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Investigating morphology and performance of cellulose acetate butyrate electrospun nanofiber membranes for tomato industry wastewater treatment

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ABSTRACT

In this research, cellulose acetate butyrate (CAB) electrospun nanofiber membrane (ENM) was prepared by electrospinning method in order to separate the contaminants of an industrial wastewater. The influence of various electrospinning parameters on morphology and average fiber diameter of the membranes were investigated by scanning electron microscopy and image analysis. The permeability of the membranes was evaluated by measuring pure water flux. In order to investigate the performance of the prepared membranes for tomato wastewater treatment, the rejection of the pollution indices and flux were determined. The results demonstrated the potential of using CAB nanofiber membrane for wastewater treatment. The appropriate electrospinning conditions led to preparation of the membranes with high water permeability in the range of microfiltration, proper retention efficiency in the range of ultrafiltration and noticeable antifouling property.

Keywords: Cellulose acetate butyrate; Electrospun membrane; Nanofiber; Wastewater treatment

1. Introduction

Nowadays with the rapid growth of industry, agriculture and population, there is an increasing trend in water consumption, and the demand on the treatment of wastewater is intensified. While inadequate access to clean water is the most serious problem in many countries around the world [1], wastewater reuse is recognized as a positive and practical means for solving the water shortage problem [2]. The technology for wastewater treatment includes very different options. Among those membrane processes are the most promising, and are thought to be an appropriate candidate and key element of advanced wastewater reuse [3].

A serious challenge of the membrane technology is the trade-off between membrane permeability and selectivity, i.e., when permeability is high, selectivity is low and vice versa. The high energy consumption is a major drawback that prevents the wide application of pressure-driven liquid filtration. Furthermore, membrane fouling is another problem by which energy consumption is increased and the lifetime of membrane is reduced [4].

Electrospinning is a simple, versatile and inexpensive method to fabricate ultrafine fibers from various materials [4–6]. Polymer nanofibers are an important class of nanomaterials, which have attracted increasing attention during the last decade [7]. Due to the unique properties of flexibility in surface functionalities, superior mechanical performance and good biocompatibility, nanofibers have been proposed as an optimal candidate for many applications, such as catalysis, tissue engineering, protective clothing, electronics, drug delivery, vessel engineering, filtration, agriculture and nanosensors [8–10]. In particular, electrospun nanofiber membranes (ENMs) have a unique potential in membrane technology because they have a huge surface area per unit volume, high porosity with excellent pore interconnectivity and submicron pore sizes because of small diameters

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of nanofibers [5,11–15]. Because of these unique features, ENMs have been applied for wastewater treatment in various industrial sectors such as chemical, pharmaceutical and food industries [16]. Thus, even though ENMs have been commercially employed only for air filtration at present, they have a great potential in water treatment and separation of non-aqueous solutions [4,5].

A number of advantages of ENMs over the conventional membranes have been reported in the literature. For example, the low transmembrane pressure (TMP) and the high permeability are the desired properties by an ideal membrane, and the ENMs have both of these properties [17,18]. Application of ENMs is more useful and effective to minimize the fouling [17,19]. ENMs are a competitive option because the other commercial membranes have higher production cost than ENMs (50 €/m^2 compared with an estimated 20 €/m^2 for ENMs), and in most cases, a high TMP is required [20]. Generally, ENMs have become an adequate candidate because of their high permeability, low pressure drop, acceptable selectivity and low production and operating cost [19,21].

