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Preparation of PPSU Hollow Fiber Nanofiltration Membranes for Nanofiltration Application

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Abstract

PPSU hollow fiber nanofiltration membranes are prepared by applying two concentrations and various extrusion pressures according to the phase inversion method. Cross-sectional area and outer structures were characterized by using scanning electron microscope (SEM) and atomic force microscopy (AFM). In additional to the pore size distribution, either the mean roughness or the mean pore size of the PPSU hollow fiber surfaces was evaluated by AFM. It was found that the morphology of the PPSU fibers had both sponge-like and finger-like structures through different extrusion pressures and PPSU concentrations. The mean pore size and mean roughness for inner and outer surfaces were seen to be decreased with the increase of extrusion pressure at two different PPSU concentrations. Moreover, the lead ion rejection was significantly improved from 19 to 78 % with increasing the extrusion pressure from 2.5 to 3 bar at 25 wt.% PPSU concentration.

Key words: PPSU, hollow fiber membrane, nanofiltration, morphology, heavy metals removal.

Introduction

Although there are more than 124 types of the polymers and most of the traditional polymersused for the preparation of membranes, some of the polymers are still not widely investigated to apply for this purpose such as the PPSU hollow fiber nanofiltration membranes. However, several researchers referred to the use of PPSU for the preparation of membranes for different applications. For example, Hwang [1], had improved the permeability of polymer membranes and the efficiency of filtration process throughout the water treatment by means of preparing the novel composite of membranes via adding activated carbon (AC) /

polyethylene glycol (PEG) with different ratios into both polyetherimide (PEI) polymers and polyphenyl sulfone (PPSU). The results showed that the adding up of AC was affected in a straight line the distribution of pore size, the morphology of membrane, the chemical properties, and the porosity. It also appears that increasing in the AC concentration was guided to improve both the filtration flux and the permeability of membrane (i.e. AC/PPSU/PEI/PEG composite). Alternatively, the addition of hydrophilic pore-formation agent (i.e. PEG) can also be led toenhance both the porosity and the surface hydrophilicity of the novel composite of membranes. Furthermore, Siavash [2], had prepared the PPSU as a result of its prospect utilize in the solvent resistant nanofiltration (SRNF) membranes. These membranes had made according to the dry wet spinning method in the midst of three special concentrations of PPSU (22.5%, 25% and 27.5% W/W) with the present of N-methyl-2-pyrrolidone (NMP) as a dope solution. They were cautiously distinguished consistent with their permeability, porosity, morphology, and selective properties. It was discovered that the fiber had a classical asymmetric structure with almost dense skin layer and porous substructure. In addition, an increase in the quantity of macro-voids inside the porous substructure was clearly detected once the concentration of polymer was diminished. Whereas, it seems that raising of polymer concentration within the generated dope was caused an increase in the rejection of dyes with a decrease in the permeability of iso-propanol. In terms of improvement the rejection, the membrane is exhibited a typical opportunity for favorable apply in nanofiltration applications, although the permeability is reduced and it therefore needs supplementary optimization. Additionally, by measuring the alter in the length of the fiber prior to- and subsequent to- the solvent treating for ten days, the effects of the membranes exposure on the acetone, isopropanol, diethyl ether, toluene, n-hexane, and ethyl acetate have been investigated. Generally, the stability of membranes was confirmed in most of solvents with the exception of methyl ethyl ketone. The permeability in n-heptane, n-hexane, iso-propanol, ethanol, methanol, toluene and acetone has been analyzed and excluding the last two solvents, the membranes demonstrate an excellent stability during the permeation test. Zhong [3], exploited a sulfonated polyphenylene sulfone (sPPSU) in order to fabricate a novel positively charged nanofiltration (NF) membranes, which is supported with both fully sponge-like morphology and hydrophilic properties as a result of the employ of UV-induced grafting. The obtained NF membranes - molecular weight cut off (MWCO) of 1627–1674 Da - with a high pure water permeability and an effective pore diameter of 1.13–1.20 nm were seen to be successfully developed as a result of employing two dissimilar categories of positively charged grafting monomers. According to the salt rejections results over the novel nanofiltration membranes, it seems to be followed the order of R(MgC_{l2})4R(NaCl)4R($MgSO_4$)4R(Na_2SO_4). A high removal to $MgCl_2$ of up to 95.20% was also acquired. Besides, these newly evolved NF1 and NF2 membranes are shown an interesting probability for the elimination of dye from wastewater treatment. Siavash [4], also prepared NF flat sheet membranes derived from polyphenylsulfone (PPSU). The synthesis methods depend upon phase

inversion with three diverse compositions of PPSU (i.e. 17 wt.%, 21 wt.%, and 25 wt.%) in dimethylacetamide (DMA), N-methyl-2-pyrrolidone (NMP) and a mixture of dimethylformamide (DMF) and NMP. It was observed that the morphology of membranes has a regular asymmetric structure characterized by a dense skin top layer and a porous substructure. It seems once again, the promoting in the amount of macro-voids within the membrane substructure is created as soon as the polymer concentration is reduced. Theperformance of the elaborated membranes was checked by evaluation both permeability of methanol and repudiation of dissolved dye (Rose Bengal). It was noticed that an increase in the polymer concentration was led to decrease in the methanol permeability, even as a rejection of Rose Bengal (RB) was enhanced. In addition, By investigating the rejection of RB before and after solvent contact and determining the flux of methanol, the impact of acetone, iso-propanol, diethyl ether, toluene, n-hexane, and ethyl acetate on the membranes has also been studied. In most of these solvents exclusive of acetone and toluene, the membranes were seen to be stable with a dramatically changed in the performance of membranes for both diethyl ether and ethyl acetate, on the other hand a minor effect and higher stability was observed for iso-propanol in n-hexane.

