SYNTHESIS, CHARACTERIZATION AND OPTIMIZATION OF POLYACRYLONITRILE ELECTROSPUN NANOFIBER MEMBRANES

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A thesis submitted in fulfilment of the
requirements for the award of the degree of
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To my parents, my wife; Melia Marleny and beloved daughters,
Siti Manisa Putri Mataram, Challysta Puan Mataram and my son,
Muhammad Azka Mataram for their supports and understandings

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ABSTRACT

The control of electrospinning process parameters, such as high electric potential, flow rate, screen distance and concentration becomes increasingly difficult. Electrospinning is capable of producing fibers in nanosize diameter range due to the increase of mechanical forces to drive the fiber formation process. Polyacrylonitrile (PAN) nanofiber membrane produced by electrospinnning was structurally developed to improve the performance of wastewater treatment. The dispersion of silica nanoparticle concentration in dope solution of 1 wt.% has changed the structural and mechanical properties of fibers. The fiber structure was examined in terms of pore size, contact angle, tensile strength, Young's modulus, fourier transform infrared spectrometer (FTIR), and scanning electronic microscopy (SEM). The results indicated that the increase of polymer concentration and flow rate, the average fiber diameter increases. On the other hand, the increase of screen distance and electric potential decreased average fibers diameter. Young's modulus and tensile strength increased by the addition of silica content at 1 wt.% and decreased with the increase of the silica content of 2 wt.%. The further addition of silica particles concentration produced more brittle and fragile PAN/silica composite fibers. The effect of silver functionalized membranes to pathogen removal was also studied and the tests were performed in a flow through system. Response Surface Methodology (RSM) was also performed to investigate the influence of the variables on the quality and quantity of permeate to attain the optimized conditions for preparing electrospun PAN fibers. Results from RSM were used to assess the interaction factors, namely, screen distance, polymer concentration and voltage. The quadratic models based on the responses resulted in potential of pore size, contact angle, young modulus and clean water permeation (CWP) to suitable chemical oxygen demand (COD), total suspended solids (TSS), ammonia nitrogen (NH_3-N) and e. coli removal efficiencies. The results showed high removal of TSS, COD, NH_3-N and e. coli at 96.18%, 91.82%, 68.89%, and no detectable, respectively. Therefore, it can be concluded that electrospun nanofibers membrane can be promising alternative materials in water filtration, especially as membrane for antibacterial and stand-alone microfiltration unit.

ABSTRAK

Kawalan parameter proses pemintalan elektro, seperti potensi elektrik yang tinggi, kadar aliran, jarak skrin dan kepekatan menjadi semakin sukar Pemintalan elektro mampu untuk menghasilkan gentian dalam lingkungan bersaiz nano diameter disebabkan oleh peningkatan daya mekanikal untuk memacu proses pembentukan gentian. Membran nanogentian poliakrilonitril (PAN) yang dihasilkan daripada pemintalan elekro dibangunkan secara struktur bagi meningkatkan prestasi rawatan air sisa. Penyerakan kepekatan nano zarah silika di dalam larutan dop 1 % berat mengubah struktur dan sifat-sifat mekanikal gentian. Struktur gentian telah diperiksa dari segi saiz liang, sudut sentuh, kekuatan tegangan, modulus Young, spektrometer inframerah transformasi Fourier (FTIR), dan mikroskop imbasan elektronik (SEM). Keputusan menunjukkan bahawa peningkatan kepekatan polimer dan kadar alir larutan dop menaikkan purata diameter gentian. Sebaliknya, peningkatan jarak skrin dan potensi elektrik menurunkan purata diameter gentian. Modulus Young dan kekuatan tegangan meningkat mengikut peningkatkan kandungan silika pada 1 % berat tetapi menurun dengan peningkatan kandungan silika 2 % berat. Penambahan kepekatan zarah silika menghasilkan gentian PAN / silika komposit yang lebih rapuh dan mudah pecah. Nilai tambahan membran berfungsian perak untuk penyingkiran patogen juga telah dikaji dan ujian telah dilakukan dalam sistem beraliran terus. Kaedah respons permukaan (RSM) juga telah dilakukan untuk mengkaji pengaruh pembolehubah terhadap kualiti dan kuantiti meresap untuk mencapai syarat yang dioptimumkan bagi penyediaan PAN gentian pemintalan elektro. Hasil dari RSM digunakan untuk menilai faktor interaksi iaitu, jarak skrin, kepekatan polimer, voltan. Model kuadratik yang dihasil berdasarkan respon telah diguna dan menghasilkan saiz liang yang berpotensi, sudut sesentuh, modulus Young dan penyerapan air bersih (CWP) terhadap permintaan oksigen berkimia (COD) yang sesuai, jumlah pepejal terampai (TSS), nitrogen ammonia (NH_3-N) dan kecekapan penyahan e. Coli, yang berkesan. Keputusan menunjukkan penyahan untuk memberangsangkan TSS, COD, NH_3-N dan e. coli pada 96.18%, 91.82%, 68.89%, dan tidak dikesan. Oleh itu, boleh disimpulkan bahawa gentian nano membran pemintalan elektro boleh menjadi bahan alternatif yang berpotensi dalam penapisan air, terutamanya sebagai membran antibakteria dan penapisan mikro unit bersendirian.

