Cellulose Acetate Ultrafine Fibers Prepared by Electrospinning Technique

Miss Tanarinthorn Puangparn
Miss Monchawan Weerasombut

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Name	Miss Tanarinthorn Puangparn 45050901 Miss Monchawan Weerasombut 45050909
Department	Chemistry
Program	Polymer Science and Technology
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Special Project Advisor	Assoc.Prof.Dr. Ittipol Jangchud
Special Project Co-advisor	Assoc.Prof.Dr. Pitt Supaphol

ABSTRACT

Electrospinning or electrostatic spinning is a process by which a high electrical potential is applied to a polymer solution to jet out fibers from needle to collector plate with diameters in the sub-micrometer down to nanometer ranges. In this research work, effects of various solution parameters (such as concentration, electrical conductivity and solvent system) and process parameters (such as applied voltage and distance from needle to collector plate) on morphological appearance and average sizes of electrospun cellulose acetate fibers were investigated using scanning electron microscope (SEM). For the investigation of solution parameters based on solution properties, it was found that these properties were important factors on morphology and the diameter of the fibers obtained. The lower the concentration, the smaller the diameter of fibers. For process parameters, the morphological appearance and the diameter between fibers obtained from higher or lower applied voltage were different. The electrospun fibers from higher applied voltage had bigger diameter than those of lower applied voltage. Swelling test showed a minor effect to the CA ultrafine fibers.

Finally, in this research, it was found that the optimum conditions for CA electrospinning process were 16% w/v with 12 kV by controlling the solvent ratio of 2:1 (acetone:DMAc).

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Tanarinthorn Puangpam
Monchawan Weerasombut

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

FIGURE **PAGE**

FIGURE **PAGE**

FIGURE **PAGE**

CHAPTER 1
INTRODUCTION

1.1 Background

Ultrafine fiber is an exciting new class of materials used for several value-added applications such as medical, filtration, barrier, wipes, personal care, composite, garments, insulation and energy storage. Special properties of nanofibers make them suitable for a wide range of applications from medical to consumer products and industrial to high-tech applications for aerospace, capacitors, transistors, drug delivery systems, battery separators, energy storage, fuel cells and information technology [1,2]. Ultrafine fibers produced by electrospinning technology come from polymer solutions. The polymer used is the most important parameter that defines the properties of the final ultrafine fibrous fabric [3].

Electrospinning is a technique to spin polymer fibers with diameter in nanometer scales by using electrical field to apply both polymer solution and a collector plate to force a polymer jet out of the needle tip during spinning. For electrospinning of polymer fibers to have diameter in nanometer scale, some parameters must be concerned such as applied voltage, polymer solution concentration, distance between needle tip and collector plate and also electrical conductivity of polymer solution [4].

Cellulose acetate (CA) is derived from cellulose by deconstructing wood pulp into a purified fluffy white cellulose. The cellulose is then reacted with acetic acid and acetic anhydride in the presence of sulfuric acid. It is then put through a controlled, partial hydrolysis to remove the sulfate and a sufficient number of acetate groups to give the product the desired properties. The anhydroglucose unit is the fundamental repeating structure of cellulose and has three hydroxyl groups which can react to form acetate esters. The most common form of cellulose acetate fiber has an acetate group on approximately two of every three hydroxyls. This cellulose diacetate is known as secondary acetate, or simply as "acetate" [5].

Cellulose acetate (CA) is an amorphous thermoplastic material in cellulosic resin family. It is obtained by introducing the acetyl radicals of acetic acid into cellulose (as cotton or wood fibers) to produce a tough plastic material.

Cellulose acetate has been widely used as a base for photographic and motion picture film, microfilm, aerial film, transparent overlays and for sound recording [6].

1.2 Objectives

1. To prepare cellulose acetate (CA) ultrafine fibers by using electrospinning technique.
2. To study some properties of the prepared CA ultrafine fibers including physical and morphological properties.

1.3 Scopes of Study

1. Literature reviews of the theory and publication involved this research.
2. To study some factors affecting the electrospinning process such as concentration of polymer solution, amount of voltage, the distance between needle tip and collector plate, and also electrical conductivity of polymer solution.
3. To characterize the properties of the fibers, such as fiber diameters, swelling, and weight loss.

1.4 Expected Results

1. To gain appropriate conditions in preparing CA ultrafine fibers with electrospinning technique.
2. Knowledge and findings in this work can be applied to use in other applications such as drug delivery, membrane and synthesized membrane.

CHAPTER 2

THEORY AND LITERATURE REVIEWS

2.1 Electrospinning Process

Nanofibers are polymer fibers that having diameters of less than one micron or less in the nanoscales. When the diameters of polymer fiber materials are down from micrometers (10^{-6}m) to nanometers (10^{-9}mm), they appear to have several amazing characteristics such as very large surface area to volume ratio, flexibility in surface functionalities, and superior mechanical performance (e.g. stiffness and tensile strength) compared with other materials. These outstanding properties make the polymer nanofibers to be optimal candidates for many important applications such as medical and filtration applications. An electrospinning technique has been used to produce polymer nanofibers.

Electrospinning is the process that using electrostatic force to generate the fine filament. The electrospinning set up is shown in Figure 2.1 which consists of an adjustable DC power supply for applying the voltage, a syringe is connected to a stainless steel needle using a teflon tube having an inner diameter of 1.0 mm, and collector wrapped with aluminum foil [6].

In the electrospinning process a high voltage is used to create an electrically charged jet of polymer solution or melt, which dries or solidifies to leave polymer fibers. One electrode is placed into the spinning solution/melt and the other attached to a collector. Electric field is subjected to the end of a capillary tube that contains the polymer fluid held by its surface tension. This induces a charge on the surface of the liquid. Mutual charge repulsion causes a force directly opposite to the surface tension. As the intensity of the electric field is increased, the hemispherical surface of the fluid at the tip of the capillary tube elongates to form a conical shape known as the Taylor cone. With increasing field, a critical value is attained when the repulsive electrostatic force overcomes the surface tension and a charged jet of fluid is ejected from the tip of the Taylor cone. The discharged polymer solution jet undergoes a whipping process where in the solvent evaporates, leaving behind a charged polymer fiber, which lays itself randomly on a grounded collecting metal screen. In the case of the melt the discharged jet solidifies when it travels in the air and is collected on the collector [7].

The coulombic repulsion force between charges of the same polarity produced in the polymer solution or melt by the emitting electrode destabilizes the hemispherical droplet of the polymer solution or melt located at the tip of the nozzle to finally form a droplet with a conical shape (i.e. the Taylor cone). With further increase in the electrostatic field strength beyond a critical value, the coulombic repulsion force finally exceeds that of the surface tension which results in the ejection of an electrically charged stream of the polymer solution or melt (the charged jet).

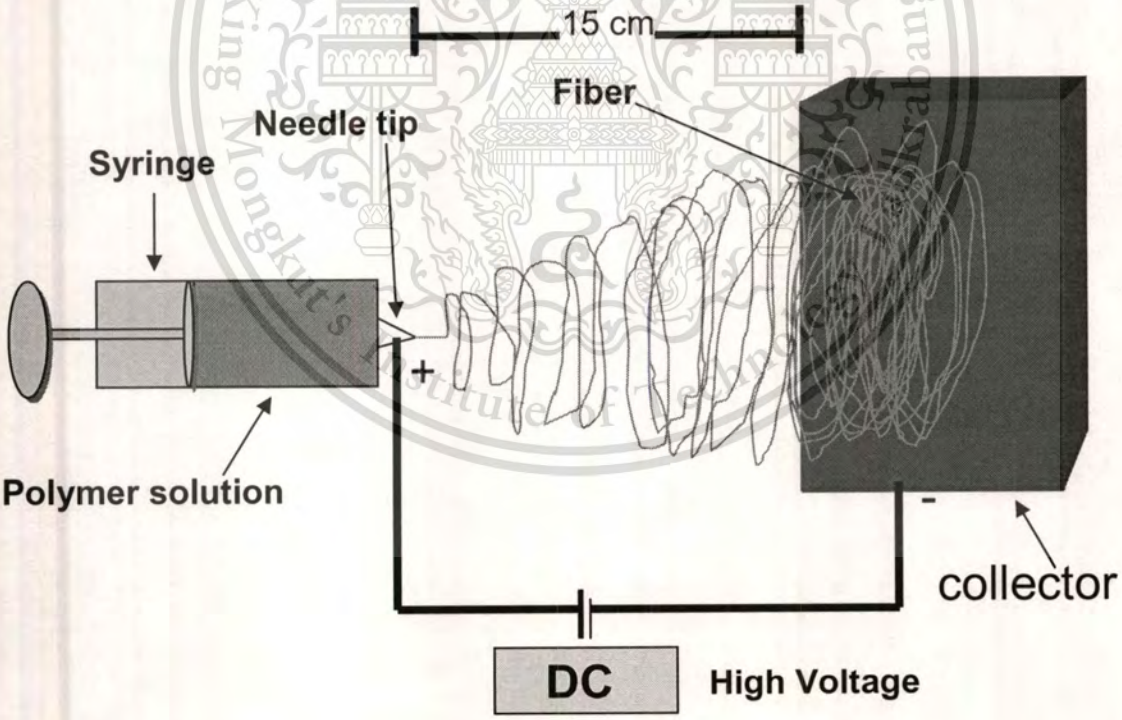


Figure 2.1 Electrospinning process set-up

There are six major forces acting on an infinitesimal segment of the charged jet :

1) Body or gravitational forces
2) Electrostatic forces which carry the charged jet from the nozzle to the target
3) Coulombic repulsion forces which try to push apart adjacent charged species present within the jet segment and are responsible for the stretching of the charged jet during its flight to the target
4) Viscoelastic forces which try to prevent the charged jet from being stretched
5) Surface tension which also acts against the stretching of the surface of the charged jet
6) Drag forces from the friction between the charged jet and the surrounding air [8]

System Parameters:

1. Molecular weight, molecular-weight distribution (MWD) and architecture such as branched, linear etc. of the polymer
2. Solution properties (viscosity, conductivity and surface tension)

Process Parameters:

1. Electric potential, flow rate and concentration
2. Distance between the needle tip and collector
3. Ambient parameters temperature, humidity and air velocity in the chamber
4. Motion of target screen [9]

One of the most important quantities related with electrospinning is the fiber diameter. The fiber diameters will depend primarily on the jet sizes as well as on the polymer contents in the jets. It has been recognized that during the traveling of a solution extent is the applied electrical voltage. In general, a higher applied voltage ejects more fluid in a jet, resulting in a larger fiber diameter [10].

Another problem encountered in electrospinning is defects such as "beads" occur in polymer nanofibers. It has been found that the polymer concentration also affects the formation of the beads. In general, the higher polymer concentration results in fewer beads [11].

Figure 2.2 SEM photographs of electrospun nanofibers from different polymer concentration solutions [12]

An addition of salts will result in a higher charge density on the surface of the solution jet during the electrospinning, bringing more electric charges to the jet. As the charges carried by the jet increased, higher elongation forces would be imposed to the jet under the electrical field, resulting in smaller bead and thinner fiber diameters [13].

2.2 Applications of Polymer Nanofibers

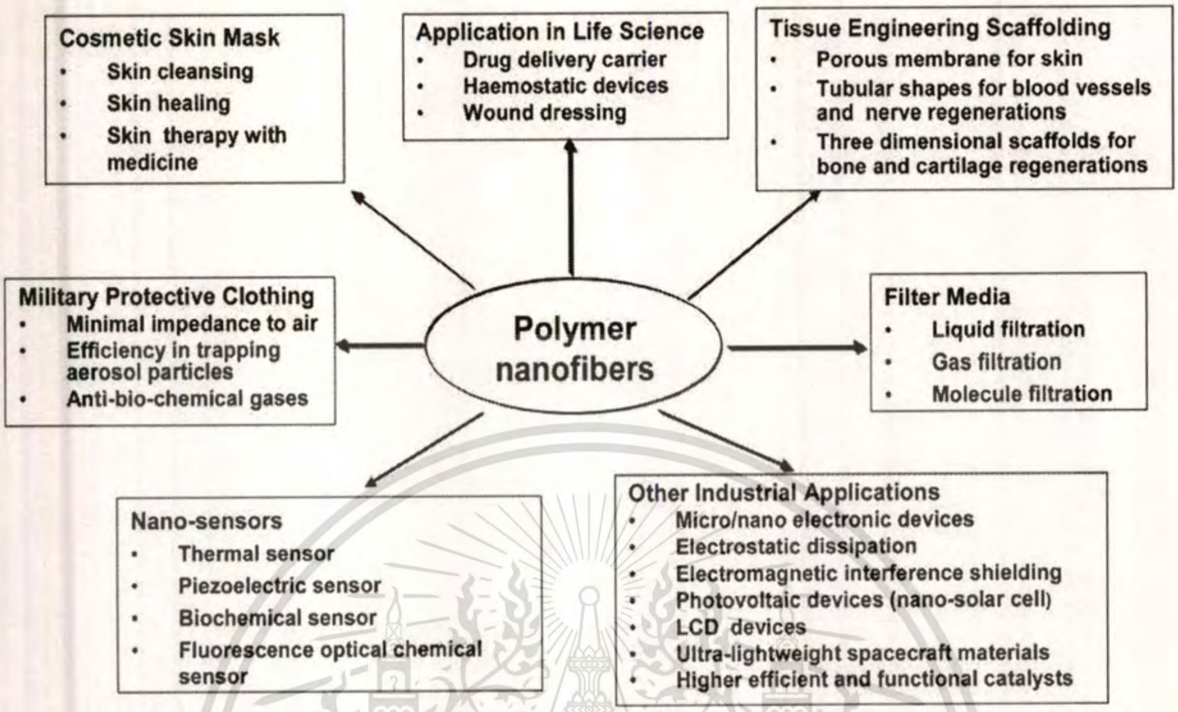


Figure 2.3 Applications of polymer nanofibers [14]

2.2.1 Filtration Application

Filtration is necessary in many engineering fields [15]. In general, due to the very high surface area to volume ratio and resulting high surface cohesion, tiny particles of the order of <0.5 mm can be easily trapped in the electrospun nanofibrous structured filters and hence the filtration efficiency can be improved [16,17].

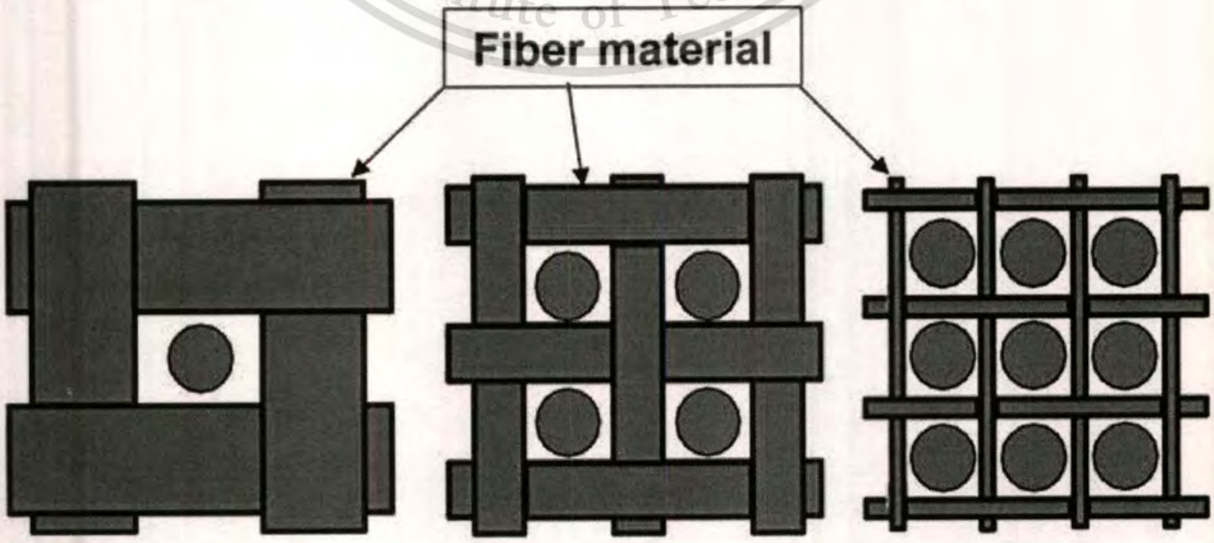


Figure 2.4 The efficiency of a filter increases with decrease in fiber diameter [14]

2.2.2 Biomedical Application

-Medical prostheses

Polymer nanofibers fabricated via electrospinning have been proposed for a number of soft tissue prostheses applications such as blood vessel, vascular, breast, etc [18-24]. In addition, electrospun biocompatible polymer nanofibers can also be deposited as a thin porous film onto a hard tissue prosthetic device designed to be implanted into the human body [25-28]. This coating film with gradient fibrous structure works as an interphase between the prosthetic device and the host tissues, and is expected to efficiently reduce the stiffness mismatch at the tissue/device interphase and hence prevent the device failure after the implantation [14].

