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Hydrophilic polyethersulfone-based microfiltration membranes by electrospinning of polymer blends

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ABSTRACT

Polyethersulfone (PES) is considered one of the most popular polymers used for the fabrication of microfiltration membranes. In this study, we demonstrate the preparation of PES nanofibrous membranes via electrospinning technique for water purification. The amphiphilic polyethyleneoxide/polypropylenoxide multiblock copolymer (Tetronic 901) was used to improve the hydrophilicity of the relatively hydrophobic PES membrane. The water contact angle measurements confirmed the improvement of the hydrophilicity of the blended membrane surfaces. The effect of Tetronic additive on the morphology of the electrospun nanofibers as well as on the stability and performance of the prepared membranes was studied. The results indicated that the water permeability of the blended membranes was higher than that of original PES membrane. After membranes cleaning, the permeability loss decreased from 68% for PES membrane to only 34% for the 3 wt% Tetronic-containing PES membrane, indicating less fouling tendency of the polymer blend membranes.

Keywords: PES; Electrospinning; Microfiltration membrane; Tetronic; Hydrophilicity; Filtration

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1. Introduction

Microfiltration (MF) is a commonly used separation process for the removal of suspended solids, including large microorganisms such as bacteria and protozoa. The wide applications of MF (e.g., in dairy and pharmaceutical industries [1]) motivate the development of new materials to be used in manufacturing of MF membranes. The promising structural features of nanofibrous materials make them suitable for MF process. The high porosity of these materials implies a high permeability and the interconnected pores are able to compensate fouling to some degree. Besides, smaller pore size results in a higher retention. Consequently, water and air purification can be effectively achieved using nanofibrous membranes [2,3].

Different processing techniques are used to prepare nanofibrous membranes, such as template synthesis [4], drawing [5], self-assembly [6], phase separation [7] and electrospinning [8,9]. Among these methods, electrospinning is considered a suitable method for mass production of continuous nanofibers from various polymers and allows some control on fiber dimensions [10]. In electrospinning, a polymer solution

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or melt is subjected to electrostatic forces in an arrangement of electrodes to which high voltage is applied. One electrode is a capillary tip, which also delivers the polymer solution, and the second electrode (collector) is a plate. The electric field is extremely inhomogeneous due to this geometry. As a result, a charge separation is produced at the end of the capillary tip which accelerates the polymer jet towards the collector. Fiber formation occurs through evaporation of the solvent during the travel to the collector, while the strong acceleration leads to stretching and thinning of the jet producing fibers [11].

Many varieties of polymer nanofibers have been prepared by electrospinning techniques, such as polyvinyl alcohol (PVA), polyvinylidenfluoride (PVDF), polyethersulfone (PES), etc. [12–14]. Because of their thermal, chemical and mechanical strength as well as outstanding membrane forming properties, sulfone polymers (e.g., PES) have been used frequently for the fabrication of high-performance commercial MF membranes. However, these membranes still have low water flux caused by the hydrophobic properties of their materials [15,16]. Many investigations have confirmed that increasing membrane surface hydrophilicity could effectively increase the water flux and reduce membrane fouling. Consequently, many methods such as blending, adsorption, coating, as well as chemical and radiation-induced grafting, have been developed to modify membrane surfaces using hydrophilic modifiers [17,18]. Poly(ethylene oxide) (PEO) has been commonly recognized as a kind of very effective surface modifier due to its high hydrophilicity and charge neutrality [19]. However, the main disadvantage of PEO is that the adsorbed polymer may not be able to retain on the surface permanently. Hence PEO-enriched surfaces with greater stability could be efficiently achieved by blending small amounts of PEO-containing block copolymers comprising hydrophobic anchor blocks into the hydrophobic polymer matrix [20]. Pluronic and Tetronic are typical commercial amphiphilic copolymers bearing hydrophilic PEO and hydrophobic polypropylene oxide (PPO) segments [21]. The hydrophobic PPO units in Pluronic or Tetronic ensure them to be firmly held in the polymer matrix, while the hydrophilic PEO segments provide the membranes surface with higher hydrophilicity [22]. In a previous work, it had been shown that Pluronic copolymers can efficiently modify PES membrane surfaces [23]. The branched architecture of Tetronics (Fig. 1) in which four arms of PPO–PEO blocks are linked to a central ethylenediamine-based group [24] could produce better blended membranes than the linear shaped Pluronics. Thus, Tetronics can enhance the surface hydrophilicity and, consequently, improve the membrane water permeability. Rahman et al. [25] reported the influence of the addition of Tetronic 1307 on the structure formation of hollow-fiber PES membranes prepared by nonsolvent induced phase separation method. The results showed that the addition of Tetronic was useful for improving the water permeability and for obtaining a hydrophilic membrane surface.

