just that there are fibers whose shape is not yet perfect because they are like widened ribbons. These results indicate that a voltage of 10 kV is still not ideal for the formation of nylon fibers. Apart from that, the fibers produced are not straight and tend to be irregular. This indicates that electrical energy is still not able to drive the nylon solution optimally. Likewise, the size of the fibers produced varies from 693 nm to 2674 nm with an average diameter of 1277.8 nm + 97.71 nm. The polarization microscope image and fiber size distribution can be seen in Figure 17.

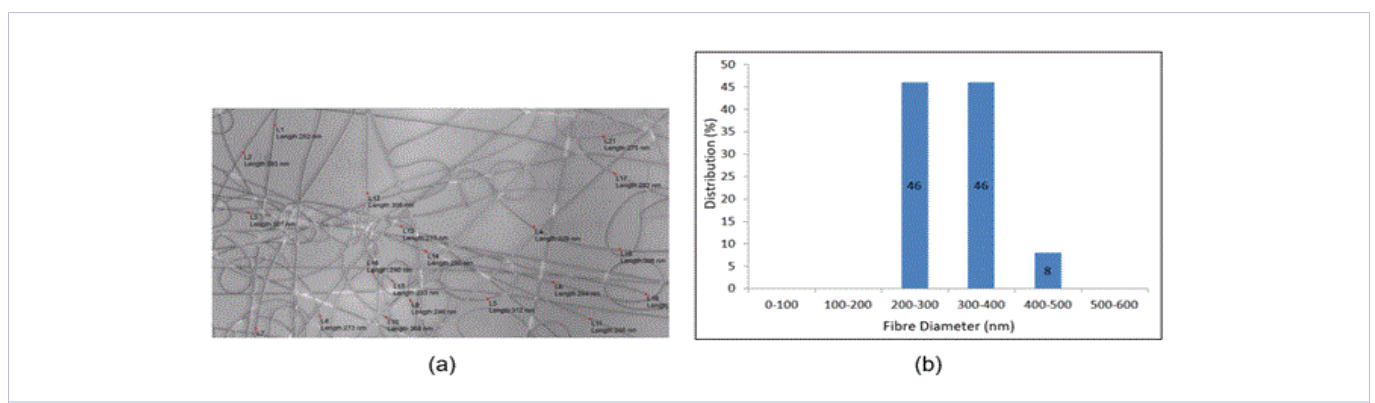
b). At a voltage of 15 kV, the fibers formed tend to be free from beads and the fibers appear more even. The size of the fibers produced varies from 205 nm to 468 nm with an average diameter of 305.2 nm + 45.51 nm. The polarization microscope image and fiber size distribution can be seen in Figure 18.

c). At a voltage of 20 kV, the fibers formed are free from beads and the fibers appear flatter and more uniform. The size of the fibers produced varies from 175 nm to 437 nm with an average diameter of 300.9 nm + 46.36 nm. The polarization microscope image and fiber size distribution can be seen in Figure 19.

From the results of observations of nanofibers produced using an electrospinning machine, it is clear that the influence of



**Figure 17:** Fiber Size Distribution measured with a Polarizing Microscope at a voltage of 10kV, nanofiber (a) and Fiber Size Distribution (b).



**Figure 18:** Fiber Size Distribution measured with a Polarizing Microscope at a voltage of 15kV, nanofiber (a) and Fiber Size Distribution (b)..

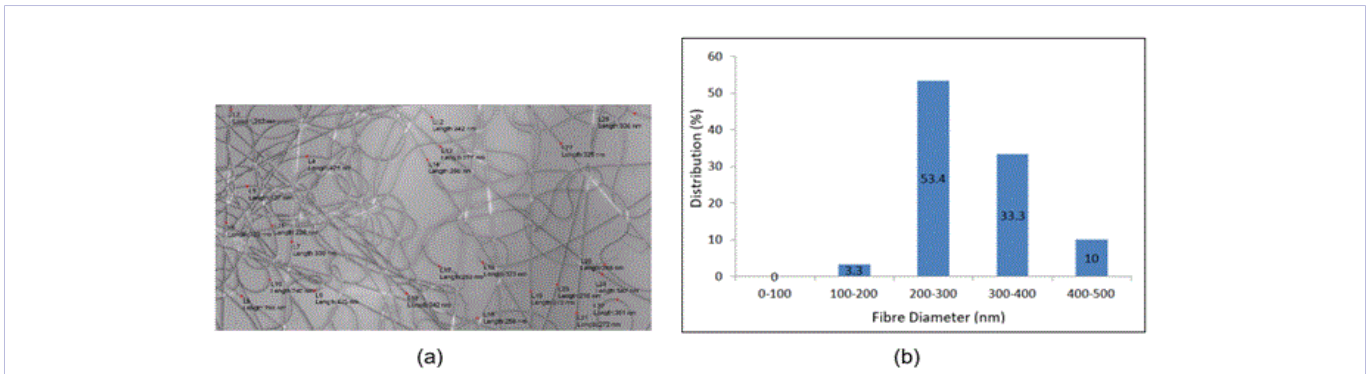


Figure 19: Fiber Size Distribution measured with a Polarizing Microscope at a voltage of 20kV, nanofiber (a) and Fiber Size Distribution (b)

