Preparation of Sulfonated Poly(ethylene terephthalate) Submicron Fibrous Membranes for Removal of Basic Dyes

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ABSTRACT: In this investigation, a nanofibrous sulfonated poly(ethylene terephthalate) (SPET) membrane was prepared by electrospinning of the SPET solution in trifluoroaceticacid (TFA)/dichloromethane (DCM) mixture. The produced nanofibers had diameter ranging from 300 nm to 1 μm. The performance of this membrane's separation process was evaluated under different operating conditions. The influence of the electrospinning and filtration process parameters, such as concentration, applied voltage, deposition time, operating pressure, and filtration time on rejection of C.I. Basic Blue 3 were studied. The dead-end recirculation ultrafiltration set-up was employed. Under optimum conditions (concentration of 20% (w/v) of SPET, applied voltage of 20 kV and deposition time of 3 h) the removal of the aforementioned dye was 98%. Operating pressure has posed a significant influence on the membrane's separation performance, whereas the operating time had some effect on the separation performance. Two equilibrium adsorption isotherms: Langmuir and Freundlich were fitted to the dyes' equilibrium sorption data on SPET membrane at different feed concentrations. The Freundlich isotherm was found to well represent the measured adsorption data based on the higher coefficient of determination (r^2). © 2012 Wiley Periodicals, Inc. J Appl Polym Sci 124: E190–E198, 2012

Key words: nanofibrous membrane; electrospinning; filtration; cationic dye; adsorption isotherms

INTRODUCTION

Textile, paper, carpet, leather, and printing industries produce large volumes of wastewater polluted with dyes.[1–3] Among all dyeing processes, direct dyes on viscose and basic dyes on acrylic materials release the highest quantities of dye molecules in the wastewater.[4] Basic dye is a class of highly colored organic substances with positive charge delocalized throughout the chromophoric structure and has affinity towards textile materials which have negatively charged functional groups. Since these dyes are hardly degradable, also they resist to aerobic digestion and are stable to heat, light, and oxidizing agents; hence, wastewater containing basic dye is difficult to purify.[5]

There are various methods for removal of dye molecules from wastewater, such as ozonation,[6,7] biological treatment,[8,9] adsorption,[10–12] ion exchange,[13,14] coagulation,[15] membrane processes,[16–19] and, etc. Among all the mentioned methods, the membrane processes seem to be more effective than other methods.[20]

A membrane in quintessence acts as a barrier separating two discrete phases. Its main function is to differentiate species which come into contact within one phase (feed) and transport them across to the other (permeate). This process has been frequently carried out under a driving force such as pressure or concentration gradient. Once a membrane is created, two key factors define its performance: flux and selectivity. Selectivity is controlled by the surface properties of membrane which discriminates the type of species passing through it. Whereas flux expresses the rate at which species are transported across the membrane. These two factors are affected by the structural and morphological possessions of the membrane such as porosity, pore size and distribution, wettability, pressure drop across the membrane (transmembrane pressure), and thickness.[21]

Recently, pressure-driven membrane processes, such as reverse osmosis, nanofiltration, and ultrafiltration have been investigated for the treatment of dye containing industrial wastewater. In spite of the fact that dyes are completely removed, the flux of reverse osmosis is very low and the process is not cost-effective. Although nanofiltration does not attain the retention behavior compared to reverse osmosis, but its flux is found to be more acceptable for water reuse. However, the major disadvantage of

nanofiltration is the flux diminishment due to adsorption of organic compounds on the membrane surface.[22]

Porous polymeric ultrafiltration membrane manufactured by the classic method (phase immersion method) has its inherent restrictions, e.g., low flux and high fouling tendency. These phenomena are due to geometric structure of pores, the analogous pore size distribution, and undesirable macro-void formation across the whole membrane thickness.[23]

Nanofibrous membrane which made during an electrospinning method, offer unique properties such as high specific surface area, good interconnectivity of pores, high fluxes, and potential to incorporate active chemistry or functionality on nanoscale.[24]

A combined technique based on polymeric nanofibrous membrane through an ultrafiltration method and adsorption is a new process for dye wastewater treatment. Banat and Al-Bastaki[25] simultaneously used an integrated method of adsorption by means of activated carbon and ultrafiltration. The combined procedure attained better rejection of dyes than that, by ultrafiltration process alone.[26]

The chemical and physical properties of poly(ethylene terephthalate) (PET) can be changed upon modification with ionic groups such as sulfonic group.[27] Sodium sulfonated containing PET ionomers, i.e., polymers containing small amounts (2–3 mol %) of sulfonated units, are known for long time and have been originally commercialized by DuPont as textile fibers. The sulfonated PET fibers are of lower strength, but the anionic nature of these side groups provide desirable binding sites for basic dyes.[1]

In this article, nanofibrous sulfonated poly(ethylene terephthalate) (SPET) membrane was prepared and applied through a dead-end recirculation ultrafiltration process to treat dye containing wastewater.

