UNIVERSITI TEKNIKAL MALAYSIA MELAKA

ELECTROSPINNING PVA NANOFIBRES STUDY ON DIFFERENT PHYSICAL PARAMETERS USING FACTORIAL DESIGN METHOD

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Faculty of Mechanical and Manufacturing Engineering Technology

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A thesis submitted
in fulfillment of the requirements for the degree of
Bachelor of Mechanical Engineering Technology (Refrigeration and Air Conditioning System) with Honours

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2022

DECLARATION

I declare that this Choose an item. entitled “Electrospinning PVA Nanofibres Study on Different Physical Parameters Using Factorial Design Method” is the result of my own research except as cited in the references. The Choose an item. has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature : 

Name : Ida Najiha Binti Junus

Date : 18 January 2022

APPROVAL

I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the Bachelor of Mechanical Engineering Technology (Refrigeration and Air Conditioning System) with Honours.

Signature : 

Supervisor Name : Ts. Dr. Amir Abdullah Bin Muhamad Damanhuri

Date : 28/1/2022

DEDICATION

To my beloved parents,

Junus Bin Mamat and Masnorlila Binti Mat Idris.

Thank you for your gentleness in caring for me, supporting, advising, and loving me.

Thank you to my supervisor, Ts. Dr. Amir Abdullah Bin Muhamad Damanhuri, Azmil Arif Bin Mohamad Wazir, and also to the friends for your help.

Thank you to the Faculty of Mechanical Engineering for providing the opportunity to use machines and equipment to conduct this research.

ABSTRACT

Nowadays, electrospinning is a simple and fast technique for producing fibers with nano-scale diameters from various types of polymers. These nanofibers exhibit several important characteristics such as a surface area with volume ratio, high porosity, and superior mechanical properties. Nanofiber air filtration applications for the industry are of concern at present. The use of Design of Experiment (DOE) software was used as an application to generate 19 samples. In this study, 19 samples were used containing a variety of concentrations, distances, voltages, and feed rates. It is also known as the input variable factor. Consumption concentration between 8wt% to 12wt%, Distance between 10cm to 20cm, voltage between 10kV to 20kV and feed rate is between 1mL/h to 2mL/h. Polyvinyl Alcohol (PVA) has been used with tap water as a solvent. Electrospinning samples were used to study the Average Fiber Diameter (AFD) using a Scanning Electron Microscope (SEM). SEM sample results show AFD is from between 325nm to 992nm. Furthermore, to study the degree of water absorption of the sample, 5µl of distilled water was dripped onto the sample. Next, the picture was taken using Contact Angle (CA) measurements and analyzed using ImageJ software. The results of the study have also shown that the CA sample yield is between 49.07º to 80.83º. After data analysis and experiments, it was found that the sample containing parameters for 15kv voltage, 15cm distance, 10wt% concentration, and 1.5mL/h feed rate was the optimal sample with 500nm data for SEM and 67.23º for CA. Furthermore, the results of studies for DOE software have shown that AFD results are influenced by concentration, distance, voltage, and feed rate. In addition, the results of the study also show that CA results are influenced by distance, concentration, voltage, and feed rate. Both reactions of these output variables i.e. AFD and CA show that the feed rate is less influential of the sample compared to the others. ANOVA for the factorial model showed that it was significant for AFD is 0.0077 and CA is 0.0204. This is because it shows the interference during the experiment is less than 0.0500. Moreover, the Regression Analysis (R^2) also showed a good value when AFD reached 0.999 and CA reached 0.0204 as it approached 1.0000. A value of the coefficient of determination R^2 approaching 1.0000 is justified because it proves that most of the variability in the response variables is adequately explained by the variability of all factors. Having made experiments and analysis proved that the use of PVA is not suitable for air filtration applications because it is categorized in the hydrophilic category which has an angle between 0º to 90º.