voltage on the formation of nanofibers is very large. Nanofibers smaller than 300 nm are produced from voltages between 15kV (N15) and 20kV (N20). This is shown where at a voltage of 20kV, the number of nanofibers with a diameter smaller than 300 nm produced is +56.4% and +46% is produced at a voltage of 15kV. However, both voltage levels (15kV and 20kV) have been able to produce nano-sized fiber diameters (<300 nm). It is possible that the difference between the voltage of 15 kV and 20kV is that there is a greater drawdown in the N20 (20kV) sample than in the N15 (15kV) sample.

#### Effect of Flow Rate on Fiber Size

In this experiment, 2 variations of flow rate were used, namely 0.5 ml/hour and 1.5 ml/hour at the same voltage, namely 15 kV.

a). At a flow rate of 0.5 ml/hour with a voltage of 15 kV, the fibers produced tend to be free from beads and the fibers appear flatter. The size of the fibers produced varies from 156 nm to 361 nm with an average diameter of 251.6 nm + 39.38 nm. The fiber size distribution can be seen in Figure 20.

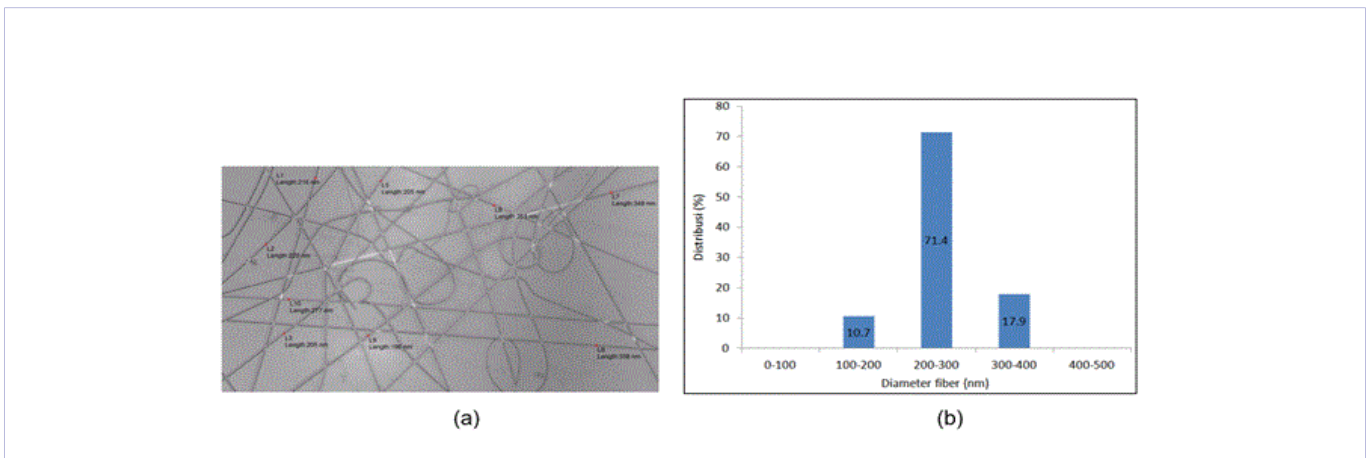
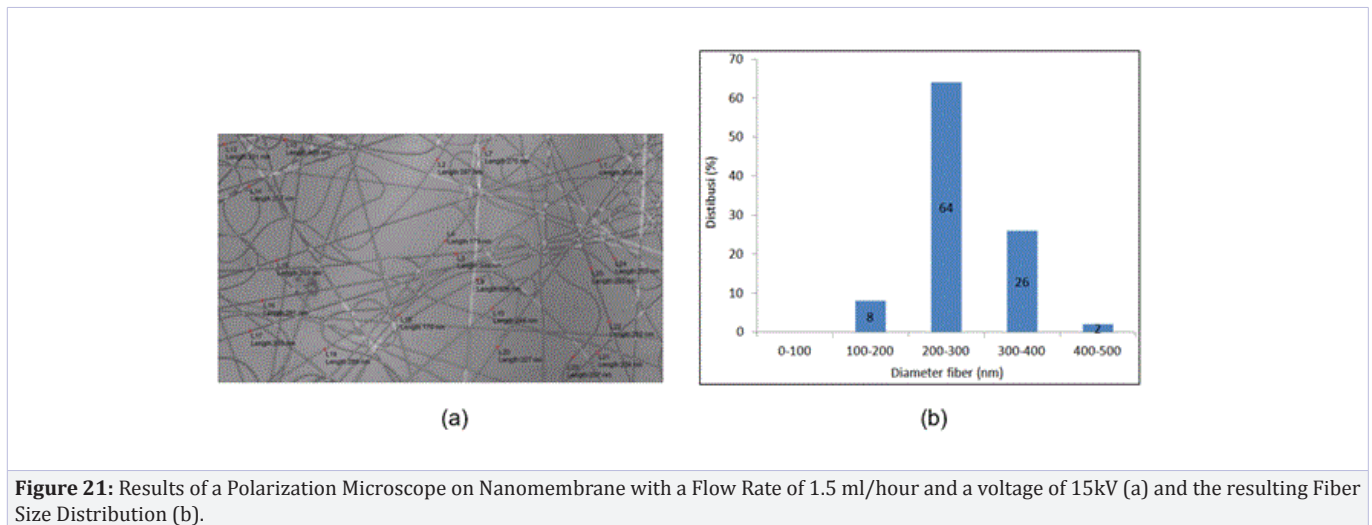


