TENSILE BEHAVIOR OF ELECTROSPUN POLYACRYLONITRILE (PAN) NANOFIBER MEMBRANE FOR WATER DESALINATION PROCESS

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A report submitted in fulfillment of the requirements for the degree of Bachelor of Mechanical Engineering

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

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DECLARATION

I declare that this project entitled "Tensile Behavior of Electrospun Polyacrylonitrile (PAN) Nanofiber Membrane for Water Desalination Process" is the result of my own work except as cited in the references.

Signature :

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Date : 20 JUNE 2021

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APPROVAL

I hereby declare that I have read this project report and in my opinion this is sufficient in term of scope and quality for the award of the degree of Bachelor of Mechanical Engineering.

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ABSTRACT

Desalination is one of the advanced technologies to manage the freshwater shortage and membrane distillation (MD) is one of the powerful application for desalinating water. MD is a non-isothermal membrane-based desalination method controlled by the temperature difference created within the membrane. There is also the limitation issued as the membrane breakage can occur when mechanical strength is insufficient due to unable to withstand the stress and pressure applied during the MD procedure. To build a membrane that is ideal for long-term performance, comprehensive research is required to investigate the relationship between the MD and mechanical properties. The polyacrylonitrile (PAN) and electrospinning are the polymer and technique chosen to generate the desired feature of nanofiber membrane that is compatible with water desalination application. The preparation of the solution and fabrication of nanofiber were discussed. The parameters during the electrospinning process are controlled because it gives an impact on the result. The characterization process is done using scanning electron microscopy (SEM) to assess the morphological characteristic and fiber diameter of the fibers. The average diameter of the fiber was decreased as the tip-to-collector distance increased. A comparative study for tensile strength was conducted to compare the result of tensile behavior. Tensile strength and Young's modulus were increased while the strain of the nanofiber was decreased if the fillers were added. This study aims to show a brief introduction to electrospun PAN nanofiber and a comprehension to achieve the required properties in polymeric membranes used in MD for desalination purposes.

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CHAPTER 1

INTRODUCTION

1.1 Background of the study

The demand for freshwater resources is already crucial in many regions and will be one of the major concerns as the world's population continue to grow each of every day. Due to various growing scarcities in many nations, it is already widely believed the world will confront a serious water problem in the next generations. Various human rights organizations have issued graphics in the past few years, all of which illustrate that growing scarcities of water are causing even more nations around the globe to become progressively water-stressed (Biswas, A.K, 1999). In this modern decade, technologies can be utilized to fulfill the world's demand.

One of the improving solutions to solve this problem is with treating the saline water with the desalination process. Saline water is water that carry a high concentration of dissolved salt. Recent research on United States Geological Survey, there is a 1% weight of dissolved salt in the water that has a concentration of 10,000 parts per million. The method that removes minerals components from saline water is called desalination. Tliti, I. and Alkanhal, T. A. (2019) state that several methods including distillation, chemical disinfectant treatment, sand filtration, reverse osmosis, (RO), nanofiltration (NF) are familiar widely around the world to provide humans with all-important freshwater. RO and NF are membrane separation technologies that need driving forces such as pressure and electric potential. In this process, the water containing dissolved salt molecules is forced through a filter which only the smaller water molecules can get through the membrane

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holes. Furthermore, membrane separation is considered a new method that has benefits including low power consumption, chemical-free, and low-temperature operation. Besides RO and NF, another latest technology that is still in process of being developed is membrane distillation (MD). It is a non-isothermal membrane-based technology that uses a sufficiently hydrophobic microporous membrane to desalinate seawater. The temperature difference formed throughout the membrane is used to operate MD (Ravi et al., 2020). Although the membrane separation technologies have many benefits, it also has its disadvantages such as permeation flux decreased by membrane pore blockage. Abdelrasoul et al. (2013) found this circumstance called membrane fouling which mentions membrane pore blockage during the filtration process on the surface and pores of the membrane. The production rate of permeate is reduced meanwhile the operation difficulty of the membrane filtration is increased both caused by pore blockage. For more membrane growth and implementations, this is the most challenging issue.

