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Electrospinning of cellulose acetate nanofiber membrane using methyl ethyl ketone and N, N-Dimethylacetamide as solvents



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HIGHLIGHTS

G R A P H I C A L A B S T R A C T

- Cellulose acetate nanofibers electrospun using high boiling point solvent mixture of MEK and DMAc.
- Formation of cylindrical and beaded CA nanofibers mapped vs. solvent ratios and polymer concentration.
- Sessile drop experiments proved hydrophilic nature & good wettability.
- \bullet Membranes had DI water flux of 10,197.044 $\rm Lm^{-2}h^{-1}$ for initial 50 ml.
- Membranes could be reused four times without rupture and suitable for water filtration application.



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ABSTRACT

Cellulose acetate (CA) nanofiber membrane was prepared by electrospinning method using solvent mixtures of methyl ethyl ketone (MEK) and N, N, - dimethylacetamide (DMAc) in different ratios (2:1, 1:1, 1:2) and also different concentration of CA (7–19%). MEK was selected in place of acetone due to its high boiling point, thereby, minimises the evaporation loss of the solvent enabling the longer duration of electrospinning. The morphology of electrospun nanofibers was observed by Scanning Electron Microscope (SEM). It was observed that cylindrical fibers formed at higher concentration of polymer with increase of DMAc. Fiber diameters were in the range of 40–500 nm with large diameters formed at higher polymer concentration. Contact angle measurement revealed that membranes have good wetting property. The water flux measurements of membranes were carried out under gravity. A water flux of 10,197 $\rm Lm^{-2}h^{-1}$ was measured initially and was reduced subsequently to 365-200 $\rm Lm^{-2}h^{-1}$. The membranes could be reused up to four times without rupture. The above experiments suggest that MEK and DMAc could be an alternate solvent system in addition to other systems.

1. Introduction

Cellulose Acetate (CA) is a cheap, naturally degradable polymer with

glass transition temperature of 190 °C, therefore, suitable for membrane applications involving low to medium temperature fluids [1–3]. Due to its good hydrophilic property, non-toxicity, chemical and thermal

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Table 1

Physical properties of solvents [20].

	Molecular weight (g/ mol)	Boiling point (°C)	Absolute Viscosity (cP)	Surface tension (dyn/cm)	Polarity (water 100)
MEK	72	80	0.41	24.6	32.7
DMAc	87	166	0.92	34	40.1

stability, moderate mechanical properties, low cost etc. [3,4], CA is an excellent material for water filter membranes. As an example, N. Chitpong, & S. M. Husson [5] used electrospun cellulose acetate nanofiber membranes for removal of cadmium. Similarly, inclusion of CA nanofibers in cigarette filter tip increased the efficiency of tar removal from 47.7% to 71.6% [6].

Cellulose acetate has been widely used as membranes for wound dressing [4], reverse osmosis [7], dialysis [8], ultra-filtration [9] etc. Electrospinning is a suitable process to obtain high aspect ratio nanofibers even at industrial scale [10]. Numerous polymeric, as well as ceramic fibers, have been produced by using electrospinning process [10–13]. Generally, the process involves the application of high voltage to a polymer droplet making it to eject a thin jet. A syringe used to carry and push the solution to be electrospun at uniform rate. The syringe needle acts as an electrode and metallic stand warped with aluminium foil that is grounded acts as counter electrode. The jet is emitted when externally applied electrical field crosses a critical value [14] and jet subdivides into micro to nanofibers as it travels towards metallic collector due to forces between similar charges in polymer jet.

Many researchers studied electrospun cellulose acetate nanofibers using different solvent systems [15–19]. The various solvents used for electrospinning were: acetone, DMAc, DMF, TFE, chloroform, methanol, water or their mixtures in various ratios, acetone been the common solvent. Since, the boiling point (56 $^{\circ}$ C) of acetone is low, it evaporates quickly making difficult in electrospinning for longer duration required large scale production of membranes for various applications. It was felt that an alternate solvent system with a high boiling point solvent in place of acetone is necessary.

Therefore, methyl ethyl ketone having boiling point of 80 °C was considered along with DMAc in this study. The electrospinning was carried out at different MEK: DMAc ratios as well as at different polymer concentration. The morphology of nanofibers (beads and cylindrical) obtained at different conditions was correlated/mapped w.r.t. the solvent ratios and concentration of polymer. In addition, water flux studies under gravity of the membranes and their reusability were also studied.

2. Experimental

2.1. Materials

Cellulose Acetate (CA) (acetyl group 29–45%) white powder, Methyl Ethyl Ketone (MEK) and N, N-Dimethylacetamide (DMAc) solvents were procured from Loba Chemie, India. The properties of solvents are given in Table 1 [20].