Even though ENMs are often found to be applicable for the particle removal from water, they can also be used in many membrane processes like ultrafiltration (UF), nanofiltration (NF), reverse osmosis (RO), membrane distillation and membrane bioreactor (MBR) [12,17]. Gopal et al. [22,23] reported that ENMs reported that ENMs have potential as pre-filter for particulate removal. Aussawasathien et al. [24] prepared nylon-6 ENMs and employed them as pre-filter for removal of micron and submicron particles from aqueous media. Sang et al. [25] developed chlorinated polyvinyl chloride ENM by high-voltage electrospinning process and investigated the removal of divalent metal cations including Cu^{2+}, Pb^{2+} and Cd^{2+} from the simulated groundwater. Veleirinho and Lopes-da-Silva [26] prepared self-supporting nanofibrous poly(ethylene terephthalate) membrane and investigated the application of this type of membranes in apple juice clarification process. Bjorge et al. [20] evaluated the use of nanofibrous microfiltration (MF) membranes in water filtration applications: (1) for pathogens removal, (2) in a lab-scale submerged MBR, and (3) as a stand-alone filter for water treatment. Uyar et al. [27] prepared polystyrene fibrous membranes and reported that they show potential for efficient removal of organic compound (e.g., phenolphthalein) from aqueous solution. Homaeigohar et al. [17] developed polyethersulfone (PES) ENMs with non-woven sublayer for polystyrene suspended particle filtration from aqueous media. They demonstrated that the filtration performance of the ENMs is highly dependent on size distribution of the suspended particles. Bilad et al. [19] studied the feasibility and optimization of low-cost ENMs as potential MBR membranes. They observed that the ENMs showed performances comparable with those of the tested commercial membranes in the short and long term. Cooper et al. [28] reported the development of chitosan-polycaprolactone (PCL) ENMs to utilize the natural antibacterial property of chitosan. They demonstrated the potential of chitosan-PCL nanofibrous membranes as pre-filters for water filtration with intrinsic antibacterial advantages. Huang et al. [29] demonstrated a chemical modification capable of modifying the mechanical properties of polyacylonitrile (PAN) and polysulfone (PSF) ENMs. The chemical modification involved the polydopamine (PDA), a hydrophilic polymer and improved hydrophilization and strengthening of ENMs. Obaid et al. [30] prepared modified PSF electrospun nanofiber membrane and evaluated it in oil/water separation. The membrane modification was achieved by using NaOH nanoparticles as an additive in the polymer solution and formation of a thin layer from a polyamide polymer on the surface of the electrospun membrane. Despite a lot of studies about using nanofibrous membranes for the filtration of wastewater, there isn't sufficient information about their performance for real wastewater treatment. The use of real wastewater in order to evaluate the performance of ENMs is rare in the literature, and it is the serious omission of previous research.

Cellulose acetate butyrate (CAB) is one of the cellulose derivatives that has improved dimensional stability, weathers well and is more chemical and moisture resistant than cellulose acetate [31]. CAB is considered as one of the appropriate materials for different applications because it is one of the toughest cellulosic plastics, and has good transparency, colorability, weatherability, electrical properties and resistance to inorganic chemicals [32]. Also, it is considered as one of the appropriate membrane materials because of its good resistance to fouling, chlorine tolerance and chemical stability [33].

To the best of the authors' knowledge, preparation of ENMs from CAB and investigation of the effect of electrospinning parameters on its morphology and performance have not been reported in the previous studies. Also, the use of real wastewater with a wide range of contaminants in the performance evaluation of ENMs is rare in the literature, and this, we believe, is the missing link between the academic research and practical application. In the present study, CAB ENMs were prepared in order to separate the contaminants from an industrial tomato wastewater. The effects of various electrospinning parameters, i.e., polymer concentration, applied voltage, rotational speed of collector, and distance between the needle tip and collector, on the morphology and the pure water flux (PWF) of the CAB ENMs were investigated. The performance of the prepared ENMs in the filtration of tomato wastewater was evaluated in terms of permeate flux and the rejection of chemical oxygen demand (COD), total dissolved solids (TDS) and turbidity. The antifouling property and fouling resistance of prepared membranes after wastewater treatment were evaluated.

2. Experimental

2.1. Materials

CAB (Mn ~ 65,000; acetyl content = 28.0–31.0 wt.%; butyryl content = 16.5–19.0 wt.%; 0.9–1.3 wt.% hydroxyl), was purchased from Sigma-Aldrich (USA) was used as polymer for preparation of the electrospinning dope. Dimethylformamide (DMF) and acetone were supplied from Akkim (Turkey) and Merck (Germany), respectively, were used as solvents. Non-woven polyester was obtained from Awa Paper (Awa Paper Manufacturing Co. Ltd., Japan) was used as the sublayer of the ENMs. The wastewater used in the present study was obtained from a local tomato processing plant. The wastewater characteristics are presented in Table 1.