Due to their unique features, the electrospun nanofiber is recently being preferred for highly efficient nanofiltration material production. Tliti, I. and Alkanhal, T. A. (2019) state that the need for beneficial breakthrough filtration technology of recognition that relies on advanced materials for filtering application which use nanofiber membranes. Electrospinning is an advanced technique to produce nanofiber. According to Subbiah et al. (2005), electrospinning has obtained popularity because of its flexibility in spinning a broad range of polymeric fiber applications and its performance in submicron fiber processing. Despite the fact there is a various application available for nanofiber membrane fabrication, electrospinning has many benefits over all of them. This is because this process straightforward and can simply manage the fiber morphology and fiber orientation due to its low and minimum beginning investment. In electrospinning, the fibers can be generated in a short time and their morphology can be managed by process

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parameters (Subbiah et al., 2005). However, during the electrospinning process, the loaded forces on the fiber can cause temporary or permanent mechanical failure. It is important to recognize either the fiber is strong enough to withstand the external and internal forces exerted throughout the process (Abdelrasoul et al., 2013). This study is aimed to discuss and evaluate further mechanical properties specialized in the tensile behavior of electrospun polyacrylonitrile (PAN) nanofibres membrane for the water desalination process.

1.2 Problem Statements

Membrane distillation (MD) in the water desalination process has grown outstandingly in the past few decades due to its benefits compared to other water treatment applications. The concept of the MD process is quite straightforward in which the membrane performs as a filter that will allow water to flow through, while at the same time it captures suspended solids and other substances. Throughout the process, the saline water is heated and the membrane only produces pure water vapor that has being evaporated at the surface of the membrane.

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Although there are many advantages of this technology such as environment-friendly and have a very simple flow sheet, there are also unavoidable risks. The risk included is the decrease of permeation flux due to blockage that occurred at the membrane pore. The fouling will take place due to the presence of blockage which can greatly limit the permeation rate through the membranes and make them essentially incompatible for such applications. Inorganic fouling, colloidal fouling, biological fouling, and organic fouling are four groups of fouling based on fouling material. By implementing some form of water treatment and choosing a membrane material that resists the adsorption of organic material to the membrane, it can reduce organic fouling problems. The characteristic of the solution or material such as the concentration of solid, properties of particles, pH, and strength of ionic also contributes to the membrane fouling (Abdelrasoul et al., 2013). Fouling of membrane is the relationship between the membrane and the solution. Hence, the solution properties should be analyzed thoroughly so that a suitable method can be selected. The next risk is the pore collapse and membrane breakage that can occur when mechanical strength is lacking due to the mechanical behavior that unable to withstand the force and pressure exerted during the MD operation (Ravi et al., 2020). This situation will affect the performance of the membrane.

It is essential to determine the thickness of the membrane whether it is best to be thicker or thinner because it will affect the mechanical behavior such as tensile strength. The force and the pressure exerted also have their roles. As an example, the liquid entry pressure (LEP) should be lower than the pressure exerted on the feed channel to avoid membrane fouling. According to Ravi et al, 2020, one of the challenges of researchers is to achieve a good balance between LEP and permeability of membranes. The authors also found that thicker membranes provide greater mechanical strength but reduced permeability. Hence, a comprehension study is needed to analyze the relationship between the MD and mechanical properties to produce the membrane that is excellent for long term performance. The suitable material and techniques are chosen to produce the desired characteristic of nanofiber membrane that is compatible with water desalination application.

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1.3 Objectives

1. To fabricate electrospun polyacrylonitrile (PAN) nanofiber membrane
2. To analyze the morphological characteristics of electrospun polyacrylonitrile (PAN) nanofiber membrane
3. To implement a comparative study for tensile behavior of electrospun polyacrylonitrile (PAN) nanofiber membrane

1.4 Research scopes

The research scopes were determined as below:

i. The selected material is polyacrylonitrile polymer (PAN) and the solvent used for dissolving the PAN powder is dimethylformamide (DMF).

ii. The technique chosen is electrospinning which will produce an electrospun PAN membrane.

iii. The electrospinning variable parameter for morphological characterization is tip-to-collector distance with 10 cm, 15 cm, and 20 cm with a fixed running time of 5 minutes. The voltage and rotation per minute applied are 15 kV and 120 RPM.

iv. The morphological characteristics and fiber diameter of electrospun PAN nanofiber were analyzed using Scanning Microscopy Electron (SEM) and Image J software.

v. A comparative study was conducted to compare the result of tensile behavior. Tensile strength , Young's modulus and strain result being reviewed.