Under operating conditions, the SPET membrane which became negatively charged, could be attached to cationic dyes due to electrostatic attractions. Consequently, dye molecules can be rejected through an adsorption-filtration process. The removal of C. I. Basic Blue 3 was investigated to evaluate the SPET nanofibrous membrane performance.

EXPERIMENTAL

Electrospinning process

Five different concentrations (20, 22, 24, 26, and 28% w/v) of SPET (Shaoxing Global Chemical Fiber, China) solutions were prepared in trifluoroaceticacid (TFA)/dichloromethane (DCM) mixtures (70 : 30 v/v). The solvents were purchased from Merck (Germany). A syringe pump (Terumo, Japan) (traverse speed: 4 cm/min) was utilized to supply a constant

flow rate of 0.263 mL/h polymer solution during electrospinning. A voltage of 20 kV (Emersun, China) was loaded to draw the nanofibers from the needle (diameter: 0.5 mm). The fibers were collected on a rotated drum (diameter 7 cm, rotating speed: 70 rpm) covered with polyurethane mesh which coated by activated carbon particles. The distance between the collector and the needle was 15 cm. Electrospinning method was conducted for the concentration of 20% at three different voltages (14, 17, and 20 kV) and five deposition times (2, 3, 4, 5, and 6 h).

Membrane characterization

The morphology of electrospun fibers was observed using a scanning electron microscopy (SEM) (Philips, Netherland) and the diameter of nanofibers was determined from SEM image using the Measurement software. At least 100 nanofibers were chosen within each sample layer and their diameters were compared with image's scale. The average of the results was used as the nanofibers diameter. The thickness of the membrane was measured using a thickness variation indicator.

The porosity of the membrane was determined by using the bulk density of SPET pellet and the membrane density $(1 - \rho_{e\text{-spun}}/\rho_{pellet})$.[28] The density value (1.27 g/cm^3) of SPET pellet used in this experiment was measured by densitometer, (METTLER TOLEDO PR503, Germany). The electrospun membrane density was calculated by measuring the membrane size, thickness, and the mass of the membrane. Before any measurements, all samples were dried under vacuum at room temperature until the masses become constant.

Experimental set-up

Figure 1 shows a schematic diagram of the experimental set-up. In the set-up, the circulated dead-end process was selected. In this method the feed (1)
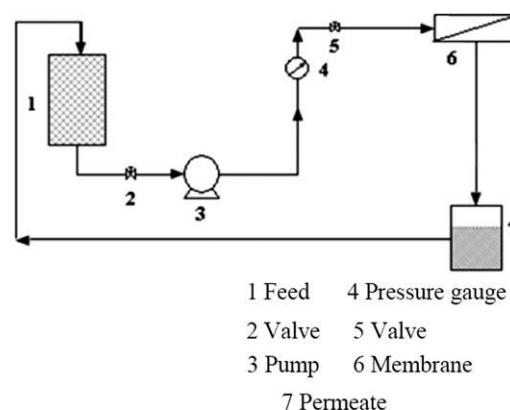

1 Feed	4 Pressure gauge
2 Valve	5 Valve
3 Pump	6 Membrane
7 Permeate	

Figure 1 Schematic diagram of the experimental set-up.

was pumped (3) to the membrane cell (6) from the tank. The system consisted of the regulator valve (2) to control the applied pressure. The permeate flow (7) was taken out of the membrane and sent to the feed tank for the next circulation. This cycle has been repeated continuously for a determined time of filtration process. The membrane cell consisted of a lid and a base, which the membrane with the area of 40 cm^2 put between these two parts. There were two ducts connected to two hoses on the lid part for feed inlet and one hole in the bottom of station part under the membrane to exit the permeate flow out of the cell.

The membrane settled on an aluminum honey comb layer to protect against deformation under pressure of water. Lid part and base part of the cell were linked together with eight screws to prevent the leakage of wastewater solution. An O-ring was placed between two parts of the cell. In the flow line there was a pressure gauge (4) (0–10 bar) to show the applied pressure of filtration process. The tank was a plastic vessel with the capacity of 5 L.

Filtration procedure

Before starting the experiments, the membrane was pretreated with double distilled water at higher pressure (2 bar) for 10 min to provide required hydrophilicity for passage of water through the membrane.