Hwang [5], had synthesized an antifouling ultra-filtration membrane for humic acid (HA) separation by employing a blending technique - using a non-solvent induced phase separation method. In order to figure the PPSU/PEI blend membranes, a positively charged/hydrophilic polyetherimide (PEI) was in that case blended with negatively charged/hydrophobic polyphenyl sulfone (PPSU). The specifications of membrane surface (i.e. surface charge, roughness, hydrophilicity, and morphology) were tailored according to the blending ratio with the aim of improvement the resistance of the membrane against fouling. For instance owing to the effect of electrostatic repulsion, the blend membranes with a weak negative charge were seen to be had a high-quality resistance to the negatively charged HA. The hydrophilic PEI is segregated to the membrane/water interfaces with increasing the PEI content due to change the surface charge from negative to neutral and constructs a hydrophilic surface. In this study, PPSU hollow fiber nanofiltration membranes are prepared in proportion to the phase inversion method. The effects of two different PPSU concentrations under various extrusion pressures on the morphology of PPSU hollow fibers and their separation performance were neatly investigated. SEM and AFM techniques were used to characterize the hollow fiber membranes. A lead ion was also utilized to test the performance of the PPSU membranes.

Experimental Work

1. Materials

Polyphenyl sulfone (PPSU) achieving from Solvay (Belgium) was used as a polymer material. 1-methyl-2-pyrrolidone (NMP, 99.5%) was employed as a polymer solvent and provided by Sigma-Aldrich (Germany). $pb(NO_3)_2$ heavy metals was also utilized to explore the membrane separation performance, which is supplied by BDH (England).

2. PPSU Membrane Preparation

Grains of PPSU with concentration of 25 and 29 wt.% were dissolved over

magnetic stirrer inside 75 and 71 wt.% of NMP solvent, respectively at room temperature for two days. The final homogeneous PPSU solution was transferred to a vertical column and left for 24 h to remove the air bubble from the PPSU solution as shown in Figure 1.

After that the Nitrogen gas was employed to compressed the PPSU solution to the spinneret (polymer feed side) at different extrusion pressures. The water as a bore fluid was pumped at a rate of 3 ml/min by applied precision Gear pump (Information Technology Engineering Co., Guro-Gu, South Korea) to the spinneret (water feed top side) as shown in Figure 1. The two fluids were brought in to contact at the exit point of the spinneret and then entered the tap water coagulation bath at 36 °C after air gap distance of 3.5 cm. Subsequently, the nascent hollow fiber was drawn with a suitable speed without any starching in the nascent hollow fiber. The produced hollow fiber was kept in the water vessel for 24 h to remove the remaining NMP from the hollow fiber. Finally the hollow fiber was treated with glycerol solution for 48 h in order to avoid the structure collapse of the hollow fiber.

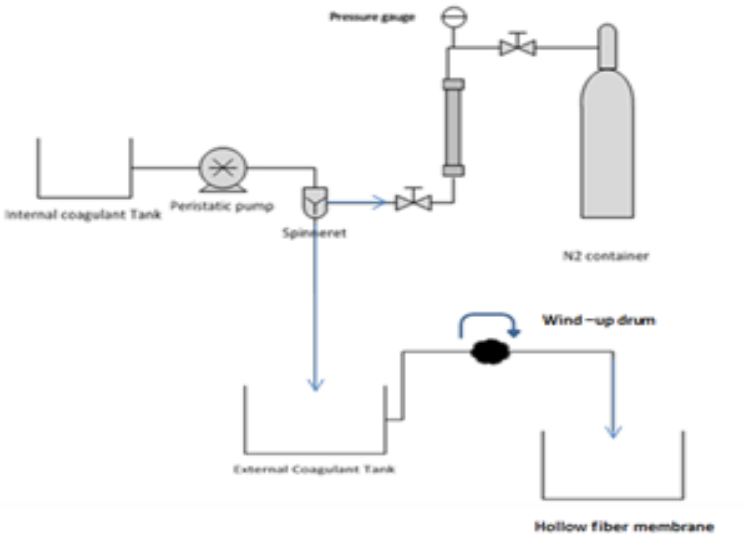


Fig. 1: PPSU hollow fiber membrane spinning system

3. SEM Measurement

One of the powerful techniques for characterization the structural morphology of the membrane is the scanning electron microscope (SEM). The structure of the prepared membranes in this work was investigated using SEM (Model: Quanta FENG 200, FEI Company) at the University of Technology, Baghdad, Iraq. In order to measure the cross-section structure, the each membrane sample freezes and breaks in liquid nitrogen, to make a clean and uniform configuration without any damage in the structure of the membranes. With the aim of measuring the structure of the inner and outer surfaces, the membranes were therefore dried under vacuum; and then coated with a thin gold layer by means of a sputter apparatus.