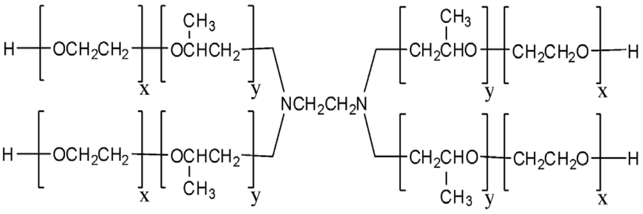
Fig. 1. General chemical structure of Tetronics.

To the best of our knowledge, preparation of PES MF membranes modified with Tetronic 901 copolymer using electrospinning technique has not been done before. Accordingly, PES/Tetronic 901 nanofibrous MF membranes were prepared using the electrospinning technique in this work. The membrane characteristics including membrane morphology, pore size distribution, surface hydrophilicity, chemical composition and membrane performance were investigated.

2. Experimental procedure

2.1. Materials

Polyethersulfone Ultrason E6020P (MW = 58,000 g mol^{-1} and density = 1.37 g cm^{-3}) was purchased from BASF (Germany). Prior to using the material, it was dried at 120°C for at least 4 h. Tetronic 901 block copolymer (TET; MW = 4,700 g mol^{-1}, PEO:PPO = 3:18) was purchased from BASF (Mount Olive, NJ, USA).

A poly(ethylene terephthalate) (PET) nonwoven (Type Novatexx 2429, Freudenberg Filtration Technologies SE & Co. KG, Weinheim, Germany) served as the substrate. *N*-methyl-2-pyrrolidone (NMP) was purchased from Merck (Hohenbrunn, Germany). Whey protein isolate (WPI) from Davisco Food Inc., USA. Milli-Q water (Millipore, Burlington, MA, USA) was used as model compound to assess the filtration performance of the prepared membranes.

2.2. Viscosity measurements

The viscosities of polymer solutions (PES and TET/PES) were measured by Physica MCR 301 rheometer (Anton Paar, Germany) with A CP25 – 2/TG cone-plate geometry. All viscosity measurements as a function of the shear rate were performed at 25°C.

2.3. Preparation of nanofibrous MF membranes by electrospinning

PES and TET/PES nanofibrous membranes were prepared according to the established electrospinning method for the preparation of unmodified PES nanofiber membranes [26]. Briefly, PES solution (22 wt% in NMP) was fed into a capillary needle (inner diameter = 0.8 mm) at a constant rate of 20 μL min^{-1} by means of a syringe pump (Harvard Apparatus, USA). The distance between the needle and the collector was set at 10 cm and the humidity was kept constant (RH = 65% ± 5%) during the preparation process. The voltage between the needle and the collector was adjusted at 18 kV using Heinzinger power supply. PES was electrospun on aluminum foil (as the control substrate) and PET nonwoven mesh. The modified TET/PES membranes were spun from PES/NMP solutions to which Tetronic 901 was added in concentrations of 3 and 5 wt% under the same electrospinning conditions.

2.4. Characterization of the prepared membranes

The surface morphologies of the membranes were characterized by scanning electron microscope (SEM; Quanta 400 FEG instrument (FEI)). K 550 sputter coater (Emitech, UK) was used to cover the outer surface of the sample with gold/palladium layer for enhancing the image quality. The diameters of the nanofibers were determined from the SEM images using the ImageJ software available online.