2.2. Sample preparation

2.2.1. Preparation of homogeneous viscous CA solution

To study the effect of single solvent, CA solutions in MEK were prepared at 8 and 10% (w/v) and solutions of CA in DMAc were prepared at 10 and 15% (w/v) on a magnetic stirrer (IKA C-MAG HS4) at 500–750 rpm. Also, to study the effect of the mixed solvent system, three different mixtures of MEK and DMAc in the volume ratio of 2:1, 1:1 and 1:2 (v/v) were prepared by stirring for few minutes to achieve a homogeneous solvent. In these homogenized solvent systems (10 ml), different CA concentration solutions for e.g. 7-19% (w/v) in steps of 2% (w/v) were prepared by dissolving appropriate amount of CA with constant stirring at room temperature until a clear viscous solution was obtained.

2.2.2. Electrospinning of CA nanofibers

The solution as prepared above was filled in a 5 ml syringe with a fine needle (0.55 mm needle). The syringe was placed in a lab-scale electrospinning unit (M/s Espin Nanotech, Kanpur) and high voltage was applied slowly to the needle. The schematic diagram of electrospinning setup is presented in Fig. 1.

A grounded metallic stand ($12.5 \text{ cm} \times 12.5 \text{ cm}$) warped with Aluminium foil (0.05 mm thickness) connected to neutral was used for collection of fibers. Membranes for flux measurement were prepared on an Aluminium roller collector (diameter: 80 mm, $800 \pm 70 \text{ rpm}$) mounted on a sliding table (7 cm). Electrospinning conditions are presented in Table 2. After electrospinning, the samples were stored in a desiccator for further characterization.



Fig. 1. Schematic diagram of the electrospinning setup.

Table 2

Electrospinning conditions used in this work.

Solvent ratio:	2:1 & 1:1 (v/v) MEK: DMAc	1:2 v/v MEK: DMAc
Electrospinning conditions:	Tip to collector distance = 15 cm ; High Voltage = 15 kV ; RH = $60-70\%$; Flow rate of solution = $0.5-1 \text{ ml/h}$.	Tip to collector distance = 10 cm, High Voltage = 19 kV RH = $60-70\%$ Flow rate of solution = 2 ml/h.

2.3. Sample characterizations

The morphology and diameter of the electrospun fibers examined on Carl Zeiss EVO 18 scanning electron microscope (SEM). The FTIR absorbance spectra of the fiber membrane were recorded by keeping membrane sample in the holder and scanning 16 times in the wave-number range from 400 to 4000 cm⁻¹ with a resolution of 4 cm^{-1} on PerkinElmer Frontier MIR/FIR System. The DSC was carried out on PerkinElmer DSC-7. About 4.2 mg of nanofiber sample measured and put into aluminum pan and lid arrangement. The heated rate of $10 \,^{\circ}\text{C/min}$ used in the temperature range of $25 \,^{\circ}\text{C}$ -290 °C.

2.3.1. Water contact angle measurement

The surface wetting properties of the nanofiber membranes were determined by sessile drop method Surface Electro Optics, Korea (Phoenix 300+) contact angle analyzer. About 8 µL DI water droplet was dropped manually on the membrane surface and contact angle was measured over time by recording multiple images on the system.

2.3.2. Water flux measurement

In a previous study, water flux for PAN nanofiber membranes was measured and was found that the flux decreases rapidly above 125 μ m [21]. Therefore, $80 \pm 10 \,\mu$ m thick membrane from 11% CA in 2:1 MEK DMAc was prepared by electrospinning for 3 h and a 47 mm circular membrane sample (area: 1734 mm²) was punched out. The circular membrane was placed in a stainless steel filter holder of effective filtration area 1160 mm². 150 ml DI water was passed through the membrane and time was measured at different volumes of flow. Two

samples were tested and the flux was calculated for the membranes by Eq. (1)

$$Water flux = \frac{Volume \ of \ Water \ (\Delta v)}{(Effective \ area \ of \ Filtration^* Time \ for \ filling \ \Delta v \ water)}$$
(1)

2.3.3. Reusability study

A reusability study was carried out in order to know the suitability of membrane for practical water filter application. In this experiment, one of the above samples was reused 3 more times under gravity by drying in an oven (at 120 °C for 1.5–2hr) to ensure all water trapped inside the membrane is removed each time. The dry weight of the sample before each use was about 0.019 \pm 0.001 g. The time taken for 150 ml DI water to flow through the membrane each reuse was used to calculate water flux by Eq. (1). The water level was maintained to full by refilling water every few minutes into the filter cup (320 ml) throughout the experiments.