Filtration experiments were performed using Estrol Turquoise Blue N-G dye solution and the solute concentration was measured using UV–visible spectrometer (Carry 300) at 655 nm wavelength of light. The separation experiments were conducted at four different pressures (1.2, 1.5, 1.6, and 1.7 bar) and five operating time (60, 100, 140, 180, and 220 min).

All experiments were performed at pH value of 4.3 at 25°C. Estrol Turquoise Blue N-G is a basic dye of mono-oxazines class and is highly water soluble.

Permeate flux was obtained by measuring the volume every 5 min and can be calculated by eq. (1):

$$\text{Flux} = \frac{V}{St} \tag{1}$$

Where V is the volume of permeate, S is the effective area of the membrane, and t is the permeate collection time.

The rejection of the dye, R, is defined by eq. (2):

$$R\% = (1 - \frac{C_P}{C_0}) \tag{2}$$

where C_p and C_0 represent the dye concentration in permeates and feed, respectively.

RESULTS AND DISCUSSION

Electrospinning process parameters

The following variables including concentration of polymer solution, applied electric field, and deposition time have been examined. Their relationships with the dye rejection and membrane morphology are summarized below.

Effect of concentration

When polymer concentration increases the influence of the solution viscosity also increases. Under the same electrostatic forces and the other solvent properties being constant, this effect prevents the polymer jet from being stretched, resulting in fibers with larger diameters.[29]

The effect of polymer concentration on the membrane morphology was investigated for five different concentrations. The SEM images of the electrospun membranes from the SPET solutions of different concentrations are shown in Figure 2.

The average diameters of the SPET nanofibers ranged from 308 to 1043 nm (Table I), depending on polymer concentration. Increasing SPET concentration leads to an increase in fiber uniformity and higher regular morphology. This effect is mainly attributed to the increase in solution viscosity. The solution viscosity increases from 94.5 cP for the concentration of 20% w/v to 635 cP at the concentration of 28% w/v. It opposes to the surface tension effect acting against the interface increasing. At the concentrations less than 20%, droplet and beaded fibers were observed. The ratio of solvents was selected with considering the formation of finer, more uniform flattening electrospun fibers.[30] On the other hand, bonding between fibers that could be expected from the solvent with higher dielectric constant (TFA = 42.1 and DCM = 8.9) and lower surface tension (TFA = 13.4 and DCM = 27.2 ($\times 10^3$ J/m^2)) was also considered.

It was observed from Figure 3 that the rejection of dye underwent a slight reduction between concentrations of 20 and 26%. From this point onwards, it increased slowly to just above 50% in the concentration of 28%. In contrast, the membrane porosity decreased steadily from 86.2% to 73.2%, between the concentrations of 20 and 28% (Table I). The fiber fineness is one of the most important concerns for the nanofibrous membrane performance.[31] In general, due to the very high surface area to volume ratio (specific area) and the resulting high surface cohesion in finer fibers, tiny particles can be easily trapped in the electrospun nanofibrous filters and hence the filtration efficiency can be improved. Unpredictably, with decreasing the porosity with concentration, a significant fall was observed in
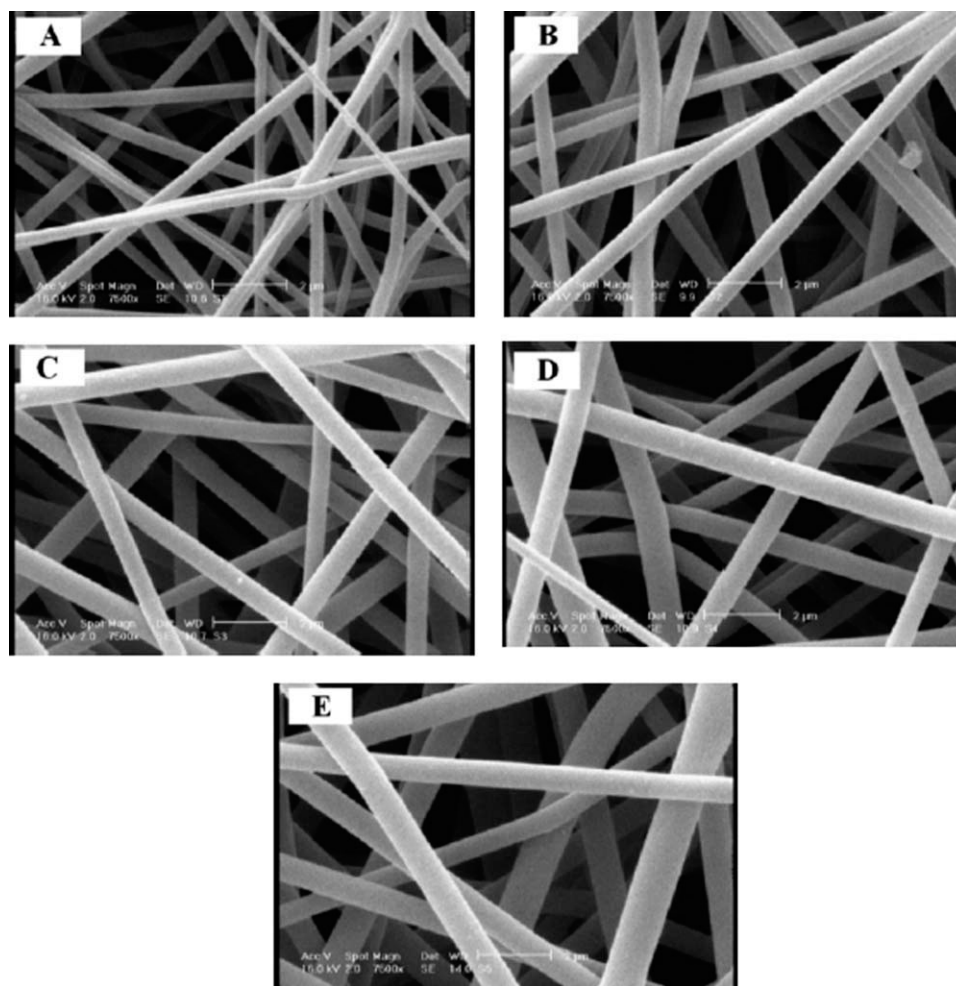
Figure 2 SEM images (magnification 7500×) showing the morphology of fibers electrospun from SPET solutions in TFA/DCM 70 : 30, at concentration of (A) 20%, (B) 22%, (C) 24%, (D) 26%, and (E) 28% (voltage of 20 kV, constant spinning distance of 15 cm, deposition time of 2 h).