-Tissue template

For the treatment of tissues or organs in malfunction in a human body, human cells can attach and organize well around fibers with diameters smaller than those of the cells. In this regard, nanoscale fibrous scaffolds can provide an optimal template for cells to seed, migrate, and grow [29].

- Wound dressing

Polymer nanofibers can also be used for the treatment of wounds or burns of a human skin, as well as designed for haemostatic devices with some unique characteristics. With the aid of electric field, fine fibers of biodegradable polymers can be directly sprayed/spun onto the injured location of skin to form a fibrous mat dressing which can let wounds heal by encouraging the formation of normal skin growth and eliminate the formation of scar tissue which would occur in a traditional treatment [30-33].

- Drug delivery and pharmaceutical composition

In general, the smaller the dimensions of the drug and the coating material required to encapsulate the drug, the better the drug to be absorbed by human being. Drug delivery with polymer nanofibers is based on the principle that dissolution rate of a particulate drug increases with increasing surface area of both the drug and the corresponding carrier if needed [34].

2.2.3 Protective Clothing Application

The protective clothing in military is mostly expected to help maximize the survivability, sustainability, and combat effectiveness of the individual soldier system against extreme weather conditions, ballistics, and NBC (nuclear, biological, and chemical) warfare [35]. Electrospinning results in nanofibers laid down in a layer that has high porosity but very small pore size, providing good resistance to the penetration of chemical harm agents in aerosol form [36].

2.3 Cellulose Acetate

Cellulose acetate (CA) is used for transparent, translucent and opaque objects (e.g. typewriter keys, calculators, switches, car wheel coverings).

Furthermore, it is especially suitable for coatings applications requiring high melting-point, toughness, clarity, and good resistance to ultraviolet light, chemicals, oils, and greases.

Cellulose acetate is an amorphous thermoplastic material belonging to the cellulosic resin family. It is obtained by introducing the acetyl radical of acetic acid into cellulose (as cotton or wood fibers) to produce a tough plastic material [6].

Cellulose acetate is manufactured by reacting cellulose with acetic anhydride using sulfuric acid as a catalyst. This slow burning base material is frequently used for motion picture films and in sheet form for overlay cells in animation.

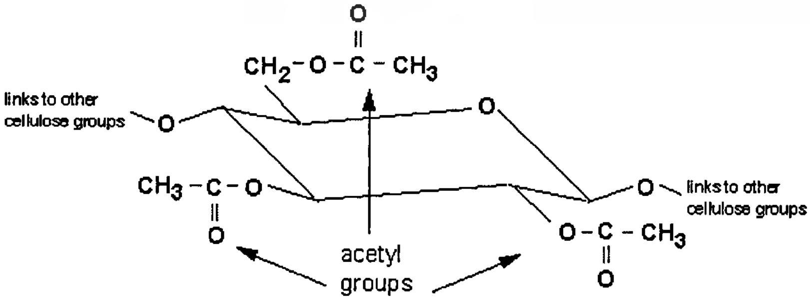


Figure 2.5 Structure of cellulose acetate

Cellulose will react under anhydrous conditions, in the presence of an acid catalyst, with acetic anhydride to form cellulose triacetate according to the following simplified equation:

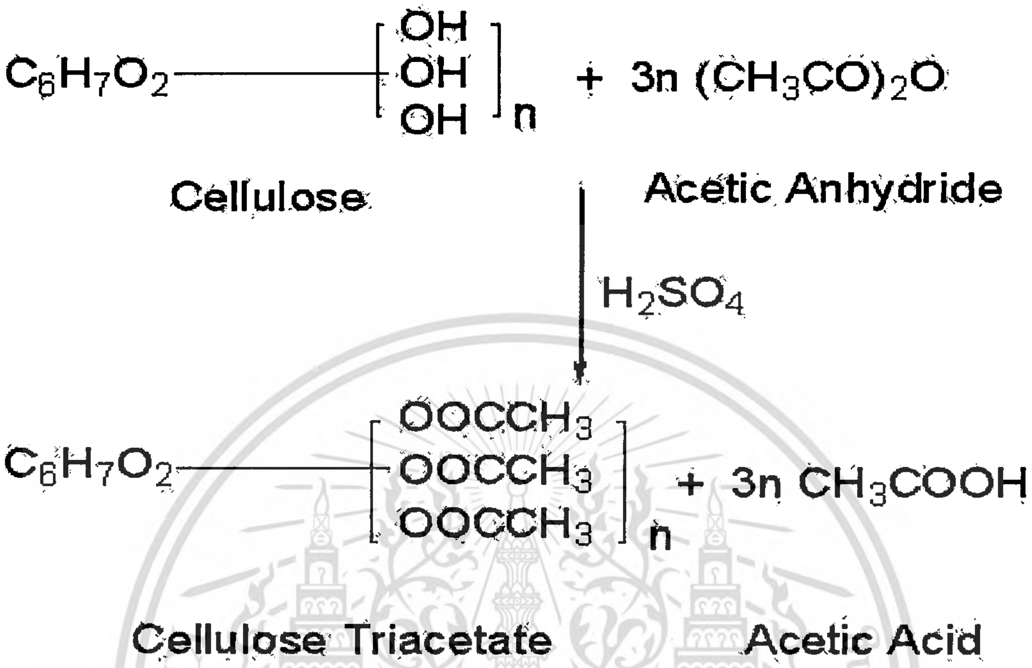


Figure 2.6 The mechanism of cellulose acetate

The fully acetylated cellulose triacetate is then hydrolyzed to give cellulose acetate according to the following simplified equation [37].

Literature Reviews

Na Yu, Shao Changlu, Liu Yichun, Guan Hongyu, Yang Xinghua [38] found a method for making inorganic nanofibers by combining sol–gel processing with electrospinning technique. Extrathin fibers of poly(vinyl alcohol) (PVA)/lithium chloride/manganese acetate composite fibers were prepared. After calcination of the precursor fibers at 600 °C, the spinel lithiummanganese oxide ($LiMn_2O_4$) nanofibers with a diameter of 100–200 nm, were successfully obtained. The fibers were investigated by TG–DTA, XRD, FT-IR, and SEM, respectively. The results showed that the crystalline phase and morphology of the fibers were largely influenced by the calcination temperature. One of the attractive features associated with this method was that the obtained nanofiber mats possessed high surface areas and small pore sizes. The electrospun nanofibers of $LiMn_2O_4$ was described in order to obtain a kind of material with improved electrode properties due to its high surface area and 1D (Quasi-one-dimensional) nanostructural properties.

Guan Hongyu, Shao Changlu, Liu Yichun, Na Yu, Yang. Xinghua [39] prepared electrospun nanofibers of $NiCo_2O_4$ to improve its electrocatalytic. The spinel $NiCo_2O_4$ nanofibers with diameters of 50–100 nm were prepared by high temperature calcinations of simple inorganic-polymer composite fibers, which were obtained by electrospinning of the PVA/cobalt acetate/nickel acetate composite precursor. The crystallinity, purity, and surface morphology of the as-prepared $NiCo_2O_4$ nanofibers were investigated by XRD, FT-IR, SEM, respectively.

Yanga Xinghua, ShaoChanglu, Liua Yichun, Mub Rixiang, Guana Hongyu [40] studied thin polyvinyl alcohol/cerium nitrate composite fibers which were prepared by using sol–gel processing and electrospinning technique. This method was used to obtain nanofiber mats with high surface areas and small pore sizes which are beneficial in a variety of applications. After calcinations of the above precursor fibers, CeO_2 nanofibers with a diameter of 50–150 nm were successfully obtained. The fibers were characterized by Raman spectroscopy, thermogravimetric analysis, differential thermal analysis, X-ray diffraction, Fourier transform infrared spectroscopy and scanning electron microscopy. In this work, nanofibers of CeO_2 with improved catalytic activity and storage capacity due to its high surface areas and small pore sizes were prepared.

Kim Bumsu, Park Hyun, Lee Sung-Hwan, M.Wolfgang Sigmund [41] studied poly(acrylic acid) (PAA) nanofibers fabricated by electrospinning into NaCl solution. Effects of the ionic strength of NaCl on the morphology of PAA nanofibers was investigated. The smallest diameter of the PAA nanofiber was prepared with 0.01 M NaCl PAA solutions. At the concentration 1 M NaCl, PAA nanofibers could not be fabricated. The viscosity and conductivity changes were measured with changing the concentration of NaCl. It was suggested that diameter variations may be caused by conductivity and polymer chain conformation changes with varying the concentration of NaCl.

N. Dharmaraj, H.C. Park, C.K. Kim, H.Y. Kim, D.R. Lee [42] described the preparation and characterization of nickel titanate nanofibers produced by the calcination of nickel titanate/PVAc composite fibers obtained through electrospinning. Nickel titanate/poly(vinyl acetate) (PVAc) composite nanofibers were prepared by sol–gel processing and electrospinning technique. Rhombohedral nickel titanate fibers with 150–200 nm diameter were obtained by high temperature calcination of the composite fibers. Structural, morphological and crystalline phase features of electrospun $NiTiO_3$ fibers were studied by SEM, AFM, XRD and FT-IR. The fibrous nature of nickel tiatanate was stable even after calcination at 1273 K.

Yu Wang, Serrano Santiago, J. Jorge Santiago-Avile′s [43] studied Raman characterization of eletrospun carbon nanofibers Ultrafine fibers spun from polyacrylonitrile (PAN)/N,N-dimethyl formamide (DMF) precursor solution using a homemade electrospinning setup, and then pyrolyzed in vacuum at 873, 1073, 1273 and 1473 K, respectively. The pyrolyzed fibers, with diameter of the order of 100 nm as revealed by a scanning electron microscope (SEM), were characterized using Raman microspectrometry. Their Raman scattering spectra manifested D and G peaks, characteristic of disordered carbon and graphite, respectively. PAN-based carbon nanofibers were prepared by electrostatic deposition and subsequent vacuum pyrolysis. Raman characterization revealed D and G peaks, characteristic of the disordered carbon and graphite in the nanofibers. The graphitic crystallite size and mole fraction were estimated between 1.5 and 2.6 nm, and between 0.25 and 0.37, respectively, both increasing with the pyrolyzing temperature. The graphite mole fraction was found to obey an Arrhenius relation: $x_G = x_0 \exp(-Q/RT)$, where $x_0 = 0{:}691 \pm 0{:}081$ and $Q = 7360 \pm 110$ J/mol.

Changlu Shao, Xinghua Yang, Hongyu Guan, Yichun Liu, Jian Gong [44] studied nanofibers of PVA/nickel acetate/zinc acetate composite prepared by using sol–gel processing and electrospinning technique. By high temperature calcinations of the above precursor fibers, nanofibers of NiO/ZnO composite with diameters of 50–150 nm could be successfully obtained. The fibers were characterized by TG/DTA, IR, XRD and SEM. The results showed that the crystallinity and the morphology of the fibers were largely influenced by the calcination temperature. Nanofibers of NiO/ZnO composite with diameters of 50–150 nm were prepared by using sol–gel processing and electrospinning technique. This route might open a new door to make nanofibers of mixed metal oxide composite. By modifying the parameters of sol–gel or electrospinning processing, one could also expect to be able to make nanofibers of composite materials with smaller diameter.

Changlu Shao, Hongyu Guan, Yichun Liu, Xiliang Li, and Xinghua Yang [45] prepared Mn_2O_3 and Mn_3O_4 nanofibers via an electrospinning technique. Electrospun fibers of PVA/manganese acetate composite by using sol–gel processing and electrospinning technique were studied. Nanofibers of Mn_2O_3 and Mn_3O_4 were obtained by calcinations of the precursor fibers under different temperatures. After calcinations of the above precursor fibers, Mn_2O_3 and Mn_3O_4 nanofibers with a diameter of 50–200 nm could be successfully obtained. The fibers were characterized by TG–DTA, Scanning electron microscopy, FT-IR, WAXD, respectively. The results showed that the crystalline phase and morphology of nanofibers were largely influenced by the calcination temperature.

Periasamy Viswanathamurthi, Narayan Bhattarai, Hak Yong Kim, Douk Rae Lee [46] studied about vanadium pentoxide nanofibers by electrospinning. New vanadate fibers with submicron diameters were prepared by electrospinning using vanadium sol and poly(vinylacetate) (PVAC) solutions followed by thermal treatment. The fibers were characterized by SEM, AFM, XRD and IR spectra.

Yuh Junhan, C.Juan Nino and M.Wolfgang Sigmund [47] synthesized barium titanate nanofibers with perovskite structure by electrospinning. Solution–gel as precursors was utilized along with high molecular weight polymers yielding electrospun nanofibers that was dried and annealed. After that polycrystalline barium titanate fibers was obtained after annealing at 750 °C for 1 h. Typical fiber morphology was 80–190 nm in diameter and over 0.1 mm in length. In this experiment morphologies and dimensions of nanofibers to analyze the microstructure and crystal structure of the nanofibers were studied. In this experiment they demonstrated that the electrospinning technique offered a simple and versatile method for synthesizing long nanofibers of complex ferroelectric ceramics.

Zuwei Ma, M. Kotaki and S. Ramakrishna [48] studied the feasibility of applying cellulose nanofiber membrane prepared by electrospinning as affinity membrane. Cellulose acetate (CA) solution (0.16 g/ml) in a mixture solvent of acetone/DMF/trifluoroethylene (3:1:1) was electrospun into nonwoven fiber mesh with the fiber diameter ranging from 200 nm to 1 μm. CA nanofiber mesh was heated under 208 °C for 1 h to improve structural integrity and mechanical strength, and then treated in 0.1 M NaOH solution in H_2O/ethanol (4:1) for 24 h to obtain regenerated cellulose (RC) nanofiber mesh. The RC nanofiber membrane was further surface functionalized with Cibacron Blue F3GA (CB), a general affinity dye ligand for separation of many biomolecules. Chemical and physical properties of the materials were characterized by SEM, DSC and ATR-FTIR. Water filtration properties of the novel RC membrane were studied and compared with a commercial micro-filtration membrane. The CB derived RC nanofiber membrane has a CB content of 130 μmol/g, and capture capacity of 13 mg/g for bovine serum albumin (BSA) and 4 mg/g for bilirubin. The membrane showed reusability after regeneration with elution buffer. Dynamic adsorption of BSA on the nanofiber membrane was studied by breakthrough curve measurements.

CHAPTER 3

EXPERIMENTAL DETAILS

This work can be divided into 4 parts:

Part 1 Effects of concentration and viscosity of cellulose acetate solution

First, cellulose acetate solutions in 2:1 (acetone:DMAc) solvent with different concentrations of 12, 14, 16, 18 and 20% w/v were prepared. Each solution was measured the viscosity, conductivity and surface tension by viscometer, conductivity meter and tensiometer.

After that, all of solutions were spun by electrospinning process for 10 minutes by giving the fix distance between needle tip and collector (15 cm) and applied voltage is constant (12 kV). Finally, morphology of the fibers was studied by using SEM.

Part 2 Effects of the applied voltage

From Part 1, the scanning electron micrographs of CA electrospun mats at every concentrations were compared. It was found that, at 16% w/v concentration of cellulose acetate solutions can be produced the smallest diameter without beads.

Choosing 16% w/v CA in 2:1 (acetone: DMAc), the best condition to produce electrospun mats to study the applied voltage.

First, cellulose acetate solution in (2:1) acetone:DMAc solvent at 16% w/v was prepared. Then, the viscosity, conductivity and surface tension were measured. After that, 8, 12, 16, 20 and 24 kV of voltage were applied to solutions. And then, all of solutions were spun by electrospinning process for 10 minutes by giving the same distance between needle tip and collector (15 cm) with Part 1. Finally, morphology of the fibers were studied by using SEM.

Part 3 Effects of solvent system

First, the different ratios of (acetone:DMAc) solvent are 1:1 and 3:1 were prepared. After that, 16% w/v concentration of cellulose acetate solution was prepared. Then, the viscosity, conductivity and surface tension were measured. And all of solutions were spun by electrospinning process for 10 minutes by giving the applied voltage is constant (12 kV). Finally, morphology of the fibers were studied by using SEM.

Part 4 Study of swelling

First, electrospun mats from part 1 to 3 were immerged into distilled water for 24 hours. And then, the swelling of cellulose acetate electrospun mats were observed at room temperature. Finally, the degree of swelling and percent of weight loss were calculated.

