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Preliminary study of cellulose acetate nanofibre produced through the tri-solvent technique

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Abstract

This paper presents a preliminary study of the multi-solvent effect on the formation of cellulose acetate nanofibre through the electrospinning method. Due to cellulose acetate nanofibre's potential application in various fields, a different solvent was suggested to produce consistent quality of cellulose acetate nanofibre. The solvent utilised in the study were acetone and water, while ethanol was added to the formulation. An attempt to add ethanol to acetone/water mix-solvent was performed to determine whether the tri-solvent technique could produce the nanofibre. The produced nanofibre was characterised using SEM to study the nanofibre morphology. The tri-solvent approach efficiently produced cellulose acetate nanofibre with relatively low electrostatic field strength (EFS) with non-beaded nanofibre.

Keywords: electrospinning, nanofibre, multi-solvent, electrostatic field strength (EFS)

Full length article *Corresponding Author, e-mail:shahruln@outlook.com

1. Introduction

Nanofibre has been defined as a fibre with a diameter ranging from 1 to 1000 nm and a length to diameter ratio greater than 100 [1]. Researchers have suggested several nanofibre methods, such as chemical vapour deposition (CVD), electrospinning, melt air spinning, conjugate spinning, and meltblown [2]. Electrospinning has emerged as one of the essential processes in producing nanofibre due to a wide range of materials such as synthetic and natural polymers, metals, ceramics, and composite systems [3].In addition, electrospinning is a simple, fast and inexpensive process with high adaptability and versatility and durability in the production of multifunctional nanofibres up to a nanoscale diameter [4]. The electrospun nanofibre has wide applications such as in tissue engineering and drug delivery, oil-water separation, oil fence, flood protection, membrane distillation, water retaining in soil, air and water purification filters, acoustic insulation, protection against cold, snow shovelling, battery separator, flying carpet, superconducting fibres, scaffold for tissue engineering, cosmetics, disposable diaper, and others future applications [5]–[8]. Cellulose acetate (CA) derived from the acetylation process becomes a crucial starting material to produce CA nanofibre [9]. Cellulose polymer has excellent thermal stability, chemical resistance, biodegradability and others [10]. The advantage of CA is its eco-compatibility in terms of biodegradability as a function of the synergy between photodegradation, biodegradation and the physical design of consumer products. Due to its excellent properties, cellulose acetate nanofibre can be used as membranes, biosensors, chemosensors, protective clothes and reinforced nanocomposites [11]. CA nanofibre has comparatively high modulus, flexural and tensile strength. Thorough studies by previous researchers on how to prepare a cellulose solution by dissolving in acetone, acetone, chloroform, N, N-dimethylformamide(DMF), dichloromethane (DCM), methanol (MeOH), formic acid, and pyridine [12]. A mixed-solvent has also been proposed consisting of acetone-dimethylacetamide, chloroform-methanol, and dichloromethane-methanol [13].

Son et al. [14] proposed an acetone-water mix-solvent with an optimised 10-15% water ratio. Acetone is relatively less hazardous than other solvents such as DMF, DCM and methanol. Therefore, in this preliminary study, an acetone-water and acetone-water-ethanol mixed-solvent solution of CA was compared in producing the cellulose acetate nanofibre was conducted. Other parameters that need to be studied to create the desired cellulose acetate nanofibre

are needle distance, collecter speed, electrostatic strength, surface tension, and the solution feed rate [4].

Alcohol is known to dissolve antimicrobial compounds such as pyrrolidone. In this preliminary work, the ethanol was added to prepare for the antimicrobial to be dissolved in the solution for the later study. As a result, alcohol is expected to act as a carrier to homogeneous mix the compound in the produced nanofibre.

2. Materials and methods

2.1. Materials

CA (acetyl content 39.8%, Mw = 30,000), ethanol and acetone were purchased from Aldrich Co. and used as received. Distilled water was used as the co-solvent in the multi-solvent solution. The water mentioned in this paper is referred to as distilled water.

2.2. Methods

The CA concentration was fixed at 5% w/v dissolve in solution, as shown in Table 1 below.

