# POLYURETHANE NANOFIBERS COATINGS ON THE X-RAY FILM BY USING ELECTROSPINNING TECHNIQUES

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# POLYURETHANE NANOFIBERS COATINGS ON THE X-RAY FILM BY USING ELECTROSPINNING TECHNIQUE

NURUL NADIRAH BINTI SUTERIS

Thesis submitted in fulfillment of the requirements
for the award of the degree of
Bachelor of Applied Science (Honor) Material Technology

Faculty of Industrial Sciences& Technology
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JANUARY 2017

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## DEDICATION

I dedicated this work to my respected supervisor, Prof Jose Rajan, my family and friends. A special feeling of gratitude towards both of my parents, Suteris Bin Masron and Nirwati Binti Hussin who always sbeen supporting me and thanks to all my siblings, Nurul Najwa Bint Suteris (sister), Muhamad Syazwan Bin Suteris (brother), Nurul Nadana Binti Suteris (sister), Muhammad Syarafuddin Bin Suteris (brother), Muhammad Syahmi Bin Suteris (brother) and Khairunnisa Madihah Binti Suteris (sister), that keep on giving words of encouragement to me.

I also dedicated this work to all my friends that has helping me throughout the process and very appreciate them a lot especially with friends under the same supervisor, Nurul Athirah Binti Ismail.

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I would like to thank all the lecturers and friends that always guided and helped me during this project.

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## ABSTRACT

This thesis is about synthesizing polyurethane (PU) solution. The purpose of conducting this research are to prepare a polymer nanofibers using electrospinning technique. Electrospinning is the technique of nanofabrication of polymer nanofibers. The synthesization of the solution is dissolved in N,N-dimethylformamide (DMF) with 12 wt.% and the solutions is mixed using magnetic stirrer. After that, the polyurethane nanofibers was obtained by using electrospinning machine with a voltage of 30kV, a flow range of 1.00 ml/h and working distance of 8 cm. The results demonstrated that the SEM images is more clearer as the magnification increases. Attenuated total reflection – fourier transform infrares (ATR-FTIR) shows that the PU electrospun has maintained it's molecular structure during the electrospinning process. The spinnable parameters are very important in this research to ensure that the polyurethane can be coated properly on the X-ray film.

# ABSTRAK

Thesis ini melaporkan kajian mengenai mensentisis cecair polimer Poliuretana Tujuan kajian ini diadakan adalah untuk menyediakan serat nano untuk polimer dengan menggunakan teknik putaran elektro. Teknik putaran elektro merupakan suatu teknik penyediaan serat nano. Pelarut N,N-dimethylformamide (DMF) digunakan untuk proses sentisis dengan menggunakan peratus 12 wt% dan dialrutkan menggunakan pengacau magnet. Selepas itu, serat nano poliuretana boleh didapati melalui proses putaran lektro dengan menggunakan voltan sebanyak 30 kV, kadar aliran sebanyak 1.00 ml/sejam dan jarak kerja sejauh 8 cm. Keputusan kajian imbasan mikroskop elekron menunjukkan bahawa semakin besar kadar pembesaran, semakin jelas keputusan gambar yang didapati. Keputusan kajian pantulan fourier pengubah inframerah menunjukkan bahawa polimer poliuretana mempunyai struktur molekul yang seimbang. Parameter pemboleh ubah adalah sangat penting di dalam kajian ini kerana poliuretana haruslah disalut dengan sebaliknya diatas lapisan filem x-ray.

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## LIST OF SYMBOLS

| | | |
|---|---|---|
| ~ | - | approximately |
| % | - | percent |
| λ | - | wavelength |
| $\eta$ | - | coulombic efficiency |
| $^{o}$C | - | degree celcius |
| g | - | grams |
| µ | - | kinematic viscosity |
| $h$ | - | jet radius (m) |
| $Z$ | - | distance from the nozzle to the collector |
| $\rho$ | - | density (kg/m$^3$) |
| $Q$ | - | flow rate (m$^3$/s) |
| $E_{\infty}$ | - | external electric field (V/m) |
| $I$ | - | electric current (A) |
| $\gamma$ | - | surface tension of the fluid (N/m) |
| $\varepsilon$ | - | dielectric permittivity (F/m) |
| $x$ | - | dimensionless whipping instability |
| $R$ | - | electrical resistance of the jet (Ω) |
| $U$ | - | applied voltage (V) |
| $rs$ | - | resistivity of the solution (Ω m) |
| S | - | section of the jet (m$^2$) |

## LIST OF ABBREVIATIONS

| | | |
|---|---|---|
| SEM | - | Scanning Electron Microscope |
| ATR-FTIR | - | Attenuated Total Reflection-Fourier Transform Infrared |
| PU | - | Polyurethane |
| DMF | - | N,N-dimethylformamide |

# CHAPTER 1

# INTRODUCTION

## 1.1 INTRODUCTION

With the improvement of our nanotechnology, many works become more curious in the research of nanoscale materials. Electrospinning technique has shown much more interest recently due to its uniqueness and wide potential for many areas. The nanoscale fibers are generally affects by the strong field on polymer solution or melt. Over the few decades ago, a quite number of polymers have been conducted for various applications and the number of research is still increasing (Nandana et al., 2010).