ABSTRAK

Pada masa kini, elektrospin adalah teknik yang mudah dan cepat untuk menghasilkan serat dengan diameter skala nano dari pelbagai jenis polimer. Nanofiber ini menunjukkan beberapa ciri penting seperti luas permukaan dengan nisbah isipadu, keliangan tinggi, dan sifat mekanik yang unggul. Aplikasi penapisan udara Nanofiber untuk industri menjadi perhatian buat masa ini. Penggunaan perisian Design of Experiment (DOE) telah digunakan sebagai aplikasi untuk menghasilkan 19 sampel. Dalam kajian ini, telah menggunakan 19 sampel yang mengandungi kepelbagaian kepekatan, jarak, voltan dan kadar suapan. Ia turut dikenali sebagai faktor pembolehubah input. Penggunaan kepekatan antara 8wt% hingga 12wt%, Jarak antara 10cm hingga 20cm, voltan antara 10kV hingga 20kV dan kadar suapan adalah antara1mL/h hingga 2mL/h. Polyvinyl Alcohol (PVA) telah digunakan bersama air paip sebagai pelarut. Sampel electrospinning telah digunakan untuk mengkaji Purata Diameter Gentian (AFD) dengan menggunakan Scanning Electron Microscope (SEM). Hasil sampel SEM menunjukkan AFD adalah daripada antara 325nm hingga 992nm. Tambahan pula, untuk mengkaji tahap penyerapan air terhadap sampel, 5µl air suling telah dititiskan ke atas sampel. Seterusnya, penangkapan gambar telah diambil dengan menggunakan pengukuran Sudut Sentuhan (CA) dan dianalisis menggunakan perisian ImageJ. Hasil kajian juga telah menunjukkan bahawa hasil sampel CA adalah antara 49.07º hingga 80.83º. Setelah dibuat analisis data dan eksperimen didapati bahawa sampel yang mengandungi parameter bagi voltan 15kv, jarak 15cm, kepekatan 10wt% dan kadar suapan 1.5mL/h adalah sampel yang optimum yang mempunyai data 500nm bagi SEM dan 67.23º bagi CA. Tambahan pula, hasil kajian bagi perisian DOE pula telah menunjukkan bahawa keputusan AFD dipengaruhi oleh kepekatan, jarak, voltan dan kadar suapan. Di samping itu, hasil kajian juga menunjukkan bahawa keputusan CA dipengaruhi oleh jarak, kepekatan, voltan dan kadar suapan. Bagi kedua-dua tindakbalas pembolehubah output ini iaitu AFD dan CA menunjukkan bahawa kadar suapan kurang mempengaruhi sampel berbanding dengan yang lainnya. ANOVA bagi model faktorial menunjukkan bahawa ia adalah signifikan bagi AFD 0.0077 dan CA 0.0204. Hal ini kerana ia menunjukkan gangguan semasa eksperimen adalah kurang daripada 0.0500. Selain itu, Regression Analysis (R^2) juga menunjukkan nilai yang baik apabila AFD mencapai 0.999 dan CA mencapai 0.0204 kerana ia menghampiri 1.0000. Nilai pekali penentuan R^2 menghampiri 1.0000 adalah wajar kerana ia membuktikan bahawa kebanyakan kebolehubahan di dalam pembolehubah bergerak balas adalah dijelaskan dengan secukupnya oleh kebolehubahan semua faktor. Setelah dibuat eksperimen dan analisis membuktikan bahawa penggunaan PVA adalah tidak sesuai untuk aplikasi penapisan udara kerana ia dikategorikan dalam kategori hidrofilik yang mempunyai sudut antara 0º hingga 90º.

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LIST OF SYMBOLS AND ABBREVIATIONS

DOE	-	Design of Experiment
AFD	-	Average Fiber Diameter
CA	-	Contact Angle
SEM	-	Scanning Electron Microscope
Pt	-	Platinum
Ir	-	Iridium
Ag	-	Silver
Cr	-	Chromium
Au	-	Gold
Au/Pd	-	Gold/Palladium
HEPA	-	High-Efficiency Particulate Air
PLA	-	Poly (Lactic Acid)
CS	-	Chitosan
PVDF	-	Polyvinylidene Fluoride
POE	-	Polyethylene Oxide
ASHRAE	-	The American Society of Heating, Refrigerating and Air Conditioning Engineers
PAN	-	Polyacrylonitrile
PMMA	-	Poly(methyl methacrylate)
ANOVA	-	Analysis of variance
PAA	-	Polyacrylic acid
PE	-	Polyethylene
PS	-	Polystyrene
PVC	-	Polyvinyl chloride
PTFE	-	Polytetrafluoroethylene
PDMS	-	Polydimethylsiloxane
R^2	-	Regression Analysis

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CHAPTER 1

INTRODUCTION

1.1 Background

Nanofiber membranes are common in air filtration applications in the age of globalization. This is due to the nanofiber's distinct properties and advantages. Mechanical strength, high porosity, and other properties are among the characteristics that distinguish each polymer. In comparison to microfiber, the nanofiber is also more flexible. Nanofiber technology is a technique for creating, processing, manufacturing, and utilizing nanoscale fibers (Frank K. Ko and Yuqin Wan, 2014). Nanofiber is a very fine fiber with a diameter ranging from 1 nanometer to 1000 nanometers. A variety of techniques and methods have been developed over hundreds of years to create this small strand material. Drawing, self-assembly, template synthesis, phase separation, and electrospinning are some of the techniques that can be used to make or process nanofibers (Munir & Ali, 2013). Several different polymers, either natural polymers or synthetic polymers, are used to make these nanofibers. Each polymer has its own set of physical characteristics and applications. It is dependent on the polymer that will be used in the experiment.