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LIST OF ABBREVIATIONS

ANOVA	-	Analysis of Variance
COD	-	Chemical Oxygen Demand
DMF	-	Dimethylformamide
FTIR	-	Fourier Transform Infra-Red Spectroscopy
H_2O	-	Water
MF	-	Microfiltration
NH_3-N	-	Ammonia Nitrogen
NF	-	Nanofiltration
PAN	-	Polyacrylonitrile
RSM	-	Response Surface Methodology
S.D.	-	Standard Deviation
Ti_2O	-	Titanium Dioxide
TMP	-	Transmembrane Pressure
TSS	-	Total Suspended Solid
UF	-	Ultrafiltration

LIST OF SYMBOLS

a	–	Stokes–Einstein radius (m)
Ak/Δx	–	Ratio of membrane porosity to membrane thickness (m^{-1})
A	–	Membrane surface area (m^2)
Cm	–	Concentration of solute in the fluid at the feed ($mol.m^{-3}$)
Cp	–	Concentration of solute in the permeate solution ($mol.m^{-3}$)
dp	–	Pore diameter (nm)
D	–	Diameter of a tube (m)
Js	–	Averaged solute flux over membrane surface ($mol.m^{-2}.s^{-1}$)
Jpwp	–	Pure water permeability ($m^3.m^{-2}.h^{-1}$ or $m.s^{-1}$)
Jv	–	Permeate flux ($m^3.m^{-2}.h^{-1}$ or $m.s^{-1}$)
k	–	Boltzmann's constant (1.38 $kg.m^2.s^{-2}.K^{-1}$)
M	–	Molecular weight ($g.mol^{-1}$)
Ms	–	Molecular weight of the solvent ($g.mol^{-1}$)
n	–	Stokes–Einstein coefficient (dimensionless)
ρ	–	Density of liquid ($kg.m^{-3}$)
P'	–	Local solute permeability (m2. S^{-1})
P	–	Solute permeability ($m.s^{-1}$)
Pf	–	Feed pressure (bar)
Pr	–	Retentate pressure (bar)
Pp	–	Permeate pressure (bar)
rp	–	Pore radius (nm)
rs	–	Stokes radius (nm)
Re	–	Reynolds number (dimensionless)
Qp	–	Permeate flow ($kg.m^{-2}$)
T	–	Temperature (^{0}C)

Greek letters

ε	–	Porosity of the membrane (%)
η	–	Solution viscosity ($N.s.m^{-2}$)
ℓ	–	Membrane thickness (m)
μ_s	–	Geometric mean diameter of solute molecule at R = 50% (nm)
μ	–	Solvent viscosity (water viscosity at 25°C, 0.894×10^{-3} $kg.m^{-1}.s^{-1}$)

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CHAPTER 1

INTRODUCTION

1.1 Research background

Electrospinning is a straight forward method of fiber preparation that relies on electrostatic forces to produce fibers with diameters typically in the nanometer size range from either polymer solutions or melts (Ji and Zhang, 2008). They are important industrially and have a wide range of applications, from sports equipment to the aerospace industry (Gu *et al*., 2008). Nanofibers, like other one-dimensional (1D) nanostructures, such as nanowires, nanotubesand molecular wires, are receiving increasing attention because of their large length to diameter ratio. Their potential applications are in nanocomposites, high temperature catalysis, templates for nanotubes, high temperature filters, rechargeable batteries, supercapacitors, and bottom-up assembly applications in nanoelectronics and other applications (Moon and Farris, 2009).

