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Phosphate removal from water by polysulfone ultrafiltration membrane using PVP as a hydrophilic modifier

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ABSTRACT

The phosphate removal from aqueous solution was studied with a dead-end filtration process using ultrafiltration membranes. Polysulfone (PSf) ultrafiltration membranes were prepared by blending PSf with polyvinylpyrrolidone (PVP). The prepared membranes were characterized by water content, contact angle, SEM, EDX, AFM, and FTIR. The hydrophilicity and porosity of membrane improved considerably and water contact angle declined with the incorporation of PVP. While pure PSf membrane did not have any flux, PSf/PVP 3 wt.% (UF2) shows 9.6 L/m^2 h permeate flux. The impact of diverse operating parameters, such as PVP concentration, pressure, and pH of the feed solution on the removal of phosphate was examined. A high phosphate removal (93.6%) from aqueous solution was observed for UF2 membrane and 87.2% for UF3 membrane at feed solution pH 2. It was found that the phosphate rejection dropped as the pressure and PVP concentration increased. A reduction in pH of the feed solution gave a higher phosphorus rejection.

Keywords: Polysulfone; Polyvinylpyrrolidone; Ultrafiltration; Phosphate removal

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1. Introduction

Phosphorus is a noteworthy component for industrial and agricultural products and a vital supplement in the marine environment. Phosphates regularly exist in urban/industrial wastewater and groundwater. Civil sewage and municipal wastewater contain 1–5 mg/l of phosphorus, whereas industrial wastewater contains 10–50 mg/L of phosphate [1]. The presence of phosphate in surface water may prompt eutrophication. Thus, phosphate exclusion from wastewater is very important [2,3].

Reverse osmosis and nanofiltration (NF) membranes were used to separate the heavy metals and organic pollutants from water and it consumes more energy, i.e. high operating pressure than UF and MF membranes. Ultrafiltration (UF) is a low pressure-driven membrane separation method used for the removal of inorganic micropollutants. The technology of ultrafiltration (UF) membrane has tremendous applications in different industrial procedures, for examples, in pharmaceutical, food, biotechnological, seawater desalination, and pure water generation [4–7]. The porosity, hydrophilicity, mechanical strength, and surface roughness of UF membrane plays an important role in membrane separation

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process [8]. Polysulfone (PSf) is generally utilized as a material for the UF membrane preparation on account of its excellent offset among the chemical and mechanical properties [9,10]. However, the utilization of PSf membranes is limited because of its hydrophobicity. Hydrophobic properties of PSf membranes bring about low water flux and considerable membrane fouling. Hence, diverse strategies have been explored to produce hydrophilic PSf membranes, for example, blending with hydrophilic polymers [11–13] and grafting with other polymers [14,15]. Because of its simplicity, polymer blending is an appealing procedure for the outline of new PSf membranes [10–12].

Although there are numerous literatures reporting phosphate removal by nanofiltration and adsorption/ultrafiltration system, only a few researches reported the phosphate removal by flat sheet ultrafiltration membranes. Cathie et al. [16] accomplished more than 99% phosphate removal from a feed solution containing 120 mg/l of phosphate by means of NF90 commercial nanofiltration membrane. Zelmanov and Semiat [17] employed an adsorption/ultrafiltration framework for phosphate removal from aqueous solution. Phosphate concentration of less than 0.1 mg/l was achieved by this system, which is satisfactory for wastewater pretreatment. Muthumareeswaran and Agarwal [18] studied the retention properties of phosphate at various feed concentrations and to investigate the effect of pH on the rejection of phosphate ion through modified polyacrylonitrile (PAN) UF membrane. PAN UF membrane showed more than 90% rejection at pH 7 for feed solution containing 10 ppm of phosphate. Noordman et al. [19] prepared and examined the performance of ZrO_2 UF membrane for phosphate removal from water. The results proved that ZrO_2 UF membrane gave more than 90% rejection of phosphate ion. Upon further studies, Disha et al. [20] studied the potential of phosphate recovery by PEI/PSS bilayers ultrafiltration membrane. Their results showed that this particular combination of bilayers yielded high flux membranes that allowed selective removal of $H_2PO_4^-$ in the presence of Cl^- at low pressure (0.28 bar). For capacity extension of one of the drinking water production plants of Amsterdam water supply, four treatment schemes applying UF are considered by Hofman et al. [21]. The main purposes of the UF process are phosphate removal, removal of suspended solids and colloidal matter; and hygienic water quality improvement [21]. In this study, wet/wet-phase inversion technique between solvent (NMP) and nonsolvent (water) was applied for the preparation of PSf/PVP ultrafiltration membrane. Some literatures applied same method for the preparation of ultrafiltration membrane [22–25]. The aim of this work is to explore a new way of removing phosphate from aqueous solution through a dead-end UF system. Hence, PSf/PVP blend membranes were prepared and performance of membranes was studied in different PVP loadings, feed pH, and pressure.