Design of Experiment (DOE) or also known as sample optimization is one of the tools used to study the sample systematically and in detail with various types of internal problems that may arise. It is often used in the fields of development, production, and research (Ahmed Badr Eldin, 2011). So, it proves that if an experiment is done randomly then the results that will be obtained are also random. In addition, some studies that use this software state that the results of the experiment are dependent on the conditions during the

experiment or the variables of an experiment (Sundberg, 1994). The software consists of two data factors namely replication run and repetitive run. It has multiple responses taken at the same factor level. In this study, both data factors were involved. There are 16 samples of repeated runs and 3 more samples are replication runs and the responses sought in this software are Average Fiber Diameter (AFD) and Contact Angle (CA). In addition, some studies also involve the same factor of 10 repeated run samples and 5 more replication run samples, it also studied the responses of AFD and Porosity of Medium (Dehghan et al., 2016).

This study was conducted to study the morphology effect of Average Fiber Diameter (AFD) by using the electrospinning process. Solution variables, processing variables, and ambient variables are all factors that can influence the electrospinning process. The term "solution variable" refers to the properties of the polymer or solvent system, such as viscosity, dielectric constant, conductivity, surface tension, the molecular weight of the polymer, dipole moment, and polymer concentration. The polymer concentration has the greatest impact because the fiber diameter increases as the polymer concentration increases. This is due to an increase in viscosity corresponding to an increase in the concentration of the polymer in solution (Tribuzi Morais, 2011).

A processing variable is a collection of parameters that can be used in a process, such as voltage, collector end distance, feed rate, and collector type. The applied voltage has a significant effect on the fiber geometry. Voltage affects the fiber diameter, but the extent of that effect depends on the concentration of the polymer solution as well as the collector tip of the distance (Yördem et al., 2008). Because are poorly studied, ambient variables are more difficult to control and predict. Temperature, humidity, atmospheric type, and pressure can all affect fiber conformation, even if their use is minimal. The fiber diameter shrinks as the

temperature increases. Low humidity, causing the solvent to evaporate faster and dry faster. In this study, parameters such as distance, power supply voltage, concentration, and feed rate were studied using DOE software to obtain AFD and CA responses.

In addition, Polyvinyl Alcohol (PVA) was chosen as the solution because it is a water-soluble synthetic polymer (Satoh, 2014). PVA is a semi-crystalline, heat-stable hydrophilic polymer, with excellent chemical properties. It is also odorless, non-toxic, and resistant to oils and greases, and it is often used in filtration air. Its unique physical properties, such as strength, flexibility, and ability to function in a high-oxygen environment, have made it the best choice for use. Due to these properties, PVA is increasingly used in medical, cosmetic, food, pharmaceutical, and packaging applications (Qin & Wang, 2006). There are two types of PVA available in the market, namely powder and pellets. In this study, PVA-type pellets were used. During the process of making this PVA solution, tap water has been mixed with PVA, and it can be used only for 2 days after the process of making PVA is completed.

Before the experiments were carried out, concentration calculations were performed first. It is very important to ensure that the amount of polymer and water is sufficient for a given concentration. Incorrect calculations will affect the effect on the sample and the results obtained will not be accurate. Analytical Balance is a machine that has been used to weigh the amount of polymer and tap water needed for this experiment. Next, the Overhead Mixer machine was also used to dissolve the compounds between the polymer and tap water. The process of this compound takes 3 hours for one concentration.

Due to simple instrumentation, continuous processing, lower cost compared to other available methods, and ability to make fiber diameters from a few nm to a few microns, electrospinning is the most widely used method in the industry (Kumbar et al., 2008). Depending on the jet formation produced, electrospinning methods can be classified into several different types of categories. There are two ways nanofibers can be made through 4 electrospinning techniques namely needleless and needle-based. In this study, the needlebased electrospinning process was used because it is a continuous and cost-effective process, its diameter can be controlled from a few nm to a few microns, has a programmable needle pump, and the ability to monitor the pump as well as the injection rate.