Fig. 2. SEM micrographs showing effect of polymer concentration and solvent ratio on morphology of nanofibers.



Fig. 4. SEM micrographs (3,000x) and their corresponding diameter distribution of nanofibers electrospun from 2:1 MEK: DMAc solvent system with CA concentrations (a) 7% (b) 9% and (c) 11% (w/v) (d) 13% and (e)15% CA.



Fig. 5. SEM micrographs (3,000x) and their corresponding diameter distribution of nanofibers electrospun from 1:1 MEK: DMAc solvent system with CA concentrations (a) 11% (b) 13% & (c) 15% (w/v).

3. Results and discussion

3.1. Morphology of nanofibers

3.1.1. Single solvent system

Solutions of CA (10 & 20%) in DMAc were clear and viscous enough for electrospinning and produced only particles/beads, but not the fibers. Liu and Heish [15], and Tungaprapa et al. [18] also reported the inability to produce fibers in single solvent of DMAc. Similarly, it was



Fig. 7. Variation of fiber diameter as a function of concentration of CA solutions and solvent mixing ratio.



Fig. 6. SEM micrographs (5,000x) and their corresponding diameter distribution of nanofibers electrospun from 1:2 MEK: DMAc solvent system with CA concentrations (a) 17% (b) 19% w/v).

Table 3

The morphology of electrospun cellulose acetate nanofibers with different cellulose acetate concentrations in different solvent systems.

Solvent system	Beaded fiber	Cylindrical fiber	References
Acetone	short fibers <5%, 9–13% long fibers	Few fibers 17–21%, blocking of tip	[15,17,18]
2:1 Acetone: DMAc	10–14%	15–20%	[15,18,19]
2:1 MEK: DMAc	<11%	11–15%	In this work
1:1 Acetone: DMAc	<16%	>16%	[18]
1:1 MEK: DMAc	<15%	15–17%	In this work
1:2 Acetone: DMAc	<20%	25%	[19]
1:2 MEK: DMAc	<17%	19%	In this work

difficult to prepare fibers of cellulose acetate using MEK solvent. In the former case, dielectric constant and surface tensions are too high while in the latter case, they are too low [Table .1]. Therefore, it was felt that a mixture of the two solvent ie. MEK and DMAc should aid the fiber formation during electrospinning.

3.1.2. Mixed solvent system

SEM images in Fig. 2 display the effect of solvent mixture ratios and also the effect of CA concentration on formation and morphology of beads/fibers. In all the solvent mixtures, with increase in polymer concentration, morphology changed from beaded fibers to cylindrical fibers.

3.1.2.1. Mechanism of fiber formation under different solvent conditions. In the current investigation, polymer concentration was varied systematically in different solvent compositions and results are mapped in Fig. 3. It was seen that, solvent system with higher surface tensions (higher DMAc content) required higher polymer concentrations to form cylindrical fibers. For instance, it was observed 11%, 15%, and 19% (w/v) CA concentrations were required to form cylindrical fibers in 2:1, 1:1 and 1:2 (v/v) MEK: DMAc solvents respectively. This is because higher polymer concentrations provide higher viscosity necessary to balance the higher surface tension force (in higher DMAc). Also, higher surface tension of solvent containing higher DMAc promoted bead formation due to higher polarity and higher intermolecular forces between DMAc molecules. For example, with constant CA (13%) and changing the solvent system from 2:1 to 1:1 MEK: DMAc, produced beaded fibers. These observations are concurrent with literature [18,19,22,23].

SEM images and diameter distribution of nanofibers electrospun from 2:1, 1:1 and 1:2 ratios of MEK and DMAc solutions are presented in



Fig. 9. (a) Contact angle vs. time and (b) shapes of droplet on electrospun cellulose acetate nanofiber membrane.



Fig. 10. Flux vs. volume of flow of DI water.



Fig. 8. (a) FTIR absorbance spectra (b) DSC thermogram of electrospun CA nanofiber.



Fig. 11. Flux vs. Number of uses of cellulose acetate nanofiber membrane.

Fig. 4, Fig. 5 and Fig. 6 respectively. In all the cases, the fiber diameters were in the range of 40 nm–500 nm and fiber diameters increased with increase in cellulose acetate concentration due to increasing viscosity with chain entanglements [18,24,25]. For membrane preparation and water flux measurement, 11% CA in 2:1 (v/v) MEK and DMAc was selected due to its low CA concentration that maintain optimum viscosity required for electrospinning.