Electrospinning is a process which provides forming of polymeric scaffolds for tissue engineering of the cardiovascular system. This process produced fibers with nm- to μm- scale in diameter. The combination of polyurethanes (PU) properties with an electrospinning process, gives a lot of possibilities of forming novel PU materials that gives a lot of advantages (J. Kucinska et al., 2015).

There are some properties in PUs such as protection for surfaces and versatility. A primary benefit of using PU coatings is the protection it provides the surface itself. When applied, the coating will create what is important on impermeable barrier between the elements and the cover for the object. For versatility, PU coatings can be applied to many material surfaces including metal, wood and plastic. PU has come a long way since the last few decades, it can provides many benefits and advantages. No wonder it has become a mainstream choice in surface coating applications.

In this research, PU is used as nanofibers coating on the x-ray films. A polyester urethane (PU) having an amino acid derived diol (N-Bocserinol) as chain extender was synthesized. After Boc cleavage, the polyurethanes were used to produce aligned fibers to mimic the aligned morphologies of soft tissue such as nerve (Chiara et al., 2015). Polyurethanes can be formed in many ways including elastomers and coatings. Polyurethane coatings have a good impact resistance to the surface of material. These coatings are important for used on surfaces of materials.

X-ray film is made up from a gelatin-covered polyester base. An emulsion are coated on the both sides of the film that contains tin silver halide crystals that are sensitive to visible light X-rays, gamma rays, heat, moisture and pressure.

The coating of the PU on the X-ray films is by using electrospinning process. Electrospinning become the most frequently used method for nanofiebrs production. These polymers are often used for the production of nanofibrous scaffolds because of their functional characteristics such as collagen, fibrinogen, starch and many mores which can improve the overall capability of the scaffold (Pavlo et al., 2004).

## 1.2 PROBLEM STATEMENT

X-ray film cannot be used if outdated as it may fog and might disturb its diagnostic usefulness.A thin layer of adhesive is used to achieve firm attachment between the emulsion and base. The emulsion is protected from scratches, pressure or contamination during use by a thin layer of gelatin called ‘supercoating’. The thickness of a radiographic film is about 0.25mm.

With these in concerns, we aim to propose developing nanofibrous PU films on X-ray films.

. **Figure 1.2** Outdated X-ray Film

## 1.3 OBJECTIVES OF RESEARCH

Main objectives in this proposed research:

Objective of the research is to coat Polyurethane (PU) nanofibers on the x-ray film by using electrospinning technique.

## 1.4 SCOPE OF THE STUDY

Besides all the uniqueness of electrospinning process, there are some boundaries such small size of the pores and un-even size of nanofiber wires. There are few attempts in these directions are being made to improve the design of the nanofibers produced (Xiaomin Shi et al., 2015). In general, the electrospinning process shows incredible promise for better enhancement of nanofiber scaffolds.

Electrospinning process is impossible with the high concentration or the viscosity, this is because of the solutions that will be difficult to melt in liquid jet. As the concentration of solution increases, the fiber diameter will also be increases due to the higher viscosity that resisted the jet extension.

Different polymers will eventually have different viscosity range. Solution viscosity parameter is influencing the fiber diameter. As higher the viscosity, fiber diameter will be larger. As the viscosity is increases, the diameter of the beads will also increase (K. H. Akbar et al., 2012)

Surface tension depends a lot on solvent composition, but the dependence will not affect the solution concentration. Different solvents will results in different surface tensions. But a solvent that has a lower surface tension will not be suitable for electrospinning process. The formation of nanofibers, beads and droplet are affected by the surface tension of the polymer solutions (K. H. Akbar et al., 2012)

# CHAPTER 2

# LITERATURE REVIEW

## 2.1 HISTORY OF ELECTROSPINNING

Electrospinning is a technique to obtain unlimited possibilities in nanofibers modeling if the given electrospinning conditions are well established. Diversification of chain extenders causes the biocompatibility and stability of the electrospun PU nanofibers is increases after implantation. It will also improve the mechanical properties of the scaffold. This whole process can also be achieved by crosslinking the polyurethane (Kucinska et al., 2014).