rejection values. It could be explained from an increase in pores diameter as a result of lower specific area in higher concentrated membrane. Increasing the rejection from 26 to 28% was perhaps due to

formation of dead-end pores in the primary layers of electrospun membrane as a result of increasing the fibers thickness.[32] Rejection of 85% was obtained for concentration of 20% so that this concentration could be selected for other experiments as well.

Effect of applied voltage

The jet formation is a self-accelerating procedure. As soon as an electric field is applied on the droplet of the polymer solution at the tip of the needle, the surface of the liquid becomes charged via the movement of ions through the liquid. When the electric field is high enough to overcome the forces related with the surface tension, a quasi-stable, straight, and electrically charged jet is ejected. The balance between the surface tension and the electric force is critical to determine the primary cone shape of the polymer solution at the tip of the needle and fiber fineness. To study this effect, the applied voltage was increased from 14 to 20 kV while keeping a fixed feed rate (0.263 mL/h) and deposition time of
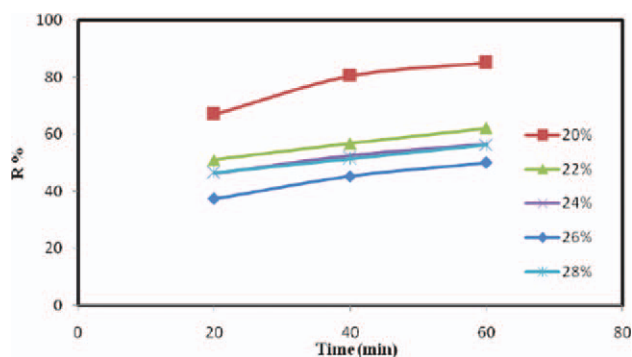
TABLE I
The Variations of General Characteristics of Nanofibrous Membrane with Polymer Concentration (Membrane Thickness: 28 μm, Applied Voltage: 20 kV)

Polymer concentration (% w/v)	Average diameter (nm)[a]	CV%[b]	Membrane density (g/cm³)[c]	Porosity (%)
20	308	25	0.17	86.22
22	469	19	0.23	81.88
24	657	18	0.25	80.31
26	785	26	0.30	76.37
28	1043	18	0.34	73.22

[a] Average values of one hundred measurements.
[b] The coefficient of variation (ratio of the standard deviation to the mean).
[c] Measured for electrospun mats with similar thickness. The deposition time was controlled to get SPET membrane with different thickness.

Figure 3 Effect of SPET concentration on rejection of dye (membrane thickness: 28 μm, pressure: 1.2 bar, filtration time: 1 h, dye concentration: 0.019 g/L). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

2 h. The average fiber diameter calculated from 100 measurements changed from 470 to 308 nm (Table II) and this indicates that the fiber diameter slumped as the voltage increased.

