Morphological Investigation of Electrospun Nanofibers Cellulose Acetate-based Membrane

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Abstract: Electrospinning technique had been used to produce ultra-fine fiber with diameter in nano-scale by dissolving polymer precursor in suitable solvents. In this study, electrospinning was used to produce cellulose acetate nanofibers with distance 20 cm, flowrate 0.10-0.15 ml/h and voltage 8 kV. Cellulose acetate was dissolved in acetone:DMSO (2:8 and 3:7). The morphologies of the spun fibers were investigated using a scanning electron microscope (SEM). The functional group of the fibers was analyzed using a Fourier Transform Infrared (FTIR). The result of FTIR showed that C=O functional group at wavenumber 1751 cm^{-1}. The morphology of cellulose acetate dissolved in acetone:DMSO (3:7) wassmoother than acetone:DMSO(2:8).

1 INTRODUCTION

About the population of the world, 11% of themis lack clean water access. The World health Organization (WHO) in 2012 anticipates that the shortage of clean water can involve with 4 million lives by 2050. Although water consists of more than 70% on earth surface, 97% is sea water (Shirazi, Kargari and Shirazi, 2012). In addition, almost 3% of the water stuck in the ground or inside glaciers and ice. Hence, the world only leaves less than 1% of water that can be depleted, the desire of gaining more productive and economical water filtration and cleaning methods are required to raise demand for water.

Membrane-based technology has gained popularity for more than a last century due to its high separation efficiency, inexpensive and easy to operate. The membrane work on principle two phases separation which only let the phase with suitable size of membrane porous. Membrane can be classified into porous and dense depending of their structure (Takht Ravanchi, Kaghazchi and Kargari, 2009). The nature of membrane transport and selectivity depends heavily on the structure of its porous (Ahmed, Lalia and Hashaikeh, 2015).

Nanofibers are part of a nanomaterial which has very unique and interesting properties due its nanoscale diameters and large aspect ratio(Huang *et al.*, 2003). They can be produced by various technique such as synthesis templates, phase separation(Ichimori *et al.*, 2013), self-assembly, steam-explosion(Gea *et al.*, 2018), and electrospinning(Arkoun *et al.*, 2017). Among three of them, electrospinning develops significantly due its simple and reliable technique for converting various polymer into with controllable morphology(Ahmed, Lalia and Hashaikeh, 2015) .Electrospinning is a technique used to produce a continuous of nanofibers in non-woven form. The process spinning fibers diameters ranging from 80 to several hundred nanometers. The report on electrospinning continuous to increase due to its efficient equipment and usage, simple, fast and economical (Daels *et al.*, 2011).

One of nanofiber applications is water filtration. For flat-shaped nanofiber, it can be applied as a water filtration membrane or microfiltration. Due to the higher porosity and interconnected open pore

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structure provide higher permeability properties for filtration of water than conventional materials (Bjorge *et al.*, 2009). The purpose of this study is to produce electrospun nanofiber membranes of cellulose acetate and investigate its morphology.

Cellulose acetate is an easily dissolved polymer in acetone, but the acetone is highly volatile with a boiling point of about 56ºC. In electrospinning the use of volatile solvent causes the fibers to evaporate before reaching the collector, it would block the tip of the needle and stop the spinning process(Son *et al.*, 2004). Thus, acetone needs to be lowered in its volatility by adding other solvents to form binary solvent including acetone-water (Son *et al.*, 2004; Quirós *et al.*, 2016), acetone-ethanol (Baptista *et al.*, 2011), and acetone-DMAc (Tungprapa *et al.*, 2007). The use of water, ethanol, acetic acid, and DMAc mixed with acetone has many disadvantages such as the ratio of water should be smaller, the volatility of ethanol approaching acetone, and the corrosive DMAc and acetic acid harm the electrospinning device. In this study, we were interested in using acetone and DMSO as binary solvent to dissolve cellulose acetate. Then cellulose acetate solution was fabricated into nanofiber by using electrospinning.

2 MATERIALS AND METHODS

2.1 Materials and Devices

Table 1 : Summary of chemical materials used in this study

Properties	Cellulose acetate	Acetone	DMSO
Form Density (g/ml) at 25ºC	Solid 1.3	Liquid 0.791	Liquid 1.1
Boiling Temperature ºC	no data	56	189
Molecular weight (g/mol)	Mn 30000 by GPC	58.08	78.13

Chemical materials included cellulose acetate (CA), acetone and dimethyl sulfoxide (DMSO) were obtained from Sigma Aldrich.Devices usedinclude terumo 10 cc srynge and electrospinning series device from Nanolab instrument (NLi) Company Malaysia. The summary of chemical is written on Table 1.