3.1 Flow Chart

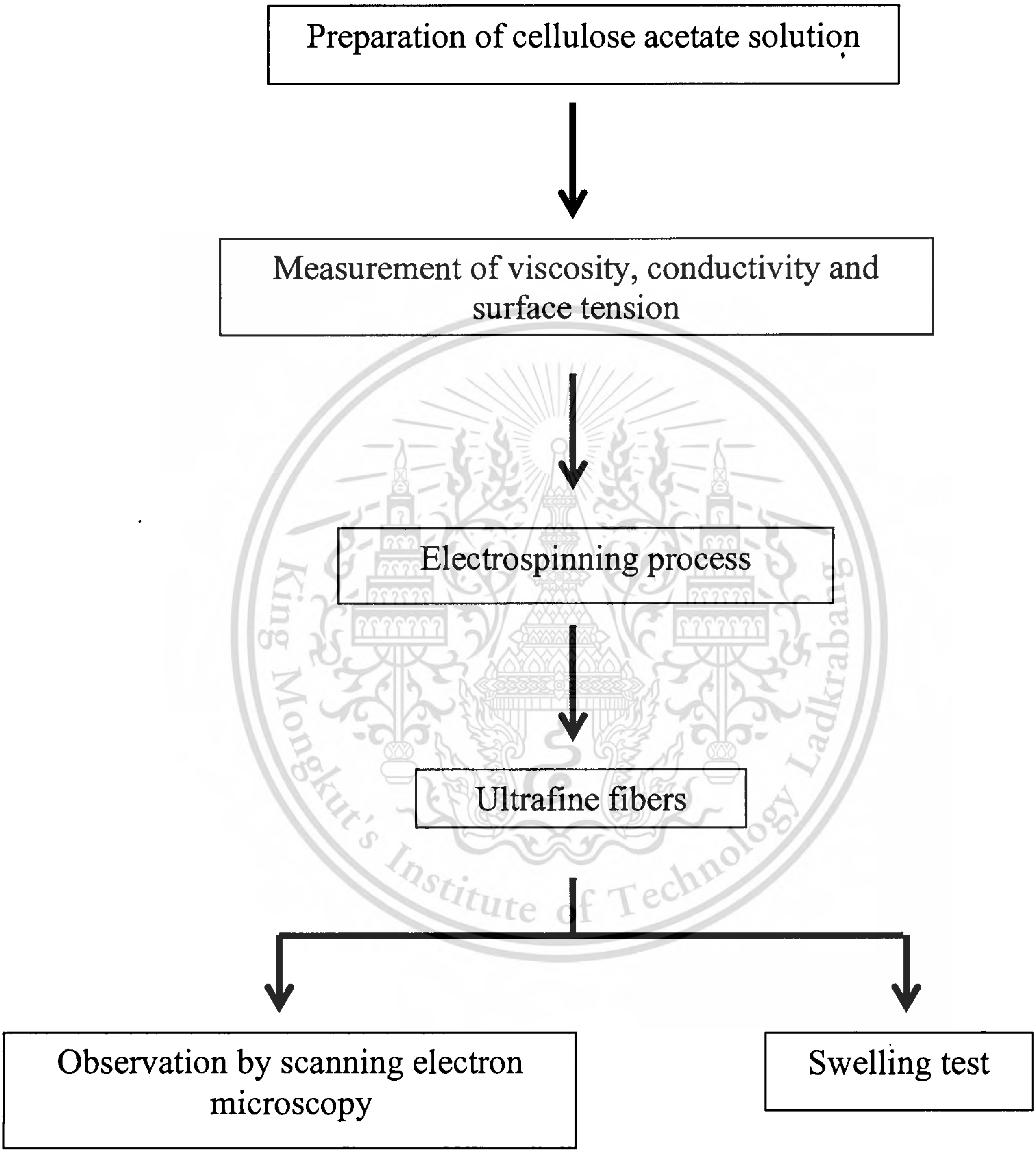


Figure 3.1 Diagram of this research

3.2 Chemicals

1. Cellulose acetate, (acetyl content: 39.8%, molecular weight: 30000 (GPC), was purchased from Aldrich Chemical Co.
2. Dimethylacetamide (DMAc) with analytical grade from Lab Scan Company
3. Acetone with analytical grade from Carloerba Co., ltd
4. Distilled water

3.3 Equipments

1. Beaker
2. Stirrer
3. 5 ml syringe
4. Aluminuim foil
5. Oven
6. Balance (Model TC-254 by Denver Instrument Company)
7. High voltage power supply (MODEL D-ES 30 PN/M629, Gamma High Voltage Research)
8. Nozzle
9. Collector
10. Scanning electron microscope (LEO 1455 VP from LEO electron Microscope Company)
11. Viscometer (Brookfield DV-III programmable viscometer)
12. Tensiometer (scs Sciencific)
13. Conductivity meter (1.04k, 4130 JENWAY)

3.4 Procedure

1. Preparation of cellulose acetate solution
    1.1 Dissolve the cellulose acetate powder with 12, 14, 16, 18 and 20% w/v concentration in acetone/DMAc solvent (Part 1) and 16% w/v concentration in acetone/DMAc solvent (Part 2 and 3)
    1.2 Stir the solution until homogeneously.

1.3 Measure the viscosity, conductivity and surface tension of cellulose acetate solution.

2. Preparation of fibers for the electrospinning technique

    2.1 Set up the electrospinning as Figure 3.2 which consists of

- Syringe
- High voltage power supply
- Collector

Given

- Distance between needle tip and collector is 15 cm

Part 1 Effects of concentration and viscosity of cellulose acetate

Apply 12 kV of voltage to 12, 14, 16, 18 and 20 % w/v concentration of cellulose acetate solution for 10 minutes.

Part 2 Effects of applied voltage

Apply 8, 12, 16, 20 and 24 kV of voltage to 16% w/v concentration of cellulose acetate solution for 10 minutes.

- Dry the electrospun mat in the oven for 1 night and observe the morphology by scanning electron microscopy (SEM).

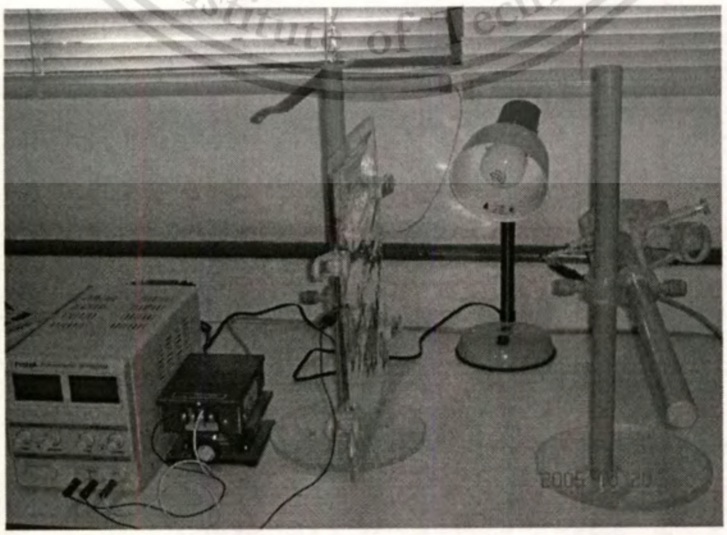


Figure 3.2 Electrospinning process set-up

Part 3 Effects of solvent system

Using different solvent ratios (acetone:DMAc) by varying amount of ratios are 1:1, 2:1 and 3:1 and apply 12 kV of voltage to 16% w/v concentration of cellulose acetate solution for 10 minutes.

Part 4 Study of swelling

1. Weigh the electrospun mats
2. Immerge the electrospun mats into distilled water for 24 hours at room temperature
3. Calculate the degree of swelling

$$\mathbf{S\ (\%) = [(W_t - W_0) / W_0] \times 100}$$

W_t = Final weight of electrospun mat

W_0 = Initial weight of electrospun mat

4. Dry the electrospun mat in the oven at 50°C for 1 day
5. Weigh the dried eletrospun mat again
6. Calculate the percent of weight loss

$$\mathbf{Weight\ loss(\%) = [(W_0 - W_t) / W_0] \times 100}$$

W_0= Initial weight of dry electrospun mat

W_t= Final weight of dry electrospun mat

3. Preparation of electrospun mat for observing morphology by scanning electron microscope (SEM)
    - 3.1 Cut the electrospun mat on the aluminium foil into square shape
    - 3.2 Place the electrospun mat on the stub
    - 3.3 Coat the electrospun with gold
    - 3.4 SEM analysis

4. Measurement the diameter of electrospun
    - 4.1 Using Semafore® 4 measures diameter of each fiber
    - 4.2 Calculate the average and standard deviation of electrospun

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Effects of concentration and viscosity of cellulose acetate Solution

In the electrospinning from a polymer solution, various solution properties affect morphology of the obtained fibers. Some of these properties include solution concentration, viscosity, conductivity and surface tension. So properties of each solution should be studied.

It has been shown to some extents that the solution properties (i.e., viscosity, conductivity and surface tension) play an important role in the morphological appearance of the obtained electrospun cellulose acetate ultrafine fibers, cellulose acetate solution in the range of 12 to 20 %w/v were characterized for their viscosities, surface tensions and conductivity values. It was found that the viscosity value of the solution was found to tremendously increase from 260 to 928 cP with increasing cellulose acetate concentration from 14 to 20 %w/v. At 12 %w/v the viscosity cannot be measured because it had too low concentration. The surface tensions and conductivity values were found to quite constant.

Qualitatively, at viscosities lower than 260 cP, the results showed that when the concentrations of the solution less than 12% w/v only droplets were presented. In electrospinning process, the viscosity of solution plays an important role in determining the range of concentrations from which continuous fibers can be obtained.

The formation of the droplets can be described based on the analysis of forces acting on small segment of a charge jet. In this case, six types of forces were considered, i.e., 1) body or gravitational force, 2) electrostatic force which carries the charged jet from the nozzle to the target, 3) coulombic repulsion force which tries to push apart adjacent charged species being present within the jet segment and is responsible for the stretching of the charged jet during its flight to the target,
4) viscoelastic force which tries to prevent the charged jet from being stretched,

5) surface tension which also acts against the stretching of the surface of the charged jet, 6) drag force from the friction between the charged jet and the surrounding air.

The interplay between three most important forces being responsible for the formation of elongated jet was considered, i.e., coulombic force, viscoelastic force and surface tension. At the low viscosity values, the viscoelastic force was comparatively smaller than the coulombic force. This resulted in the over-stretching of charged jet, hence, the break-up of charged jet into many small spherical droplets (so called “beads”) as a result of the viscosity. On the contrary, for solutions of higher viscosity, the viscoelastic force became larger in comparison with the coulombic force (due mainly to the increased number of chain entanglements). The increase in the viscoelastic force was sufficient to prevent a charged jet from breaking up into small droplets and to allow the electrostatic stress to further elongate the jet which finally thin down the diameter of the jet tremendously.

The relationship between the solution viscosity and the solution concentration for cellulose acetate solution could be approximated with an exponential growth equation as shown in Figure 4.1. The selection of the exponential growth equation to describe the data was based solely on the quality of the fitting that the equation provided.

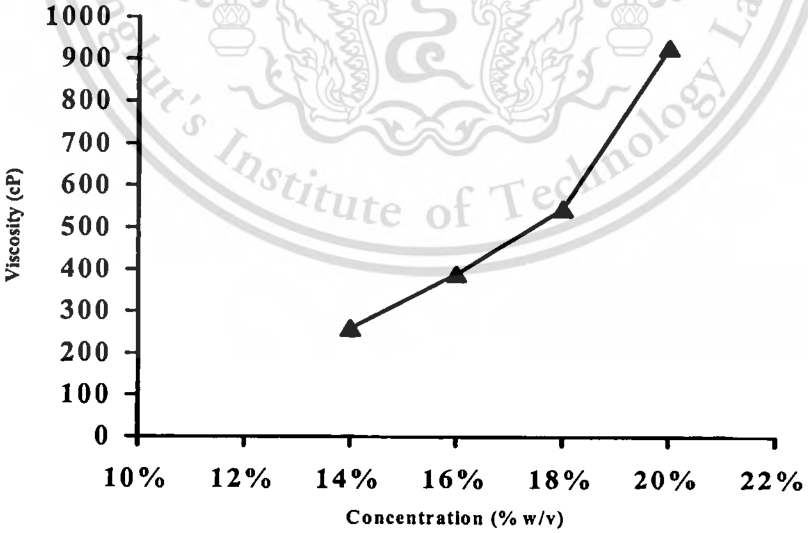


Figure 4.1 Graph between viscosity and concentration of cellulose acetate solution in 2:1 solvent ratio (acetone:DMAc)

(For the concentration at 12% of cellulose acetate cannot be measured)

The surface tension values for solutions of cellulose acetate were found to monotonically increase but slightly with increasing cellulose acetate concentration. Specifically, the values for all of the solution investigated were found in range between 28 to 30 mN/m (see in Figure 4.2).

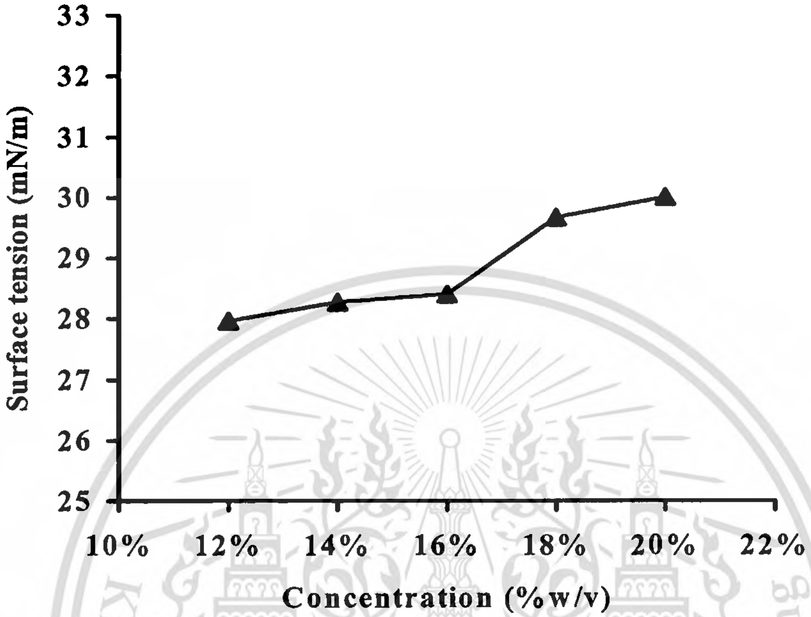


Figure 4.2 Graph between surface tension and concentration of cellulose acetate solution in 2:1 solvent ratio (acetone:DMAc)

The conductivity value for solutions of cellulose acetate were found to decrease initially to reach a minimum at a concentration of 14% w/v and increase with further increase in the concentration solutions. Specifically, the value for all of the solutions studied ranges between 4.81 and 4.60 mS/cm.

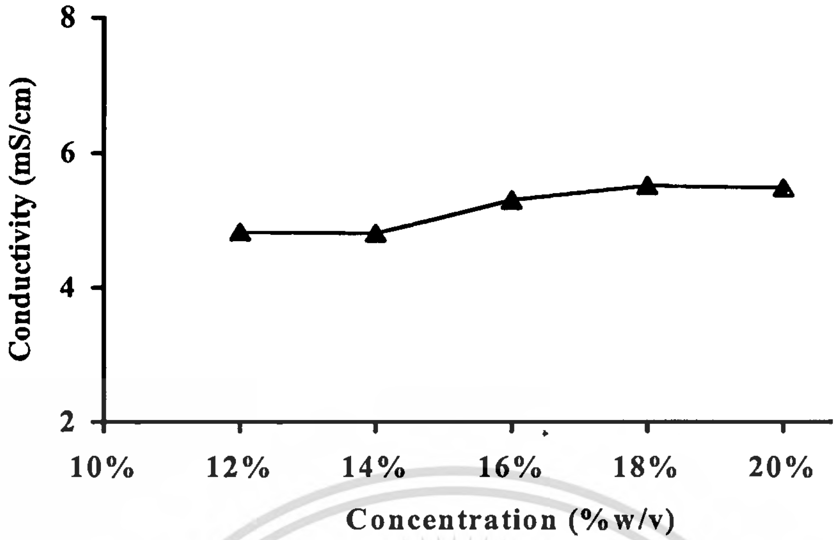


Figure 4.3 Graph between conductivity and concentration of cellulose acetate solution in 2:1 ratio of solvent (acetone:DMAc)

The results obtained illustrate that an increase in the cellulose acetate concentration resulted in a significant increase in the viscosity, a slight increase in the surface tension and conductivity of the solution and the viscosity of the resulting the solution increased at appreciably with increasing chains entanglement of the dissolved polymer. Both of surface tension and the conductivity values were relatively less affected. The significant increase in the viscosity of the solutions with increasing cellulose acetate concentration is obviously due to the increased molecular entanglements.