Table 1
Characteristics of tomato processing wastewater

Index	Value
COD (mg/l)	708
TDS (mg/l)	1,278
Turbidity (NTU)	392
pH	4.5

2.2. Preparation of electrospun membrane

A specified quantity of CAB was dissolved in 2:1 (v/v) acetone/DMF solvent by a stirrer rotating at 200 rpm for 24 h at room temperature. Then the polymer solution was kept for 12 h without stirring to remove air bubbles. The electrospinning apparatus was supplied from Asian Nanostructures Technology Company (ANSTCO, Iran). The spinning dope so prepared was extruded by means of syringe pump from a syringe needle (0.7 mm inner diameter) at a flow rate of 0.5 mL/h and collected on a rotating drum, which was placed 15–25 cm away from the needle tip and with a polyester non-woven sheet wrapped on its surface. A high voltage in the range of 16–18 kV was applied between the needle tip and the rotating drum. The electrospinning was conducted at an ambient temperature condition. Table 2 summarizes the spinning conditions of different ENMs spun in this work.

2.3. Scanning electron microscopy

Morphology of CAB ENMs was observed by a scanning electron microscope (KYKY-EM3200, KYKY Technology Development Ltd., China). The samples were sputter-coated with a thin film of gold. The photomicrographs were taken under very high vacuum conditions at 25 kV. The diameter size distribution and average diameter of the electrospun nanofibers were determined by image analysis using the ImageJ 1.47 software [34].

2.4. Porosity and pore size measurement

For the porosity measurement, a square piece of the ENM with a known surface area was cut and weighed by an electronic balance with a resolution of 0.1 mg. The thickness was measured by a digital micrometer with a resolution of 0.001 mm. Then, its apparent density (ρ) was calculated from the obtained mass and volume. The average ρ value of three pieces was used in Eq. (1) to calculate the porosity (ε) of the ENM [35,36]:

$$\varepsilon = \frac{\rho_0 - \rho}{\rho_0} \times 100 \quad (1)$$

where ρ_0 is the density of the CAB, which is 1.25 g/cm^3 [37].

Mean pore radius (*r*) of the ENM was calculated by using Eq. (2) [35,36,38,39]:

$$r = \frac{\sqrt{\pi}}{4}\left(\frac{\pi}{2\log\left(\frac{1}{\varepsilon}\right)} - 1\right)d \quad (2)$$

Table 2
CAB percentage of the polymer solutions and the applied experimental conditions

Membrane	CAB (wt.%)	Applied voltage (kV)	Distance between the tip of needle and collector (cm)	Rotational speed of collector (rpm)
M-1	10	16	15	300
M-2	12	16	15	300
M-3	14	16	15	300
M-4	16	16	15	300
M-5	12	16	15	1,000
M-6	12	18	15	1,000
M-7	12	18	25	1,000

where *d* (nm) is the average fiber diameter obtained in Section 2.3.

2.5. PWF measurement

PWF was determined using an experimental setup in a batch mode. The permeation cell (liquid volume 350 ml, effective membrane area 20 cm^2) was made from stainless steel. The details and schematic representation of the setup can be found elsewhere [40,41]. The permeation experiment was conducted at TMP of 6 psi, and PWF, *J* (L/m^{2}h), was calculated by the following equation [42]:

$$J = \frac{Q}{A \cdot \Delta t} \quad (3)$$

where *Q* is the permeate volume (L) collected during the sampling time Δ*t* (h), through the effective membrane area *A* (m^2). The PWF measurements were repeated three times to ensure its accuracy.

2.6. Evaluation of wastewater treatment

The wastewater treatment experiments were conducted at room temperature and TMP of 6 psi. The permeate flux of each membrane was evaluated according to Eq. (3). Furthermore, the rejection percentage of three pollution indices was evaluated from Eq. (4):

$$\text{Rejection percentage} = \left(1 - \frac{C_p}{C_f}\right) \times 100 \quad (4)$$

where C_p and C_f represent TDS, COD and turbidity of permeate and feed, respectively.

TDS value was measured by electrical conductivity meter (Extech EC-400, Extech Instruments, USA). A thermoreactor (RD125, Lovibond, Germany) for heating and digestion of COD vials solution, as well as COD photometer, supplied from Lovibond Tintometer (Germany), were used to measure COD. Lutron Electronic turbidity meter (TU-2016, Taiwan) was used to measure turbidity.

2.7. Analysis of membrane fouling

After filtration of the wastewater, the membranes were washed with distilled water, and then PWF of the fouled membranes was measured. To evaluate fouling resistance of the membranes, flux recovery (FR), which indicates the recycling property of membrane, was calculated by using Eq. (5) [42]:

$$\% FR = \left(\frac{j_{w2}}{j_{w1}}\right) \times 100 \tag{5}$$

where j_{w2} and j_{w1} are the PWF after and before the tomato wastewater treatment experiment.