2.2 Preparation of Electrospun Nanofibers

Cellulose acetate (CA) solution was prepared with concentration 17.5% and 20% (w/v) by dissolvingCA in acetone:DMSO 2:8 and 3:7 (v:v)under reflux at 40-50 ºC for 2 hours.TheCA solution wasthen transferred into10 cc terumo srynge. The electrospinning was run with flowrate 0.10-0.15 ml/h, distance 20 cm and voltage 8 kV at room temperature.The spun-nanofibers were collected using a flat collector. Electrospinning set up is illustrated in Figure 1.

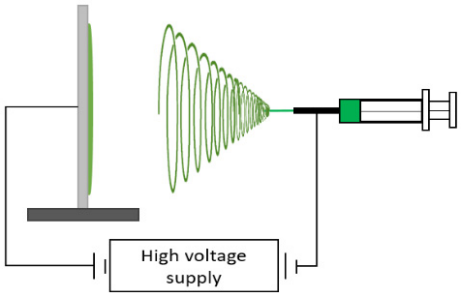

Figure 1: Electrospinning set up.

2.3 Characterization

2.3.1 Scanning Electron Microscope

The morphology of the fibers was analyzed using a scanning electron microscope Jeol 6060. To reduce charging during analysis, the fibers were sputter-coated with a layer of gold alloy.

2.3.2 Fourier Transform Infrared

The functional group of raw cellulose acetate and spun-fibers was analyzed using an FTIR Spectrometer (Nicolet 8700, Thermo Scientific). The instrument was operated in a transmission-mode with a resolution of 2 cm^{-1} and 100 scans.

3 RESULTS AND DISCUSSION

3.1 Morphology Analysis

The morphology of electrospun nanofibers was investigated using SEM. Figure 2 and Figure 3 show the micrographs of CA nanofibers in acetone:DMSO(2:8 and 3:7). The average diameter of the spun fibres is written in Table 2.

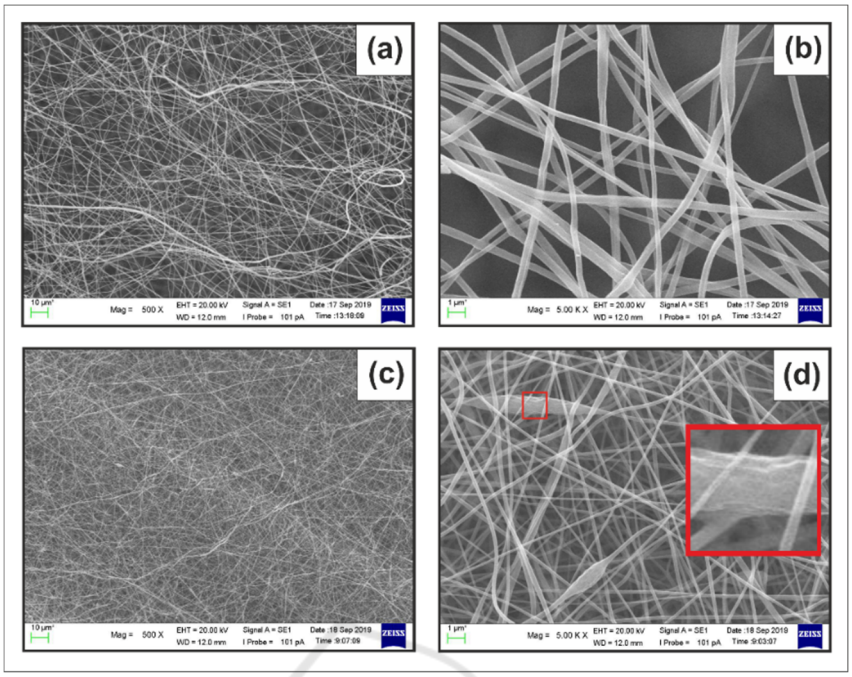

Figure 2: The SEM image of spun fiber: (a)-(b) CA 17.5 % and (c)-(d) CA 20 % (w/v) in acetone:DMSO 2:8 (v:v)