4.2 Effects of Electrospinning Process

The basic principles of the electrospinning process are concerned with the application of a high electrical potential to a polymer solution across a finite distance between a nozzle and a collector plate. The polarity of the emitting electrode (i.e. the one that is in contact with the polymer solution) can be either positive or negative. When an electrostatic field is applied, charges are built up on surface of a droplet of the polymer solution at the tip of the nozzle. The charges destabilize the hemispherical shape of the droplet into a cone shape at a electrostatic field, the electrostatic force overcomes the surface tension, causing a charge stream of polymer solution (i.e. a charged jet) to be ejected from the tip of the cone. The charge jet travels in a straight line at fixed the distance as its diameter thins down appreciably, before undergoing a bending instability during which the diameter of the jet continues to decrease tremendously. Finally, fibers are collected on a grounded collector plate look like sticky and fluffy cotton as shown in Figure 4.4.

Figure 4.4 Cellulose acetate electrospun fibers on the aluminium foil

4.2.1 Effects of Concentration and Viscosity

It was shown that the solution properties (i.e. viscosity and concentration) were important factors characterizing the morphology of the fibers obtained. Among the three properties, solution viscosity, solution conductivity and solution viscosity, solution viscosity was found to have the greatest effect. The solutions with high enough viscosities (CA concentrations between 14 and 24% in the 2:1 acetone:DMAc solvent mixture) support continuous formation of electrospun mats. These concentrations correspond to viscosities between 260 and 928 cP.

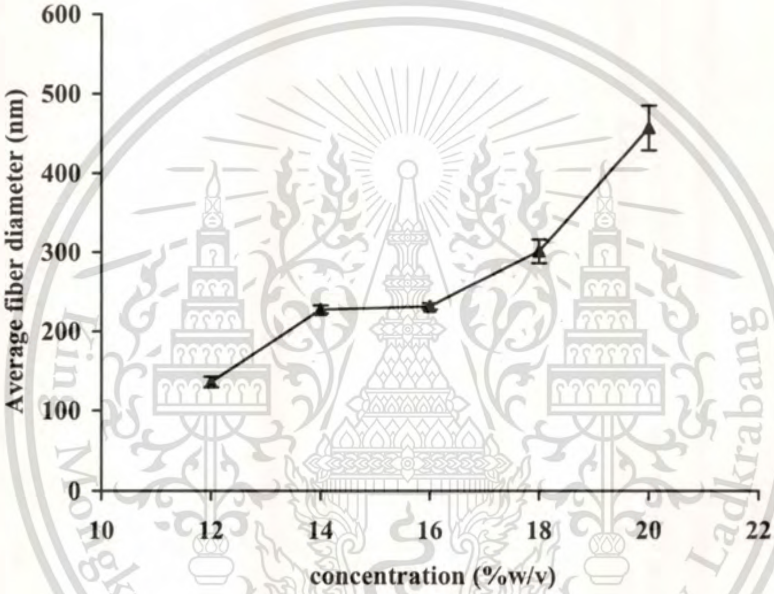


Figure 4.5 Graph plot between average diameter and concentration of cellulose acetate solution in 2:1 solvent ratio (acetone:DMAc) with 12 kV of applied voltage

At a given concentration, fibers obtained from cellulose acetate of higher molecular entanglement appeared to be larger in diameter because of higher concentration of CA solution make stronger of chain entanglement. The result was less elongation of polymer chains as increasing the concentration of polymer solution. It was observed that the average diameter of the fiber from cellulose acetate of different concentrations had a common relationship with the solution viscosities which could be approximated by exponential growth equations are shown in Figures 4.5 and 4.6.

Table 4.1 Viscosity, conductivity, surface tension and diameter of 12, 14,16, 18 and 20% w/v cellulose acetate solution in 2:1 (acetone:DMAc) solvent

Concentration (%w/v)	Viscosity (cP)	Conductivity (ms/cm)	Surface tension (mN/m)	Diameter (nm)	Magnification (SEM)
12	n/a	4.83	28.0	136±7	10000
14	260	4.81	28.3	227±5	10000
16	390	5.30	28.4	231±4	10000
18	545	5.51	29.7	301±6	10000
20	928	5.48	30.0	369±28	10000

Note: At 12 %w/v concentration cannot measure viscosity

Qualitatively, diameters of the obtained fibers were found to increase with increasing viscosity of solutions.

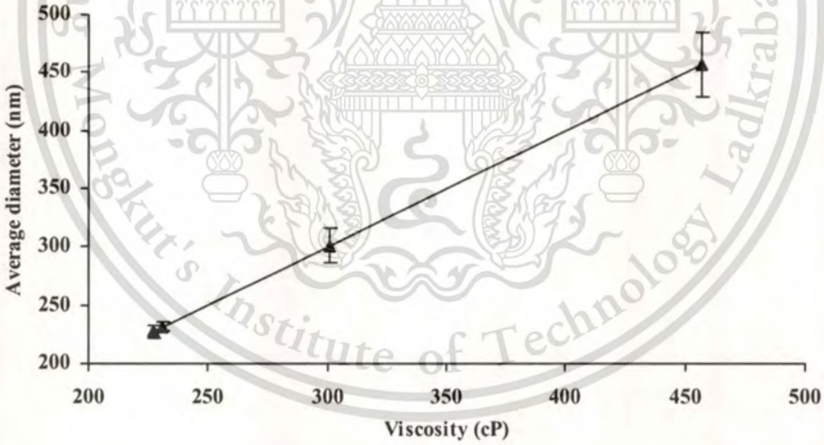
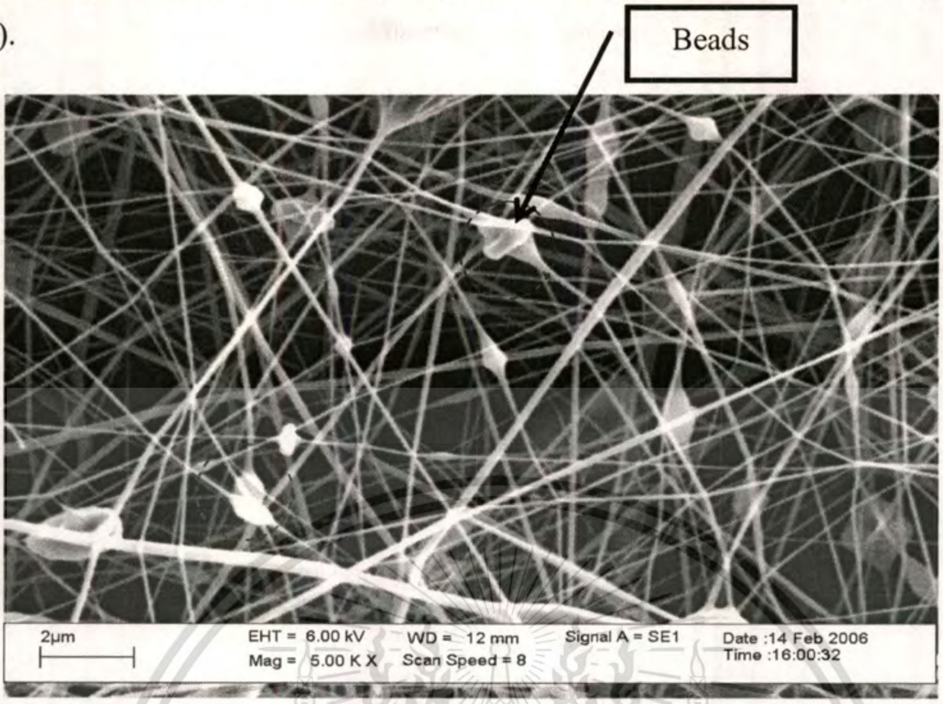
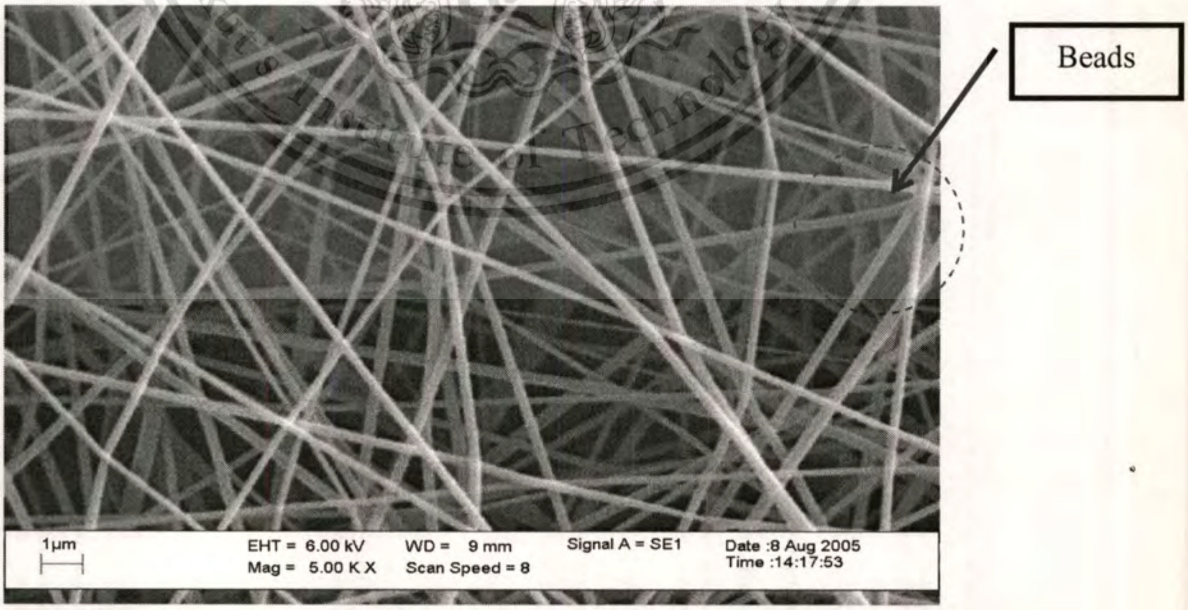


Figure 4.6 Graph between average diameter and viscosity of cellulose acetate solution in 2:1 solvent ratio (acetone:DMAc) with 12 kV of applied voltage

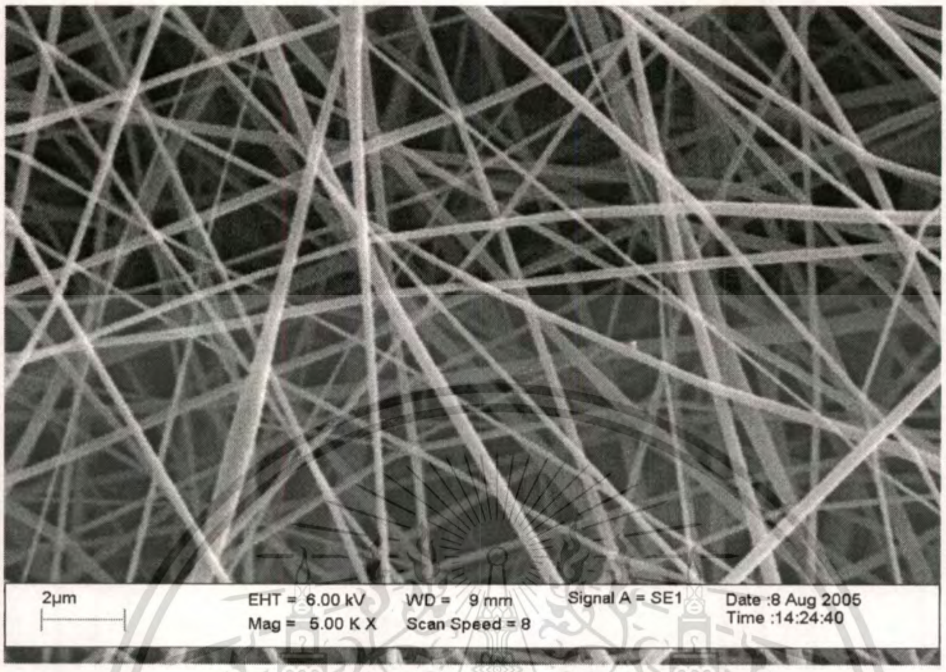
Figure 4.7 shows selected scanning electron micrographs of products obtained from the electrospinning of cellulose acetate in 2:1 ratio of solvent (acetone:DMAc) for five different concentrations. Evidently, beads were more prevalent when the viscosity of the cellulose acetate solution was low (see Figure 4.7a). With an increase in the viscosity of solution to 260 cP, only uniform fibers were obtained (see Figure 4.7 b-e).



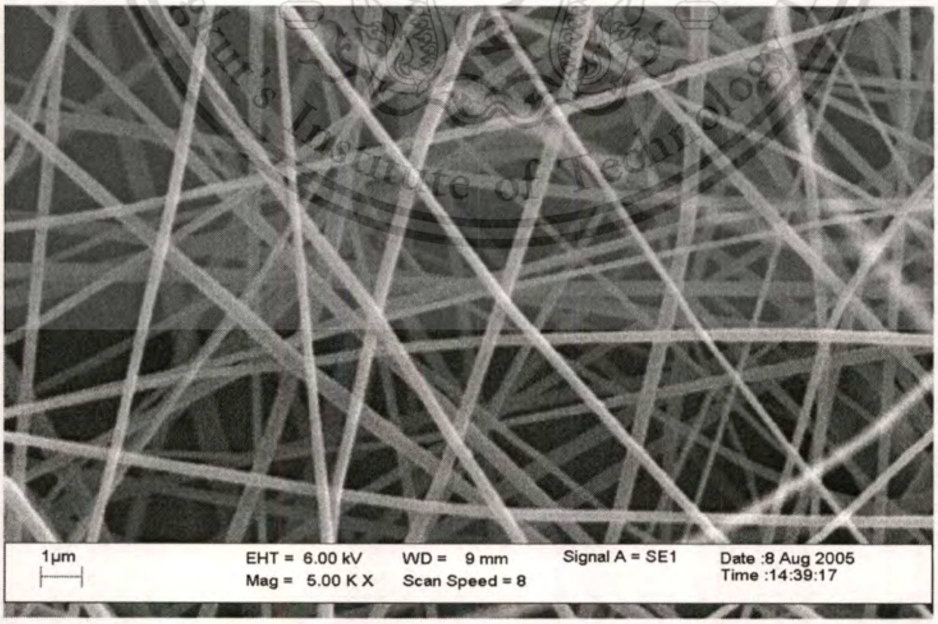
(a)



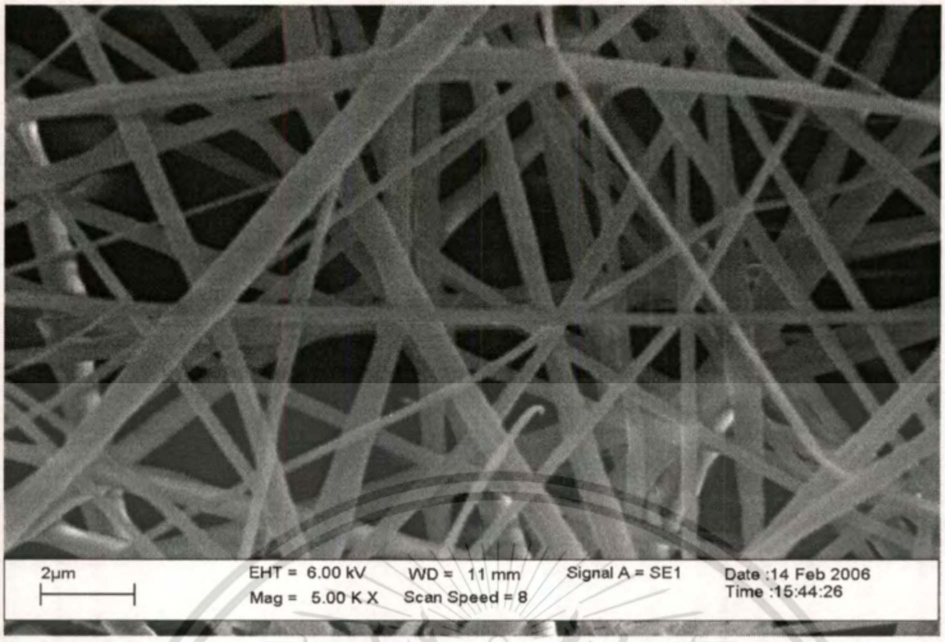
(b)


2µm
EHT = 6.00 kV
Mag = 5.00 K X
WD = 9 mm
Scan Speed = 8
Signal A = SE1
Date :8 Aug 2005
Time :14:24:40

(c)


1µm
EHT = 6.00 kV
Mag = 5.00 K X
WD = 9 mm
Scan Speed = 8
Signal A = SE1
Date :8 Aug 2005
Time :14:39:17

(d)



(e)

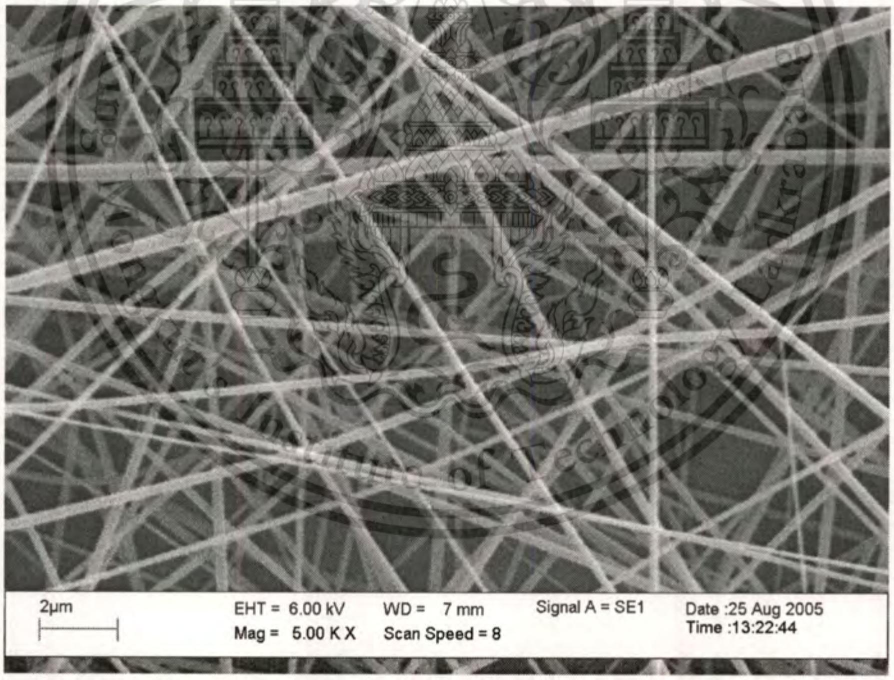
Figure 4.7 Scanning electron micrographs of electrospun materials obtained from solutions of cellulose acetate in acetone:DMAc (2:1) as solvent ratio at concentration: (a) 12% w/v (b) 14% w/v (c) 16% w/v (d) 18% w/v (e) 20% w/v with 12kV of applied voltage (magnification = 5000x)

From the morphological observation, it can be assumed that the concentration at 16% gave an excellent electrospun fibers with uniform ultrafine fibers without beads compared to other concentrations. This concentration was selected to spin the fibers in other studies.