As shown in Figure 4, with increasing the voltage, charge accumulation on the fibers becomes significant and this improves repulsion between the fibers. Consequently, it will change the trend in fiber arrangement as revealed by SEM images. Barhate et al.,[33] studied the effect of electric field strength on the structure of polyacrylonitrile nanofibrous membrane and observed that the permeability and pore size decreased as the electric field strength increased. They also indicated that an enhanced drawing rate as a result of increasing the applied

TABLE II
The Variations of Fibers Diameter with Applied Voltage

Applied voltage (kV)	Average diameter (nm)[a]	CV%[b]
14	470	23
17	360	28
20	308	25

[a] Average values of one hundred measurements for electrospun mats with similar thickness.
[b] The coefficient of variation (ratio of the standard deviation to the mean).

voltage would increase the number of fiber crossings. Therefore, the high extent of fiber crossing would reduce the pore size and improve the interconnectivity of pores. Regardless of the above-mentioned effects, increasing the applied voltage will cause the nanofibers to be arranged more randomly and irregularly. These phenomena increase the possibility of contact between the dye molecules and fiber walls. Consequently, the dye adsorption can be improved with applied voltage (Fig. 5). The best rejection (85%) was reached for applying voltage of 20 kV.

Effect of deposition time

Membrane thickness is one of the effective membrane parameters on dye rejection. In nanofibrous membrane, membrane thickness is in principle determined by electrospinning time (deposition time). To study this effect, the deposition time was
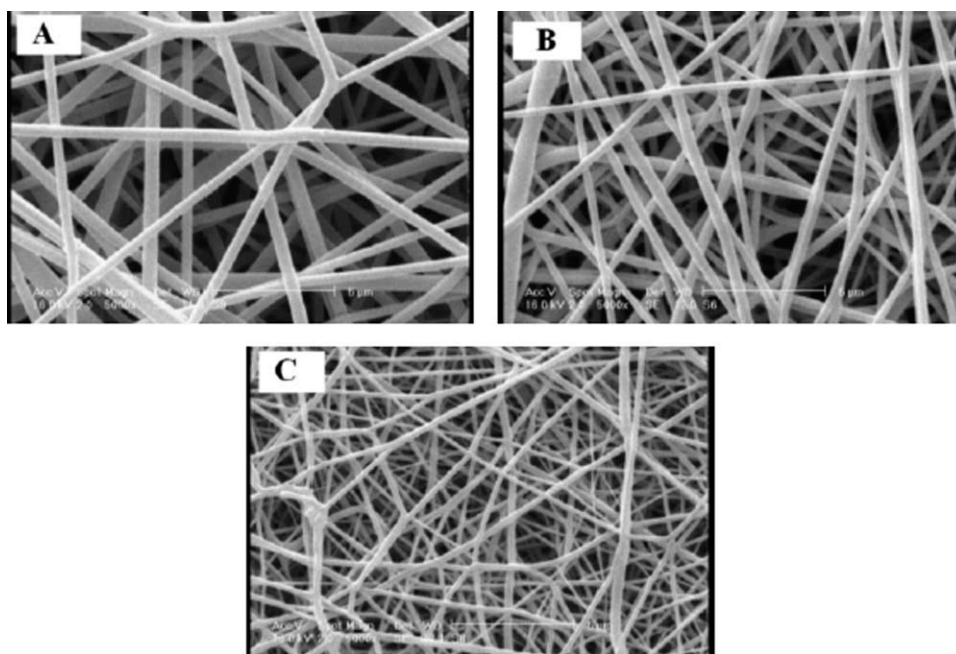