The process of electrospinning is very simple and provides reasonable cost technology which produced high surface area of the nanofibers and porosity. Spun fibers are commonly used in various applications. The nanofibers coating have been characterized by few techniques such as scanning electron microscopy and UV Vis spectrometer (Nandana et al., 2009).

Polyurethane has been applied in many applications thanks to their uniqueness polymer structure. Moreover, functional groups can be easily incorporated into the polymeric chain, reducing the need of additional functionalization procedures on the polymer nanofibers with possible modification of morphology and bulk properties.

Since the development of PU, the quality of it has greatly been increasing. Many research has been continue to help make the materials much more superior. For

example, by changing the starting pre-polymers they can develop polyurethane fibers which have even better unique characteristics.

Future of the polyurethanes will improve the production process which results in faster, low cost, and more environmentally friendly polyurethanes. A recent study in polyurethane has been discovered that production of polyurethanes can be replaced from toluene diisocyanates with less-volatile polymeric isocyanates..

## 2.2 SPINNABLE PARAMETERS

The spinnable parameter that involved in the research should be considered. The important parameters are the concentration of the polymer, flow rate, distance of the tip of the needle from the current collector and applied voltage. (Tan S. H. et al., 2005) stated that when the flow rate, concentration of polymers and applied voltage are lower, the diameter of the fibers will smaller due to the effect of jet elongation.

The concentrations of the polymer solution also act as an important duty during the formation of the fibers while using the electrospinning technique. When the concentration of the solution is too high, it will results with the formation of a mixture of beads. On the other hand, when the concentration is too low, the fibers obtained will results like polymeric micro (nano)-particles (Li Z. et al., 2013).

(Li Z. et al., 2013) is also stated that the flow rate of the polymer solution play an important duty in the formation of the nanofibers. When the flow rate is too high, the thick diameter of nanofibers will formed. This is mostly because of the short drying time of the nanofibers to reach the current collector. The lower flow rate will be more suggested as the polymer solution will have enough time to reach the current collector.

## 2.3 NANOFIBER DIAMETER

Based on Bongdan C. et al., the nanofiber diameters can be calculated by few equations as follow below.

1) The diameter of the jet near the nozzle

$$h = \left(\frac{6\mu\rho Q^2}{\pi I E_\infty}\right)^{\frac{1}{2}} . Z^{-1} \quad (m) \qquad .....(1)$$

where :

$h$ = the jet radius [m]

$Z$ = the distance from the nozzle to the collector [m]

$\mu$ = the kinematic viscosity [$m^2/s$]

$\rho$ = the density [$kg/m^3$]

$Q$ = the flow rate [$m^3/s$]

$E_\infty$ = the external electric field [V/m]

$I$ = the electric current [A]

2) The diameter of the jet far from the nozzle

$$\boldsymbol{h = \left(\frac{\rho Q^3}{2\pi^3 I E_\infty}\right)^{\frac{1}{4}} . Z^{-\frac{1}{4}} \quad (m)} \qquad .....(2)$$

3) The shape of the jet, for the diameter, at the collector

$$h = \left[\gamma\varepsilon \left(\frac{Q}{I}\right)^2 \frac{2}{\pi(2\ln x - 3)}\right]^{\frac{1}{3}} \quad (m) \qquad .....(3)$$

where:

$\gamma$ = the surface tension of the fluid [N/m]

$\varepsilon$ = the dielectric permittivity [F/m]

$x$ = $R/h$ is the dimensionless whipping instability, with $R$ being the radius of the instability jet and $h$ the fiber diameter

$Q/I$ = the inverse of the net volume density of the charge induced in the fluid at the top electrode [$m^3/C$]

- Eq. (3) predicts that the terminal diameter of the whipping jet is controlled by the flow rate, electric current and the surface tension of the fluid.

4) From (2),

$$h = \left(\frac{\rho Q^3}{2\pi^3 I E_\infty}\right)^{\frac{1}{4}} . Z^{-\frac{1}{4}} \quad (m)$$

where:

I = $U/R$ (we consider only conduction current)

R = $r_s l/S$ is the electrical resistance of the jet

L = $z$ is the length of the jet [m]

S = $\pi h^2/4$ is the section of the jet [$m^2$]

*rs* = the resistivity of the solution [Ω m]

Thus,

$$h = \left(\frac{2 r_s \rho Q^3}{\pi^3} \frac{Z}{u^2}\right)^{\frac{1}{6}} \quad (m) \qquad \text{.....(4)}$$

5) Fiber diameter vs conductivity of solution. ( Eq. (2))

From (2),

$$h = \left(\frac{\rho Q^3}{2\pi^3 I E_\infty}\right)^{\frac{1}{4}} . Z^{-\frac{1}{4}} \quad (m)$$

where:

*I* = *U*/*R*

*E* = *U*/*Z*

*R* = the electrical resistance of the jet [Ω]

*U* = the applied voltage [V]

Thus,

$$h = \left(\frac{\rho Q^3}{2\pi^2\left(\frac{u^2}{RZ}\right)}\right)^{\frac{1}{2}} . Z^{-\frac{1}{4}} \quad (m) \qquad \text{.....(5)}$$

where :

*RZ* [Ω m] = the resistivity of the solution

1/*RZ* [S/m] = the conductivity of the solution

From (1),

$$h = \left(\frac{6\mu\rho Q^2}{\pi I E_\infty}\right)^{\frac{1}{2}} . z^{-1} \quad (m)$$

Thus,

$$h = \left( \frac{6\mu\rho Q^2}{\pi \frac{U^2}{RZ}} \right)^{\frac{1}{2}} . Z^{-1} \quad (m) \qquad \text{.....(6)}$$

## 2.4 SCANNING ELECTRON MICROSCOPY (SEM)

Scanning Electron Microscopy (SEM) is used to evaluate the morphology of the fibers that has been produced. Fiber diameters and pore size were measured on different images using Image software (Chiara et al., 2015). It is also used to determine the composition of the material and surface topography. The more greater the magnification of lenses, the image image obtained will be more clearer.

## 2.5 ATTENUATED TOTAL REFLECTION – FOURIER TRANSFORM INFRARED (ATR-FTIR)

Attenuated Total Reflection (ATR) is one accessory to measure Fourier Transform Infrared (FTIR) spectra. It is used to analysis the polymer surfaces especially if the sample is soft. The penetration depth of this technique, or the depth from which the Infrared (IR) signal is generated varies as the function of the crystal used, angle of incidence and wavelength (Seyedah et al.2014).

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# CHAPTER 3

# MATERIALS AND METHODS

## 3.1 INTRODUCTION

In this study, there are two steps involved in preparing the PU coating which are the synthesizing the PU solutions with N,N-dimethylformamide (DMF) and the preparation of PU nanofibers using electrospinning technique. There are two types of material characterization involved which are determination of PU nanofibers mat by using Scanning Electron Microscopy (SEM) and Attenuated Total Reflectance (ATR).

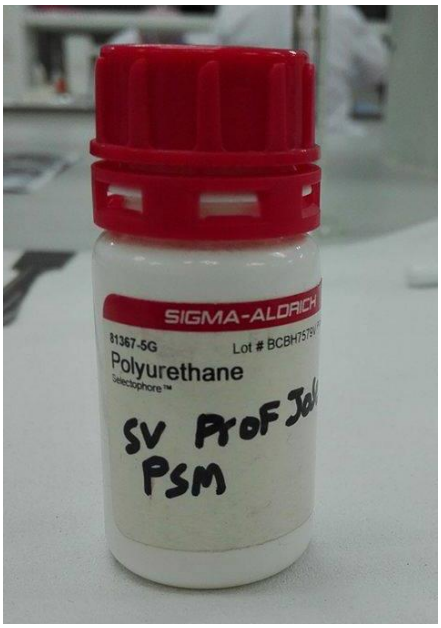


**Figure 3.1** Polyurethane

## 3.2 RESEARCH METHODOLOGY

### 3.2.1 Synthesizing PU polymeric solution

During the process, 12 wt.% of PU solution was prepared in DMF solution. The PU and DMF were mixed in Schott bottle. Magnetic stirrer was inserted into the bottle and enclosed bottle cap. The mixture was stirred at 70ºC at speed of 350 rpm for approximately 3 hours.