2. Experimental

2.1. Materials and instrumentation

PSf (M_w = 22.000 g/mol) from Sigma-Aldrich Co. (USA) was selected as a membrane material because of its superior selectivity, high mechanical strength and notable thermal resistance [26]. N-methyl-2-pyrrolidone (NMP) and polyvinylpyrrolidone (PVP) with 25.000 g/mol molecular weight from Merck (Germany) were used as a solvent and nonsolvent additive, respectively. Disodium hydrogen phosphate (Na_2HPO_4) with acidic dissociation constant (pK_a) of 12.36 and pH of 8.5 was obtained from R & M chemical. In order to adjust the pH environment of the bulk solution to acidic or basic condition, 1 M hydrochloric acid (HCl) and sodium hydroxide (NaOH) were added. Deionized water was used for all experiments. All experiments were carried out twice at room temperature (25 ± 2°C) in a batch type and dead-end UF cell (Merck) with an effective membrane filtration area of 13.8 cm^2. Fig. 1 shows the schematic diagram of the dead-end UF system used for the phosphate removal experiment.

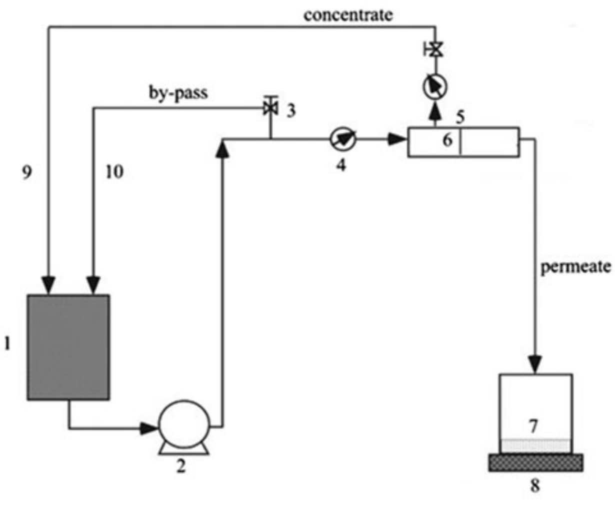

Fig. 1. Schematic diagram of the dead-end ultrafiltration system for phosphate removal from water.
Notes: (1) Feed tank, (2) pump, (3) valve, (4) pressure gauge, (5) dead end cell, (6) membrane, (7) permeate, (8) balance, (9) concentrate, and (10) by-pass.

Table 1
Preparation conditions of ultrafiltration membranes

	Casting solutions (wt.%)		
Membrane	PSf	NMP	PVP
UF0	18	82	0
UF1	18	81	1
UF2	18	79	3
UF3	18	77	5
UF4	18	75	7

2.2. Membrane preparation

In this research, pure PSf and PSf/PVP blend UF membranes were prepared by wet-phase inversion technique. Casting solutions containing 18 wt.% of PSf dissolved in NMP in the presence of four different concentrations of PVP (1, 3, 5, and 7 wt.%) were prepared by stirring for 24 h. The casting solution was cast on a glass plate by a casting knife with 80 μm thicknesses and then immersed into the coagulation bath of deionized water for immersion precipitation at room temperature. To ensure complete phase separation, the prepared membrane was stored in coagulation bath for 24 h. At the final stage, the membranes were dried at room temperature for 24 h. The composition of prepared membranes and their designation are listed in Table 1.

2.3. Feed

Ultrafiltration was carried out with 50 mg/L di-sodium hydrogen phosphate ($pK_a = 12.36$) at solution pH of 8.5 (initial pH), 7 and 2. The zeta potential of polysulfone membranes is near zero at pH 3 and become more negative with increasing pH [27]. Hence, the surface of polysulfone membrane is positively charged at $pH < 3$ and negatively charged at $pH > 3$ [27].