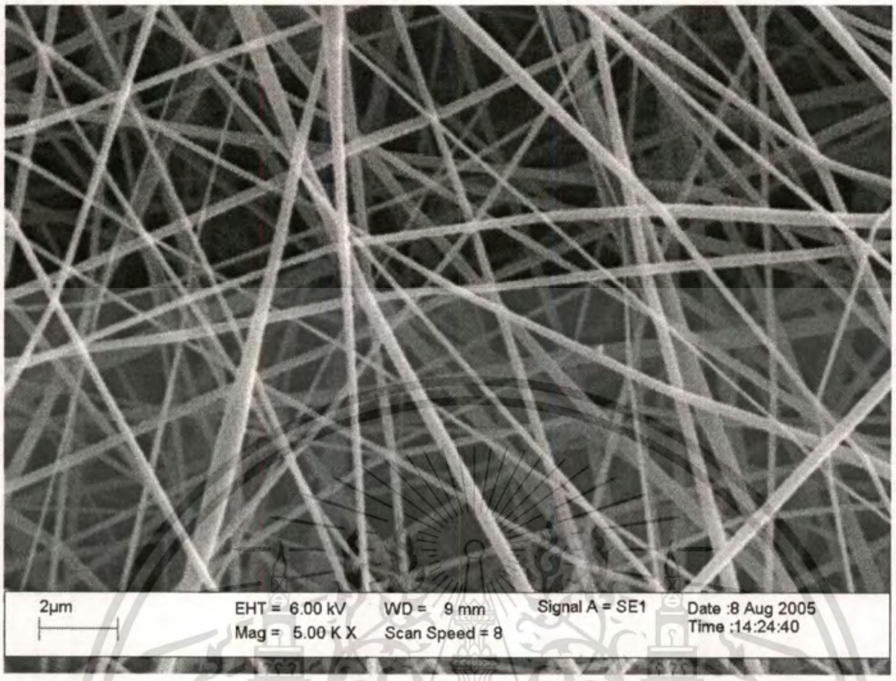
4.2.2 Effects of Applied Voltage

To investigate the effect of applied electrostatic field strength and emitting electrode polarity on morphological appearance of the obtained electrospun CA fibers, 16% w/v solution of CA in 2:1 v/v acetone:DMAc was prepared and electrospun under an application of either positive or negative emitting electrode polarity [52]. The applied electrostatic field strength was 8, 12, 16, 20 and 24 kV, respectively.

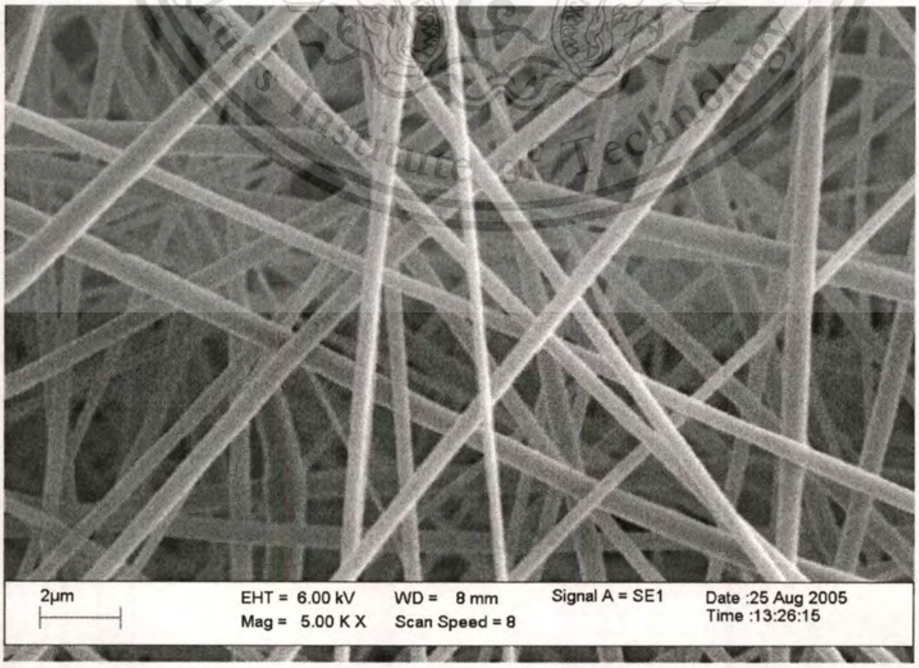
Figure 4.8 shows selected series of SEM images to illustrate the effect of applied electrostatic field strength and emitting electrode polarity on morphological appearance and size of the obtained electrospun fibers from 16% w/v CA solution in 2:1 v/v acetone:DMAc under an applied electrostatic potentials of 8, 12, 16, 20 and 24 kV under both positive and negative polarities.



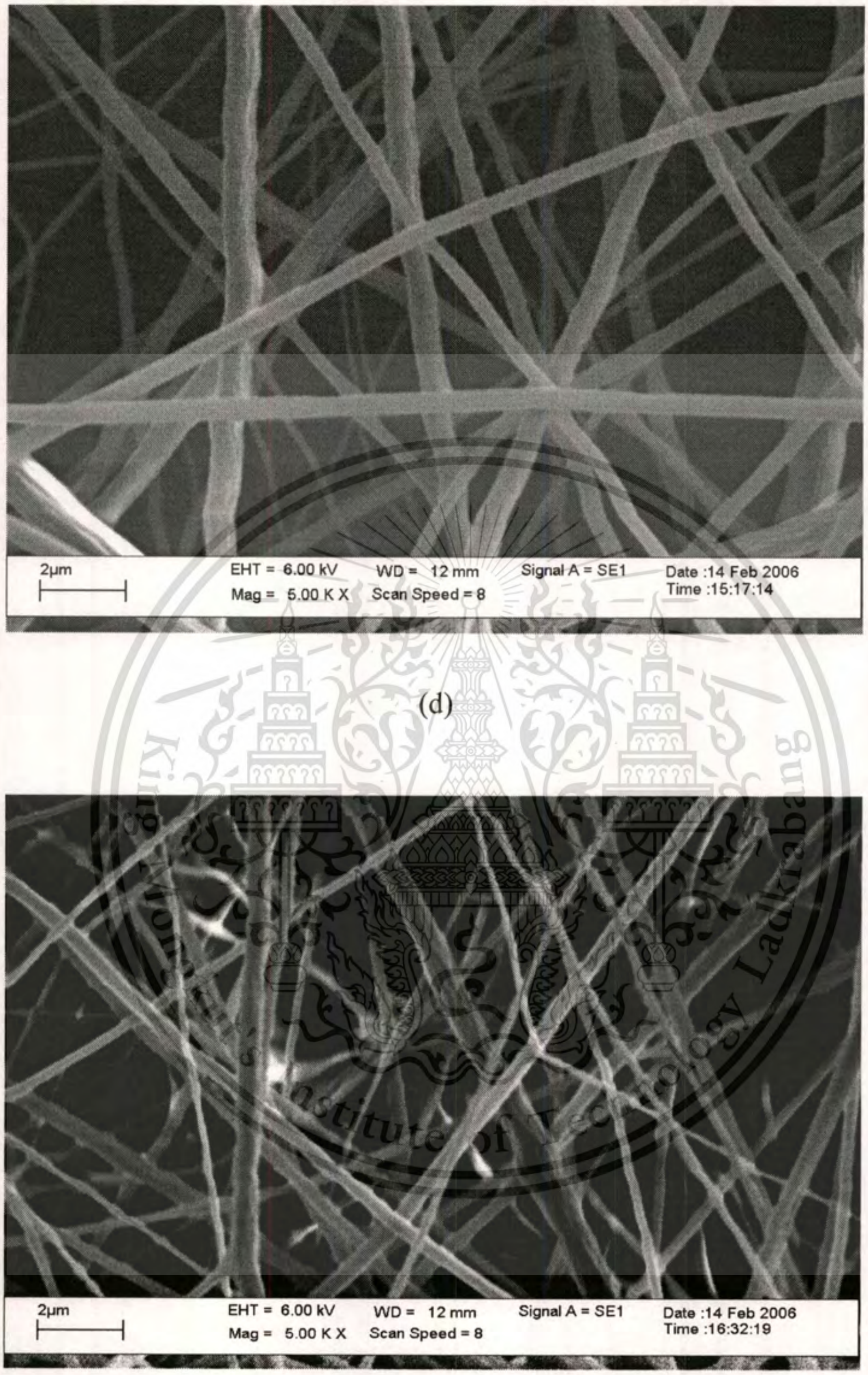
(a)


2µm
EHT = 6.00 kV
Mag = 5.00 K X
WD = 9 mm
Scan Speed = 8
Signal A = SE1
Date :8 Aug 2005
Time :14:24:40

(b)


2µm
EHT = 6.00 kV
Mag = 5.00 K X
WD = 8 mm
Scan Speed = 8
Signal A = SE1
Date :25 Aug 2005
Time :13:26:15

(c)



(d)

(e)

Figure 4.8 Scanning electron micrographs of electrospun materials obtained from 16% w/v of cellulose acetate solution in acetone:DMAc (2:1) solvent ratio at applied voltage of: (a) 8 kV (b) 12 kV (c) 16 kV (d) 20 kV (e) 24kV (magnification = 5000x)

Table 4.2 Diameters of cellulose acetate fibers by using 8, 12, 16, 20 and 24 kV applied voltage at 16% w/v concentration with 2:1 (acetone:DMAc) solvent ratio

Voltage (kV)	Diameter (nm)
8	225 ± 5
12	231 ± 9
16	382 ± 4
20	417± 14
24	466 ± 26

Obviously, smaller diameters of fibers were obtained in the electrospun fibers from smaller applied voltages of 8 and 12 kV (as shown in Figures 4.8a and 4.8b), with the larger diameters of fibers being found to increase with increasing applied voltage, with the average diameter of these fibers being found to increase from 225 nm at 8 kV to 466 nm at 24 kV as shown in Figure 4.8.

Increasing electrostatic field strength caused the electrostatic force acting on a jet segment to increase. The increased electrostatic force not only caused the jet segment to move faster to the collective screen, but also decreased the possibility for the bending instability to occur, which resulted in the decrease in the deposition area of the as-spun webs with increasing applied electrostatic field strength. As the result, faster jet segment to the collective screen caused less time for molecular elongation.

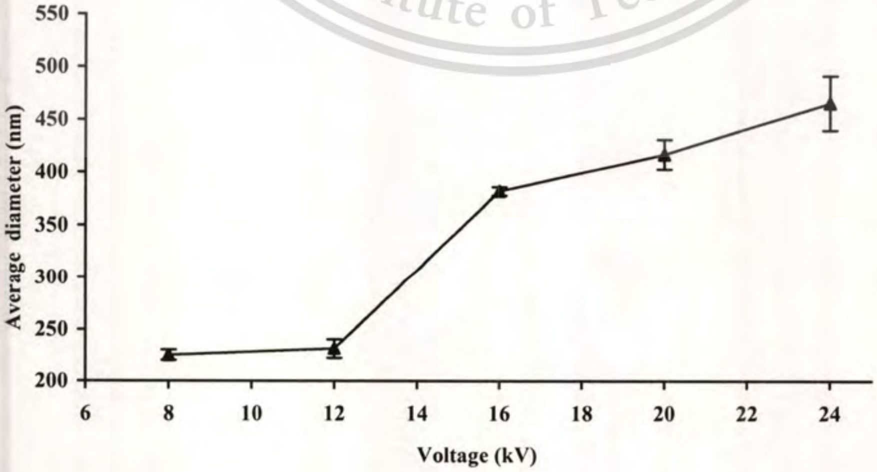


Figure 4.9 Graph between average diameter and applied voltage of 16 %w/v cellulose acetate solution concentration in 2:1 solvent ratio (acetone:DMAc)

4.2.3 Effects of Solvent System

For electrospinning process, low surface tension of the polymer solution are also desired. When applied the same electrostatic force with different surface tension of polymer solution, the fiber of the lower surface tension polymer solution can be easily and continuously jet out.

In order to investigate the effects of solvent system on morphological appearance of the cellulose acetate fibers for select the desirable electrospinning process, solution of cellulose acetate were prepared by dissolving the cellulose acetate powder in mixed solvents of acetone: dimethylacetamide (DMAc) in various compositional ratios of 1:1, 2:1 and 3:1 (v/v) prior to electrospinning process with the reason that acetone were varied because it was the lowest surface tension solvent that CA can be dissolved.

Some physical properties (i.e., viscosity, surface tension, and conductivity) of the prepared solution were measured and the results are summarized in Table 4.3.

Table 4.3 Viscosity, conductivity and surface tension with different solvent ratios of 1:1, 2:1 and 3:1 at 16% w/v concentration

Ratio of solvent (Acetone:DMAc)	Viscosity (cP)	Conductivity (ms/cm)	Surface tension (mN/m)
1:1	399	5.8	30.6
2:1	390	5.3	28.4
3:1	399	5.7	32.2

As shown in Figures 4.10 to 4.12, the viscosity, the conductivity and the surface tension of the solutions were found to quite constant and had less effect on fiber sizes.