4. Atomic Force Microscope (AFM) Measurement

With the purpose of measuring the size of pore and the distribution of pore size for the prepared membranes, PPSU hollow fiber membrane samples were introduced to the spacious surface analysis through an atomic force microscope (AFM) via contact mode with a suitable silicon tip [Angstrom Advanced Inc. (USA), model AA3000]. Measurements involved an evaluation for the topography, the deflection, and the lateral force. A statistical pore size distribution for the surfaces of PPSU hollow fiber membrane has already been established by using IMAGER 4.31 software.

5. Preparation of Heavy Metal Solution and PPSU Membrane Performance

In order to study the effect of heavy metal ions concentration on the performance of the PPSU membrane, salts solution was synthesized in single-salt solution of ($pb(NO_3)_2$ (i.e. concentration of 160 mg of $Pb(NO_3)_2$ with 1000 ml of pure water and that means 100 ppm of lead).

Before NF experiments of the heavy metals aqueous solutions, the pure water permeability experiment of the PPSU membranes was carried out at a temperature of 25±3 °C and pressure of 1.5 bar for one hour by using the NF system as shown in Figure 2.

Pure water permeability (PWP) kg/(m^2·hr.bar) was estimated along with the following equation:

$$PWP = \frac{V}{t.\ A.\ \Delta P} \qquad \ldots(1)$$

Where, V is volume of the collected permeate (L) during time t (hr), A is the effective surface area (m^2), and ΔP is the Trans membrane pressure (bar). The NF experiments of heavy metal solution were consequently completed in batch circulation mode. With the purpose of maintaining a constant concentration within feed tank, both permeate and retentate have been returned into the feed tank.

The solute separation factor, F, can be calculated by the following equation:

$$F = \frac{C_{feed} - C_{permeate}}{C_{feed}} \qquad \ldots(2)$$

Where C_{feed} and $C_{permeate}$ are the concentration of the heavy metals ions in feed and streams solution. The concentrations of heavy metals ions within the feed and permeate streams were measured using Atomic Absorption (spectrometer), PERKIN ELMER 5000.

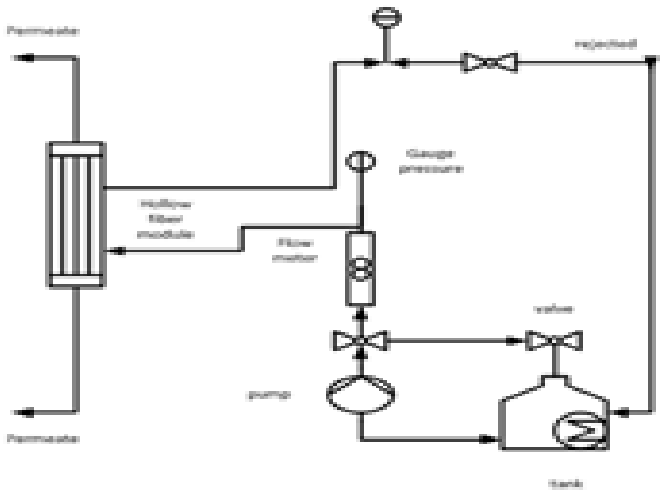


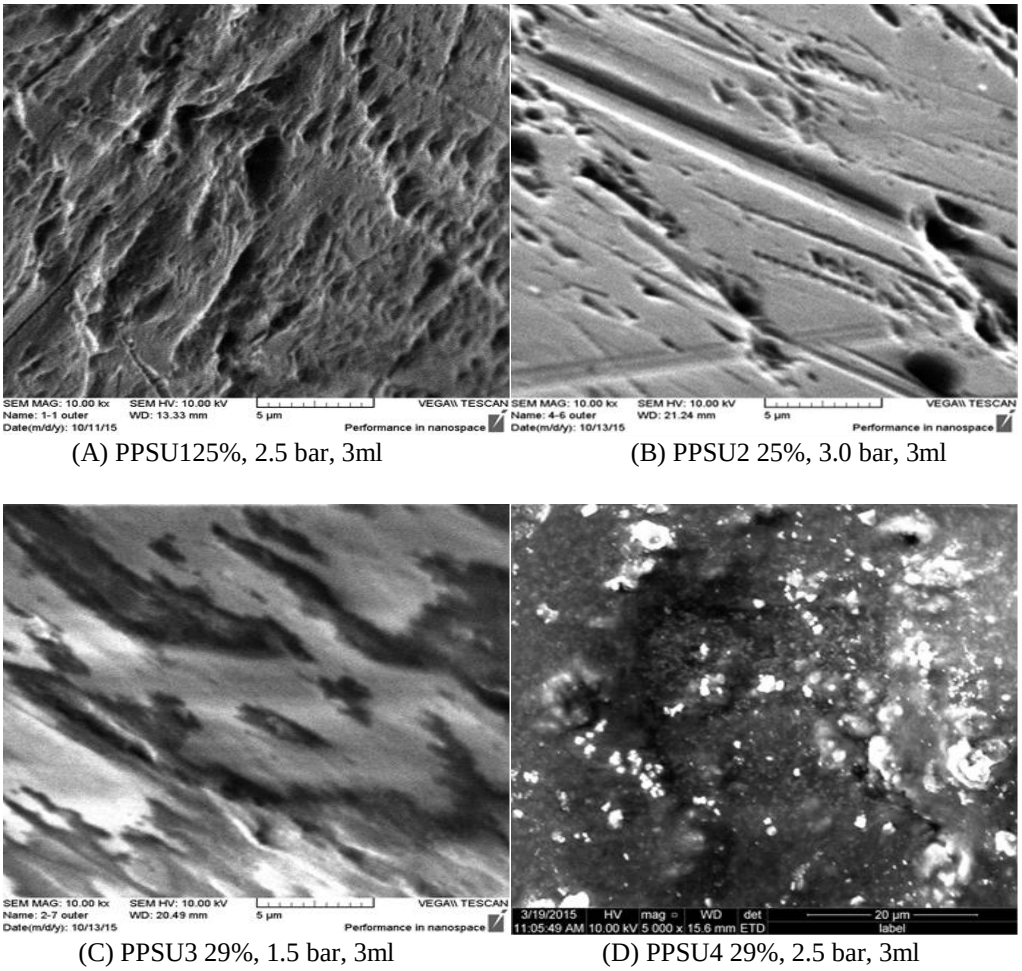
Fig. 2: PPSU hollow fiber nanofiltration membrane separation performance experimental system