2.4. Membrane water content, porosity, and contact angle

The membrane water content was measured by soaking the membrane in water for 24 h and weighing it after wiping off with tissue paper. The membranes were dried in oven, at 40°C for 24 h and weighted. Based on wet and dry weights of the membranes, the water content was calculated using the following equation:

$$\%\ \text{Water content} = \frac{W_w - W_d}{W_w} \times 100 \tag{1}$$

where W_w and W_d are the weights of wet and dry membranes respectively.

The porosity of prepared membranes was calculated using the following formula:

$$\%\ \text{Porosity} = \frac{W_w - W_d}{dAL} \times 100 \tag{2}$$

where W_w is the weight of wet membrane, W_d is the weight of dry membrane, d is density of water (g/cm^3), A is the membrane effective area (cm^2), and L is the thickness of membrane (cm). The contact angle of membranes was measured using FTA-200 dynamic contact angle analyzer according to the sessile droplet method. In order to minimize the experimental error, the average of the three different locations is reported.

2.5. The measurement of membrane performance

The flux of feed solution was measured in different feed pressure of 1–3 bar. The following equation was used for calculation of the feed flux:

$$J_w = \frac{Q}{A \cdot \Delta t} \tag{3}$$

where J_w is the flux (L/m^2 h), Q is the amount of collected permeate (L) and Δt is sampling time (h).

The phosphate rejection of prepared membranes was calculated by following equation:

$$R = \left(1 - \frac{C_p}{C_f}\right) \times 100 \tag{4}$$

where C_p and C_f are the phosphate concentrations in permeate and the feed respectively.

3. Results and discussion

3.1. FTIR evaluation of membranes

The FTIR spectra of PVP, PSf, and PSf/PVP membranes are shown in Fig. 2. The peaks in the range of 1,650–1,670 cm^{-1} was detected for the C=O functional group in PVP. This peak discloses the existence of PVP in PSf/PVP blend membrane, which is not visible in PSf spectra. The asymmetric and symmetric stretching mode of sulfonate group (–SO_2–) in PSf caused two small peaks in PSf and PSF/PVP spectra at approximately 1,293 and 1,320 cm^{-1}. Moreover, a high intensity peak around 1,240 cm^{-1} was linked to C–O–C groups in PSf and PSf/PVP spectra [28].

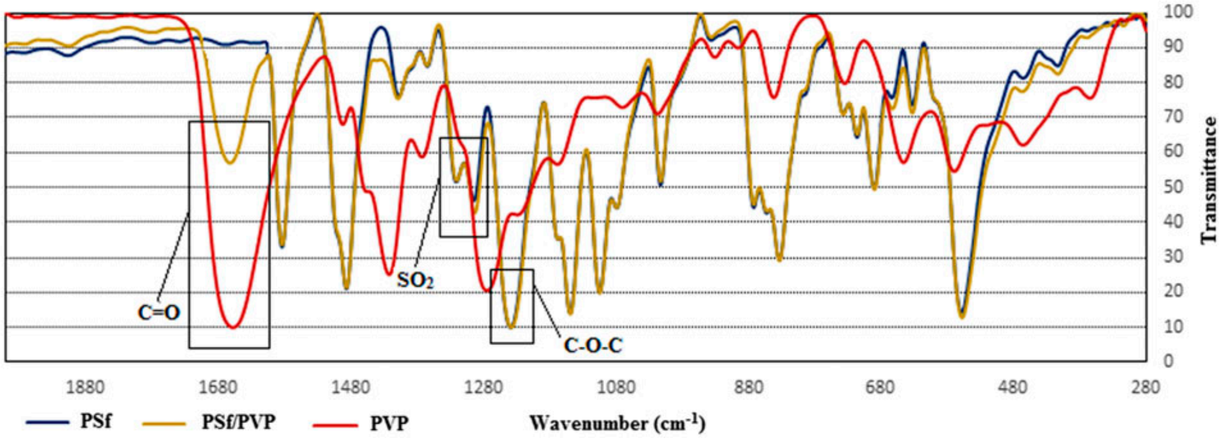

Fig. 2. FT-IR spectra of PVP, PSf, and PSf/PVP membranes.