Generally, higher FR demonstrates better antifouling property of an electrospun membrane. Furthermore, in order to analyze the membrane fouling process in details, two ratios were defined to describe the fouling resistance of the prepared membranes. R_r and R_{ir}, described by Eqs. (6) and (7) [43,44] illustrate the reversible and irreversible fouling resistances, respectively:

$$\% R_r = \frac{j_{w2} - j_p}{j_{w1}} \times 100 \tag{6}$$

$$\% R_{ir} = \frac{j_{w1} - j_{w2}}{j_{w1}} \times 100 \tag{7}$$

where j_p is the wastewater permeate flux.

3. Results and discussion

3.1. Morphological studies of the membranes

The scanning electron microscope micrograph and fiber diameter distribution of the CAB ENMs are illustrated in Fig. 1. As well, the average diameter of ENMs is given in Fig. 2. According to Fig. 1(a), beaded fibers were obtained from the spinning dope containing 10 wt.% of CAB, due to the low polymer concentration and viscosity [45,46]. When the concentration of polymer increased from 10 to 12 wt.%, smooth fibers were obtained.

In Fig. 2, the average fiber diameter increased progressively from M1 to M4, which is ascribed to the progressive increase of CAB content from M1 (10 wt.%) to M4 (16 wt.%). The high polymer concentration prevented the jet from being stretched by the Coulombic force [45–47].

In Fig. 2, the average diameter decreased from M2 to M5, due to an increase in rotational speed of the collector from 300 to 1,000 rpm, which contributed to the stretching of the solution jet [48,49]. The average diameter decreased from M5 to M6, corresponding to an increase in applied voltage from 16 to 18 kV. A higher voltage leads to greater stretching of the polymer solution due to greater Coulombic forces in the jet as well as stronger electric field [50,51]. The average diameter further decreased from M6 to M7 with increasing the distance between the tip of needle and collector from 15 to 25 cm. When the distance increases, the jet has a longer flight time, but the electric field strength decreases, which slows down the acceleration of the jet to the collector [51,52].

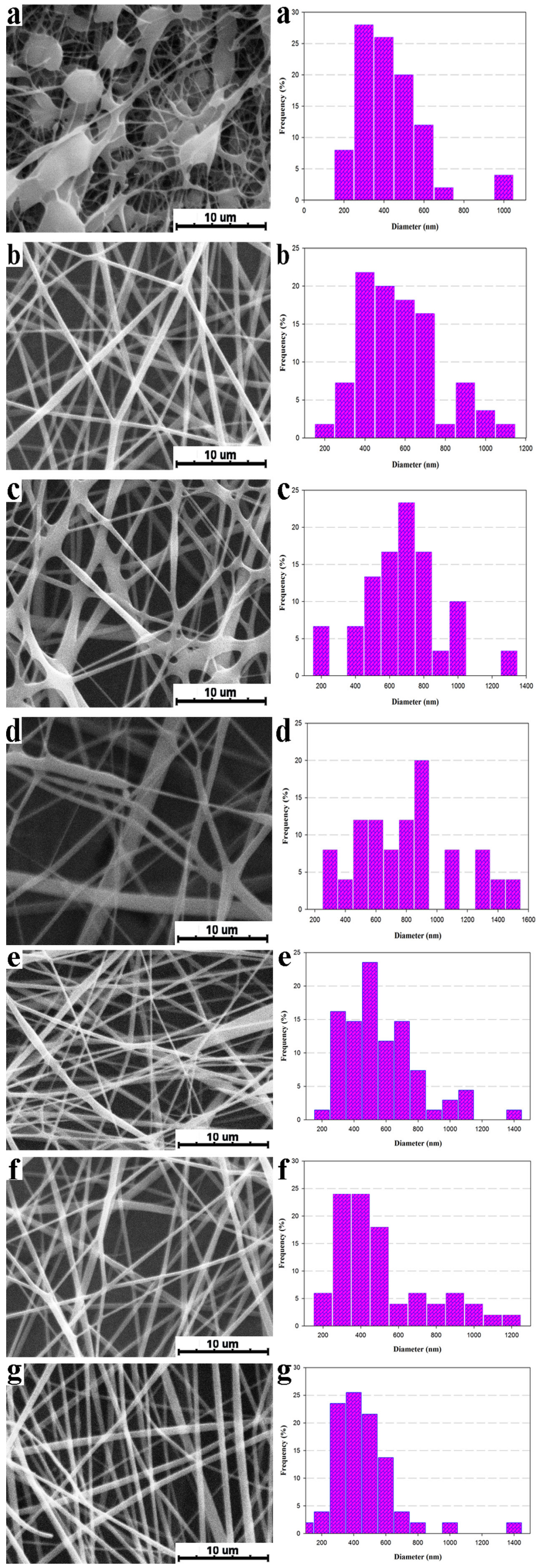

Fig. 1. Scanning electron microscope micrograph and fiber diameter distribution of the electrospun membranes: (a) M-1, (b) M-2, (c) M-3, (d) M-4, (e) M-5, (f) M-6 and (g) M-7.