Results and Discussion

1. SEM Measurement

1.1. Outer Surface of PPSU Hollow Fibers

To investigate the effects of two different PPSU concentrations and extrusion pressures and in order to study the performance of the PPSU hollow fiber membranes, the outer surface and cross-section of the PPSU hollow fibers were accurately studied using SEM analysis. The SEM images of the PPSU hollow fiber outer surface are presented in Figure 3. It can be seen that the prepared PPSU hollow fiber with 25 wt.% PPSU1 under 2.5 bar extrusion pressure present a rough and dense surface as shown in Figure 3A. Whereas in Figure 3B, it can be observed that the prepared PPSU2 hollow fiber membrane with 25 wt.% under 3.0 bar extrusion pressure at 3 ml/min bore fluid flow rate has a less rough and dense surface in comparison with that in PPSU1. This is due to the formation of aggregates during the fast speed of the hollow fiber within 3.5 cm air gap throughout the formation of the hollow fibers, Wijmans [6]. In Figure 3C and 3D similar behaviors were observed for two PPSU hollow fiber membranes that prepared from 29 wt.% and under two different extrusion pressures (i.e. 1.5 and 2.5 bar).



(A) PPSU125%, 2.5 bar, 3ml (B) PPSU2 25%, 3.0 bar, 3ml

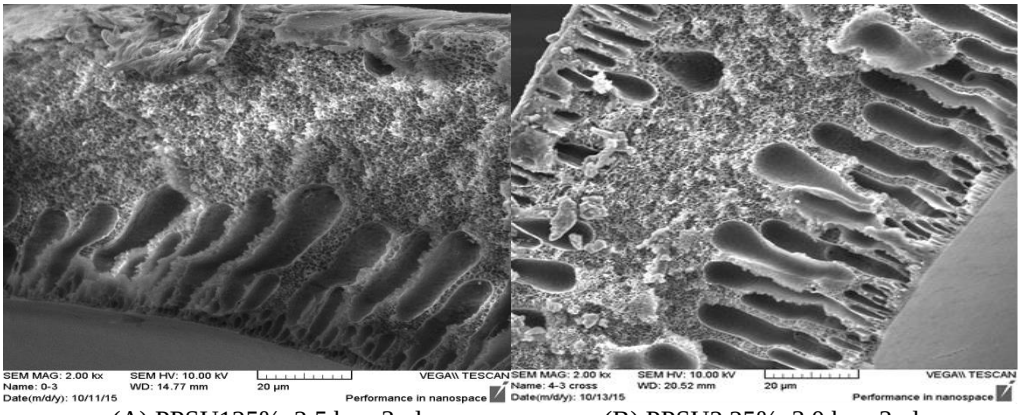
(C) PPSU3 29%, 1.5 bar, 3ml (D) PPSU4 29%, 2.5 bar, 3ml

Fig. 3: SEM images of the outer surface of PPSU hollow fiber membranes

1.2. Cross-Section of PPSU Hollow Fibers

Figure 4 presents the effect of PPSU concentration and extrusion pressure on the cross sectional structure of the PPSU hollow fiber membranes. It can be noticed that the prepared PPSU1 hollow fiber with 25 wt.% PPSU1 under 2.5 bar extrusion pressure has sponge-like structure that appearing at the entire hollow fiber, while there is only a small finger-like structure near the edge of the inner surface of the hollow fiber membrane as shown in Figure 4A. Besides, it can be observed that the PPSU hollow fiber membrane that prepared from PPSU2 with 25 wt.%, and under 3.0 bar extrusion pressure at 3 ml/min bore fluid flow rate has a little finger-like structure that appearing near from the outer edge of the hollow fiber, whilst there is only a large finger-like layer close to the edge of the inner surface of hollow fiber membrane, as well as there is also a sponge-like structure that growing within the middle of the cross-section as shown in Figure 4B. This observation can be attributed to the fast solvent exchange from the polymer solution to the internal coagulation water with enhancing of nascent hollow fiber speed during the formation of membrane, Aptel [7] – Alsalhy [8].