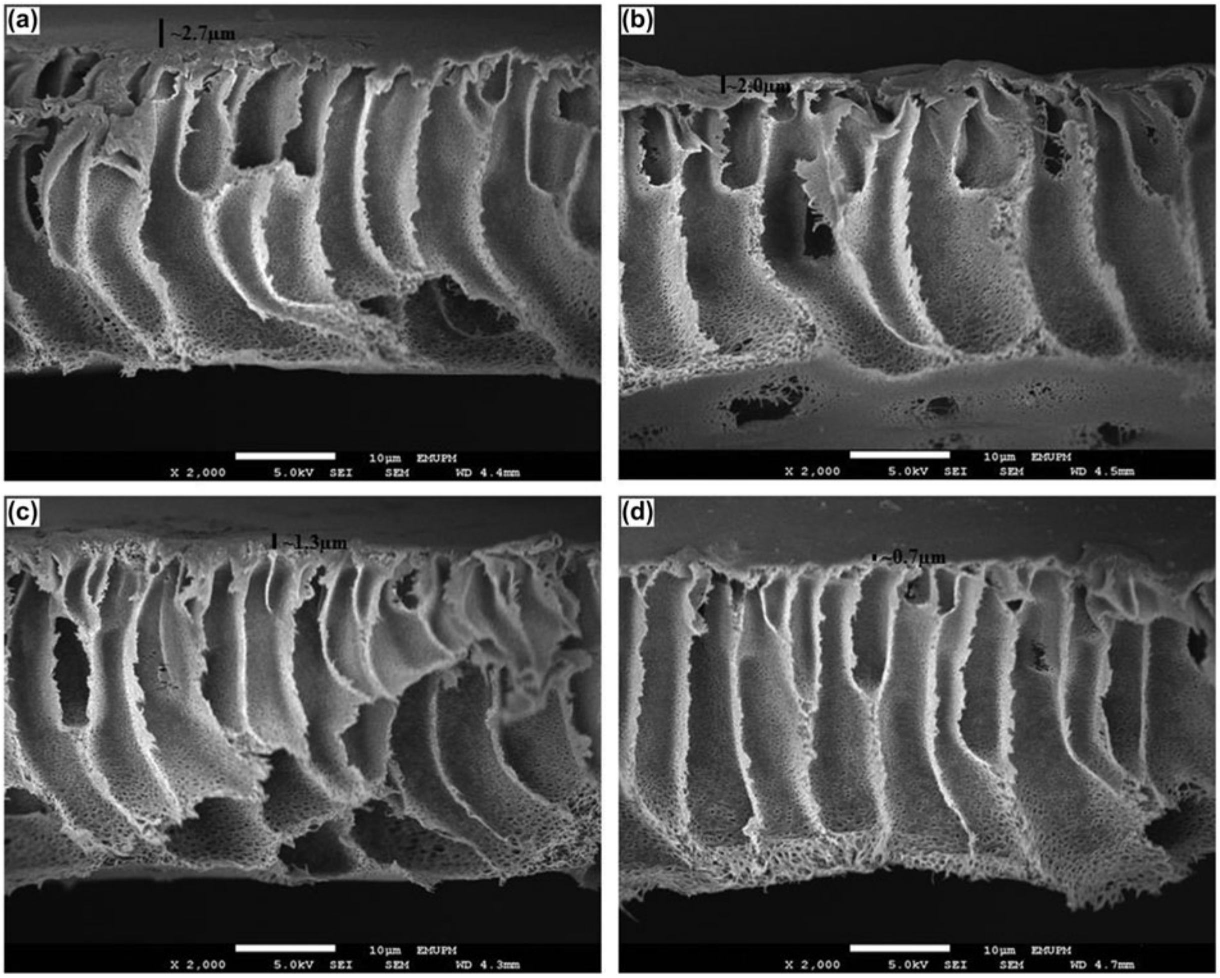

Fig. 3. SEM photographs of prepared ultrafiltration membranes (a) UF0, (b) UF2, (c) UF3, and (d) UF4.