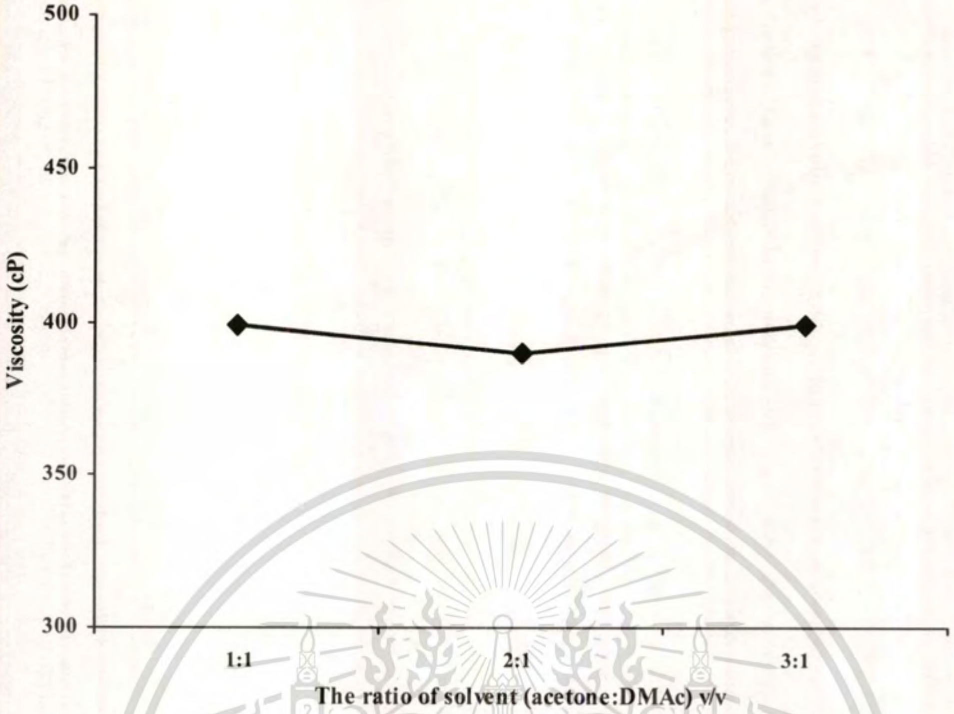


Figure 4.10 Graph between viscosity and solvent ratio of 16% w/v cellulose acetate solution concentration

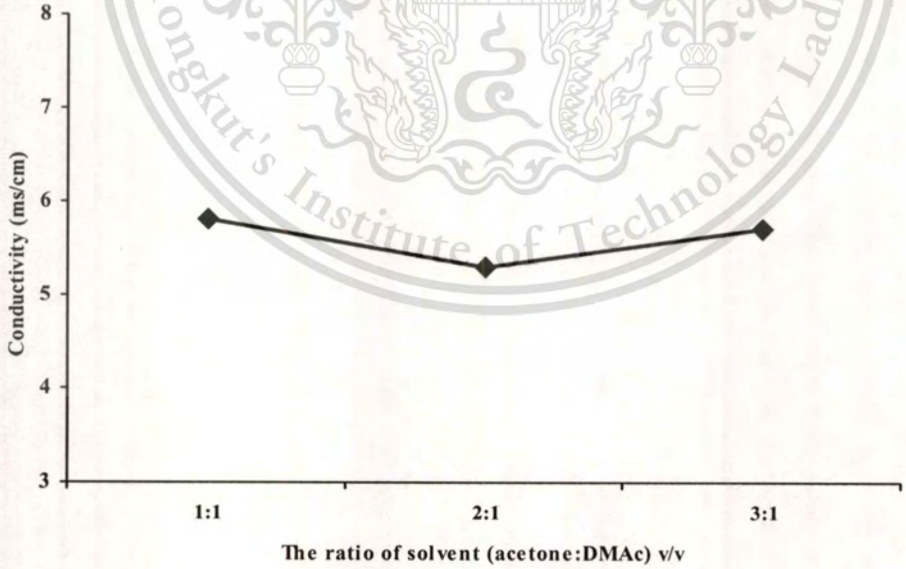


Figure 4.11 Graph between conductivity and solvent ratio of 16% w/v cellulose acetate solution concentration

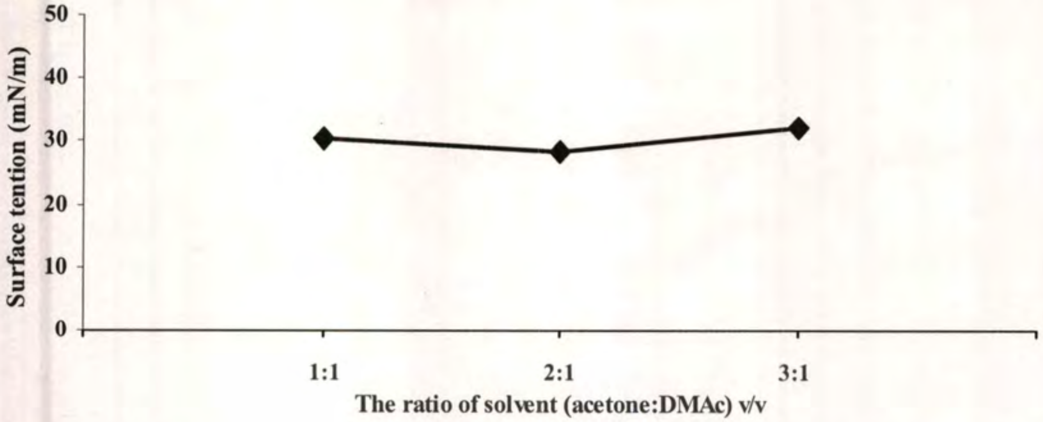
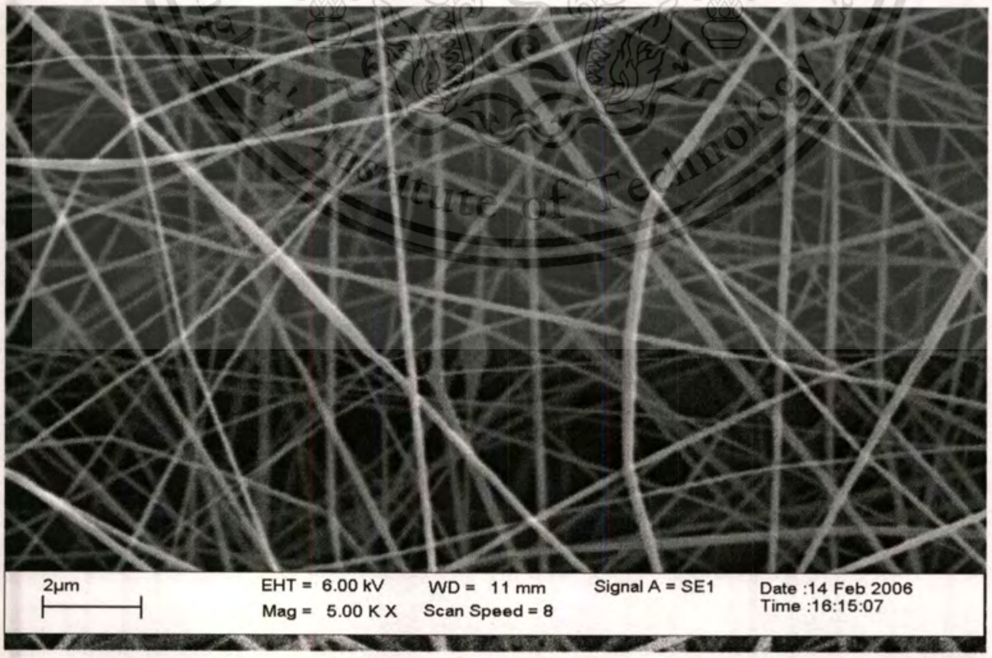
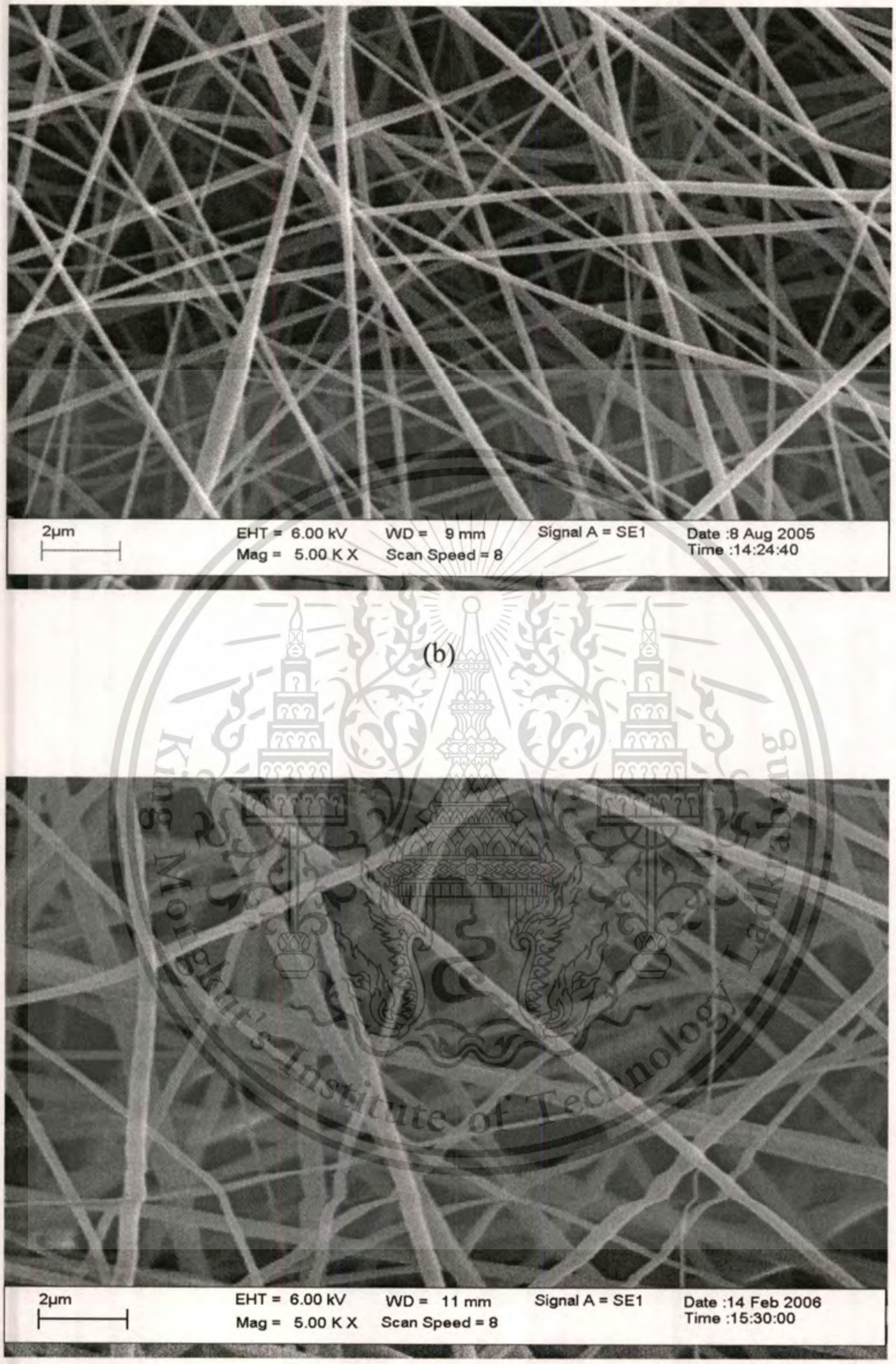


Figure 4.12 Graph between surface tension and solvent ratio of 16% w/v cellulose acetate solution concentration

Figure 4.13 shows selected scanning electron micrographs of cellulose acetate fibers obtained from the 16% w/v solution of cellulose acetate using 12 kV and in a mixed solvent of acetone:DMAc in various compositional ratios of 1:1, 2:1 and 3:1 (v/v). In the mixed solvent systems, smooth and separate fibers were obtained when the ratio of solvent 2:1 (acetone:DMAc) was used.



(a)



(b)

(c)

Figure 4.13 Scanning electron micrographs of electrospun materials obtained from solutions of cellulose acetate at 16% w/v concentration with the solvent ratio of (acetone:DMAc) at (a) 1:1 (b) 2:1 (c) 3:1

Evidently, the diameters of the obtained fibers were found to increase with increasing amount of acetone added as shown in Figure 4.12. Specifically, the average fiber diameter was increased from 160 nm for cellulose acetate solution in acetone:DMAc solvent (1:1) and small amount of beads occurred to 231 nm for cellulose acetate solution in acetone:DMAc solvent (2:1) to 322 nm for cellulose acetate solution in acetone:DMAc solvent (3:1). The increase in the average fiber diameter with increasing acetone content could be too low boiling point of acetone when compared with DMAc. This simply means that, at higher acetone contents, the DMAc can not be retard the evaporation rate of the acetone lead to the short time of chain elongation caused the bigger diameter of the electrospun as shown in Figure 4.14.

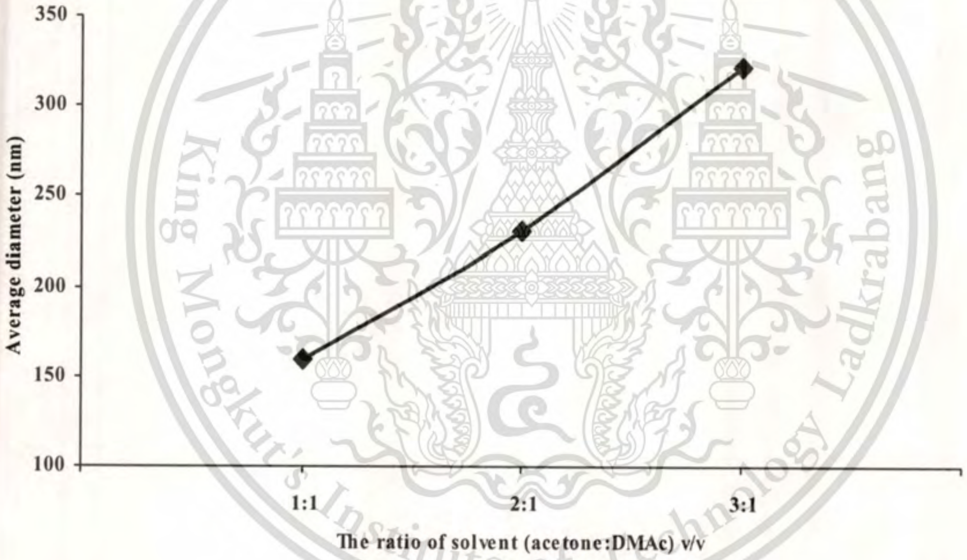


Figure 4.14 Graph between average diameter and solvent ratio of 16% w/v cellulose acetate solution

4.3 Swelling Test

In some applications of polymer ultrafine fibers have to directly contact with water such as drug delivery, filtration or military protection clothing. Thus, swelling test is necessary for this research.

4.3.1 Effects of Concentration

Cellulose acetate at 12, 14, 16, 18 and 20% w/v concentration by controlling 12 kV of applied voltage were prepared for 4 hours. Each sample was immerged into distilled water for 24 hours and dried in the oven for a day. Finally, % swelling and % weight loss were calculated as shown in Table 4.4.

Table 4.4 The percentage of swelling and weight loss of cellulose acetate fibers with 12 kV applied voltage at 12, 14,16, 18 and 20% w/v

Concentration (%w/v)	% swelling	% weight loss
12	621.8	1.72
14	805.3	1.50
16	832.2	1.00
18	846.6	1.14
20	1108.4	1.12

In electrospinng process, by increasing concentration of the solution, diameter of the electrospun fibers tended to increase. The larger the fiber diameter, the more the fiber volume fraction (V_f). This resulted in more free volume inside the fibers where the water can penetrate and absorb. Thus the percent swelling increased which increasing percent of concentration.

By increasing concentration of cellulose acetate solution, % weight loss was quite the same because cellulose acetate is insoluble in water. So % weight loss did not change too much with solution concentration.

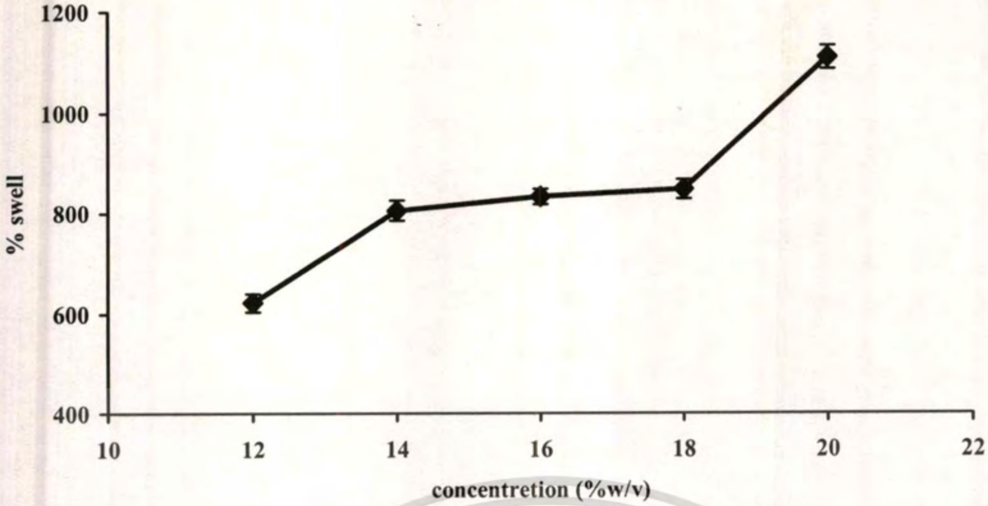


Figure 4.15 Graph between % swell and concentration of cellulose acetate solution in 2:1 solvent ratio (acetone:DMAc) with 12 kV of applied voltage

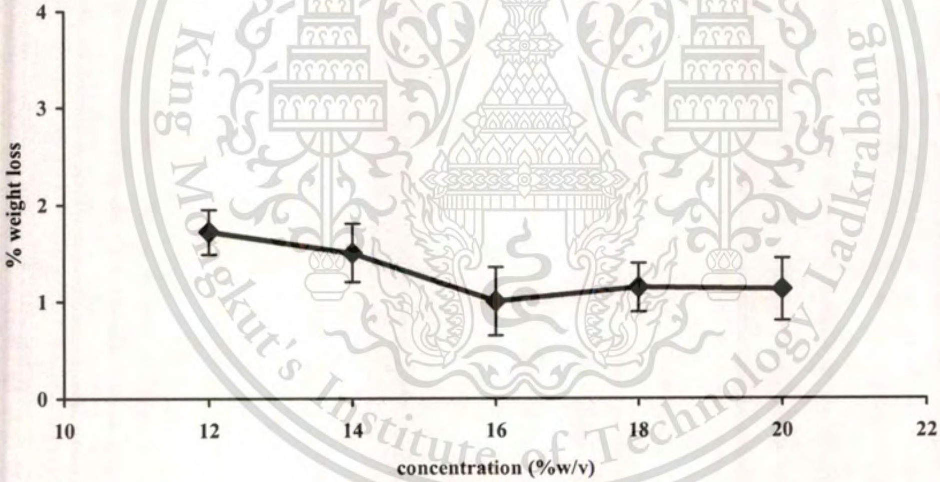


Figure 4.16 Graph between % weight loss and concentration of cellulose acetate solution in 2:1 solvent ratio (acetone:DMAc) with 12 kV of applied voltage

4.3.2 Effects of Applied Voltage

Cellulose acetate at 16 % w/v concentration in 2:1 v/v (acetone:DMAc) was electrospun by using 8, 12, 16, 20 and 24 kV of applied voltage for 4 hours. Each sample was immerged into water for 24 hours and dried in the oven for a day. Finally the % swelling and % weight loss were calculated as shown in Table 4.5.