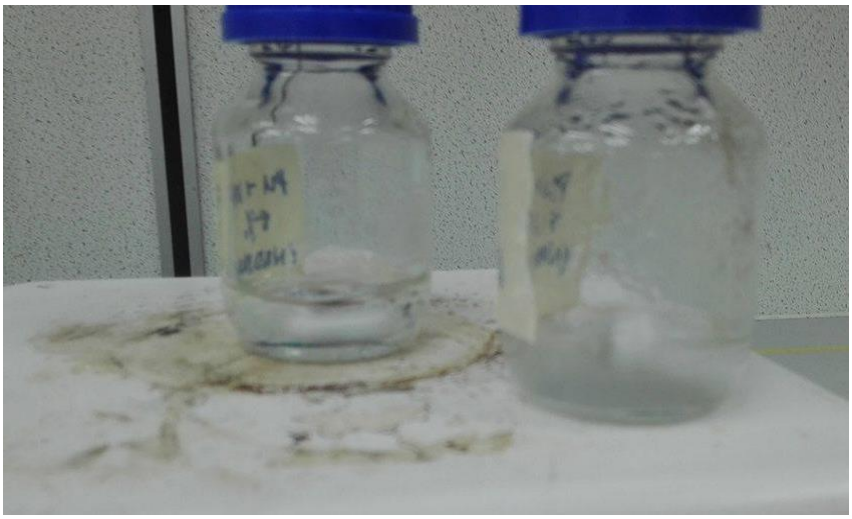

**Figure 3.2.1** Mixing of Polyurethane in DMF solution

### 3.2.2 Electrospinning of PU nanofibers

The syringe was filled with PU solution up until 3 mL and the needle was installed at the tip. X-ray film is used as a current collector was cut approximately 11cm × 11cm. The syringe was located at the syringe pump holder. The wires of power supply was connected to the needle tip and X-ray film as a current collector. The current collector distance was set to 8 cm. The syringe pump was turn on and the solution speed was set to 1.0 mL/h. The power supply was turn on and the voltage was adjusted to 30 kV. The PU solution was electrospun for approximately 3 hours.

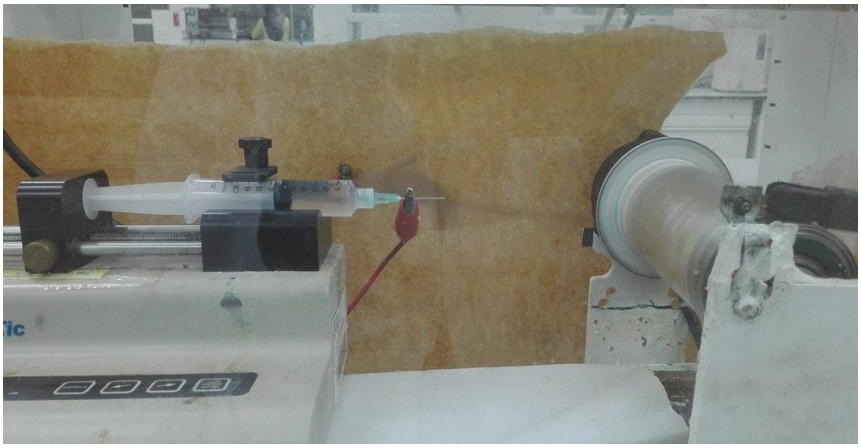

**Figure 3.2.2** Electrospinning Process

## 3.3 MATERIAL CHARACTERIZATIONS

### 3.3.1 Determination of PU nanofibers by using Scanning Electron Microscopy (SEM)


**Figure 3.3.1** SEM Equipment

SEM equipment is used to determine the composition of the material and surface topography. It also used to evaluate the morphology of the fibers that has been produced.

### 3.3.2 Attenuated Total Reflectance (ATR)

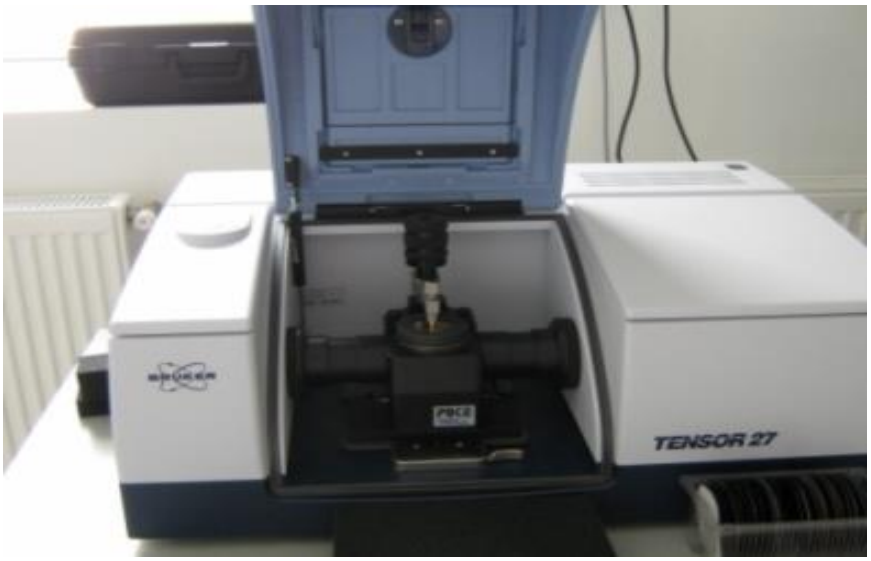


**Figure 3.3.2** ATR Equipment

ATR equipment is used to measure Fourier Transform Infrared (FTIR) spectra. It is used to analysis the polymer surfaces especially if the sample is soft.