In this study, the electrospinning process was carried out without electrode contact to the spinneret. Instead, use an electric field method that will collect enough charge to extend the droplets of solution to the plate collector (Chung, 2008). In addition, with the voltage adjustment will affect the process of electro rotation applied to the solution. A large volume of solution will be removed from the tip of the needle if using a high voltage. This is so because, referring to previous studies, by increasing the voltage is able to speed up the process on electrospinning jets (Liu et al., 2019). The use of high voltage will also cause the fibers to become thinner (Buchko et al., 1999). Figure 1.1 shows the effect in terms of voltage increase for the electrospinning process and fiber diameter.

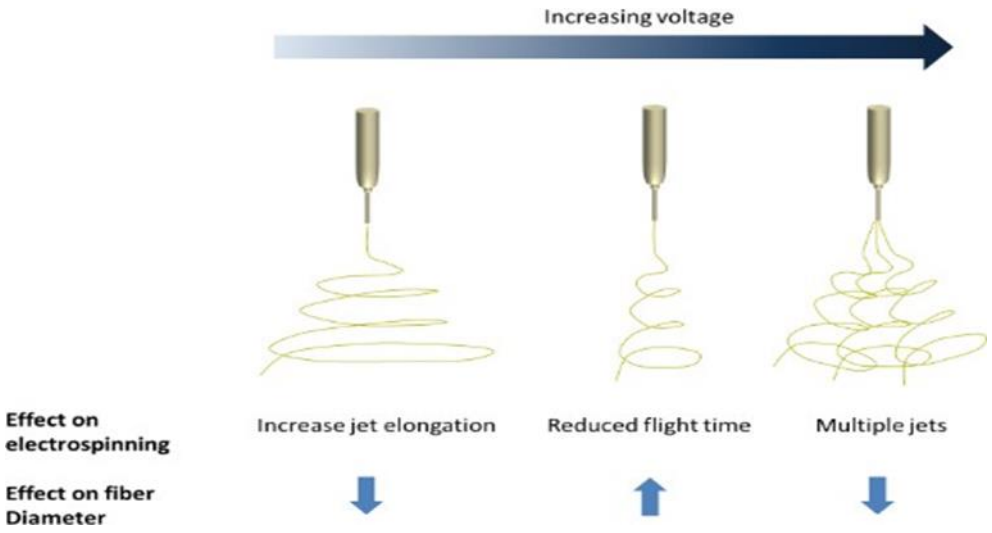

Figure 1.1 The effect in terms of voltage increase for the electrospinning process and fiber diameter (Liu et al., 2019)

Other than that, polymer concentration also plays an important role in the electrospinning process. According to previous studies, the higher concentration of polymer used, the larger of diameter electrospinning fibers (Hossain et al., 2016). The control of the polymer concentration is also the most important factor in controlling the morphological structure of any defects. For the use of too low a concentration, bead formation will occur while using too high a concentration the beads will become elongated (Sreekumar, 2020). Furthermore, the higher of polymer concentration used the higher of diameter nanofiber fibers. Figure 1.2 shows four types of electrospinning defects namely beads, fiber bonding, fiber bundles, or multiple defects.

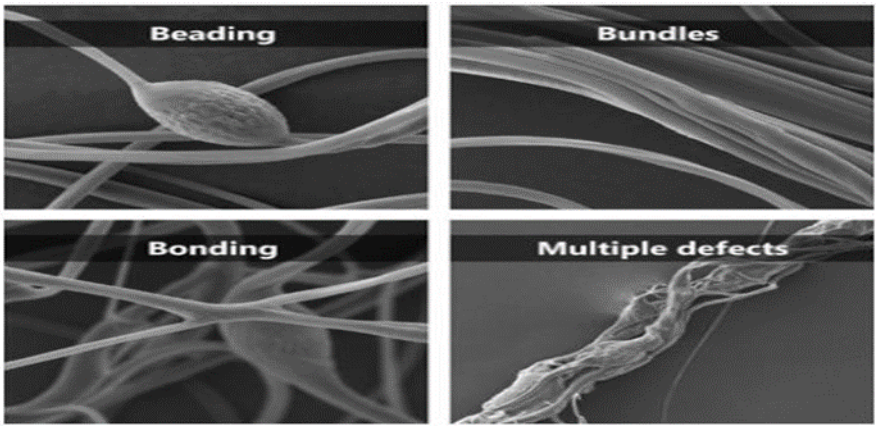

Figure 1.2 Four types of defects specific to electrospinning (Chaparro, 2021)

To produce a uniform and smooth morphological structure, the use of distance should be in the correct range. When too close a distance is used then wet and thick fibers will result because the time to evaporate is too short (Nurfaizey & Munajat, 2020). Meanwhile, the use of too far a distance will also result in the electrospinning process becoming unstable because too long a distance will weaken the strength of the electric field to carry the solvent to the collector. In addition, the use of a fixed feed rate will also cause the Taylor cone to be too small and give an instability effect to the electrospinning process (Zong et al., 2002).