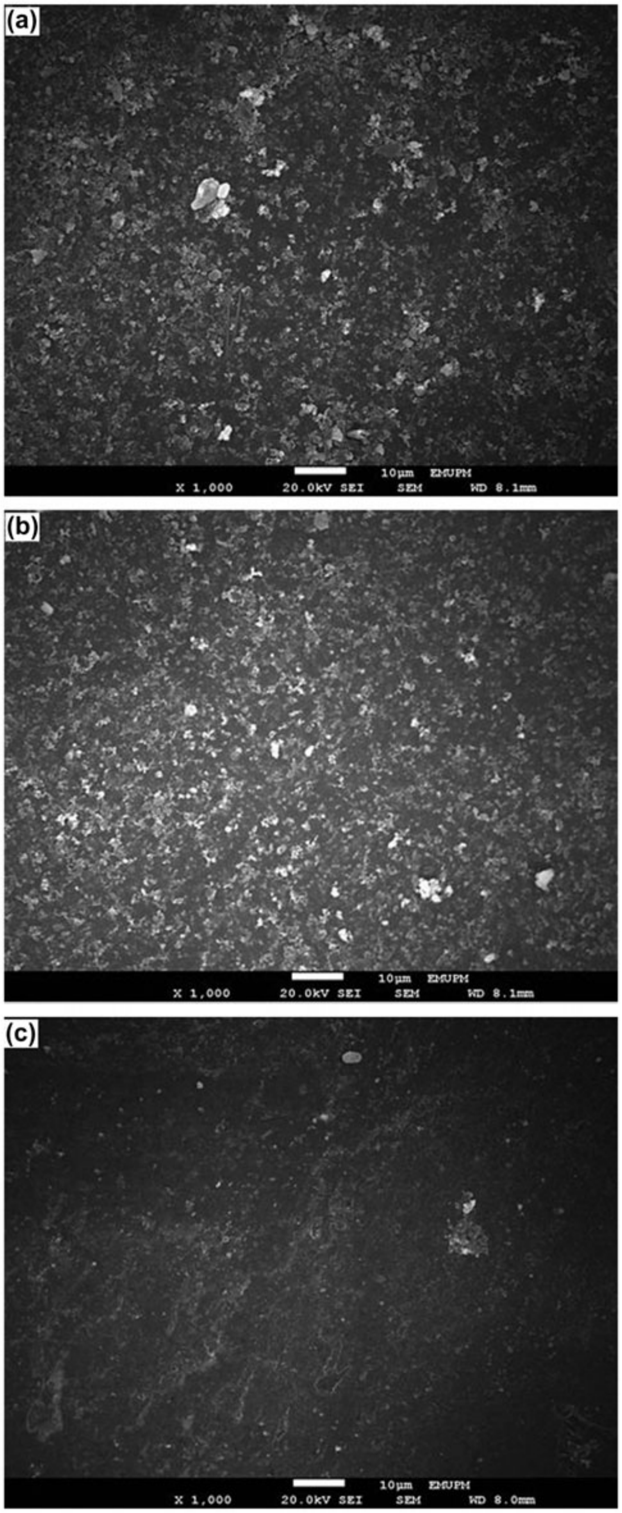

Fig. 4. Top layer analysis through FE-SEM on (a) UF0, (b) UF2, and (c) UF4.

3.2. SEM evaluation of membranes

The cross section views of prepared membranes with different PVP concentrations are shown in Fig. 3. As illustrated, all prepared UF membranes has finger like pore structure. The thickness of membrane surface layer decreased by addition of PVP to the casting solution in the following order: UF0 (~2.7 μm) > UF2 (~2.0 μm) > UF3 (~1.3 μm) > UF4 (~0.7 μm). Addition of PVP as a hydrophilic modifier, leads to instantaneous liquid–liquid phase demixing between solvent (NMP) and nonsolvent (water). Hence, membranes with thinner active layer were obtained in higher concentration of PVP [29,30].

Fig. 4 shows the variation of the top layer of the membranes by the addition of PVP to the casting solution. It was found that, in all prepared UF membranes, there is not any micropores or pinholes on the surface of membrane.

3.3. AFM evaluation of membranes

Atomic force microscopy was employed to analyze the surface morphology of prepared membranes such as the mean pore size and surface roughness parameters. The AFM apparatus was (Ambios Q-scope, Linthi-cum heights, MD). The membrane surfaces were examined in a scan size of 500 × 500 nm. The mean pore size and also surface roughness parameters of the membranes which are expressed in terms of the mean roughness (S_a), the root mean square of the Z data (S_q) and the mean difference between the highest peaks and lowest valleys (S_z) were obtained by SPM software (version 1.4.0.6). Fig. 5 illustrates the three-dimensional images of UF0, UF2, UF3, and UF4 membranes top layer. As cleared in the AFM images, the morphology of membrane top layer was significantly influenced by addition of PVP into the casting solution. The membrane roughness parameters and membrane mean pore size obtained through the AFM analysis are listed in Table 2. Based on AFM images, UF0 has a very smooth top layer and the mean pore size for this membrane was 6.30 nm. In contrast, the mean pore size as well as surface roughness parameters increased gradually by addition of PVP to the casting solution and as shown in Table 2, the UF4 blend membrane has the highest mean pore size of 11.14 among the prepared membranes.