Figure 4 SEM images (magnification 5000×) showing the morphology of fibers electrospun from SPET solutions in TFA/DCM 70 : 30, at voltage of (A) 14 kV, (B) 17 kV, (C) 20 kV, (concentration of 20%, constant spinning distance of 15 cm, membrane thickness of 28 μm).
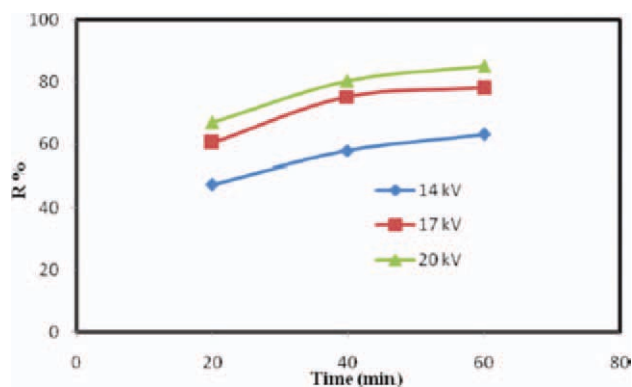
Figure 5 Effect of applied voltage on rejection of dye (membrane thickness: 28 μm, pressure: 1.2 bar, filtration time: 1 h, dye concentration: 0.019 g/L). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
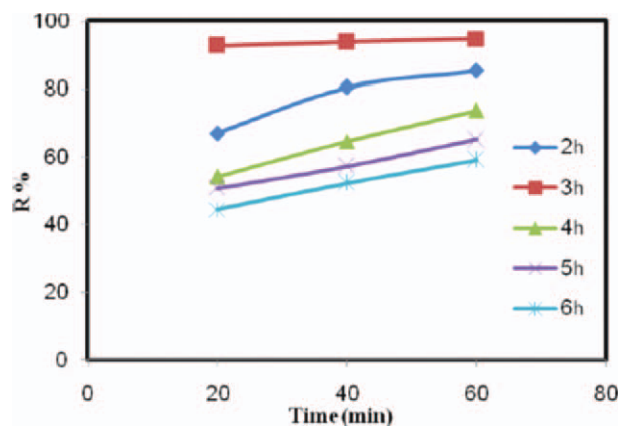

Figure 6 Effect of deposition time on rejection of dye (polymer concentration: 20%, filtration time: 1 h, dye concentration: 0.019 g/L). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

increased from 2 to 6 h and five membranes with different thicknesses were obtained. Each membrane was used at a defined applied pressure (minimum pressure for the solution passing through the membrane) for 1 h (Table III). Increasing the membrane thickness from 28 to 40 μm, accompanied with higher density of membrane layers causes dye molecules rejected up to 98% (Fig. 6). It could be explained in terms of larger extent of electrostatic forces between dye molecules and sulfonated groups. A sudden drop in dye rejection was observed at the deposition time greater than 4 h. This may be explained as follows: although the electrostatic forces and membrane density will increase with deposition time, higher applied pressures for thicker membranes are required. It also needs to be emphasized that each membrane was used at its minimum pressure. For commercial applications, the 3-h electrospun membrane is recommended.

Filtration process parameters

Effect of operating pressure

Figure 7 shows the effect of applied pressure on dye rejection during 1 h filtration. It could be observed that, the dye rejection was greater at lower pressure, in qualitative disagreement with the classical (Spiegler-kedem) convection/diffusion model. The maximum rejection of 85% was obtained at minimum applied pressure. Increasing the pressure, decreases the electrostatic forces between sulfonated mem-

brane and cationic dyes and causes the dye molecules to pass through the membrane and enter to the next cycle.

Effect of operating time

It was observed from Figure 8 that for SPET membrane the R% increased dramatically from 67% for the first sampling to 80.5%, 20 min later. From this time onwards, it underwent a slight rise to 90% at the end of 210 min filtration. Analyzing the obtained results call for comparing them to a nonadsorbable membrane such as PET. Therefore, some conditions were provided under which the PET nanofibrous membrane were prepared. The prepared membranes bear a striking resemblance to SPET ones in fibers diameter and membrane thickness (average fiber diameter of 317 nm and membrane thickness of 28 μm). In PET membrane with increasing the operating time, the rejected dye molecules on membrane
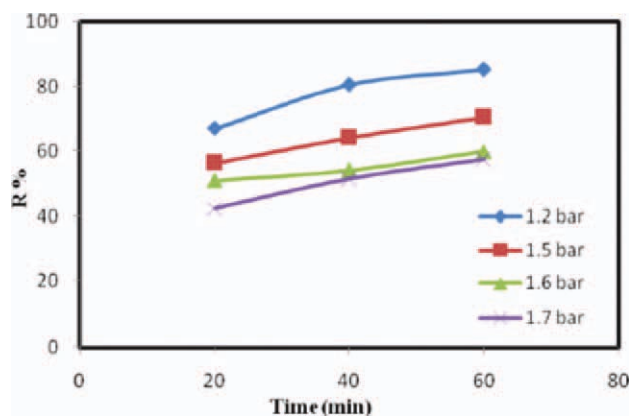