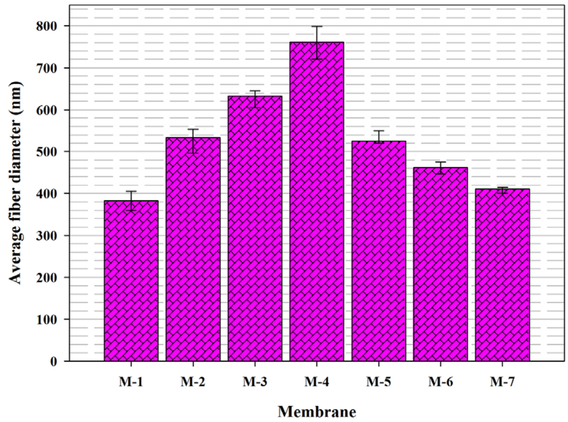

Fig. 2. Average fiber diameter of CAB electrospun membranes.

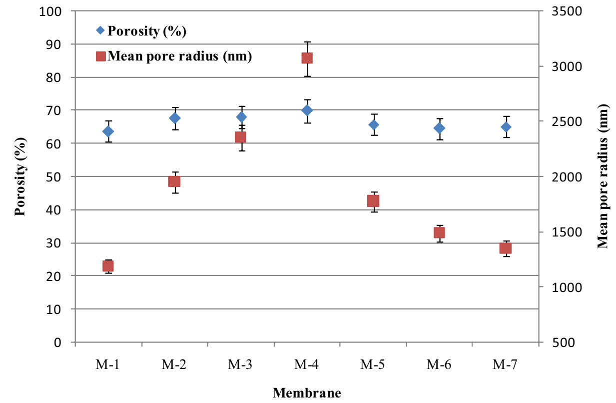

Fig. 3. Porosity and mean pore radius of CAB electrospun membranes.

3.2. Porosity and pore size

Fig. 3 shows the porosity and pore size of the ENMs. The spinning parameters have insignificant effect on the porosity (within 10%). In contrast, the spinning parameters have significant effect on the pore size. A parallel relationship is observed between the average fiber diameter (Fig. 2) and pore size, i.e., both show a maximum at M4. Also, from Figs. 2 and 3, it can be seen that the average fiber diameter of membrane M2 and M5 are approximately similar, but the porosity and mean pore radius of M2 are noticeably more than M5. It shows that the porosity affects the pore size, but the fiber diameter is the dominant parameter to manipulate the pore radius of membrane.

3.3. Pure water flux and flux recovery

Fig. 4 summarizes the PWFs before and after filtration experiments with the wastewater. Comparing Figs. 3 and 4, a relationship between the average pore size and PWF (both before and after the wastewater filtration) is obvious. For example, both average pore size and PWF show a maximum at M4.

The PWF through an electrospun membrane, which is a fibrous porous media, can be described by Happel's equation [53,54]:

$$J = \frac{1}{32(1-\varepsilon)}\left[-\ln(1-\varepsilon) + \frac{(1-\varepsilon)^2 - 1}{(1-\varepsilon)^2 + 1}\right]\frac{d^2 \Delta p}{\mu \Delta x} \tag{8}$$

where ε is the porosity; d is the average fiber diameter; μ is the dynamic viscosity; ΔP is the pressure difference across the membrane and Δx is the membrane thickness.

In Fig. 5, experimental and theoretical PWF of the prepared membranes is presented as a function of the mean pore size and fiber diameter. The PWF is increased with an increase in the pore size, which is natural particularly when the porosity is practically unchanged. Indeed, this is in the agreement with what would be expected for flow through a porous media. Moreover, the results show the proper compromise between theoretical and experimental data.