In Figure 4C and 4D, it can be distinguished that the decrease of extrusion pressure from 1.5 bar to 2.5 bar at 29 wt.% PPSU concentration is led to suddenly emerge one layer of finger-like structure near from the inner surface of hollow fiber membrane, which has already been changed to full sponge-like structure. This is maybe owing to the increase of contact time between the polymer solution and coagulation water, which results either to increase the water diffusion or penetration inside the structure of the nascent hollow fiber.



(A) PPSU125%, 2.5 bar, 3ml (B) PPSU2 25%, 3.0 bar, 3ml

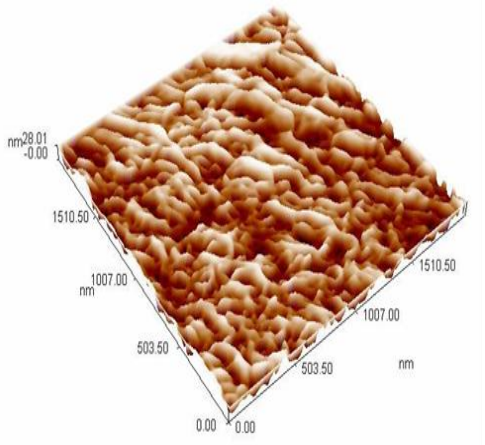
(C) PPSU3 29% 1.5 bar 3 ml min (D) PPSU429%, 2.5 bar, 3ml

Fig. 4 SEM images of the cross section of PPSU hollow fiber membranes

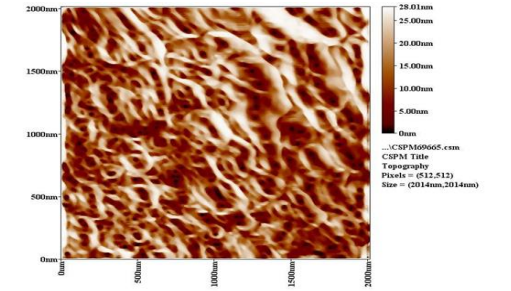
2. AFM Measurement

Figure 5 shows the 3D and 2D AFM images of the inner and outer surfaces of PPSU hollow fiber membrane, which are prepared at different extrusion pressures and two different PPSU contents. It can be recognized that the roughness of the inner surfaces is decreased either with increasing of extrusion pressure from 2.5 to 3.0 bar at 25 wt.% PPSU or with increasing the extrusion pressure from 1.5 to 2.5 bar at 29 wt.% PPSU, as illustrate in Table 2. Additionally at the same extrusion pressure (i.e., 2.5 bar) with two different PPSU concentration in dope solution (i.e., 25 wt.% and 29 wt.% PPSU), it can be recognized that the mean roughness seems to be decreased with PPSU concentration. Regarding the mean roughness of the outer surfaces, it can be said that the mean roughness of the PPSU was decreased for the outer surfaces of every PPSU hollow fibers. However regarding the pore size and the distribution of pore size for PPSU membranes, Table 1 also shows the effects of extrusion pressures at two different PPSU concentrations on the mean pore size. It can be noted that the pore size of the inner surfaces of prepared PPSU hollow fiber membrane under 2.5 and 3bar extrusion pressures at 25 wt.% PPSU in dope solution is reduced with raising of extrusion pressure. Similar behavior was observed for the pore size of the prepared membrane from 29 wt.% PPSU. Moreover, in Table 2 is shown that the pore size of outer surface for prepared PPSU hollow fibers under different extrusion pressures and PPSU concentrations has also been diminished with promoting of extrusion pressure.

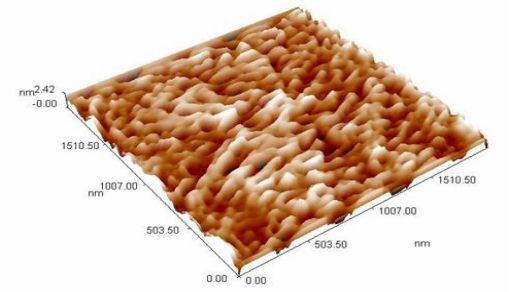
Figure 6 shows the effect of extrusion pressure and PPSU concentration in dope solution on the distribution of pore size over inner and outer surface. It can be distinguished that the pore size distribution of the inner surface of the prepared PPSU hollow fibers membrane from 25 wt.%of PPSU under 2.5 bar is between 10 and 110 nm, while the pore size distribution at 3.0 bar turn out to be narrow (about 15 and 70 nm). Furthermore, the pore size distribution of the outer surface is in the range of 30 and 130 nm at 2.5 bar, whereas the pore size distribution at 3.0 bar is between 20 and 80 nm. This means that as the extrusion pressure off increases the pore size (inner and/or outer) becomes narrower. In addition, the pore size distribution of the inner surface of PPSU hollow fiber membrane prepared from 29 wt.% at 1.5 bar is within the range of 7 and 110 nm, whilst the pore size distribution at 2.5 bar is between 23 and 90 nm. On the other hand the pore size distribution of the outer surface of the PPSU hollow fibers is about 20 to 100 nm for the prepared membrane from 29 wt.% under 1.5 bar, but the pore size distribution is between 20 and 110 nm under extrusion pressure of 2.5 bar as demonstrated in Figure 6.