The average pore size and the pore size distribution of the prepared membranes were determined by the Capillary Flow Porometer CFP (34RTG8A-X-6-L4, PMI Inc., Ithaca, NY, USA) using the "dry up-wet up" method. The wetting liquid used was 1,1,2,3,3,3-hexafluoropropene ("Galwick," PMI; surface tension 0.016 N m^{-1}). The maximum transmembrane pressure for the air flow measurements was 3 bars. The pore size distribution was estimated using the PMI software and the measurements were repeated three times using new samples prepared under the same conditions.

The OCA15 Plus goniometer (Dataphysics GmbH, Filderstadt, Germany) and the sessile drop method were used to measure the contact angle of the membranes. A drop of water was placed on the surface to be studied followed by measuring the respective angle of contact according to the method described by Weirauch et al. [27].

The membrane surface chemistry was analyzed by Fourier transform infrared (FTIR) spectroscopy using the single beam FT-IR-430 (JASCO, Japan). FTIR spectra of the samples were obtained in the spectral range 4,000–400 cm^{-1}. The presented data is the average of 64 scans performed at a resolution of 4 cm^{-1}.

In order to evaluate the release of the modified membrane materials during washing with water, all membranes were immersed in 200 mL distilled water for 2 d; then the total organic carbon was measured for the used water by Teledyne Tekmar 15-030N-200 TOC analyzer.

All experiments were carried out by using a dead-end stirred cell (Amicon cell model 8010, Millipore Corporation) connected to a feed tank (4 L). Hydraulic permeability (i.e., amount of permeate that can be produced per m^2 of the membrane at certain pressure) was measured by filtration of pure water through 25 mm diameter membrane under a hydrostatic pressure (depending on the height of water tank feeding the filtration cell, that is, ≈0.2 bar) for 3 min. The permeability was measured for three different samples of each membrane.

The filtration performance of the membranes was tested using 2,000 ppm WPI solution because WPI exists in large amounts in dairy wastewater. WPI concentrations in the permeate were determined by measuring its UV absorbance at 280 nm [1]. The apparent WPI rejection was calculated using the following equation:

$$R(\%) = \left(1 - \frac{C_P}{C_t}\right) \times 100 \tag{1}$$

where C_p and C_t (mg L^{-1}) are the protein concentrations in the permeate and the feed solutions, respectively.

Water permeability was also calculated according to Eq. (2):

$$\text{Permeability} = \frac{V}{A \times t \times \Delta P} \tag{2}$$

where V is the volume of permeate (L), A is the effective area of the membrane (m^2), t is the filtration time (h) and ΔP is the transmembrane pressure. The permeability loss (%) was calculated as shown in Eq. (3).

$$\text{Permeability loss}(\%) = \left(1 - \frac{L_V}{L_C}\right) \times 100 \tag{3}$$

where L_v is the pure water permeability of the virgin membranes and L_c is the pure water permeability of the cleaned membrane after protein filtration.

3. Results and discussion

3.1. Membrane morphology and pore size distribution

Firstly, the changes in surface morphology of the membranes with increasing the Tetronic (TET) concentration were studied. Fig. 2 shows the SEM micrographs of PES, 3 wt% TET/PES and 5 wt% TET/PES membranes. All membranes showed relatively smooth nanofibers without any beads formation. The SEM image analyses demonstrated an average fiber diameter of 1090, 700 and 610 nm for PES, 3 wt% TET/PES and 5 wt% TET/PES membranes, respectively. One important parameter of the spinning solution is the viscosity, which influences jet formation and thus may affect fiber morphology. Measurements revealed that the change in the solution viscosity with the addition of TET was insignificant as shown in Table 1. However, the SEM analysis indicated that there is a distinct change in fiber diameter distribution. With increasing TET concentration, the variation decreases and fibers get more uniform. For pure PES fibers, the SEM images showed diameters of more than 2 μm. With increasing concentration of TET, the occurrence of these thick fibers decreased considerably. The reason behind this observation might be found in electric properties of the spinning solution. According to the data in Table 1, the solution conductivity increases by adding Tetronic. This increase in solution conductivity might be due to the increased water uptake caused by the hydrophilic polymer, Tetronic. The enhanced conductivity in case of TET/PES solution led to a lower diameter of the formed fibers because the radius of the fiber jet is inversely related to the cube root of the solution conductivity [28].