One of the standard machines used to view the AFD results of samples is the Scanning Electron Microscope (SEM). However, before the above process is done, the sample must be coated first to facilitate the process of electron flow. There are several materials used to coat the samples, namely platinum (Pt), iridium (Ir), silver (Ag), chromium (Cr), gold (Au), or gold/palladium (Au/Pd) (Höflinger, 2021). In this study, the platinum coating was used. After the coating process is done, then the SEM process is done. Next, SEM images will be analyzed using ImageJ.

Furthermore, the standard machine used to view CA samples is known as the Contact Angle measurement machine. The process is very simple because the machine has been connected to a computer and sample pictures are taken through. After the sample image was obtained, it was also analyzed using ImageJ. Next, through the analysis, the sample can be known whether it is in the classification of hydrophilic, hydrophobic, or superhydrophobic. Figure 1.3 below shows the category of hydrophilic, hydrophobic, or superhydrophobic.

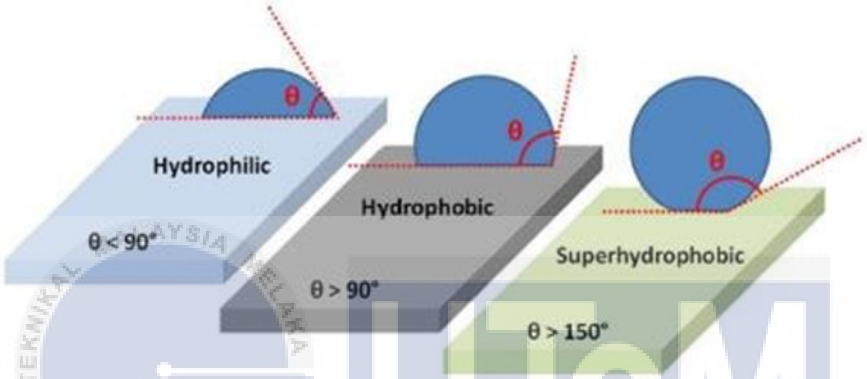

Figure 1.3 The category of hydrophilic, hydrophobic, or superhydrophobic (P. Zhang & Lv, 2015)

1.2 Problem Statement

The key to air purification is through highly efficient air filtration with low-pressure drops (Z. Shao et al., 2020). To produce good air filtration, the effect of parameters must be studied to ensure it is clean, safe to use, and environmentally friendly. This is compounded by the fact that the world now has an infectious disease (coronavirus or covid-19) that can lead to death. The number of deaths caused by this virus is increasing day by day and there seems to be no solution. Therefore, the creation of effective air filtration can prevent as well as reduce this disease from continuing to spread.

Therefore, this study was made to replace the existing polymer application filters in the market with better ones and at the same time study AFD on nanofiber membranes. It covers the study of suitable and effective polymer concentrations for air filtration. However, parameters such as collector rod speed, temperature, and relative humidity were not thoroughly studied in this study. The use of the electrospinning method has been carried out because it is capable of producing nanofibers with unmatched small sizes and high surface zone (Khude, 2017).

This study was conducted to find out the use of appropriate parameters that will not affect the morphological structure. However, some research has been done to identify the effects that will affect the morphology which includes solution parameters, processing parameters, and also ambient parameters. Where when the bead generation viscosity is low, then there will be a high increase in fiber diameter and there will also be bead loss. The use of high conductivity will affect a decrease in fiber diameter. Meanwhile, the use of high molecular weight of the polymer will have the effect of reducing the number of beads and droplets. Increasing the polymer concentration will increase the fiber diameter. Furthermore, surface tension will not affect the morphology of the fiber, wherewith high surface tension will only affect instability on the jet (Tribuzi Morais, 2011).

The processing parameter that will affect the morphology is the application of voltage, when setting a high voltage then a decrease in fiber diameter will occur. Changes in the applied voltage will change the shape of the initial fall and result in changes in the structure as well as the morphology of the fibers (Baumgarten, 1971). In addition, a minimum distance between the tip and the collector is required to create fiber uniformity. Beads will be produced when the distance is too close or too far away (J. S. Lee et al., 2004). The feed rate or flow rate must be at the correct rate because a decrease in fiber diameter