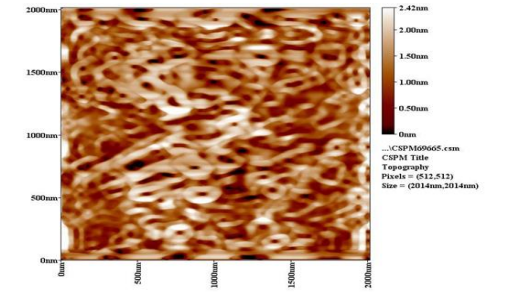
(A) 3D PPSU1 25% 2.5 bar 3 ml/min inner surface



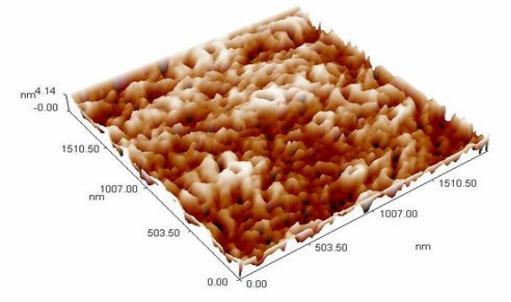
(B) 2D PPSU1 25% 2.5 bar 3 ml/min inner surface



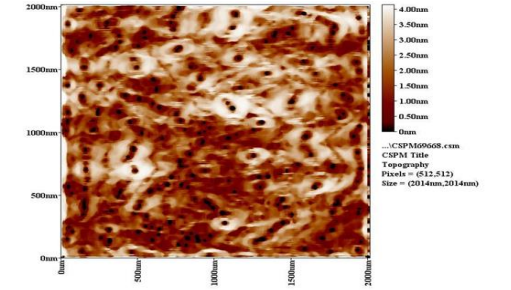
(C) 3D PPSU1 25% 2.5 bar 3 ml/min outer surface



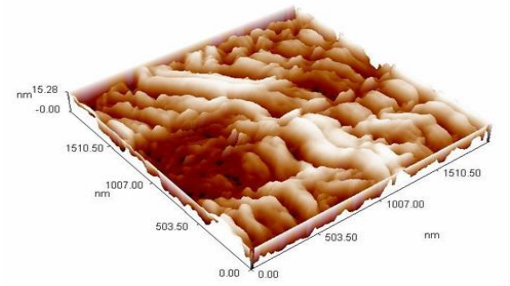
(D) 2D PPSU1 25% 2.5 bar 3 ml/min outer surface



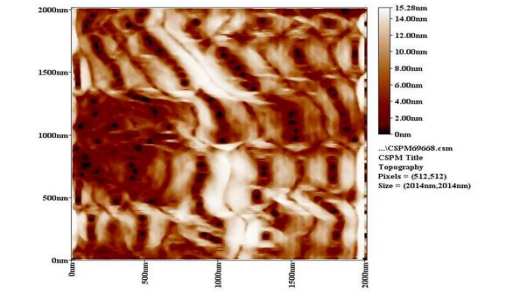
(E) 3D PPSU2 25% 3 bar 3 ml/min inner surface



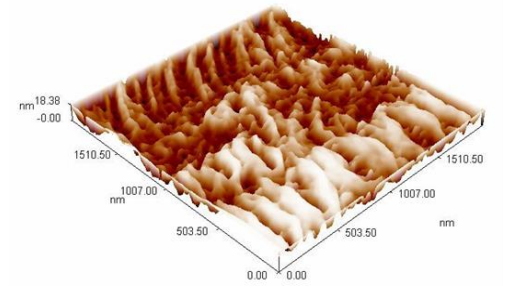
(F) 2D PPSU2 25% 3 bar 3 ml/min inner surface



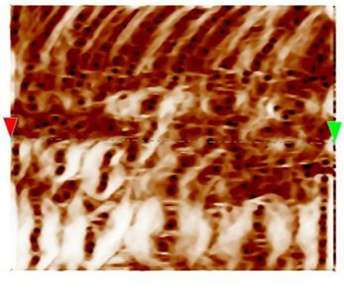
(G) 3D PPSU2 25% 3 bar 3 ml/min outer surface



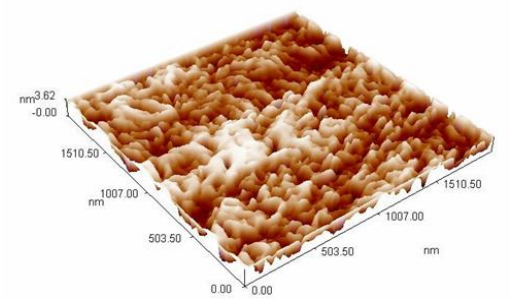
(H) 2D PPSU2 25% 3 bar 3 ml/min outer surface



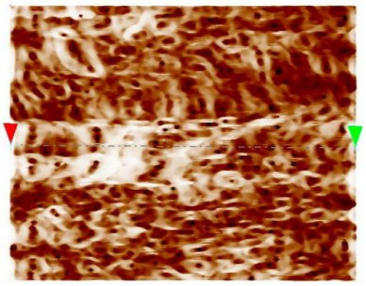
(I) 3D PPSU3 29% 1.5 bar 3 ml/min inner surface