Table 4.5 The percentage of swelling and weight loss of cellulose acetate fibers with 16% w/v CA solution at 8, 12, 16, 20 and 24 kV applied voltage

Applied Voltage (kV)	% swelling	% weight loss
8	764.3	0.94
12	775.8	1.09
16	777.2	1.55
20	882.5	0.40
24	910.5	1.43

As explained previously, by increasing the applied voltage, diameters of fibers tended to increase. Thus the % swelling was increased.

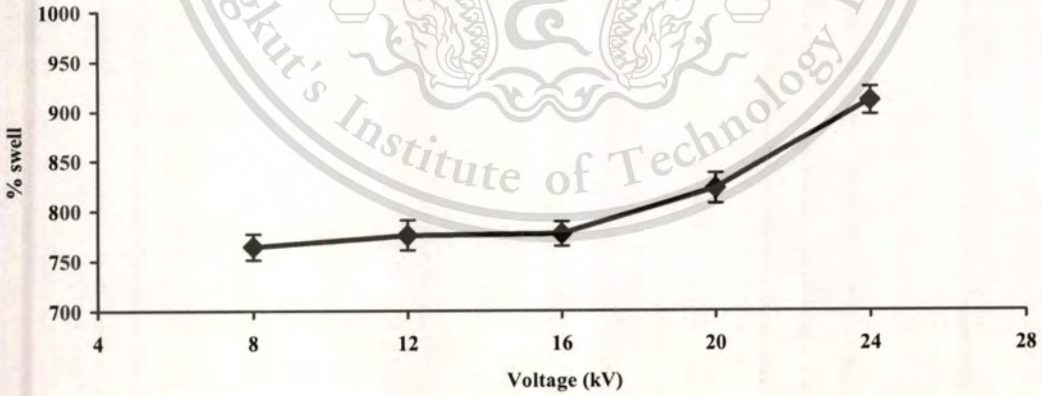


Figure 4.17 Graph between % swell and applied voltage of 16 % w/v cellulose acetate solution concentration in 2:1 solvent ratio (acetone:DMAc)

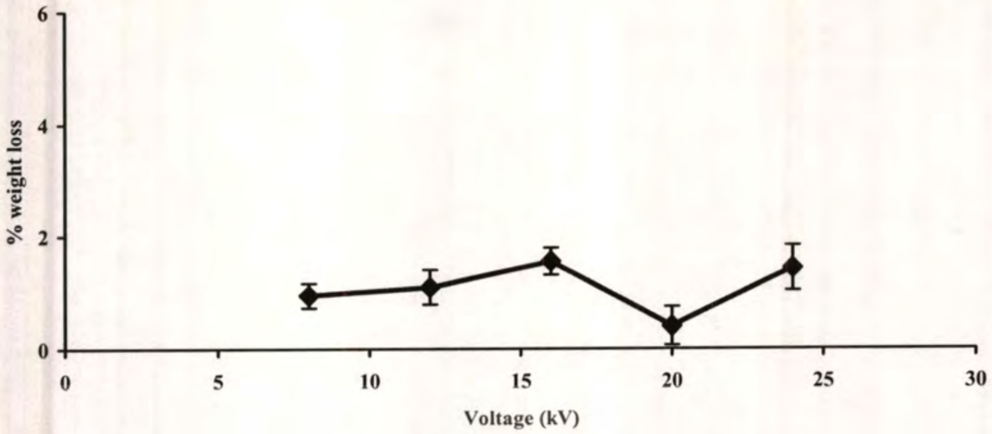


Figure 4.18 Graph between % weight loss and voltage of 16 %w/v cellulose acetate solution concentration in 2:1 solvent ratio (acetone:DMAc)

By increasing applied voltage, the deviations in the % weight loss results were large because the small samples were weighted and used to calculate.

4.3.3 Effects of Solvent Ratio

Cellulose acetate at 16% w/v concentration with the solvent ratios of 1:1, 2:1 and 3:1 (acetone:DMAc) by controlling applied voltage of 12 kV were prepared for 4 hours. Each sample was immerged into water for 24 hours and dried in the oven for a day. Finally the % swelling and % weight loss were calculated as shown in Table 4.6.

Table 4.6 The percentage of swelling and weight loss of cellulose acetate with 16% w/v concentration and 12 kV of applied voltage at 1:1, 2:1 and 3:1 (v/v) solvent ratios

Ratio of solvent (acetone:DMAc)	% swelling	% weight loss
1:1	1173.2	2.06
2:1	832.2	1.00
3:1	945.3	1.17

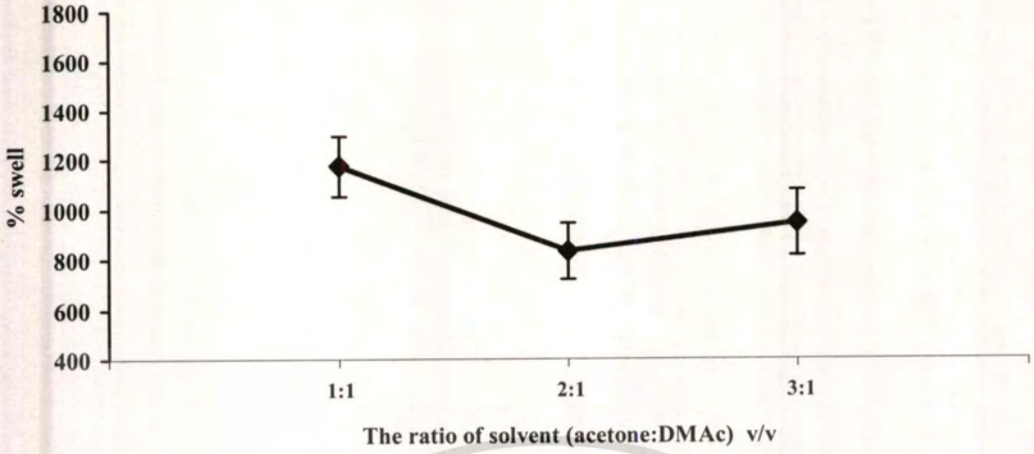


Figure 4.19 Graph between % swell and solvent ratio of 16% w/v cellulose acetate solution concentration with 12 kV of applied voltage

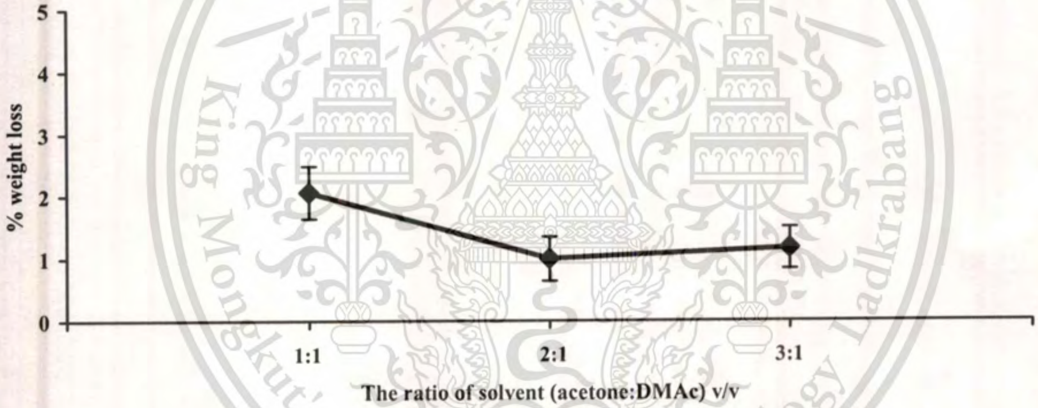


Figure 4.20 Graph between % weight loss and solvent ratio of 16% w/v cellulose acetate solution concentration with 12 kV of applied voltage

By increasing amount of solvent ratio (acetone:DMAc), diameters of fiber size were slightly affected. Swelling depended on size of diameter which is larger diameter having more free volume inside. Thus % swelling and weight loss slightly affected to solvent ratios because cellulose acetate is insoluble in water.

After CA electrospun mats were immerged in water and dried in the oven, the observation of morphologies by using SEM were studied again. It was found that the morphologies of dried electrospun mats were not changed.

These observations confirmed that the swelling test had no effect to CA electrospun mats morphology as shown in Figure 4.21.

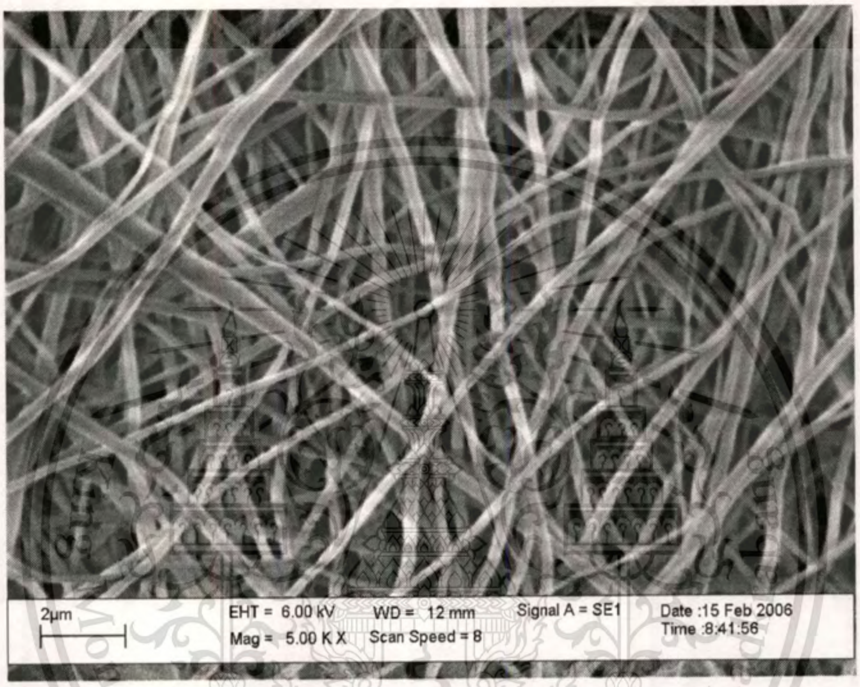


Figure 4.21 Scanning electron micrograph of dried electrospun mat obtained from solution of cellulose acetate at 16% w/v concentration, 12 kV applied voltage and 2:1 solvent ratio (acetone: DMAc)

CHAPTER 5

CONCLUSIONS

5.1 Conclusion

In this research work, electrospinning technique was used to produce ultrafine cellulose acetate fibers. The effect of some solution properties and process parameters on morphological appearance and average diameter of the cellulose acetate fibers were thoroughly investigated using scanning electron microscope (SEM). For the solution parameters, these influencing parameters were investigated: solution concentration, and the ratio of solvent (acetone:DMAc), whereas the influencing of the electric field strength or applied voltage were the process parameters that were investigated in this work.

An increase in the solution concentration caused an increase in the solution viscosity and slightly increase of conductivity and surface tension. For the relationship between the solution viscosity and the solution concentration could be approximated by an exponential growth equation. At low solution viscosities, only droplets were present. At slightly higher viscosities, a combination of droplets and smooth fibers was obtained. At some critical viscosities, droplets disappeared altogether, leaving only beaded and smooth fibers on the collector plate. Further increasing the solution viscosity resulted in the reduced number of beads and increased fiber diameters. At high enough viscosity values were necessary to result in cellulose Acetate fibers having uniform diameters.

At a constant concentration, fibers obtained from higher applied voltage appeared to be larger in diameter, but it was observed that the average diameters of the fiber obtained from cellulose acetate of different applied voltage exhibited a common relationship with the viscosities of the solution which could be approximately by an exponential growth equation.

Cellulose acetate solutions were prepared in mixed system between acetone and DMAc (Dimethylacetamide) by varied the solvent ratios to investigate the influencing of solvents. For 2:1 the ratio of acetone:DMAc solvent the solution of 16% concentration, gave the uniform electrospun fibers with the average diameters being about 231 nm, similar with the solution of cellulose acetate in 1:1 and 3:1 ratio become ultrafine fibers but for 1:1 and 3:1 not uniform and more beads occurred. So, the 2:1 acetone:DMAc mixture is the most versatile mixture because it allows CA in the 12-20% concentration range to be continuously electrospun into fibrous membranes. Thus, Fiber sizes generally decrease with decreasing CA concentrations.

For swelling test, by increasing of the solution, diameter of the electrospun fibers tented to increase. The larger the fiber diameter, the more the fiber volume fraction (V_f). This resulted in more free volume inside the fibers where the water can penetrate and absorb. Thus the percent swelling was increased when % concentration was increased.

Regardless of the percentage of weight loss, because of the initial weight and the final weight (dried fiber) of cellulose acetate electrospun quite the same. So the percentage of weight loss not effect for the cellulose acetate electrospun because of cellulose acetate not dissolve in water.

The fundamental aspects of the influencing solution and process parameters in other polymer-solvent systems are still interested. The collection of a number of data in different systems may be helpful to predict the morphological appearance and also the fiber diameter size. However, the application of these electrospun fibers products in several proposed, for example; in biomedical, in filtration device are interested as well.

5.2 Suggestion for Future Work

1. Cellulose acetate ultrafine fibers can be used for biomedical application such as drug delivery, non-woven doped drug.