The pore size of the electrospun membranes is a factor for determining the filtration performance of the membranes, that is, smaller pore size leads to higher particle separation efficiency. Table 2 summarizes the average pore sizes of PES and TET/PES membranes. Since the fiber diameters decreased by the addition of Tetronic as shown in Table 1, it was expected that the pore sizes of the TET/PES membranes will be smaller than that of pure PES membrane. However, the results in Table 2 show that pore sizes for all prepared membranes are nearly the same, probably because the prepared electrospun nanofibers are not uniform which makes pore sizes less controlled.

3.2. Chemical structure of membranes

FTIR spectroscopy was used to obtain qualitative evidence of the Tetronic 901 blending in the matrix of PES membrane. Fig. 3 shows the obtained FTIR spectra. PES showed

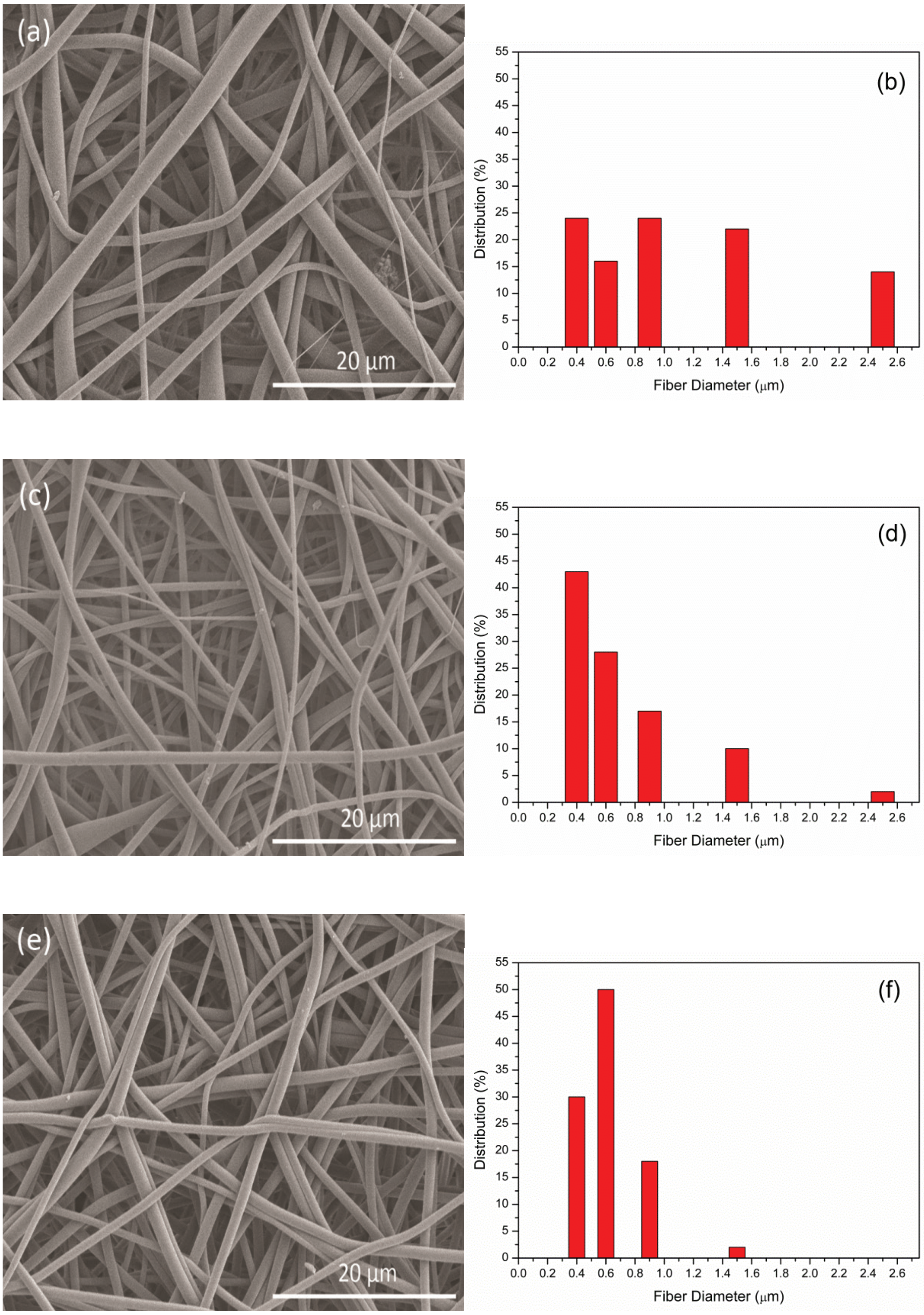

Fig. 2. SEM micrographs and diameter distribution of the electrospun nanofibers of (a,b) PES, (c,d) 3 wt% TET/PES and (e,f) 5 wt% TET/PES membranes, respectively.

three distinctive peaks, at 1,578 and 1,485 cm^{-1} originating from the benzene ring and aromatic C=C bond stretch, respectively, and the aromatic ether band at about 1,240 cm^{-1} [29]. The weak C=O band at 1,680 cm^{-1}, which appears in all samples, is characteristic for the residual NMP in the membranes. The characteristic peak of Tetronic (expected at a wavenumber of ~1,105 cm^{-1} [30]) was fully obscured by PES peaks due to the low concentration of Tetronic compared with PES. However, a significant increase in the transmittance at ~1,105 cm^{-1}, due to additional intensity of C–O bond stretch

Table 1
Viscosities and conductivities of PES and TET/PES solutions in NMP at room temperature and average diameters of the electrospun fibers

Spinning solution	Viscosity (Pa s)	Conductivity (μS/cm)	Fiber diameter (nm)
PES	4.18	1.5	1,090 ± 470
3 wt% TET/PES	4.08	4.3	700 ± 250
5 wt% TET/PES	4.11	6.2	610 ± 180

Table 2
Average pore sizes of the prepared membranes

Membrane	Average pore size (μm)
PES	3.4 ± 0.30