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1.5 Thesis outline

The thesis is split into five chapters. The thesis starts with an introduction, literature review, methodology, result, and discussion as well as a conclusion. An introduction explained the background of the selected research title including the overview of the membrane distillation (MD) process. The technique to produce nanofiber membrane also be outlined in this chapter. This chapter further highlights the problem statement, objectives, scopes, and thesis outline. Chapter 2 discussed the literature review associated with the research topic. It is including a detailed explanation of the relationship between the nanofiber and MD. The literature review also focused on the material used and the selected technique to produce nanofiber membrane which is electrospinning. The electrospinning conditions and parameters that affect the nanofiber membrane are stated. The general characteristic of PAN nanofiber membrane after being electrospun also being viewed. This chapter also includes some of the earlier research into these studies.

A thorough explanation of the methodology is explained in chapter 3. This chapter discusses the material selection as well as the preparation of the PAN solution. After the material was prepared, the fabrication of nanofibers was conducted through the electrospinning process. The variable parameter set in the process is tip-to-collector distance meanwhile, other parameters were set to be fixed. The morphological characterization procedure using SEM also being discussed in this chapter. For tensile strength, a comparative study was conducted to observed the importance Chapter 4 explained the results from the SEM analysis such as morphological characteristics and fiber diameter, An analysis of tensile strength from the earlier studies being compared and discussed. Lastly, Chapter 5 is the research conclusion as well as the proposed possible upcoming exploration.

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CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Nanofibers are ultrafine fibers with micro-to-nanometer range diameters and controlled surface morphology. Nanofibers diameters depend on the manufacturing procedure and the type of polymer implement. Interfacial polymerization and the electrospinning process can generate nanofibers. All polymers nanofibers are special because of their wide surface-area-to-volume proportion, high porosity, significant mechanical power, and the flexibleness of usability (Barbosa, M. A., 2017). Various types of polymers can produce nanofibers and have dissimilar potential for application and unalike physical characteristics.

Recently, nanofibers materials have already been marketed for some air filtration applications and it has been explored for liquid separation in particular for water treatment. It is due to their small and normal pore size and also low hydraulic resistance obtained from intrinsically high porosity (Barbosa, M. A., 2017). Also, the relatively high surface area of these materials makes it possible to use them in absorptive applications. Yalcinkaya, F. et al. (2017) states that nanofiber characteristics such as their capability to immersed in other media, bigger surface ratio, sizeable porosity, small pore size, operating simplicity, and adjustable features are more reliable than other existing polymeric membranes used for filtration of liquids. The author also states that is over 80% porosity of nanofiber in the form that increases the effectiveness of membranes filtration. One of the latest technologies of liquid separation is called membrane distillation (MD) and it is getting known in the wastewater treatment process. MD offers some of the advantages compared to other membrane separation techniques such as need minimal mechanical requirement since MD require lower temperature than other conventional distillation technique while operated.

The operating pressure can be low as well since the temperature is low. The principle is the feed stream is filled with hot liquid or saline water after being heated (B. Shahroie et al., 2016). Those liquid or saline water on the hot feed side will evaporate at the membrane's surface after being heated. The vapor entering throughout the membrane condensing at the permeate stream on the cold side (Yalcinkaya, F., 2019). The membrane between the feed stream and permeate stream performs as a filter that will allow only freshwater flow through the permeate side, while at the same time it captures suspended solids and other substances (Yalcinkaya, F., 2019). The illustration of the MD process is shown in Figure 2.1.

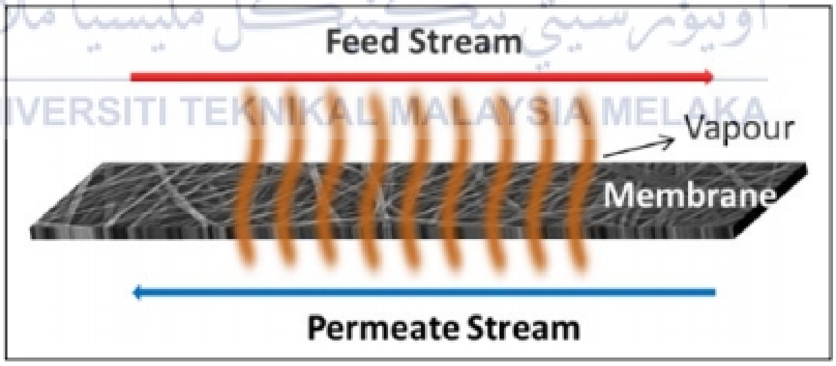

Figure 2.1: Illustration of MD process (Yalcinkaya, F., 2019)