## 3.4 FLOW CHART

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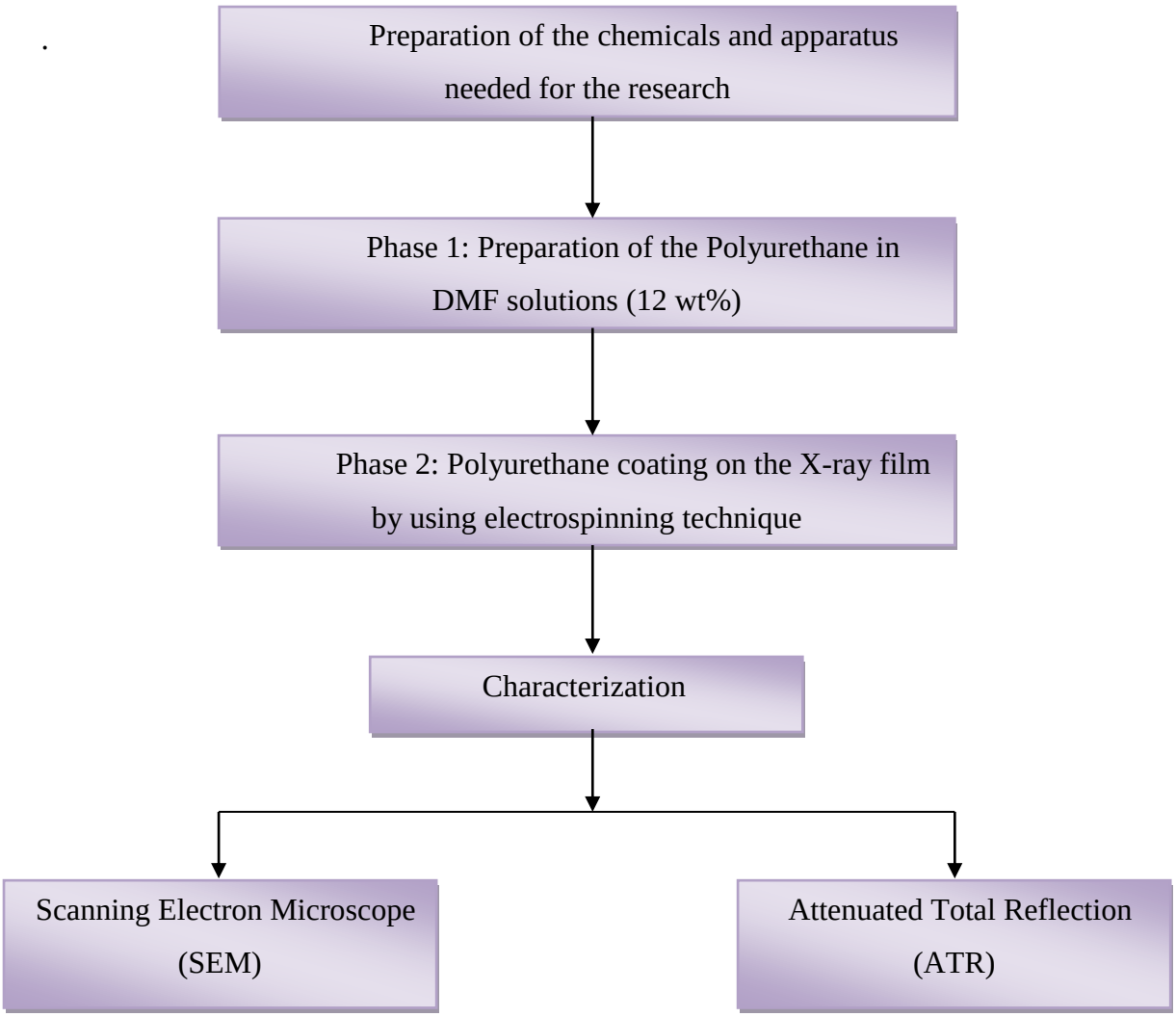