Figure 7 Effect of pressure on rejection of dye (polymer concentration: 20%, deposition time: 2 h, filtration time: 1 h, dye concentration: 0.019 g/L. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
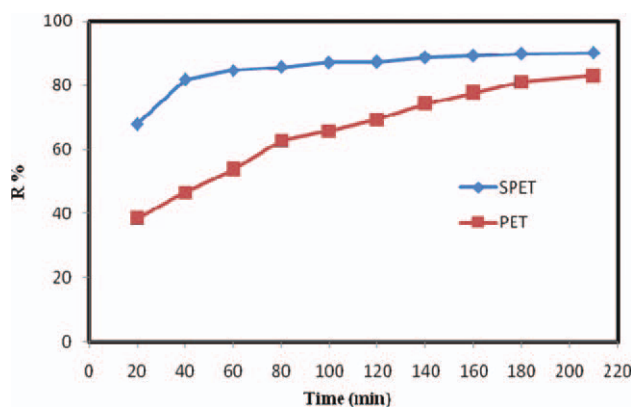
TABLE III
Effect of Deposition Time on the Membrane Thickness

Deposition time (h)	2	3	4	5	6
Membrane thickness (μm)	28	40	56	68	76
Applied pressure (bar)	1.2	1.25	1.37	1.5	1.5

Figure 8 Effect of operating time on rejection of dye in SPET membrane (deposition time: 2 h) and PET membrane (deposition time: 3 : 30 h) (polymer concentration: 20%, applied pressure: 1.2 bar, dye concentration: 0.019 g/L). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

surface formed a gel layer [Fig. 9(A,B)]. This layer may operate as an extra resist to permeate and increase the dye rejection with filtration time.[34]

By contrast in SPET membrane, first of all dye molecules are absorbed by electrostatic attractions. But it is not the end of it, from then, dye rejection could occur through the membrane pores and layers [Fig. 9(C,D)]. Generally, operating time has little influence on the membrane separation performance. With regard to the fact that every filtration process should be economically useful, it was concluded that

for commercial applications, 1-h filtration is the best alternative for operating time.

Adsorption isotherm

Existing dye molecules in the solution in contact with the membrane are assumed to be in quasi-equilibrium with the dye molecules in the adsorbed phase which is created on the membrane during filtration. Practically, a very long time is required to establish a real thermodynamic equilibrium in the system. Therefore, the equilibrium parameters of two adsorption isotherms; Langmuir, Freundlich, were determined using the experimental rejection and flux data.

The Langmuir adsorption isotherm[35] is extensively used for adsorption of dye molecules from liquid solution. The Langmuir equation is represented by eq. (3):

$$Q = Q_S \cdot \frac{kC_e}{1 + kC_e} \tag{3}$$

where Q is equilibrium adsorbed quantity per dry membrane mass (mg/g), Q_s is the final adsorbed quantity (mg/g), C_e is the adsorbate equilibrium concentration in solution (g/L) and k is a constant expressing the affinity of the membrane for the dye molecules.

The Freundlich isotherm is employed to explain multilayer adsorption with interaction between molecules on a heterogeneous sorbent surface. This isotherm can be expressed by eq. (4):
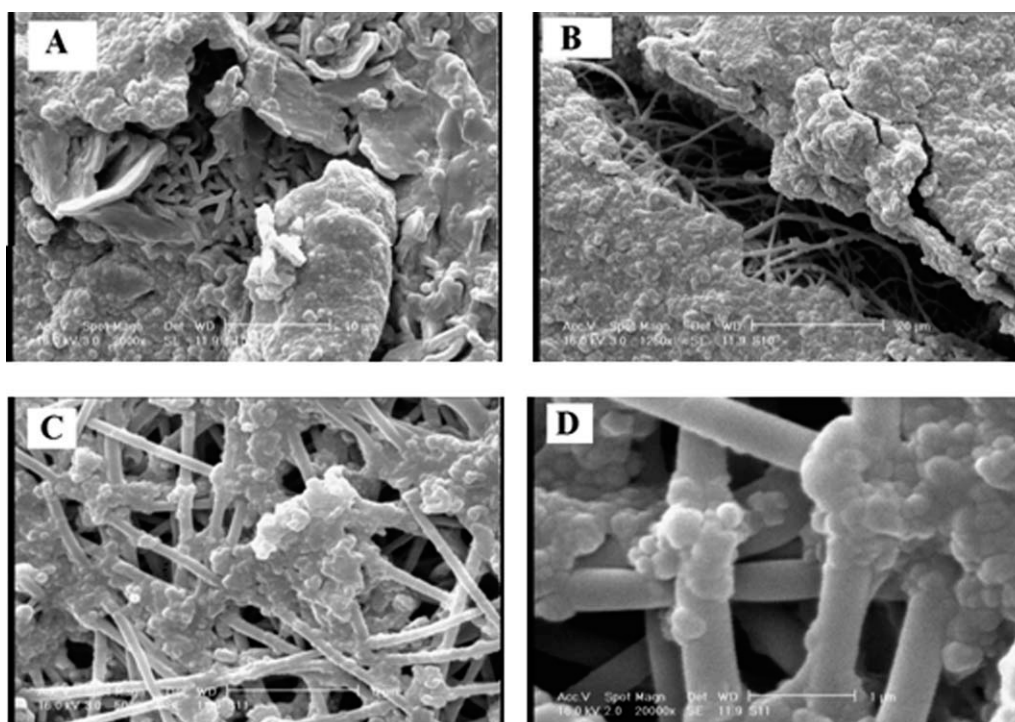