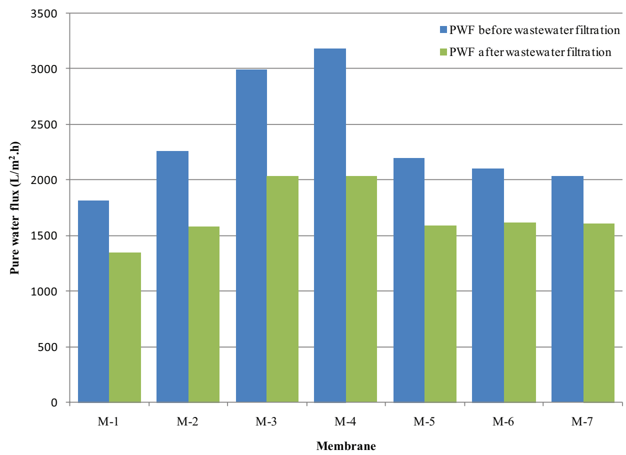

Fig. 4. PWF before and after wastewater filtration of prepared membranes.

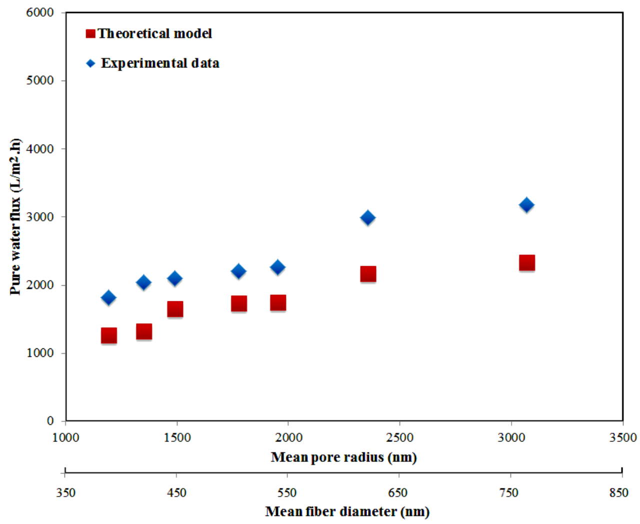

Fig. 5. PWF of CAB electrospun membranes vs. mean pore radius and fiber diameter.

3.4. Wastewater treatment performance of membranes

The process wastewater produced during sorting, cleaning and moving tomatoes constitutes the main step of the tomato processing industry and is generally high in organic contents, suspended solids and colloidal fractions that are not only slowly biodegradable but also exhibit very poor sedimentation characteristics [55–57]. In Fig. 6, the wastewater permeate flux for the prepared ENMs is presented as a function of operation time. The permeate flux of all the membranes decreases rapidly during the first 3 min, followed by a more gradual decline and finally levels off. This behavior is due to concentration polarization and fouling of the membranes. However, this low steady flux value is still acceptable from the practical production point of view and comparable with that reported in the literature as discussed below [56]. The order in the flux is M1 < M2 < M3 < M4 > M5 ≈ M6 ≈ M7, showing again a maximum at M4.

Fig. 7 shows the percentage rejection in terms of the three pollution indices. The percentage rejections are in the order of M1 > M2 > M3 > M4 < M5 < M6 < M7 for all pollution indices, which is in reverse order of PWF and wastewater permeate flux. Comparing Fig. 2 where the maximum in pore size is observed at M4, it can be concluded that the membrane selectivity decreases as the pore size increases. The noticeable point with respect to these results is that the percentage rejection was as high as that of UF membrane while the flux was as high as that of MF membrane [58].

As the particle size (constituent particles of effluent) is in submicron range, the velocity of particle migration away from the fibrous membrane surface seems to be at its minimum. The particles are adsorbed on the nanofibers surface through direct interception, inertial impaction and diffusion. Indeed, the first part of particles is entrapped within the membranes by adsorption to the nanofibers, and the next particles join them. Thus, the particles were able to join closely together, to decrease the effective pore size of the membrane significantly at the surface and construct a dense cake layer initiated from the depth of membrane and grown upward to the surface. This dense cake layer performed as the separating layer for the membrane. Thus, the membranes act as a depth and surface filter [23,35]. It was also reported that cellulose nanofibers, because of their unique properties, had adsorption capability, so they could be used for metal ions adsorption [59,60]. Whereas the UF membranes generally do not remove TDS including metal ions, the partial removal of TDS and metal ions that exist in a real wastewater by the UF membrane used in this work indicates that the high surface area and the unique properties of cellulose nanofibers