The pore sizes of the membrane and the particle filtering abilities lead to the various types of membrane separation method exist today (Yalcinkaya, F., 2019). Nanofiber is a good choice for the MD process due to several requirements such as stable thermal properties over a bigger range of temperature, suitable pore arrangement, high

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permeability, and hydrophobicity as well as have good mechanical strength (Yalcinkaya, F., 2019). In a real-world application, the membrane module that being assembled might be in any type such as hollow fiber, spiral wound, or plate-type module. The membrane needs to have good control of temperature, pore arrangement, permeability, and mechanical strength to get the maximum efficiency and at once making it one of the cost-effective technique since it requires low maintenance cost (Yalcinkaya, F., 2019).

2.2 Electrospinning

Electrospinning, adapted from electrostatic spinning is a technique familiarize to manufacture fibers with diameters varying from a few nanometers to micrometers using a high voltage power source (Jose Varghese, R., 2019). This process is used to produce polymer nanofibers, metals, ceramics, and composite materials. Electrospinning has two types of methods which are solution electrospinning (S-ES) or melt electrospinning (M-ES). Many types of polymers are used in electrospinning which may create precise nanofibers in the submicron range used for a variety of implementation.

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Variation of unnatural polymers, natural polymers, or a combination of the pair is the list of materials that have been identified to build electrospun nanofiber. Zheng and Yongmei (2019) state that about 200 and more polymers from a diversity of natural polymers have been successfully electrospun. Referred to the past findings, it is apparent that the standard diameter of electrospun fibers scales from 100 nanometres to 500 nanometres whereas, in textile and fiber technology-related research writings, fibers with a diameter of between 100 nanometre and 500 nanometres are broadly mentioned as nanofibers (Subbiah, T., 2005). In the electrospinning process, the solution flow rate was set using a syringe. The PAN solution was cautiously filled into the syringe and was electrospun with adequate tip-collector distance. The PAN nanofibrous membrane was collected on an aluminum foil placed in a collector. The polymer solution melt along the spinneret is pushed throughout the flow pump's control to produce a pendant drop at the tip of the spinneret when the high voltage potential is applied (Wei, Q. et al., 2012). Hence, because of the same applied voltage, the free charges are generated into the polymer solution. In feedback to the applied electrical field, these charged ions pass towards the opposite polarity electrode, transmitting the stretching forces produced by the electrical field to the polymer liquid. The cone-shaped known as the Taylor cone (Figure 2.2) is created due to the stretched of the fluid hemispherical surface at the spinneret tip as the applied electric field is rising.

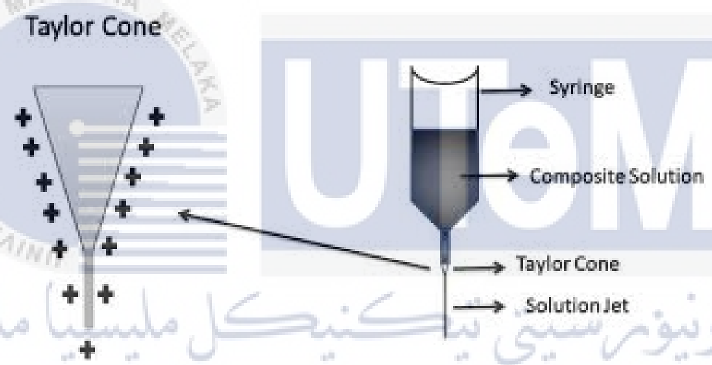

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Figure 2.2: Taylor cone (Al-Hazeem, N. Z. A., 2018)

The electrostatic force surpasses the interfacial tension since the high electrical field applied exceeds the critical value. Due to these circumstances, the solution charge jet is expelled from the Taylor cone tip and quickly expediting to the grounded collecting purposed. The process is continued until the charged jet undergoes extreme whipping and elongation, allowing solvent evaporation or melt cooling to form solid fibers on the collector (Wei, Q. et al., 2012).