Figure 9 SEM images showing the nanofibrous membrane after filtration process with different magnifications : PET membrane (A) 2000×, (B) 1250× and SPET membrane (C) 5000×, (D) 20,000×.
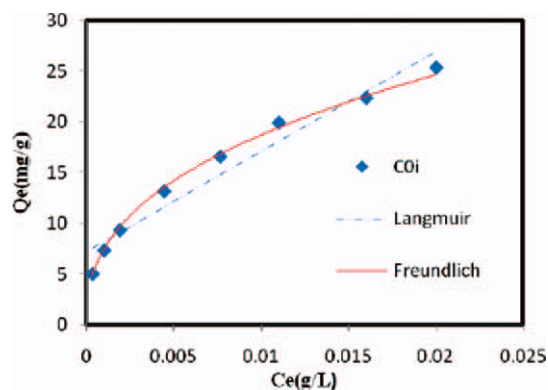
Figure 10 Isotherms for sorption of basic dye using SPET membrane (polymer concentration: 20%, deposition time: 2 h, applied voltage: 20 kV, operating pressure: 1.2 bar, filtration time: 210 min, dye concentration: 0.0095, 0.01444, 0.019, 0.0285, 0.038, 0.0475, 0.057, and 0.0665 g/L). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

$$Q = k.Ce^m \qquad (4)$$

where Q is sorption at equilibrium (mg/g), Ce is the remaining dye concentration at equilibrium (g/L), k is the relative sorption capacity (L/g), and m is an indicator of sorption intensity or surface heterogeneity. The value of m indicates the degree of nonlinearity between solution concentration and adsorbed phase as follows: if m is equal to unity, the adsorption is linear; if the value is below unity, this implies that the adsorption process is chemical and the surface is relatively homogeneous; if the value is above unity, adsorption is a physical process and the sorbent is relatively heterogeneous.[36]

Filtration was performed for eight different feed concentrations (C_0), to achieve thermodynamic equilibrium.

C_{ei} (the residual dye concentration at equilibrium for ith feed concentration) was calculated by measuring the dye concentration of permeate after 210 min filtration process for each C_{0i} using spectrometer at maximum wavelength of 655 nm. The concentration of absorbed dye (C_{ai}) is presented by eq. (5):

$$C_{ai} = C_{0i} - C_{ei} \qquad (5)$$

By using the first point where the adsorbed dye became constant at its third decimal digit with time and unit mass of membrane (M_m), Q_{ei} (mg/g) was calculated for each feed concentration by eq. (6):

$$Q_{ei} = \frac{m_{ai}}{M_m} \qquad (6)$$

where m_{ai} is quantity of dye adsorbed on the membrane at equilibrium for ith C_0.

Figure 10 depicts the isotherms fitting results for dye molecules at eight different feed concentrations when the equilibrium was obtained.

The results indicate that the two parameter, Freundlich isotherm with the higher coefficient of determination (r^2) (r^2 of the Freundlich isotherm: 0.997, r^2 of the Langmuir isotherm: 0.949) is the isotherm choice. In summary, the adsorption of basic dyes on SPET membrane is multilayer and the m-value of the Freundlich isotherm ($m = 0.3893$) is below unity. This means that the adsorption process is chemical due to the electrostatic forces between negatively charged sulfonated groups on the membrane and positively charged basic dyes. Freundlich isotherm is expressed by eq. (7):

$$Q = 117.13 \, C_e^{0.3983} \qquad (7)$$

CONCLUSIONS

SPET nanofibrous membrane was prepared and applied for removal of C. I. Basic Blue 3 dye from wastewater of textile industry. The results indicate that an excellent performance was achieved. The permeate flux was reached 5000 L/m^2 h at the lowest applied pressure (1.2 bar) for a polymer concentration of 20% with 2 h deposition time. The rejection of dye was up to 98% for similar concentration with 3 h spinning. The results show that the potential of dye rejection decreased with increasing polymer concentration in electrospinning solution and operating pressure. By contrast, it increased with increasing the applied voltage. The filtration time effect was negligible on the separation performance after 1 h filtration.

The Freunlich isotherms well described the sorption mechanism of the cationic dye molecules on the negatively charged membrane.

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