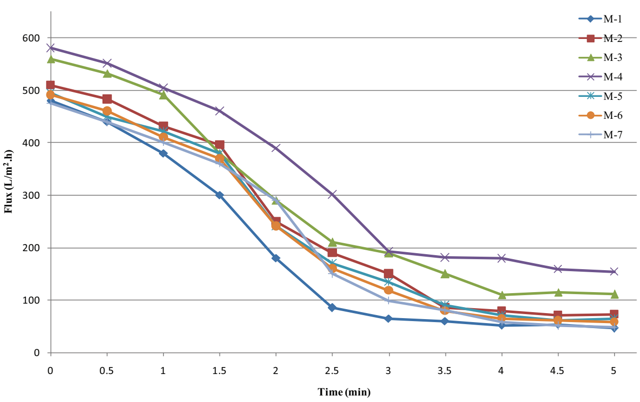

Fig. 6. Permeate flux of the wastewater through the prepared membranes.

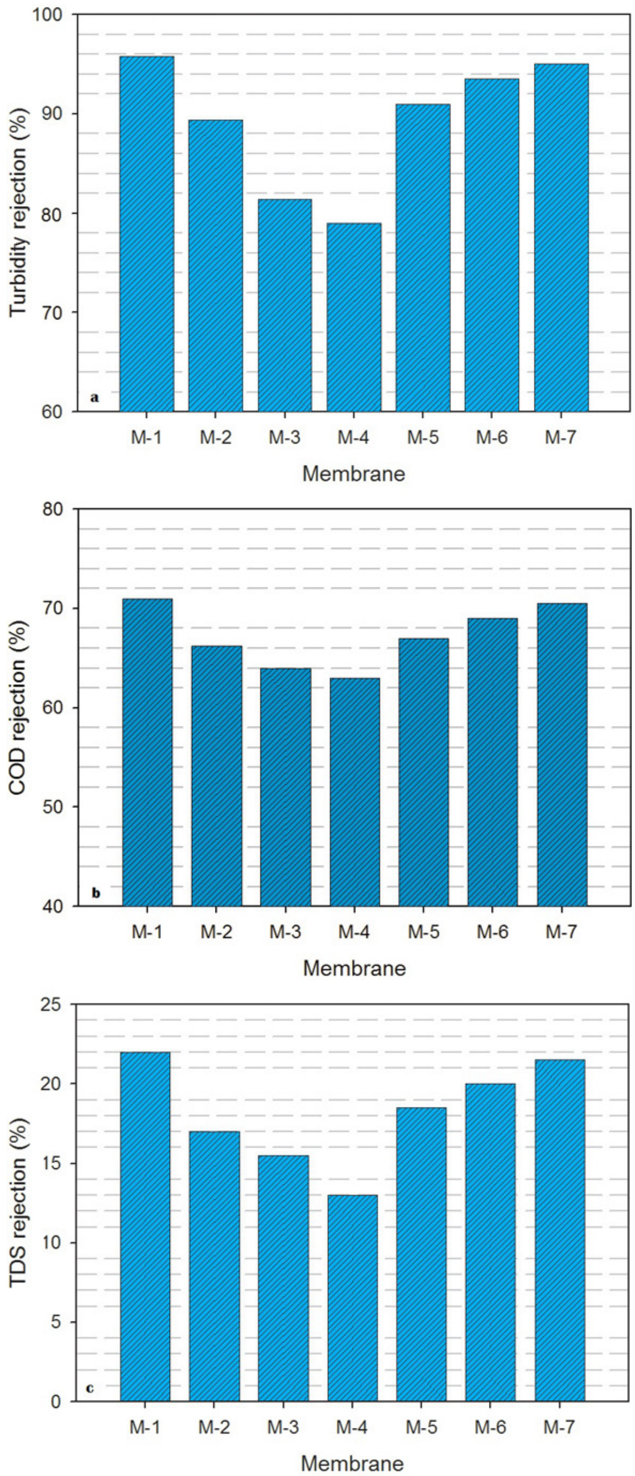

Fig. 7. Rejection of the pollution indices of the wastewater.

enhance the separation properties of the membrane. Thus, the membranes have demonstrated the limited capability for TDS rejection. Considering all the filtration results including permeability, FR and rejection of turbidity, TDS and COD, M7 seems the best among all the tested ENMs. Therefore, M7's performance was compared with that of the other research [56] on industrial tomato wastewater treatment in Table 3. Obviously, M7's performance is much better than that of the other membrane. It is interesting to note that the membrane (Desal-5 DK2540) used by the other researchers was an NF membrane despite its relatively low COD rejection.