Unlike conventional fiber spinning techniques (wet spinning, dry spinning, melt spinning, gel spinning), which are capable of producing polymer fibers with diameters down to the micrometer range, electrostatic spinning, or 'electrospinning' is a process capable of producing polymer fibers in the nanometer diameter range.

Electrospinning is a process that produces continuous ultrafine polymer fibers through the action of an external electric field imposed on a polymer solution or melt. Electrospinning is a novel and efficient fabrication process that can be utilized to assemble fibrous polymer mats composed of fiber diameters ranging from several microns to lower than 100 nm. Recently, polymer nanofibers have been attractive materials for a wide range of applications because of their large surface area to volume ratio and the unique nanometer scale architecture built by them, as shown in Figure 1.1. One of the possible applications of the nanofibers is water filtration. For this application a nanofibers flat sheet membrane can be produced. More specifically, this can be used in microfiltration. Nanofibers, due to their higher porosities and interconnected pore structures, offer a higher permeability to water filtration over conventional materials being used (Thavasi *et al.*, 2008).

Microfiltration membranes have pore sizes between 0.1 and 10 μm and trans membrane pressure (TMP) between 0.01 and 0.2 bar is used. Using these membranes it is possible to retain suspended solids and, depending on the pore size, even microorganisms such as bacteria, yeast and fungi. Earlier studies have indicated that in case of a 0.45 μm pore size a log 2–log 4 bacteria reduction could be achieved (Gomez *et al.*, 2006; Sadr *et al.*, 1999). As the membrane has a nominal pore size in the range of 0.2 to 0.4 μm, it seems very interesting to evaluate its bacteria removal capacity. In addition, the added value of silver functionalized membranes to pathogen removal was studied. These tests were performed in a flow through system as few studies have been carried out so far to test the filtration performance and disinfection efficiency of the silver impregnated nanofibers membranes.

Microbiological contamination of water sources has long been concerned to the public. According to some researchers, there were various bacterial species available (ranging from 10^2 to 10^4 mL^{-1}) in raw water as well as sewage effluents (Bonnélye *et al*., 2008; Goldman *et al*., 2009). They tend to adhere to surfaces and grow mainly at the expense of nutrients accumulated from the water phase. Microbiological contamination in any sources should be avoided at any cost since in the production of potable water, only a limited number of bacteria (depending on the type of bacteria) are acceptable. The separation process for the removal of contaminants depends not only on the nature of the microorganisms but also on the desired levels of purity.