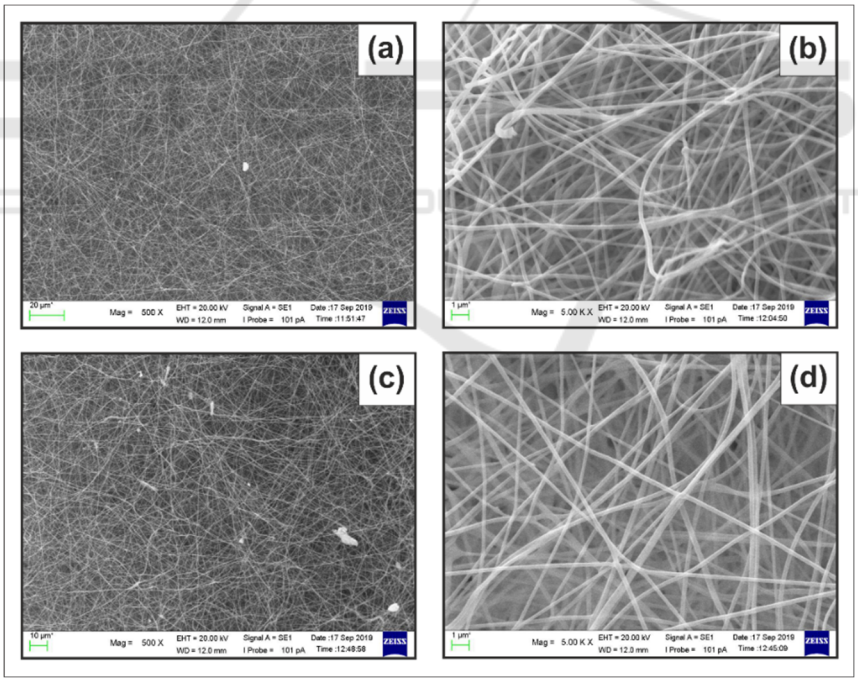

Figure 3: The SEM image of spun fiber: (a)-(b) CA 17.5 % and (c)-(d) CA 20 % (w/v) in acetone:DMSO 3:7 (v:v)

Figure 2 shows that the morphology at CA 20% was smoother than CA 17.5 %. However, it had bead fibers. The bead fiber is probably related to the electrospun jet which get capillary breakup by surface tension (Fong, Chun and Reneker, 1999). Additionally, Figure 3 shows that both the morphology at CA 17,5% had smooth and narrow morphology almost the same as the morphology of CA20% cellulose acetate. However, there were some particles left on the surface of the fiber.

The average diameters data as written in **Table 2** explains that the fiber with CA 20 % had larger

Table 2: The average diameter of CA nanofibers.

ratio of acetone : DMSO (v/v)	fibers diameters (nm)	
	17.5 % cellulose acetate	20 % cellulose acetate
2:8	301.7	152.2
3:7	165.8	212.1

diameter than CA 17.5 % in acetone:DMSO (3:7). Higher polymer concentration causes increasing of fiber diameters as reported in reference (Beachley and Wen, 2009).In addition, both of fibers in had larger diameters than the fiber at CA 20 % in acetone:DMSO (2:8).The larger ratio of volatile solution also increases diameter of fiber (Tungprapa *et al.*, 2007). However, the fiber at CA 17.5 % in acetone DMSO (2:8) had unique attention, whereas its diameter was larger among all fibers.

3.2 Fourier Transfer Infra-red Analysis

The FTIR spectra of CA nanofibers and cellulose acetate powder areshown in **Figure 4**, and the wavenumbers of each functional groupare written in **Table 3**.