**Table 3.4** Flow chart of the research

# CHAPTER 4

# RESULT AND DISCUSSION

## 4.1 SYNTHESIS AND CHARACTERIZATION OF POLYURETHANE COATINGS

### 4.1.1 Scanning Electron Microscopy (SEM) Analysis

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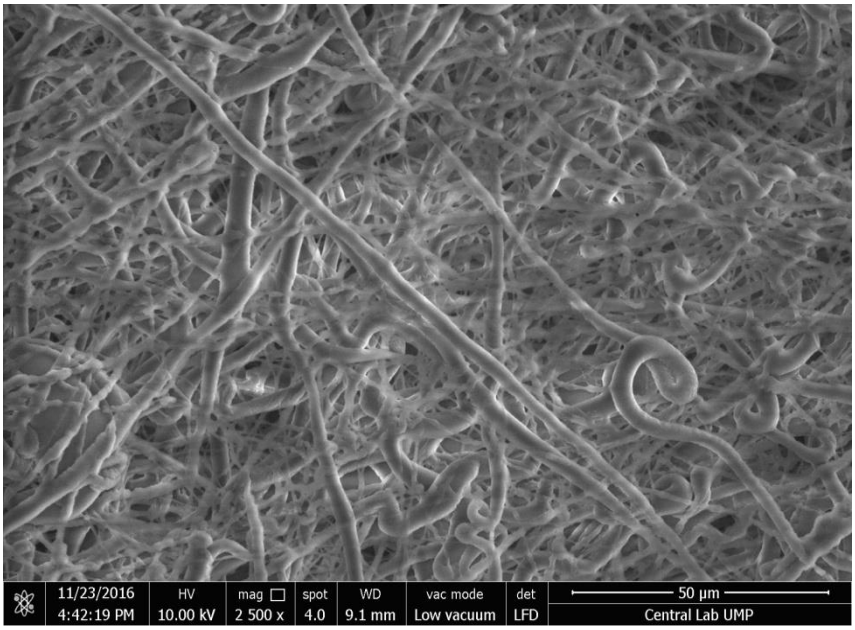


. **Figure 4.1.1 (a)** SEM image (Magnification: 2 500 X)

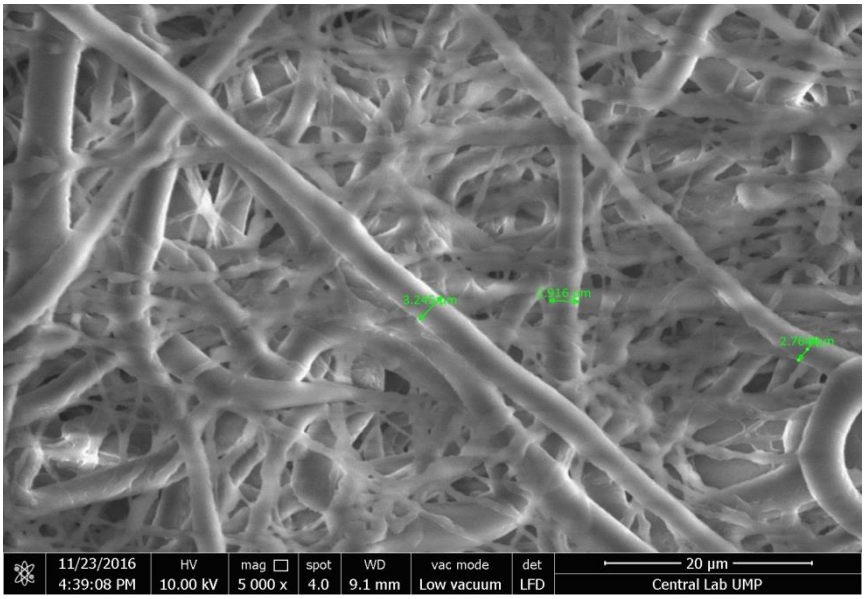


**Figure 4.1.1 (b)** SEM image (Magnification: 5 000 X)

**Figure 4.1.1 (a) and (b)**: SEM images of Polyurethane coatings on X-ray film

Based on **Figure 4.1.1 (a) and (b)** shows that the images become more clearer as the magnification increases. It shows that the diameter of the nanofibers are not constantly distributed.