Table 1: Preparation of mixed solvent

	CA weight (gram)	Mix solvent		
		Acetone	Ethanol	Water
S1	5	90	0	10
S2	5	80	10	10
S3	5	70	20	10
S4	5	60	30	10

All solutions were stirred using a magnetic stirrer for at least 5 hours.

Nfiber High Voltage DC power supply was used to generate the potential. The CA solutions were electrospun at a positive voltage of 10 kV, a working distance of 5 cm (the distance between the needle tip and the collecting plate drum), and a solution flow rate of 5 ml/h. All electrospinning procedures were carried out at room temperature. The electrospinning time was set at 10 minutes.

2.3. Viscosity characterization

The prepared solution's viscosity was characterized using the Digital spindle rotational viscometer brand Fungilab as in Figure 1. Received samples and equipment were conditioned and stabilized at room temperature before the physical property determinations (± 30 min to 1h). Samples were tested on their viscosity properties according to the parameters below and data were collected on viscosity, temperature and percentage of torque. Dial speed on the rotation was set from low (1 rpm) to high speed (up to 100 rpm) then from high to low speed. Collected data were analysed for the elongation of viscosity on their shear rate, categorize of the fluid properties and type of solution based on time dependencies.

Fig. 1. Digital spindle rotational viscometer, Fungilab

2.4. Scanning Electron Microscopy

The scanning electron microscopy (SEM) was conducted using LEO model 1530, as in Figure 2. The vacuum was set to $2x10^{-5}$ millibar to study the nanofibre surface morphology.

Fig. 2. LEO 1530 SEM

3. Results and discussion

3.1. Viscosity of the solution

The viscosity of the mixed-solvent solutions is in Table 2 below. No significant variation of viscosity was observed in S1 to S2. The CA has dissolved in the mixed-solvent formulations. However, the S4 mixed solvent showed an opaque solution. Acetone and ethanol were miscible in water.

Table 2: Viscosity of the mixed solvent

	CA weight (gram)	Mix solvent			Viscosity (cps)
		Acetone	Ethanol	Water	
S1	5	90	0	10	115
S2	5	80	10	10	101
S3	5	70	20	10	105
S4	5	60	30	10	109

3.2. Morphology of the nanofibre

SEM micrograph of cellulose nanofibre electrospun from S1 solvent as shown in Figure 3. A higher magnification of 2000x shows no bead was visible and the nanofibre alignment is almost in one direction. Figure 4 shows 200x and 2000x magnification of cellulose nanofibre electrospun from S2 solvent. The 200x SEM micrograph showed a similar pattern compared to the cellulose nanofibre electrospun from the S1 mixture. Clean and smooth nanofibre without bead appeared in the SEM micrograph. Figure 5 shows the CA nanofiber spun from S3 and S4 solutions. Both solutions produce random beads with an average diameter of 3.83 micrometres. The nanofibers appear to be stretched out from the beads.

The optimum water content for electrospinning of CA in a mixed solvent of acetone/water was at 10–15 wt.% [14]. The average diameter of CA electrospun from acetone/water (S1) is 831 nm, which is contradicted as reported by Son et al. [14]. This may be due to the lower solution feed rate and viscosity affecting nanofibre formation, as reported in previous work [15]. Nanofibre electrospun from S2 shows slight increases in diameter; however, the S3 and S4 show a decreased average diameter of the nanofibre. The random stretch of nanofibre from the beads might contribute to the reduced size. Higher ethanol concentration could alter the surface tension, leading to a mixture of electrospray and electrospun of CA polymer.

Interestingly, CA nanofibre was produced with lower EFS and needle distance between the collector drum compared to the literature [14]. However, more studies need to be conducted to verify the preliminary finding. The average diameter recorded from the SEM micrographs is summarised in Table 3 below.

Table 3. CA nanofibre average diameter electrospun from a different solution.