3 wt% TET/PES	3.6 ± 0.27
5 wt% TET/PES	3.3 ± 0.27

(in ether group of Tetronic) was observed. This confirms the presence of the additive in the membrane polymer matrix. No additional peaks were observed for the membranes prepared with the addition of Tetronic.

Table 3 shows the total organic carbon (TOC) content of the water (0.2 L) in which the prepared membranes were immersed for 2 d. It was observed that TOC of this washing water was low for all membranes, and these small amounts of TOC are probably from the residual NMP solvent which could be still present in the membranes. This assumption is supported by FTIR data. Based on these results, it can be demonstrated that the leaching out of Tetronic can be neglected indicating quite high stability of Tetronic inside the membrane polymer matrix. This is favorable for long-term and high-performance applications, which requires a good stability of the MF membranes.

3.3. Membrane surface hydrophilicity

The contact angle was measured to evaluate the hydrophilicity of membrane surface. Fig. 4(a) shows the static contact angles of the PES and the TET/PES membranes. The PES membrane prepared from pure PES solution showed the highest static contact angle of about 107°, whereas PES membranes with 3 wt% TET and 5 wt% TET contents showed contact angles of 71° and 69°, respectively. This decrease in the apparent contact angle reveals the enhancement in the hydrophilicity of PES membranes after the addition of Tetronic. The reason for this hydrophilicity is attributed to the presence of hydrophilic PEO units in the Tetronic molecule [21]. Furthermore, the water contact angles significantly changed with the contact time between the membrane and the water droplet as depicted in Fig. 4(b). The contact angles decreased with time indicating rapid droplet penetration into the membrane. The reduction in contact angle with the time is more significant in the both cases of 3 wt% TET/PES and 5 wt% TET/PES membranes than for the pure PES membranes. These results also support that the hydrophilicity of PES membrane was improved by the addition of Tetronic.