### 4.1.2 Attenuated Total Reflectance-Fourier Transform Infrared (ATR-FTIR) Analysis

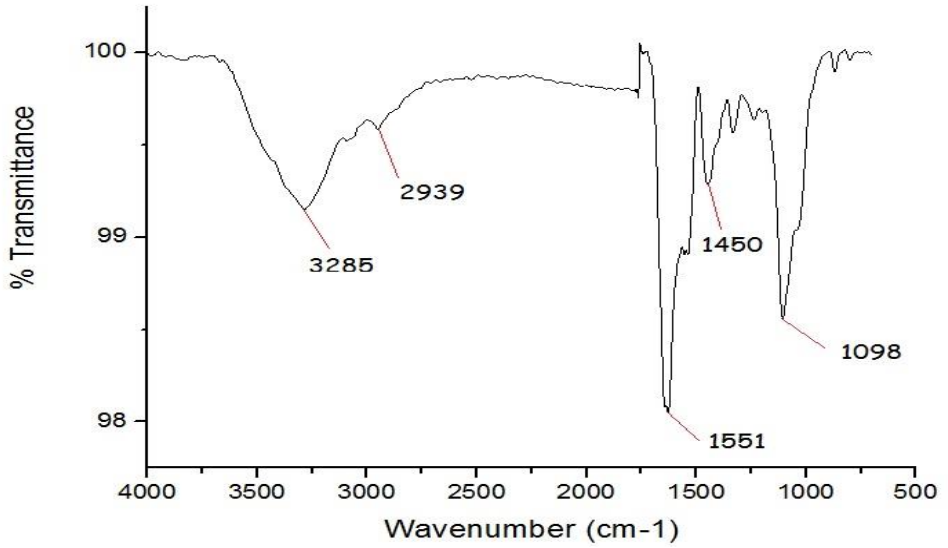


**Figure 4.1.2 (a)** ATR-FTIR spectrum for PU solutions

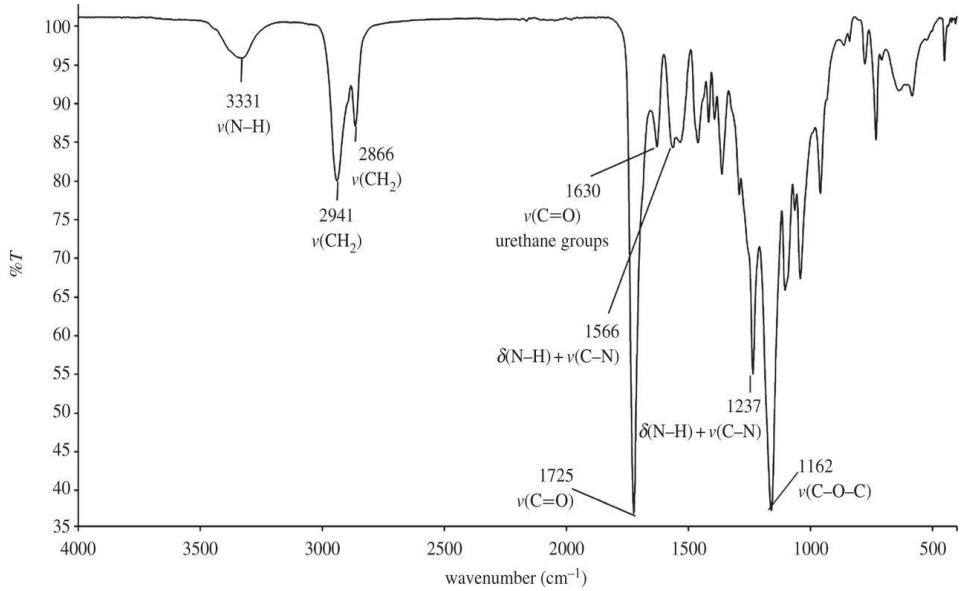


**Figure 4.1.2 (b)** ATR-FTIR spectrum for pure PU solutions (Chiono V. et al.)

ATR-FTIR spectrum of PU electrospun nanofibers and pure PU polymer are given in **Figure 4.1.2 (a) and Figure 4.1.2 (b)** respectively. Both spectra illustrate the similar general feature. It shows that the PU electrospun has maintained it's molecular structure during the electrospinning process.

Based on Figure 4.1.2 (a), the broad peak at 3140 – 3429 $cm^{-1}$ indicated stretching vibrations of the hydroxyl group due to the intermolecular and intramolecular hydrogen bonds of the O-H groups of PU. The formation of urethane linkages was confirmed by the appearances of the region at 1551 $cm^{-1}$ attributed to N-H bending vibrations (amide) and the other at 1098 $cm^{-1}$due to C-O stretching.

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# CHAPTER 5

# CONCLUSION AND RECOMMENDATION

## 5.1 CONCLUSION

Electrospinning technique is used in this research because the technique is simple, save cost and it can produced nanofibers with high crosslinking range. It can be concluded that PU polymer can properly be coated on the surface of X-ray film. The spinnable parameters are very important in this research to ensure that the polyurethane can be coated properly on the X-ray film. Polyurethane based on the DMF solutions are suitable for electrospinning process because the nanofibers can be formed on the X-ray film. The crosslinking of polyurethane nanofibers on the X-ray film is achieved so it will enhance the strength of the nanofibers film and also will enhance the durability of the X-ray film. Nanofibers have been characterized by scanning electron microscope and attenuated total reflection. Besides all the advantages of electrospinning technique for these coatings, there are few limitations such as the nanofibers took some times to be dried on the X-ray film. This is might be because of the types of the current collector used in this research which is the X-ray film.

## 5.2 RECOMMENDATIONS

In general, the favourable electrospinning parameters for this research are: a voltage of 30kV, a working distance of 8 cm and a flow range of 1.00 ml/h. The spinnable parameters are very important in this research to ensure that the polyurethane can be coated properly on the X-ray film.

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