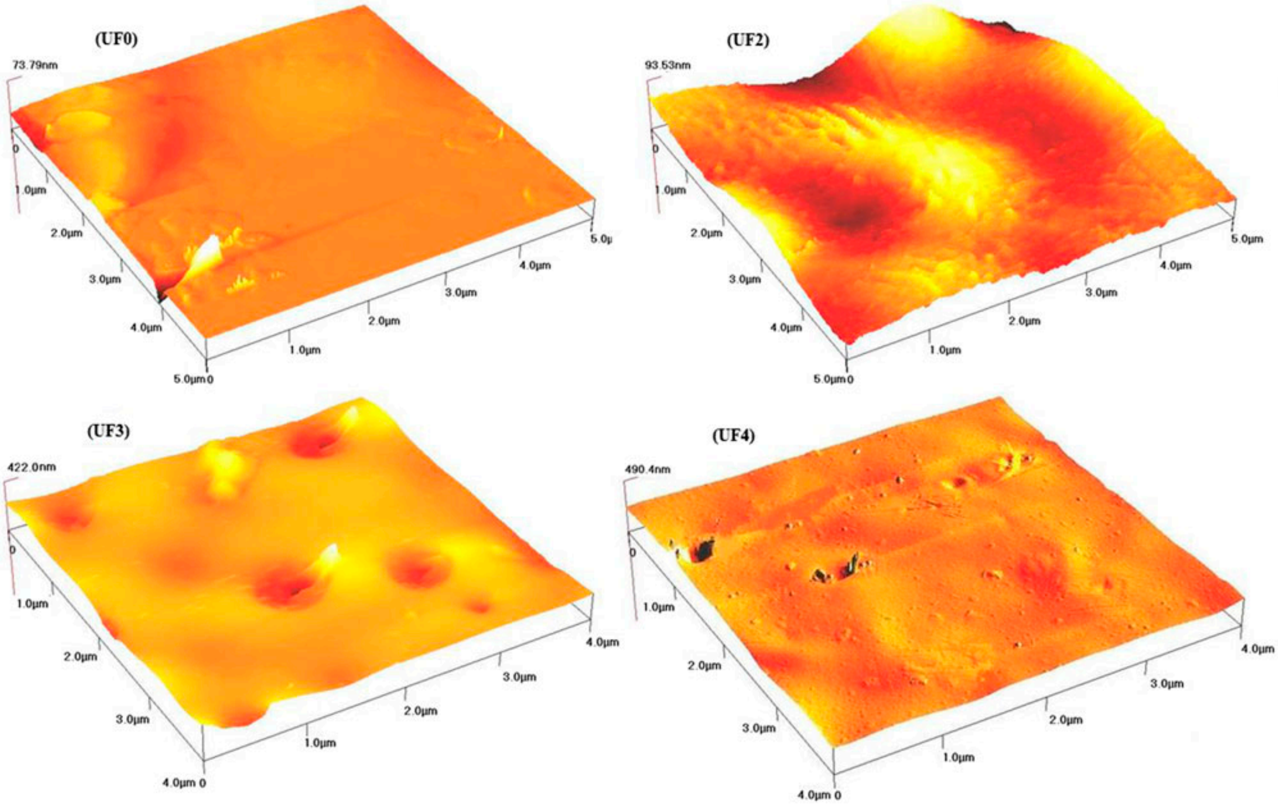

Fig. 5. Three-dimensional AFM images for UF0, UF2, UF3, and UF4.