3.5. Investigation of the antifouling property

Fig. 8 shows that FR lies between 64% and 78% for all the tested ENMs, which values seem quite acceptable for the particle filtration [61,62]. This relatively high FR is indicative of a suitable antifouling property of the membranes. ENMs do not keep the micro-particles on or within their surface permanently. They act as a screen filter, where the particles are effectively lifted up from the membrane surface by stirring, preventing them from accumulation on or within the membrane surface. The commonly applied washing was also almost enough to recover the flux of the ENMs [23]. It is interesting to note that FR showed a minimum at M4, i.e., FR was the lowest when PWF was the highest.

Fig. 9 shows reversible and irreversible fouling resistances values in order to evaluate the antifouling properties of the prepared membranes in details. With respect to Figs. 3 and 9, the irreversible and reversible resistances of the membranes were increased and decreased with an increase

Table 3
Comparison of performance of the optimum membrane with other research reported in the literature [56] on tomato wastewater treatment

Membrane	M-7	Desal-5 DK2540 [56]
TMP (bar)	0.41	4.5
Flux (L/m^2 h)	49	8.21
Turbidity rejection (%)	95	–
COD rejection (%)	70.5	61
TDS rejection (%)	21.5	–

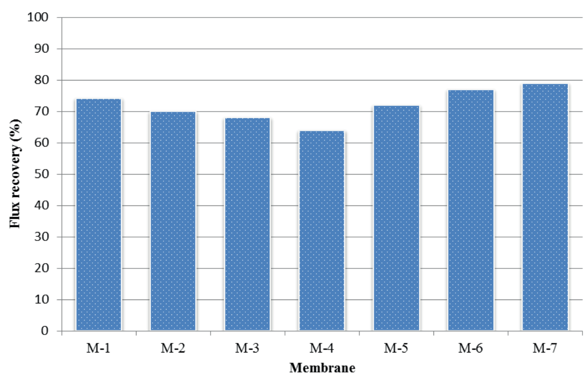

Fig. 8. Flux recovery of the prepared membranes.

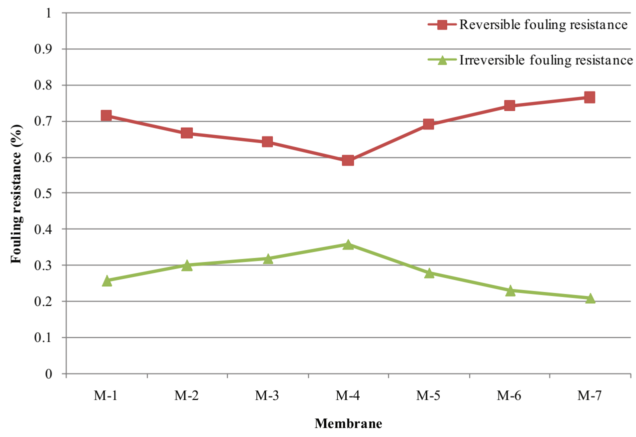

Fig. 9. Fouling resistances of the prepared membranes.

in the mean pore radius, respectively. When the pore size of ENMs is smaller than particle size, the membrane acts as a screen filter. Thus, irreversible fouling resistance decreases. However, with increasing in the mean pore radius, the probability of particle entrapment within membranes is increased [23]. Therefore, the irreversible fouling increases.

4. Conclusions

Electrospinning method was used for preparation of electrospun CAB nanofiber membranes in order to separate the contaminants of an industrial tomato wastewater. The effects of electrospinning parameters such as polymer concentration, applied voltage, distance between the tip of needle and collector, and rotational speed of collector on average fiber diameter, morphology and permeability of the membranes were investigated.

The results showed that average diameter of the fibers and the mean pore size of the membranes increased by increasing the polymer concentration and, however, decreased by increasing the applied voltage, distance between the tip of needle and collector, and rotational speed of collector. The flux of the membrane increased and the rejection of the pollution indices decreased with an increase in pore size. The portion of reversible fouling ratio and, consequently, the FR increased by decreasing the mean pore radius of ENMs. The noticeable point with the results is achieving a separation in the range of UF membrane separation while permeability is in the range of MF membrane. Also, another noteworthy result is that antifouling property of the prepared membranes is suitable.

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