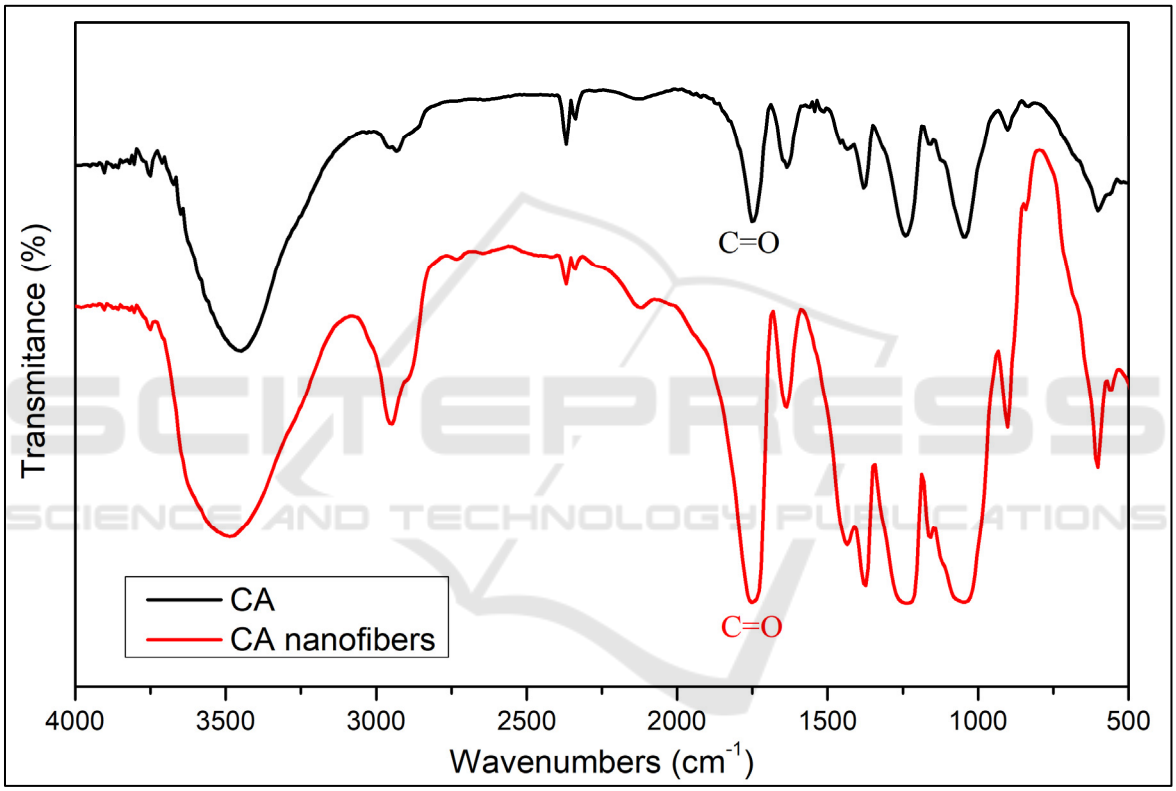

Figure 4 : FTIR spectra of CA and CA nanofibers

The FTIR spectra of CA nanofibers showed broad peak of O-H stretching at 3487 $cm^{-1}$and medium peak of CH; CH_2 or CH_3 stretching at 2947 cm^{-1}(John, Chen and Kim, 2012). Strong peak which attributes to C=O functional groupat 1435 $cm^{-1}$and C-O group at peak 1049 cm^{-1}(Ibrahim *et al.*, 2015).

Table 3. The FTIR data of CA and CA nanofiber used in this study.

Group	Wavenumbers (cm^{-1})	
	Cellulose acetate	Cellulose acetate nanofiber
O-H	3449	3487
C-H	2932	2947
C=O	1751	1751
C=C	1636	1636
CH_2	1434	1435
C-O	1042	1049

The C=O group became stronger and slightly wider after fabrication of cellulose acetate into nanofiber. It was due to the presence of C=O from acetone which appeared at 1770-1730 cm^{-1}(Hasan, Zaki and Pasupulety, 2003). The peak at 1049 cm^{-1} of nanofiber was wider than cellulose acetate. From a reference reported that the peak S=O of DMSO appears at 1070 to 1030 cm^{-1}(Awadhia and Agrawal, 2007). In this study, DMSO was used in high ratio. Hence the presence of DMSO affected the peak of IR spectrum.

4 CONCLUSION

The fabrication of nanofiber from cellulose acetate dissolved in acetone:DMSO has been done using electrospinning. The morphology of the nanofiber dissolved in acetone:DMSO (3:7) is smoother than acetone:DMSO (2:8). The peak intensity of C=O at 1751 cm^{-1}becomes stronger after fabrication of nanofiber.

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REFERENCES

Ahmed, F. E., Lalia, B. S. and Hashaikeh, R. (2015) 'A review on electrospinning for membrane fabrication: Challenges and applications', *Desalination*. Elsevier B.V., 356, pp. 15–30. doi: 10.1016/j.desal.2014.09.033.

Arkoun, M. *et al.* (2017) 'Antibacterial electrospun chitosan-based nanofibers: A bacterial membrane perforator', *Food Science and Nutrition*, 5(4), pp. 865–874. doi: 10.1002/fsn3.468.