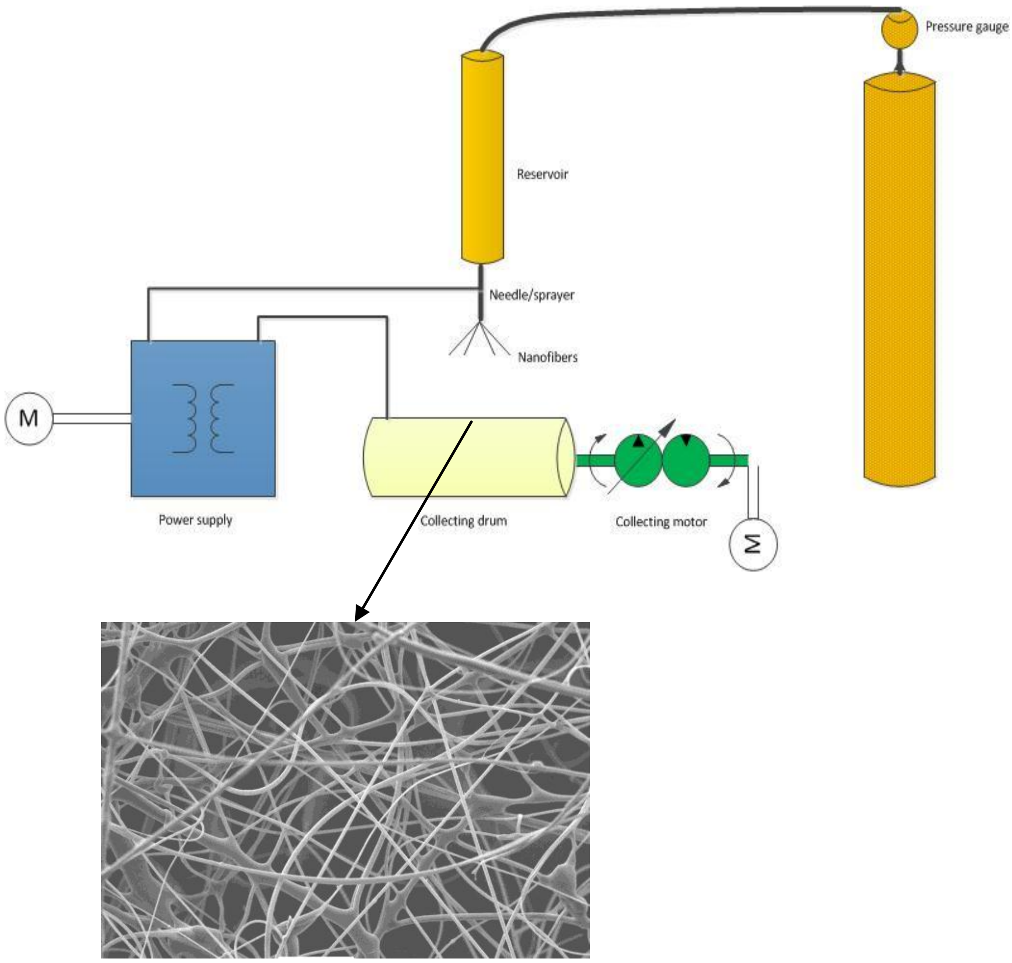

Figure 1.1: Electrospinning principle and resulting nanofibers mat (SEM picture)

Electrospinning uses electrostatic forces as the driving force to spin fibers. In the solution electrospinning process, a polymer solution held by its surface tension at the end of a capillary tube is subjected to an electric field. As the intensity of the electric field increases, the hemispherical surface of the solution at the tip of the capillary tube extends to form a cone like structure, which is also known as the Taylor cone (Shin *et al.*, 2005). When a critical point is reached with increasing voltage, a charged jet of the solution is ejected from the tip of the Taylor cone. As this charged jet moves in the air, the solvent evaporates, leaving behind a charged polymer fiber, which lays itself randomly on a collecting plate. Thus, continuous fibers are laid to form a fibrous web.

In this work, nanofibers were produced by solution electrospinning from solution using polyacrylonitrile as the polymer. Experiments were performed using this polymer and the electrospun web produced was characterized. A suitable experiment and equipment design was made in order to study the process parameters. During the process, the system made can adjust voltage and screen distance simultaneous. The process parameters investigated included the concentration of the polymer solution; the voltage and the collecting distance between the two electrodes. These parameters were optimized by using Response Surface Methodology. The structural properties of electrospun web were characterized measuring fiber diameter and its distribution, fiber orientation and pore size and its distribution. The possible use of electrospun nanofiber membrane in water filtration is in two different areas: first, membranes for pathogen removal, to be applied as a membrane for antibacterial activity; and second, membranes for the reduction of suspended solids, chemical oxygen demand, nitrogen ammonia and also pathogen removal; to be applied as stand-alone microfiltration unit.

1.2 Problem statements

Unlike conventional fiber spinning techniques, which are capable of producing fibers with diameters down to the micron size range, electrostatic spinning, or electrospinning is capable of producing fibers in the nanometer diameter size range, or "nanofibers". In electrospinning, electrostatic forces are used in addition to mechanical forces to drive the fiber forming process. Hence, the control of the process at high electric potential, flow rate, screen distance and concentration becomes increasingly difficult.