Table 2
Variations in mean pore size and surface roughness parameters of neat PSf and blend membranes

		Roughness parameters		
Membranes	Mean pore size (nm)	S_a (nm)	S_q (nm)	S_z (nm)
UF0	6.30	0.225	0.248	2.452
UF2	9.13	0.398	0.462	2.931
UF3	9.52	0.433	0.529	3.711
UF4	11.14	1.877	2.043	13.691

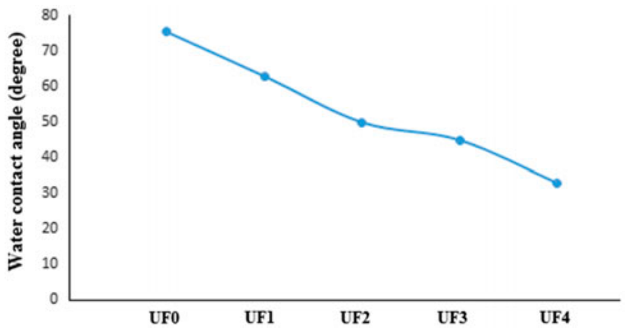

Fig. 6. Water contact angle of prepared membranes with different PVP concentrations.

Table 3
Water content and porosity of prepared membranes at different PVP concentrations

Membrane	Water content %	Porosity %
UF0	52.1	16.9
UF1	60.3	28.3
UF2	69.7	42.8
UF3	71.4	59.0
UF4	74.5	63.4

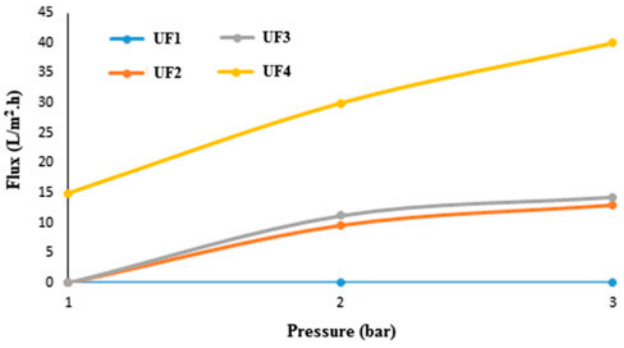

Fig. 7. Effect of pressure and PVP concentration on permeate flux of prepared membranes.

Table 4
Phosphate rejection of prepared of membranes at different feed pH

	Phosphate rejection % (pH 8.5)		Phosphate rejection % (pH 7.0)		Phosphate rejection % (pH 2.0)	
Membrane	2 bar	3 bar	2 bar	3 bar	2 bar	3 bar
UF2	83.2	60.0	84.3	61.3	93.6	69.6
UF3	52.0	44.0	53.0	45.3	87.2	60.3
UF4	37.6	28.0	38.0	28.0	43.0	35.1

3.4. Contact angle, water content and porosity measurement

Surface wetting characteristic of the PSf and PSf/PVP blend membranes was examined using contact angle measurement. Neat PSf membrane exhibited contact angle measuring 75.45˚. Lower values of contact angle indicated hydrophilic surface. From Fig. 6, it was observed that the UF4 blend membrane showed the lowest contact angle value indicating high hydrophilic top layer in this membrane. Accordingly, inclusion of PVP increased the hydrophilicity of the blend membranes thereby giving lower contact angle.

The membrane water content test was done to demonstrate the variety in hydrophilicity and porosity of the surface in different PVP concentrations. It can be seen from Table 3 that water content and porosity were enhanced with increasing amount of PVP. The highest water content and porosity perceived were 74.5 and 63.4%, respectively, for UF4. Membrane with highest content of PVP exhibit greatest water content. This confirmed that the increment of PVP concentration in the membrane, improve the hydrophilicity and porosity of the membrane.

3.5. Flux and rejection in different feed pH

Fig. 7 shows the effect of PVP on the flux of the feed solution (pH 8.5) at different feed pressures. Since PSf is a hydrophobic polymer, pure PSf membrane (UF0) did not have any feed flux from 1 to 3 bar pressure. The feed flux increased with increasing of PVP concentration in the casting solution and UF4 exhibiting the highest flux. This is related to the thinner surface layer (Fig. 3) and high membrane porosity (Table 3) at high PVP concentrations. Since the concentration of PVP in UF1 was only 1 wt.%, this membrane did not have flux until 3 bar pressure.