Awadhia, A. and Agrawal, S. L. (2007) 'Structural, thermal and electrical characterizations of PVA:DMSO:NH4SCN gel electrolytes', *Solid State Ionics*, 178(13–14), pp. 951–958. doi: 10.1016/j.ssi.2007.04.001.

Baptista, A. C. *et al.* (2011) 'Thin and flexible bio-batteries made of electrospun cellulose-based membranes', *Biosensors and Bioelectronics*. Elsevier B.V., 26(5), pp. 2742–2745. doi: 10.1016/j.bios.2010.09.055.

Beachley, V. and Wen, X. (2009) 'Effect of electrospinning parameters on the nanofiber diameter and length', *Materials Science and Engineering C*. Elsevier B.V., 29(3), pp. 663–668. doi: 10.1016/j.msec.2008.10.037.

Bjorge, D. *et al.* (2009) 'Performance assessment of electrospun nanofibers for filter applications', *Desalination*, 249(3), pp. 942–948. doi: 10.1016/j.desal.2009.06.064.

Daels, N. *et al.* (2011) 'Potential of a functionalised nanofibre microfiltration membrane as an antibacterial water filter', *Desalination*. Elsevier B.V., 275(1–3), pp. 285–290. doi: 10.1016/j.desal.2011.03.012.

Fong, H., Chun, I. and Reneker, D. H. (1999) 'Beaded nanofibers formed during electrospinning', *Polymer*, 40(16), pp. 4585–4592. doi: 10.1016/S0032-3861(99)00068-3.

Gea, S. *et al.* (2018) 'The Isolation of Nanofibre Cellulose from Oil Palm Empty Fruit Bunch Via Steam Explosion and Hydrolysis with HCl 10%', *Journal of Physics: Conference Series*, 979(1), pp. 0–8. doi: 10.1088/1742-6596/979/1/012063.

Hasan, M. A., Zaki, M. I. and Pasupulety, L. (2003) 'Oxide-catalyzed conversion of acetic acid into acetone: An FTIR spectroscopic investigation', *Applied Catalysis A: General*, 243(1), pp. 81–92. doi: 10.1016/S0926-860X(02)00539-2.

Huang, Z. M. *et al.* (2003) 'A review on polymer nanofibers by electrospinning and their applications in nanocomposites', *Composites Science and Technology*, 63(15), pp. 2223–2253. doi: 10.1016/S0266-3538(03)00178-7.

Ibrahim, M. M. *et al.* (2015) 'Role of tosyl cellulose acetate as potential carrier for controlled drug release', *Life Science Journal*, 12(10), pp. 127–133. doi: 10.7537/marslsj121015.16.

Ichimori, T. *et al.* (2013) 'Morphological diversity and nanofiber networks of poly(p-oxybenzoyl) generated by phase separation during copolymerization', *Journal of Applied Polymer Science*, 128(2), pp. 1282–1290. doi: 10.1002/app.38554.

John, A., Chen, Y. and Kim, J. (2012) 'Synthesis and characterization of cellulose acetate-calcium carbonate hybrid nanocomposite', *Composites Part B: Engineering*. Elsevier Ltd, 43(2), pp. 522–525. doi: 10.1016/j.compositesb.2011.08.021.

Quirós, J. *et al.* (2016) 'Electrospun cellulose acetate composites containing supported metal nanoparticles for antifungal membranes', *Science of the Total Environment*. Elsevier B.V., 563–564, pp. 912–920. doi: 10.1016/j.scitotenv.2015.10.072.

Shirazi, M. M. A., Kargari, A. and Shirazi, M. J. A. (2012) 'Direct contact membrane distillation for seawater desalination', *Desalination and Water Treatment*, 49(1–3), pp. 368–375. doi: 10.1080/19443994.2012.719466.

Son, W. K. *et al.* (2004) 'Preparation of antimicrobial ultrafine cellulose acetate fibers with silver nanoparticles', *Macromolecular Rapid Communications*, 25(18), pp. 1632–1637. doi: 10.1002/marc.200400323.

Takht Ravanchi, M., Kaghazchi, T. and Kargari, A. (2009) 'Application of membrane separation processes in petrochemical industry: a review', *Desalination*.

Elsevier B.V., 235(1–3), pp. 199–244. doi: 10.1016/j.desal.2007.10.042.

Tungprapa, S. *et al.* (2007) 'Electrospun cellulose acetate fibers: Effect of solvent system on morphology and fiber diameter', *Cellulose*, 14(6), pp. 563–575. doi: 10.1007/s10570-007-9113-4.