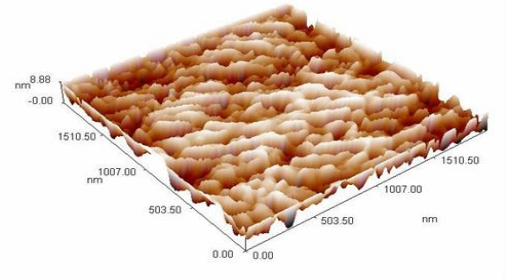
(J) 2D PPSU3 29% 1.5 bar 3 ml/min inner surface



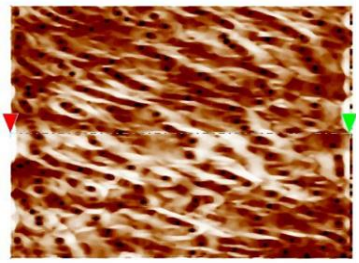
(K) 3D PPSU3 29% 1.5 bar 3 ml/min outer surface



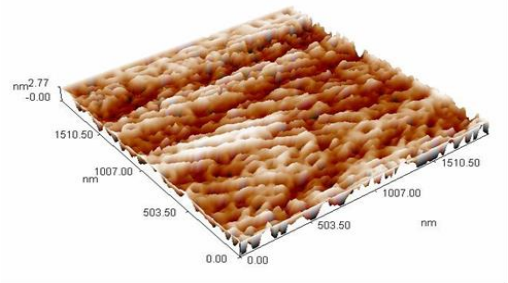
(L) 2D PPSU3 29% 1.5 bar 3 ml/min outer surface



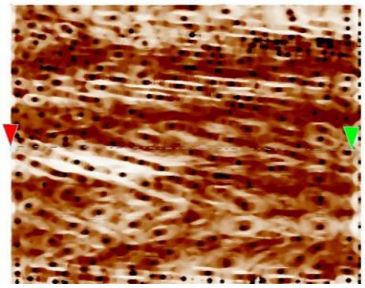
(M) 3D PPSU4 29% 2.5 bar 3 ml/min inner surface



(N) 2D PPSU4 29% 2.5 bar 3 ml/min inner surface



(O) 3D PPSU4 29% 2.5 bar 3 ml/min outer surface



(P) 2D PPSU4 29% 2.5 bar 3 ml/min outer surface

Fig. 5: AFM images of the different PPSU hollow fiber membranes

Table 1: Dope compositions and spinning conditions of the fabricated PPSU hollow fibers

Membrane code	**Dope composition (wt%)**	**Bore fluid Composition MNP/water**	**Coagulation path Temperature (˚C)**	**Extrusion Pressure (bar)**	**Bore fluid flow rate (ml/min)**	**Air gap length (cm)**
PPSU1	PPSU/NMP: (25:75)	0/100	36	2.5	3	3.5
PPSU2	PPSU/NMP: (25:75)	0/100	36	3	3	3.5
PPSU3	PPSU/NMP: (29:71)	0/100	36	1.5	3	3.5
PPSU4	PPSU/NMP: (29:71)	0/100	36	2.5	3	3.5

Table 2: Porosity, Thickness, Mean pore size and Mean roughness of PPSU hollow fibers

Membrane code	**Thickness (µm)**	**Porosity (%)**	**Mean pore size (nm)**		**Mean roughness (Ra)(nm)**	
			Inner surface	Outer surface	Inner surface	Outer surface
PPSU1	77	71	53.82	65.74	6.05	0.433
PPSU2	80	69	37.31	51.40	2.57	0.413
PPSU3	61	62	52.04	49.75	3.42	0.802
PPSU4	63	60	51.73	47.23	2.13	0.513