The rejection test of prepared membranes was carried out in three different feed pH (2, 7, and 8.5). The

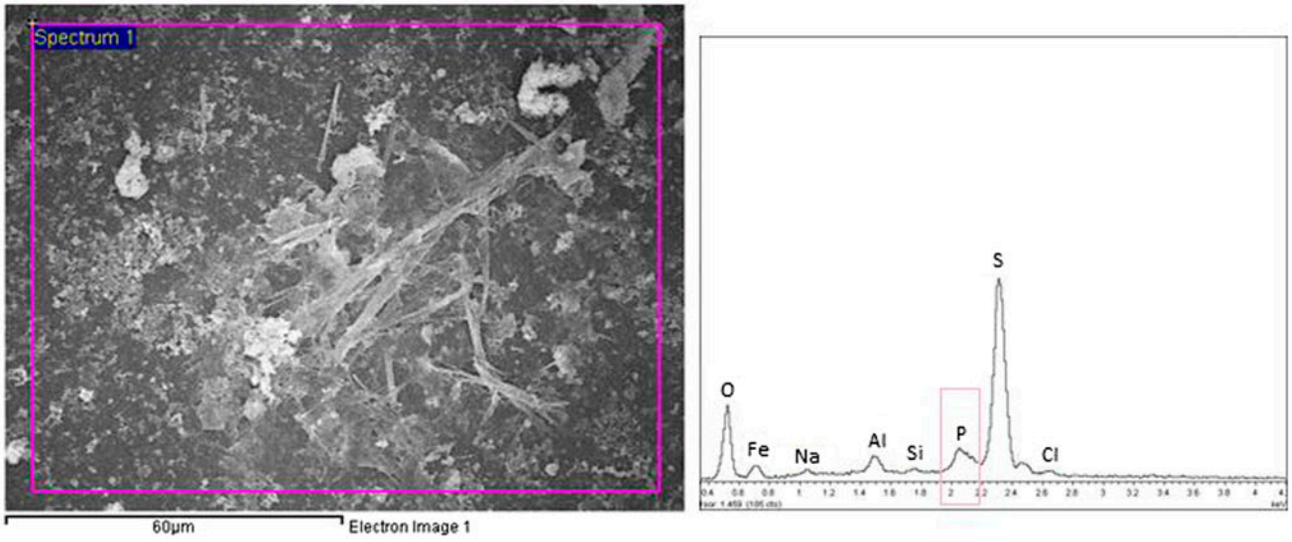

Fig. 8. Surface analysis of UF2 membrane using SEM-EDX.

initial pH of phosphate solution was 8.5. The zeta potential of PSf membrane is near zero at pH 3 (isoelectric point (IEP)), which means that the surface charge of PSf membrane is negative at $pH > 3$ and positive at $pH < 3$ [27]. The phosphate rejection values (Table 4) shows that UF2 has the highest amount of rejection among the prepared membranes at different feed pressures. The phosphate rejection capability decreased for UF3 and UF4. This can be attributed to the decreasing thickness of surface layer and the increasing pore size at higher PVP loading.

Interestingly, the performance of prepared membranes improved at lower pH. As indicated in Table 4, UF2 has phosphate rejection of 93.6% at pH 2, which was below the IEP. Since the surface of membranes was positively charged at pH value lower than IEP, there was an electrostatic attraction between positively charged membrane and the negatively charged phosphate ions, leading to a high phosphorus rejection. On the contrary, at pH 7 and 8.5, phosphorus rejection diminished because of the repulsion happened between the negatively charged membrane surface and phosphate ions [31]. For further confirmation, the used UF2 membrane was characterized by SEM-EDX (Fig. 8). The EDX spectrum revealed the peak of P, which indicates the adsorption of phosphate ions on the surface of UF2 membrane.

4. Conclusions

Phosphate removal using dead-end ultrafiltration was studied by changing the pressure, pH of the feed solution and modifier concentration. Five membranes with different PVP concentrations were prepared and characterized (UF0, UF1, UF2, UF3, and UF4). The experiments outcomes show that pressure, PVP concentration and pH had a noteworthy influence on the phosphate rejection. The phosphate rejection by UF2 membrane was highest as compared to other prepared membranes for all of the conditions studied. The phosphate rejection was noted to diminish with an increase in pressure as well as PVP concentration. While UF0 and UF1 did not have any flux in different pressures, the flux increased with increasing pressure and PVP concentration for other membranes. It was found that prepared membranes performed better in acidic condition (pH 2) rather than alkaline conditions (pH 7 and 8.5). pH value lower than IEP, gave a higher phosphate rejection, due to electrostatic attraction between positively charged membrane and the negatively charged phosphate ions. From the results acquired in this research, dead-end ultrafiltration can be considered as a possible way to remove phosphate from a liquid feed as the highest rejection of 93.6% was attained over UF2 membrane.

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