Solution	Average diameter (nm)
S1	831
S2	914
S3	541
S4	396

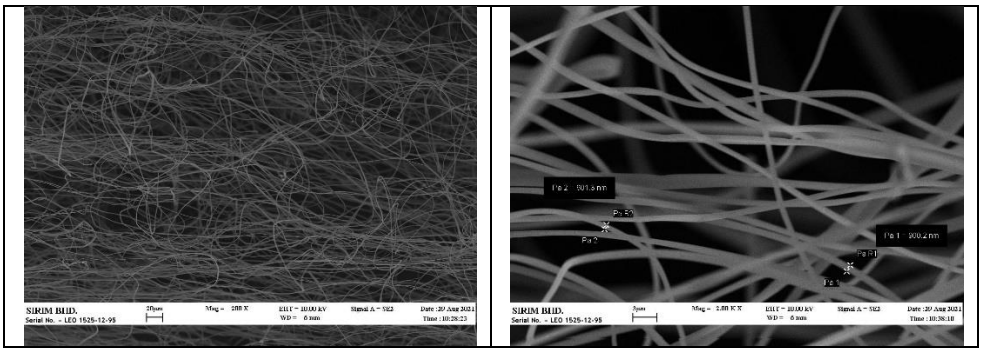


Fig. 3. CA Nanofibre electrospun from S1 solution

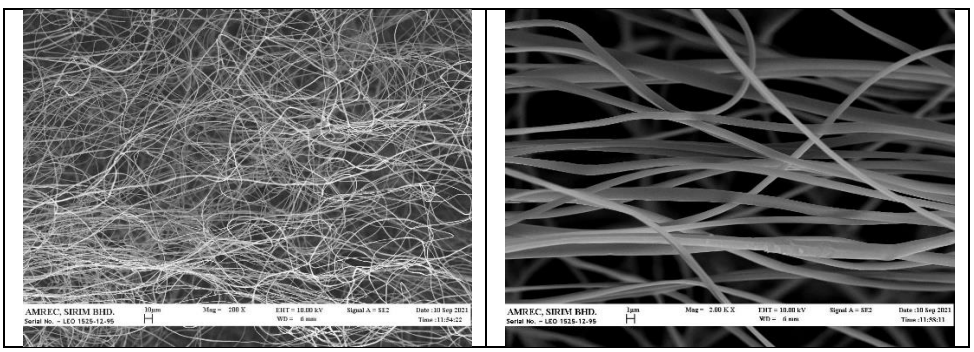
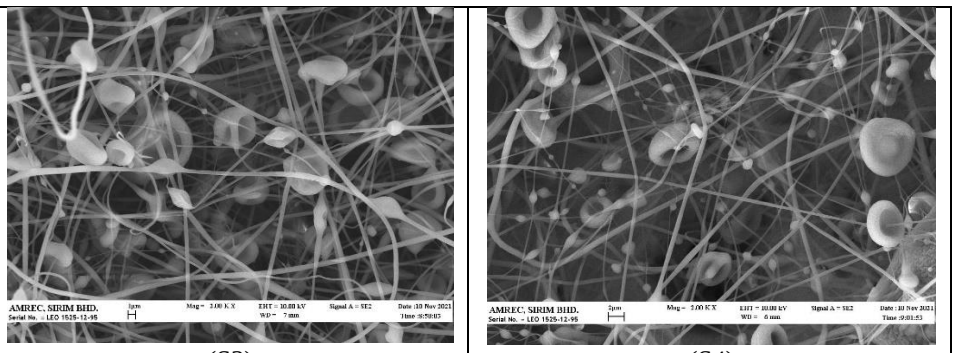


Fig. 4. CA Nanofibre electrospun from S2 solution



(S3) (S4)

Fig.5. CA Nanofibre spun from S3 and S4 solution

4. Conclusions

In summary, a mixed solvent solution of a CA polymer consisting of an acetone, water and ethanol solution successfully produced the cellulose acetate nanofibre. The S2 mix solvent mix with 5% w/v produces the nanofibre comparable to the result reported elsewhere as in the S1 mixed solvent. No extensive studies on the tri-solvent system were reported in previous research. However, detailed studies need to be conducted, including surface tension, EFS value, optimum feed rate, needle distance and roller drum rotation need to be considered.

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