The effect of concentration of CA solutions on the fiber diameters for various MEK and DMAc solvent mixtures is summarised in Fig. 7. From the horizontal line A, drawn considering points a_1 , a_2 , a_3 , it is safely concluded that polymer concentration has to be more if DMAc concentration increases for the same diameter fibers and vice-versa. Similarly, considering the vertical lines B and C drawn at different CA concentrations, the fiber diameters decreases with the increasing DMAc concentration. This is due to higher boiling point of DMAc than MEK (see Table 1), providing more time for fiber splitting before its complete evaporation. It can be seen from Table 3 that lower polymer concentrations were needed to obtain the cylindrical fibers when acetone was replaced with MEK in the same solvent ratios.

Based on the above study, it is proved that a MEK/DMAc solvent mixture produces bead/defect free cylindrical nanofibers depending on the solvent ratios and polymer concentrations. Therefore, MEK/DMAc solvent system is also an alternate to Acetone/DMAc solvent system already studied by other researchers [15,17–19]. In addition, due to low evaporation of MEK, MEK/DMAc solvent system provides longer duration for electrospinning.

3.2. FTIR spectra and DSC

The FTIR absorbance spectra of 11% CA in 2:1 MEK DMAc nanofiber membrane are shown in Fig. 8(a). CA has several side groups such as hydroxyl (O–H), methyl (C–CH₃), carbonyl (C=O) and, a main ether (C–O–C) linkage in the cellulosic back bone. The cellulose acetate spectra are confirmed by the following characteristics. A wide band around at 3486 cm⁻¹ is a result of stretching vibration of hydroxyl group. The sharp peak at 1744 cm⁻¹ is ascribed to the stretching of carbonyl group (C=O). Stretching vibrations of methylene (C–H) groups produced sharp peaks around 2,885 cm⁻¹ and 2943 cm⁻¹, whereas it's symmetric and anti-symmetric bending peaks were observed at 1,368 cm⁻¹ and 1,431 cm⁻¹, respectively. The stretching vibrations of C–O–C linkage of the cellulosic backbone were observed between 1223 cm⁻¹and 1040 cm⁻¹. The FTIR peaks obtained in this work corresponds to all major vibration modes of cellulose acetate [15,17,26], therefore, proves the nanofiber material as cellulose acetate.

The DSC thermogram of 11% CA in 2:1 MEK DMAc electrospun

cellulose acetate nanofiber is shown in Fig. 8(b). Three main thermal transitions were observed. The first endothermic transition between 50 °C and 110 °C was due to the loss of adsorbed moisture from the nanofiber surface [26]. The second was the glass transition for the electrospun nanofibers that occurs over a range of temperature. The glass transition onset is at 176 °C and transition continued till 196 °C. Therefore, mid value glass transition temperature is found to be 186 °C. This is close to the reported value in literature [27]. The third was observed after the glass transition for the melting of the fibers due to crystalline nature of cellulose acetate. The onset of melting of the CA nanofibers is at 213 °C and the melting continued until 235 °C.

3.3. Water contact angle

The water contact angle of (a) 11% CA in 2:1 MEK: DMAc and (b) droplet shapes are presented in Fig. 9. The initial contact angle of water droplet was 81° (<90°) indicating that cellulose acetate nanofiber is hydrophilic. Water droplet was spread over the membrane surface within 18 s indicating good wetting property of cellulose acetate nanofiber membrane [4].

3.4. Water flux measurement

Average flux of electrospun cellulose acetate nanofiber membrane at different volumes of flow is shown in Fig. 10. The membrane exhibited very high initial flux during the filtration of first 50 mL, then rapidly decreased in the next 25 ml. The decrease in water flux is due to the shrinkage of membrane and formation of thin film of water apparently reducing the pore diameter. Flux stabilized between 125 and 150 ml, and was in the range of $365 - 200 \text{ Lm}^{-2}\text{h}^{-1}$. This flux is comparable with the reported value by J. wang et. al. [28].

3.5. Reusability study

The flux vs. Number of uses of cellulose acetate nanofiber membrane is presented in Fig. 11. From the figure, it was observed that the flux decreased with the number of uses (four times) without rupture.

4. Conclusions

In this paper, cellulose acetate nanofibers were obtained by electrospinning CA solutions in MEK and DMAc solvent mixtures. It was found that MEK rich solvent produce cylindrical fibers at low polymer concentration. DSC study revealed glass transition temperature (T_g) and melting temperature (T_m) of the electrospun nanofibers to be around 186 °C and 226 °C respectively. Contact angle measurements proved the membrane is hydrophilic with good wettability. Water flux studies on cellulose acetate nanofiber membrane revealed that membrane flux was about 200 Lm⁻²h⁻¹ and reusability was ascertained.

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