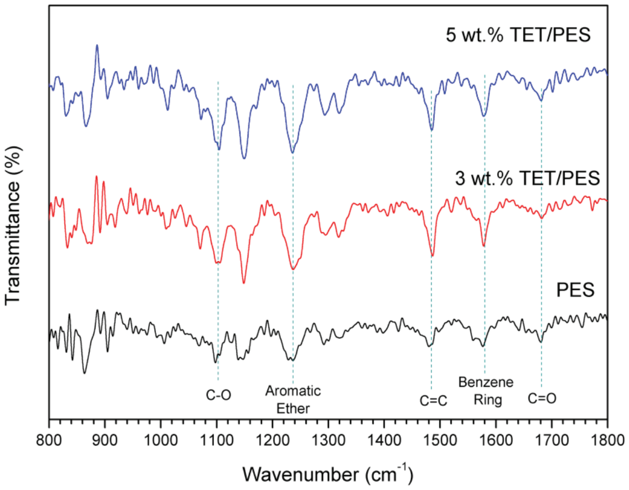

Fig. 3. FTIR spectra of the PES, 3 wt% TET/PES and 5 wt% TET/PES membranes.

Table 3
Masses of the membrane samples and TOC of the washing water of these samples

Membrane	Membrane weight (mg)	TOC content of washing water (mg L^{-1})
PES	72.5	0.42
3 wt% TET/PES	75.4	0.54
5 wt% TET/PES	78.5	0.5

3.4. Water permeability

Pure water permeability measurements were performed to demonstrate the permeability and structural stability of the prepared membranes. According to the data shown in Table 4, all membranes exhibited high permeability because of their large pore sizes which are in the known range for MF membranes. However, the water permeability of the TET/PES membranes was higher compared with that for the unmodified PES membrane. For instance, the permeability increased from 10,600 L m^{-2} h^{-1} bar^{-1} for PES to 26,200 and 25,700 L m^{-2} h^{-1} bar^{-1} for 3 wt% TET/PES and 5 wt% TET/PES, respectively. The increase in the permeability values in the case of TET/PES membranes was attributed to increasing the hydrophilicity [31] by addition of Tetronic which was proved from the contact angle measurement shown in Fig. 4.

3.5. Filtration performance of the nanofibrous MF membranes

Rejection of WPI by nanofibrous MF membranes was examined in order to evaluate the filtration performance of all membranes. 2,000 ppm of aqueous WPI suspension was prepared. Both water flux and WPI rejection of the membranes are shown in Fig. 5. As shown in the figure, the WPI rejection decreases from 79% for the unmodified PES membranes to 65% and 57% for 5 wt% TET/PES and 3 wt% TET/PES, respectively. In addition, the Tetronic modified membranes have higher flux if compared with the unmodified PES membrane. This behavior was expected because of the hydrophobic nature of the electrospun nanofibrous PES membrane. It can be fouled rapidly and cause more deposition of protein inside the pores and on the surface of the membrane, leading to a decrease in the water flux and an increase in WPI rejection. In case of blended membranes, the contact angles of 3 wt% TET/PES and 5 wt% TET/PES were nearly the same. Consequently, the increase in rejection in case of 5 wt% TET/PES membrane can be attributed to the slightly smaller pore size of this membrane (3.3 µm) compared with 3 wt% TET/PES (3.6 µm) according to the data presented in Table 2.

The disadvantage of PES membrane was the rapid fouling during filtration experiments. Measuring water permeability before and after filtration can give additional indication about the anti-fouling properties of these membranes.

Table 4 illustrates the pure water permeability of the virgin membranes and the cleaned membranes (the used membranes were immersed in distilled water with shaking for 20 min) along with the loss in permeability after usage. It was obvious that TET/PES membranes still have high water permeability and consequently low permeability loss compared with unmodified PES membranes. The addition of Tetronic plays an important role in decreasing the pore blocking tendency due to its high hydrophilic property. Therefore, the fouling of these membranes was lower compared with PES membrane without addition of Tetronic which fouled rapidly.