2 Other solvents can be used to dissolve cellulose acetate such as dimethylformamide, chloroform, methanol and etc.

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Appendix A

The properties of cellulose acetate solution

Table A.1 The viscosity of cellulose acetate solution

The concentration of cellulose acetate solution (% w/v)	Viscosity of cellulose acetate solution (cP)			Average
12	N/A			
14	259	259	261	260
16	389	391	390	390
18	544	546	544	545
20	926	929	930	928

Table A.2 The conductivity of cellulose acetate solution

The concentration of cellulose acetate solution (% w/v)	Conductivity of cellulose acetate solution (ms/cm)			Average
12	4.80	4.83	4.84	4.83
14	4.81	4.85	4.84	4.81
16	5.48	5.10	5.45	5.30
18	5.50	5.49	5.53	5.51
20	5.45	5.52	5.46	5.48

Table A.3 The surface tension of cellulose acetate solution

The concentration of cellulose acetate solution (% w/v)	Surface tension of cellulose acetate solution (mN/m)			Average
12	28.8	28.2	26.9	28.0
14	28.4	28.1	28.3	28.3
16	27.9	28.1	29.2	28.4
18	29.8	29.5	29.7	29.7
20	30.2	29.7	30.1	30.0

Table A.4 The viscosity of 16 % w/v concentration in different ratios of solvent (acetone:DMAc)

The ratio of solvent (acetone:DMAc)	Viscosity of cellulose acetate solution (cP)			Average
1:1	398.4	399.2	399.1	399
2:1	389.8	389.4	390.3	390
3:1	399.5	398.3	398.9	399

Table A.5 The conductivity of 16 % w/v concentration in different ratios of solvent (acetone:DMAc)

The ratio of solvent (acetone:DMAc)	Conductivity of cellulose acetate solution (ms/cm)			Average
1:1	5.8	5.7	5.9	5.8
2:1	5.8	6.0	5.9	5.3
3:1	5.7	5.9	5.6	5.7

Table A.6 The surface tension of 16 %w/v concentration in different ratios of solvent (acetone:DMAc)

The ratio of solvent (Acetone:DMAc)	Surface tension of cellulose acetate solution (mN/m)			Average
1:1	30.5	30.5	30.7	30.6
2:1	28.6	28.2	28.3	28.4
3:1	32.3	32.0	32.4	32.2

Appendix B

Morphology of CA ultrafine fibers

Effects of concentration

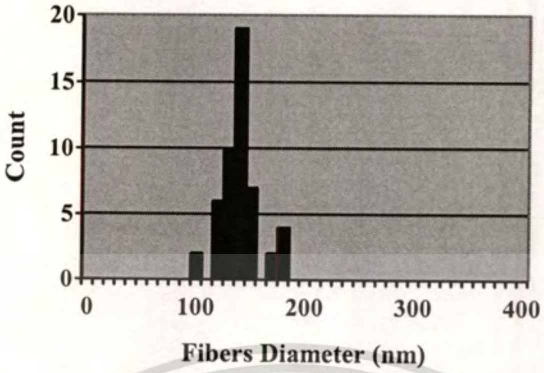

Figure B.1 Diameter of ultrafine fibers at 12% w/v concentration in 2:1 (acetone:DMAc) solvent with 12 kV of applied voltage

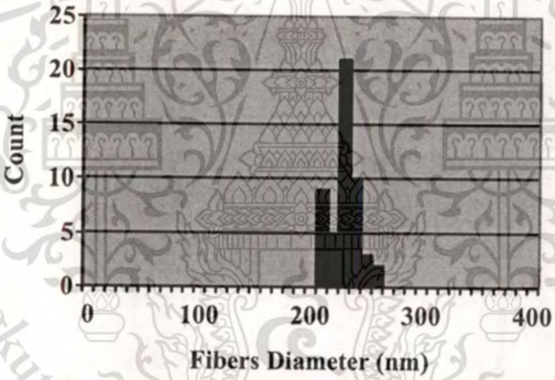

Figure B.2 Diameter of ultrafine fibers at 14% w/v concentration in 2:1 (acetone:DMAc) solvent with 12 kV of applied voltage

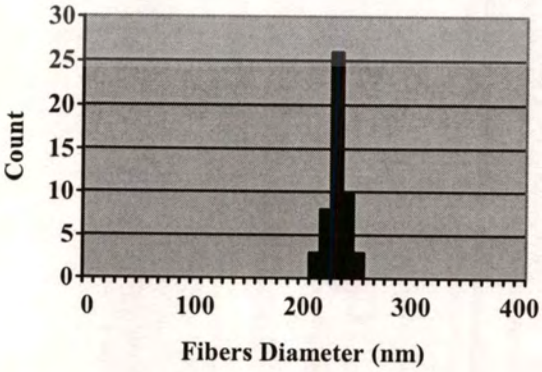

Figure B.3 Diameter of ultrafine fibers at 16% w/v concentration in 2:1 (acetone:DMAc) solvent with 12 kV of applied voltage

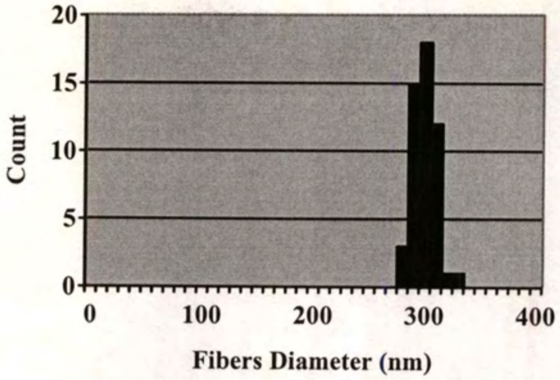

Figure B.4 Diameter of ultrafine fibers at 18% w/v concentration in 2:1 (acetone:DMAc) solvent with 12 kV of applied voltage

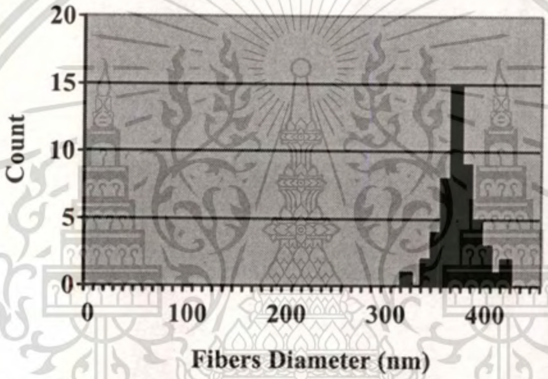

Figure B.5 Diameter of ultrafine fibers at 20% w/v concentration in 2:1 (acetone:DMAc) solvent with 12 kV of applied voltage

Effects of applied voltage

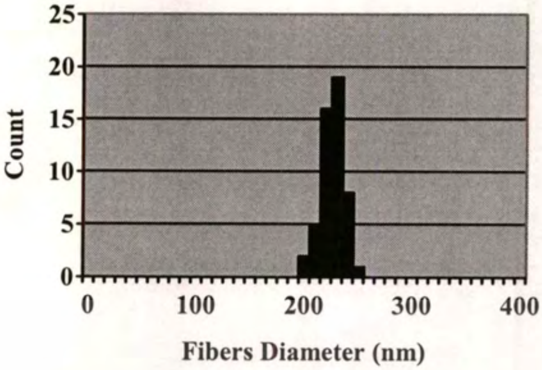

Figure B.6 Diameter of ultrafine fibers at 16% w/v concentration in 2:1 (acetone:DMAc) solvent with 8 kV applied voltage

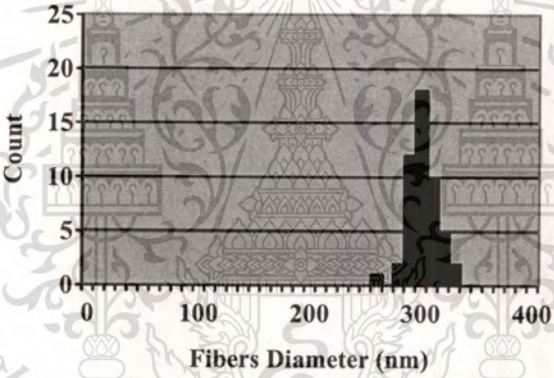

Figure B.7 Diameter of ultrafine fibers at 16% w/v concentration in 2:1 (acetone:DMAc) solvent with 12 kV applied voltage

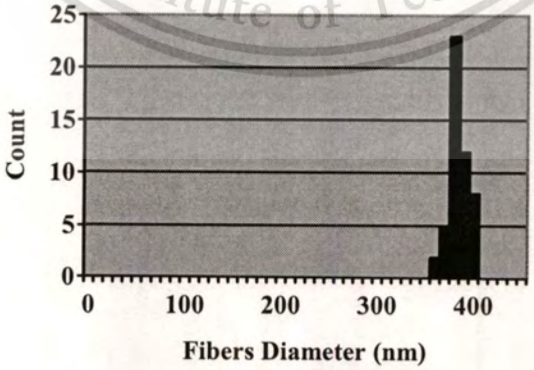

Figure B.8 Diameter of ultrafine fibers at 16% w/v concentration in 2:1 (acetone:DMAc) solvent with 16 kV applied voltage

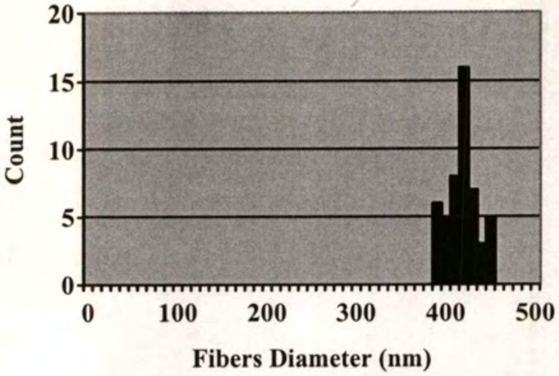

Figure B.9 Diameter of ultrafine fibers at 16% w/v concentration in 2:1 (acetone:DMAc) solvent with 20 kV applied voltage

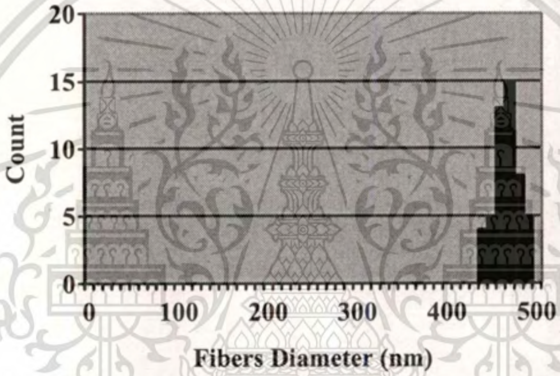

Figure B.10 Diameter of ultrafine fibers at 16% w/v concentration in 2:1 (acetone:DMAc) solvent with 24 kV applied voltage

Effects of solvent system

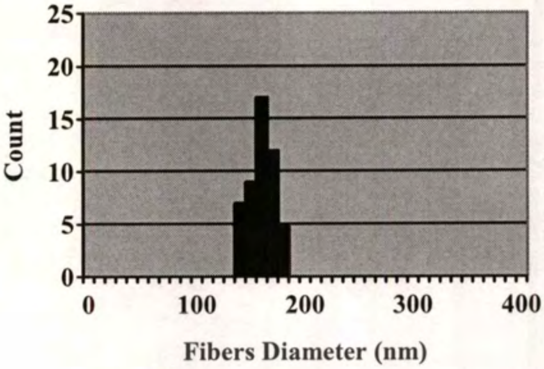

Figure B.11 Diameter of ultrafine fibers at 16% w/v concentration in 1:1 (acetone:DMAc) solvent with 12 kV applied voltage

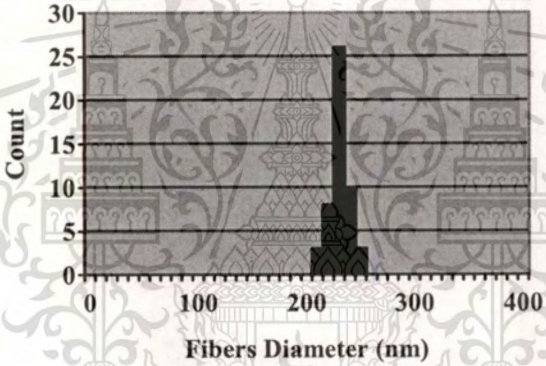

Figure B.12 Diameter of ultrafine fibers at 16% w/v concentration in 2:1 (acetone:DMAc) solvent with 12 kV of applied voltage

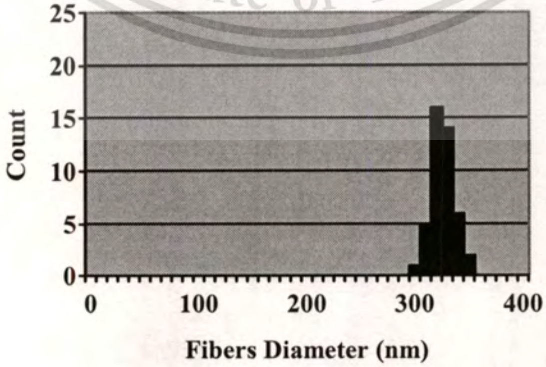

Figure B.13 Diameter of ultrafine fibers at 16% w/v concentration in 3:1 (acetone:DMAc) solvent with 12 kV of applied voltage

Appendix C

Swelling Test

Effects of concentration

Table C.1 Initial weight of cellulose acetate electrospun mats with different concentrations

The concentration of cellulose acetate solution (% w/v)	Initial weight of cellulose acetate electrospun mat (g)			Average
12	0.0241	0.0315	0.0487	0.0348
14	0.0108	0.0168	0.0124	0.0133
16	0.0443	0.0138	0.0321	0.0301
18	0.0268	0.0100	0.0161	0.0176
20	0.0161	0.0197	0.0178	0.0179

Table C.2 Dried weight (final weight) of cellulose acetate electrospun mats with different concentrations

The concentration of cellulose acetate solution (% w/v)	Dried weight of cellulose acetate electrospun mat (g)			Average
12	0.0236	0.0306	0.0485	0.0342
14	0.0102	0.0162	0.0130	0.0131
16	0.0441	0.0133	0.0319	0.0298
18	0.0153	0.0101	0.0267	0.0174
20	0.0159	0.0197	0.0174	0.0177

Table C.3 Swell weight of cellulose acetate electrospun mats with different concentrations

The concentration of cellulose acetate solution (% w/v)	Swell weight of cellulose acetate electrospun mat (g)			Average
12	0.1733	0.2381	0.3423	0.2512
14	0.0511	0.1667	0.1433	0.1204
16	0.4366	0.1021	0.3030	0.2806
18	0.1243	0.1026	0.2730	0.1666
20	0.2145	0.2212	0.2131	0.2163

Table C.4 % swelling of cellulose acetate electrospun mats with different concentrations

The concentration of cellulose acetate solution (% w/v)	% Swelling of cellulose acetate electrospun mat
12	621.8
14	805.3
16	832.2
18	846.6
20	1108.4

Table C.5 % weight loss of cellulose acetate electrospun mats with different concentrations

The concentration of cellulose acetate solution (% w/v)	% Weight loss of cellulose acetate electrospun mat
12	1.72
14	1.50
16	1.00
18	1.14
20	1.12

Effects of solvent system

Table C.6 Initial weight of cellulose acetate electrospun mats with 16 %w/v concentration with different solvent ratios (acetone:DMAc)

The ratio of solvent (acetone:DMAc) (v/v)	Initial weight of cellulose acetate electrospun mat (g)			Average
1:1	0.0203	0.0198	0.0182	0.0194
2:1	0.0184	0.0218	0.0178	0.0193
3:1	0.0299	0.0191	0.0278	0.0256

Table C.7 Dried weight (final weight) of cellulose acetate electrospun mats with 16 % w/v concentration with different solvent ratios (acetone:DMAc)

The ratio of solvent (acetone:DMAc) (v/v)	Dried weight of cellulose acetate electrospun mat (g)			Average
1:1	0.0201	0.0195	0.0175	0.0190
2:1	0.0441	0.0133	0.0319	0.0298
3:1	0.0296	0.0189	0.0275	0.0253

Table C.8 Swell weight of cellulose acetate electrospun mats with 16 %w/v concentration by different solvent ratios (acetone:DMAc)

The ratio of solvent (acetone:DMAc) (v/v)	Swell weight of cellulose acetate electrospun mat (g)			Average
1:1	0.3935	0.1492	0.1987	0.2471
2:1	0.4366	0.1021	0.3030	0.2806
3:1	0.3247	0.1950	0.2830	0.2676

Table C.9 % Swelling of cellulose acetate electrospun mats with 16 %w/v concentration by different solvent ratios (acetone:DMAc)

The ratio of solvent (acetone:DMAc) (v/v)	% Swelling of cellulose acetate electrospun mat
1:1	1173.2
2:1	832.2
3:1	945.3

Table C.10 % weight loss of cellulose acetate electrospun mats with 16 %w/v concentration by different solvent ratios (acetone:DMAc)

The ratio of solvent (acetone:DMAc) (v/v)	% Weight loss of cellulose acetate electrospun mat
1:1	2.06
2:1	1.00
3:1	1.17

Effects of applied voltage

Table C.11 Initial weight of cellulose acetate electrospun mats of 16 % w/v concentration in 2:1 (acetone:DMAc) with different applied voltages

Applied voltage (kV)	Initial weight of cellulose acetate electrospun mat (g)			Average
8	0.0348	0.0295	0.0312	0.0318
12	0.0443	0.0138	0.0247	0.0276
16	0.0184	0.0218	0.0178	0.0193
20	0.0185	0.0327	0.0245	0.0252
24	0.0146	0.0139	0.0134	0.0140

Table C.12 Dried weight of cellulose acetate electrospun mats of 16 % w/v concentration in 2:1 (acetone:DMAc) with different applied voltages

Applied voltage (kV)	Dried weight of cellulose acetate electrospun mat (g)			Average
8	0.0348	0.0296	0.0300	0.0315
12	0.0440	0.0135	0.0244	0.0273
16	0.0182	0.0215	0.0174	0.0190
20	0.0192	0.0325	0.0241	0.0253
24	0.0146	0.0136	0.0133	0.0138

Table C.13 Swell weight of cellulose acetate electrospun mats of 16 % w/v concentration in 2:1 (acetone:DMAc) with different applied voltages

Applied voltage (kV)	Swell weight of cellulose acetate electrospun mat (g)			Average
8	0.3501	0.2930	0.3210	0.3214
12	0.4366	0.1021	0.2250	0.2546
16	0.1491	0.1958	0.1631	0.1693
20	0.1644	0.2717	0.2261	0.2207
24	0.1335	0.1014	0.1282	0.1210

Table C.14 % swelling of cellulose acetate electrospun mats of 16 % w/v concentration in 2:1 (acetone:DMAc) with different applied voltages

Applied voltage	% Swelling of cellulose acetate electrospun mat
8	764.3
12	775.8
16	777.2
20	882.5
24	910.5

Table C.15 % weight loss of cellulose acetate electrospun mats of 16 % w/v concentration in 2:1 (acetone:DMAc) with different applied voltages

Applied voltage	% Weight loss of cellulose acetate electrospun mat
8	0.94
12	1.09
16	1.55
20	0.40
24	1.43