According to Wei, Q., et al. (2012) there are four major components of electrospinning apparatus:

i. **High voltage power source**

A high-voltage power source is necessary for providing an electrostatic field for electrospinning. The source needed is depending on the substance that will be electrospun.

ii. **Flow control pump**

Because flow rate influences the diameter and efficiency of the fiber, the flow control pump is used to rearrange the flow rate of the spinning fluid to create a fiber with a controllable diameter.

iii. **Spinneret**

Polymer solution or melt will pass along the small tubes that create from the spinneret. A spinneret can be arranged in various configurations depending on the form of the fibers.

iv. **Collector**

A collector can be designed to receive various fiber assemblies in different forms, as needed. It can be a rotating drum, a rotating disc, a plate, or any other shape.

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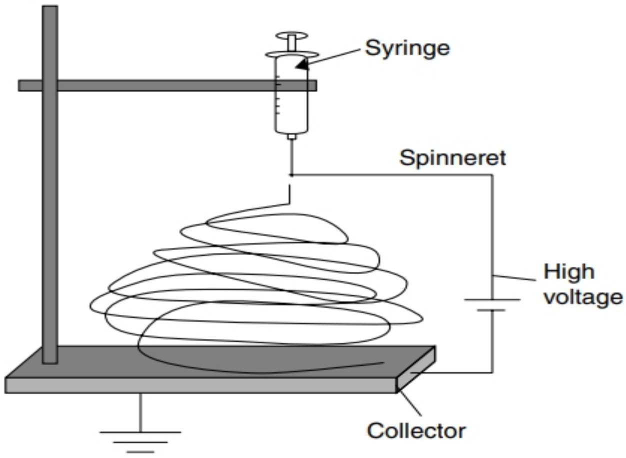

Figure 2.3: Schematic diagram of the electrospinning process (Wei, Q. et al., 2012)

A typical schematic diagram of the electrospinning process is shown in Figure 2.3. The equipment, as well as the components, can be set up and modified following the real needs. The polymer solution or melt along the spinner is pushed throughout the flow control pump to produce a pendant drop at the tip of the spinneret when the high voltage potential is applied (Wei, Q. et al., 2012). Hence, because of the same applied voltage, the free charges are generated into the polymer solution. In feedback to the applied electrical field, these charged ions pass towards the opposite polarity electrode, transmitting the stretching forces produced by the electrical field to the polymer liquid. The cone-shaped known as Taylor cone is created due to the stretched of the fluid hemispherical surface at the spinneret tip as applied electric field is rises.

The electrostatic force surpasses the interfacial tension since the high electrical field applied exceeds the critical value. Due to these circumstances, the solution charge jet is expelled from the Taylor cone tip and quickly expediting to the grounded collecting purposed. The process is continued until the charged jet undergoes extreme whipping and elongation, allowing solvent evaporation or melt cooling to form solid fibers on the collector (Wei, Q. et al., 2012). This is happened due to an unstable charged jet. A further explanation of the procedure will be included in the methodology section. The benefit of electrospinning is not counting other complicated processes, it just needs a liquid that can scatter nanoparticles and disintegrate polymers. Electrospinning is generally dependent on high electrostatic forces. Factors that affect electrospinning are the concentration of polymers, viscosity and flow rate of the solution, electrical field voltage, and air humidity.

One of the important conditions that affect nanofibers formation is solution properties. The first property is polymer molecular weight which represents the amount of bonding of polymers chain in the solution which also determines its viscosity. Earlier studies found the beads are produced instead of fibers due to low molecular weight solution. Thus, the higher molecular weight solution assists to produce more fibers with a bigger diameter (Huang et al., 2018 cited in Wei, Q. et al., 2012). Consequently, an appropriate amount of polymer chain bonding will allow a sufficient level of viscosity even the concentration of polymer is low. Uniform fiber web and surface tension limitation can be created with an adequate viscosity.

The next property is the concentration of the solution. Researchers found that low solution concentration happened due to mixing the beads and fibers. With the higher concentration, the shape of the bead that was originally spherical changed to a spindle-like shape. The diameter of fibers becomes bigger and in a uniform state (Deitzel et al., 2001 cited in Wei, Q. et al., 2012). However, the higher the concentration can lead to the failure to ensure the movement of the solution at the spinneret tip. This is happened due to the forbidden development of pursuing fibers. The third property is conductivity which is firstly controlled by the kind of polymer and solvent used. The higher conductivity is highly unbalanced which leads to drastic bending instabilities. The condition of the electrospinning process also give a big impact on nanofiber formation. The most important condition is the applied voltage. The fiber diameter will be decreased and the solvent

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