Figure 20: Fiber Size Distribution measured with a Polarizing Microscope at a voltage of 20kV, nanofiber (a) and Fiber Size Distribution (b)

b). At a flow rate of 1.5 ml/hour and a voltage of 15 kV, the fibers formed tend to be free from beads and the ribbon-like fibers begin to disappear. The size of the fibers produced varies from 153 nm to 409 nm with an average diameter of 272 nm + 40.03 nm. The fiber size distribution can be seen in Figure 21.

This experiment shows that the flow rate influences the diameter of the nanofibers, the greater the flow rate, the smaller the percentage of nanofibers with a diameter smaller than 300 nm, where at a flow rate of 0.5 ml/hour the nanofibers produce +82.1%, whereas with a flow rate of 1.5ml/hour produces +72% nanofiber. This is likely caused by differences in solution supply at the same voltage. A solution supply of 0.5 ml/hour will be

smaller than a solution supply of 1.5 ml/hour at the same voltage. As a result, there was greater withdrawal at a flow rate of 0.5 ml/hour than at a flow rate of 1.5 ml/hour. This results in the fiber diameter being smaller at a flow rate of 0.5 ml/hour compared to a flow rate of 1.5 ml/hour, so that the percentage of fibers with a diameter between 100-200 nm, at a flow rate of 0.5 ml/hour reaches 10.7% compared to a flow rate of 1.5 ml/hour of 8%. Likewise, for fiber diameters between 200-300 nm, at a flow rate of 0.5 ml/hour it reaches 71.4% compared to a flow rate of 1.5 ml/hour of 64%.



**Figure 21:** Results of a Polarization Microscope on Nanomembrane with a Flow Rate of 1.5 ml/hour and a voltage of 15kV (a) and the resulting Fiber Size Distribution (b).

## Conclusion

There are several methods for producing nano-sized fibers, one method that can produce nano-sized fibers is electrospinning. The electrospinning method is a method that utilizes a large enough voltage to produce nanoscale fibers without leaving waste or waste from the solution because the solution will evaporate when it reaches the collector. Thus, this method is more efficient, cheaper, leaves no waste and is environmentally friendly.

Initial identification of the fishing line material shows that the material is made from nylon 6 fibers. The voltage and flow rate in electrospinning greatly influence the mechanical properties of the membrane, including the morphology and size of the fibers. The higher the voltage, the thicker the membrane and the higher the strength and elongation. Meanwhile, the smaller the flow rate, the smaller the fiber size at the same voltage.

Hydroxiapatite additives influence the temperature at which material degradation occurs and also influence the tensile strength of the membrane. Hydroxiapatite also increases the percentage of TGA residue to 16.05 at a temperature of around 8000C. This indicates the possibility that hydroxyapatite containing phosphorus influences the burn-resistant and melt-resistant properties of the membrane, or in other words the phosphorus in hydroxyapatite has an effect on the burning behavior of the membrane. Further research needs to be carried out on the effect of other additives, especially their effect on anti-melting.

Membranes or filters made from nanofibers have huge prospects in several industrial fields, including health, the environment and others. When used as a filter, nanofiber membranes can filter nano-sized particles, they can also filter nano-sized bacteria or viruses.

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## Contributor

SR is responsible for implementing activity planning, preparing budget plans, YP is responsible for electrospinning machine operations, NA is responsible for carrying out tests and analyzing FTIR results, DF is responsible for carrying out tests and analyzing TGA results, MEH carries out planning and cost analysis, SeRo conducts partner collaboration exploration, MSI helps carry out work in the laboratory, L carries out formula synthesis and process solution preparation, SH as the main contributor is responsible for planning the entire program starting from preparing proposals, selecting methods, preparing material formulations, preparing test plans, analyzing results and budget preparation and reporting.

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