The regulations governing the disposal of water or wastewater are tightening and interest in removal of bacteria and also hazardous organics contaminant is growing. Contents of chemical oxygen demand (COD), total suspended solid (TSS) and ammonia nitrogen (NH_3-N) must follow 'National primary discharged standard (P. U. (A) 434, Standart B, December 10, 2009, Malaysia) as shown in Table 1.1 and Appendix H.

Table 1. 1: Composition of wastewater

Constituent, unit	National primary discharged standard (P.U. (A) 434, Standard B, December 10, 2009, Malaysia)
COD, mg/l	400
Suspended solid, mg/l	100
NH_3-N, mg/l	20

1.3 Objectives of the study

Based on the problem statements, the current study has been performed with the following objectives:

(i) To develop a new method for production of polyacrylonitrile-based activated carbon nanofibers by using electrospinning process.

(ii) To study the effects of electrospinning parameters on the structural and properties of fibers.

(iii) To study the influence of electrospinning process parameters on the structure and properties of electrospun nanofiber membranes for water filtration using response surface methodology (RSM).

1.4 Research scopes

In order to achieve the abovementioned objectives, the following scopes of study have been drawn:

(i) Formulating several spinning dope solutions with different polymer concentrations of 15, 16, 17.5, 19 and 20 wt.% at temperature of 50^{o}C for 24 hours.

(ii) Synthesizing the PAN fibers with addition of silica nanoparticles (0, 0.5, 1 and 2 wt.% of PAN) into wt.% PAN solution using dry-jet spinning system.

(iii) Characterizing the PAN fibers by Scanning Electron Microscopy (SEM), Attenuated Total Reflection Fourier Transform Infrared Spectroscopy (FTIR–ATR), and tensile strength testing.

(iv) Constructing an electrospinning system for the production of PAN nanofibers.

(v) Synthesizing the PAN nanofiber membranes with addition of 1 wt. % $AgNO_3$(silver nitrate).

(vi) Characterizing the PAN fibers with $AgNO_3$ by Scanning Electron Microscopy (SEM), contact angle, average pore size, and water permeation measurement.

(vii) Analyzing the effects of electrospinning process parameters on the quality of nanofibers membrane by using RSM to describe the individual and interactive effects of these variables.

(viii) Investigating the performance of electrospun nanofiber membranes in water filtration in terms of the antibacterial activity by using disc diffusion method and bacteria removal via the filtration of bacterial suspension, as well as the separation performance for the removal suspended solids, chemical oxygen demand, and nitrogen ammonia.

1.5 Significance of research

The significance of this research was the development of novel electrospun nanofibers for wastewater treatment which was particularly application for pathogen removal. Most of the published research works related to electrospun fibers were mainly addressing the suspended solid removal in a water and waste water. Focus of this study was bacteria removal, which involved disinfection steps, using the nanofibers has been the main focus. The antibacterial membrane extends the multi-steps options for water treatment to a stand-alone removal and disinfection of bacteria. The results obtained in the study also providing the information on the bacteria removal and bacteria killing mechanisms which lead to the most effective options in treating polluted water. Furthermore, the information on silver entrapment obtained in this study would be beneficial to the other related fields such as in medicinal and electrical field where silver is optimized in wound dressings and conducting material, respectively. In addition, the process conditioning of the water treatment process in terms of bacteria removal was conducted using pressure as low as 0.1 MPa.

1.6 Organization of the thesis

The thesis is divided into eight chapters. The first chapter presents the research background as well as the problem statement. The research objectives, scopes and significance are also highlighted in this chapter. Chapter two provides the literature review on spinning process and wastewater treatment, which includes the theories of the whole process and the options available for bacteria removal. Chapter three is dedicated to the detailed description of the research methodology. The material selection for dope preparation, membrane fabrication and performance testing conducted in this work are explained in this chapter.. Chapter 4 is about electrospinning process summary and some results from pan silica composite fiber experiments. Some results from conventional spinning process were also included. Subsequently, Chapter 5 describes the use of RSM to optimize process parameters of spun nanofibers membrane preparation. Chapter 6 discusses polyacrylonitrile nanofibers assembled by electrospinning while performance of electrospun nanofibers for wastewater treatment is investigated and discussed in chapter 7. Finally in Chapter 8 are the conclusions of the research drawn and the potential future works have been proposed.

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