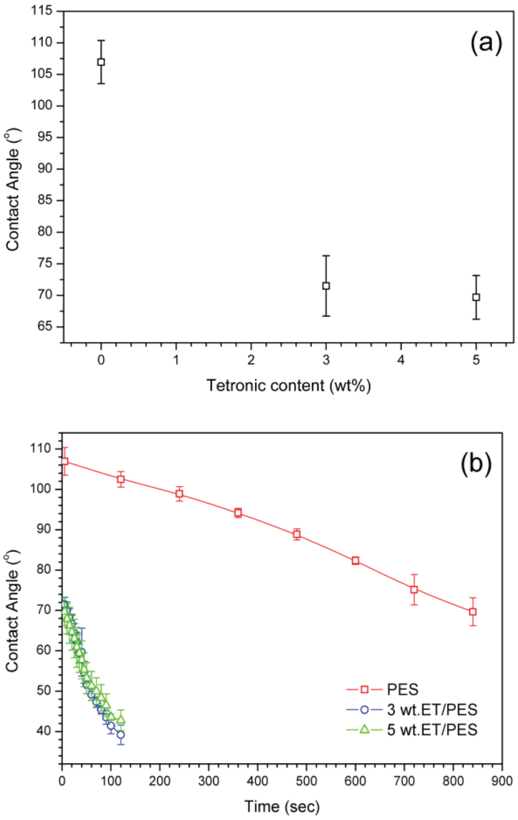

Fig. 4. Contact angle values of the prepared membranes (a) measured immediately after application of the water drop for the different membranes and (b) measured as a function of time.

4. Conclusions

High flux hydrophilic PES membranes were prepared by electrospinning technique of polymer solutions. Amphiphilic PEO-containing multiblock copolymer Tetronic 901 was chosen as modifier agent due to its stable incorporation within the membrane polymer matrix. FTIR analysis confirmed the presence of Tetronic due to the increase in the intensity of C–O bond at 1,105 cm^{-1}. The low values of TOC demonstrated the high stability of Tetronic inside the membrane polymer matrix. The contact angle measurement revealed that blending Tetronic block copolymer with PES increases the hydrophilicity of the membranes. The observable increase in the water permeability was attributed to the increase in the hydrophilicity due to addition of Tetronic copolymer. The rejection of WPI in case of unmodified PES membrane was higher than for the Tetronic modified PES membranes due to the increased surface hydrophilicity in blended-membranes. Addition of Tetronic decreases the deposition of protein in/on the membranes pores; therefore, the rejection decreases. Such large pore size MF membranes can be used as pre-filters for particulate removal in water purification or other similar processes.

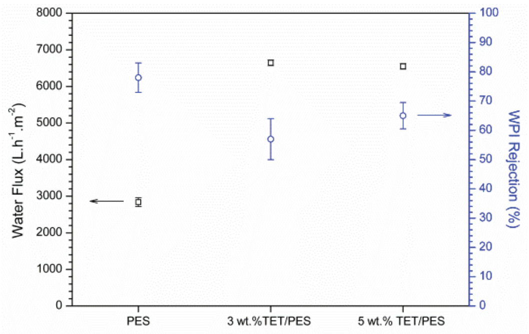

Fig. 5. Water fluxes at 1 bar and rejection of WPI.

Table 4
Pure water permeability loss for all membranes due to filtration of WPI solution

Membrane	Pure water permeability of virgin membranes (L m^{-2} h^{-1} bar^{-1})	Pure water permeability of cleaned membranes (L m^{-2} h^{-1} bar^{-1})	Permeability loss (%)
PES	$10.6 \times 10^3 \pm 5.0 \times 10^2$	$3.55 \times 10^3 \pm 6.0 \times 10^2$	67
3 wt% TET/PES	$26.2 \times 10^3 \pm 4.0 \times 10^2$	$17.2 \times 10^3 \pm 1.1 \times 10^3$	34
5 wt% TET/PES	$25.7 \times 10^3 \pm 4.5 \times 10^2$	$16.0 \times 10^3 \pm 9.0 \times 10^2$	38

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