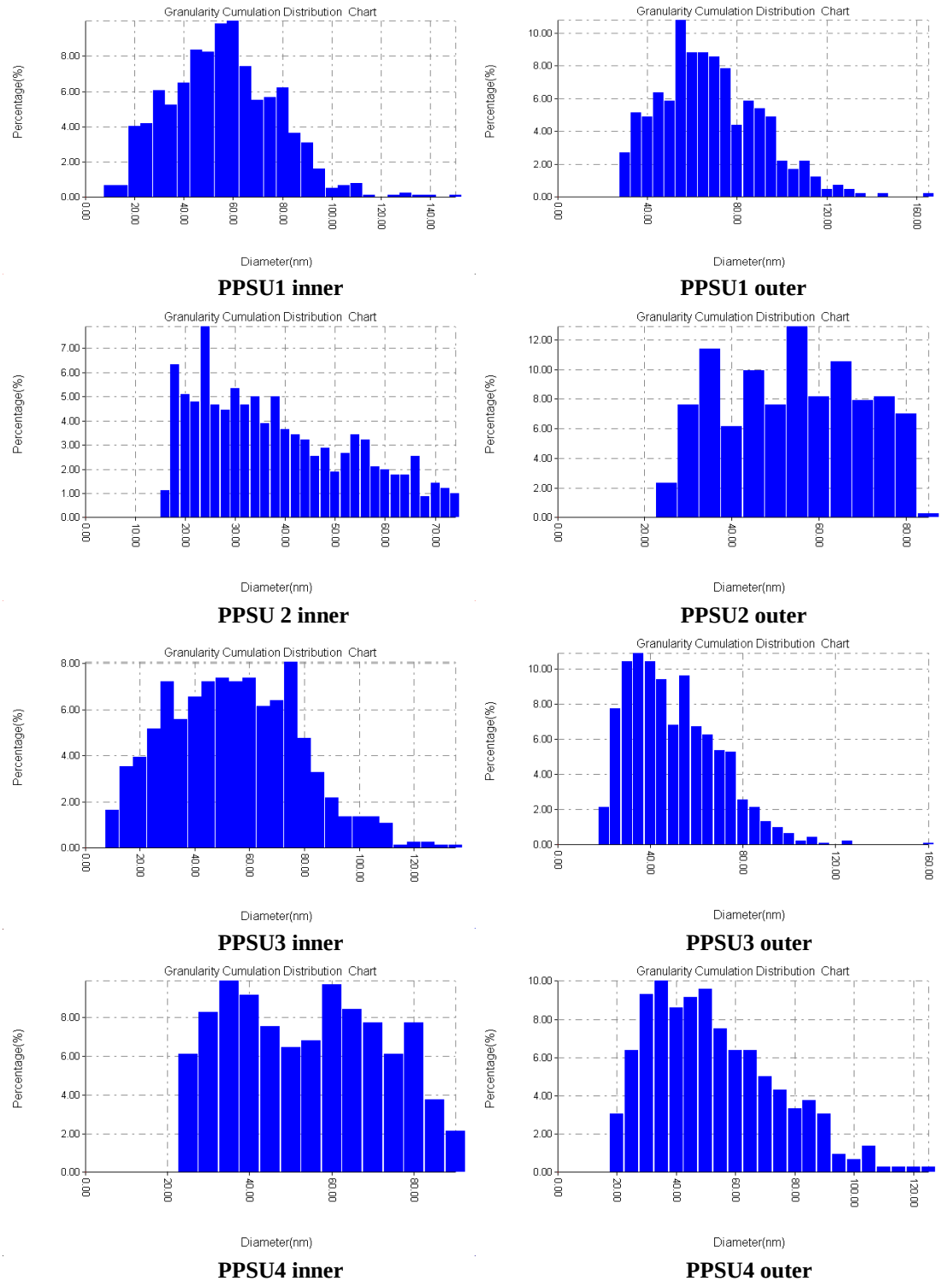


Fig. 6: Pore size distribution of the PPSU hollow fiber membranes

3. PPSU Performance

Table 3 shows the effects of two different PPSU concentrations and various extrusion pressures on both the pure water permeation flux and the heavy metal ion rejection of PPSU hollow fiber membranes.

The pure water permeation flux is 2 $L.m^{-2}.h^{-1}.bar^{-1}$ for the PPSU hollow fiber membrane under 2.5 bar, whereas increasing the extrusion pressure to 3 bar, results to an increase in the pure water permeation flux to 5 $L.m^{-2}.h^{-1}.bar^{-1}$. For the PPUS hollow fiber

membrane prepared from 29 wt. % under 1.5 bar, the pure water permeation flux is 10 $L.m^{-2}.h^{-1}.bar^{-1}$ and it is reduced to 8 $L.m^{-2}.h^{-1}.bar^{-1}$ for PPSU hollow fiber membrane prepared from 29 wt. % PPSU in the dope solution under extrusion pressure of 2.5 bar. This finding can be related to the altering of the PPSU structures during the formation of hollow fiber because of the higher amount of the polymer solution flow out from the spinneret at 2.5 bar, which might be affected by the amount of the solvent/non-solvent exchange rate of internal coagulant within the external coagulation bath. As well, this watching can be attributed to the enlargement in the thickness of the PPSU and diminishing the mean pore size with raising the extrusion pressure as summarized in Table 2.

Table 3: PPSU membrane permeation flux and separation performance

Membrane code	Permeate flow (l/ m^2bar h)	C_{Pb} in permeate side (ppm)	Pb Rejection (R_{Pb} %)
PPSU1	5	81	19%
PPSU2	2	22	78%
PPSU3	10	76	24%
PPSU4	8	84	16%

These results were agreed with that reported by Alsalhy [9], Alsalhy [10].
Likewise from Table 3, it can be perceived that there is a significant improvement in the lead ion rejection of the PPSU hollow fiber membranes with an increase in the extrusion pressure from 2.5 bar to 3 bar during the formation of the nascent hollow fiber that composed of 25 wt.% PPSU in dope solution. The Pb^+ rejection seems to be improved from 19 to 78% as a result of enhancement the extrusion pressure. Finally, using 29 wt.% PPSU concentration in the dope solution was caused a reduction in the Pb^+ rejection from 24 to 16 % as the extrusion pressure was elevated from 1.5 to 2.5 bar. This is observation can be attributed to the lower of extrusion pressures in case of 29 wt.% PPSU hollow fiber with reference to that hollow fiber prepared from 25 wt.% PPSU under extrusion pressures of 2.5 and 3 bar, which essentially leads to decrease in the thickness of the hollow fiber, and successively guides to decrease in the Pb^+ rejection.

Conclusions

Preparation of different PPSU hollow fiber NF membranes for heavy metals removal results to several conclusions, which can be summarized as follows:

- PPSU hollow fiber prepared with 25 wt.% PPSU and under extrusion pressure of 2.5 bar present a less rough and dense outer surface in comparing with that prepared under extrusion pressure of 3 bar.
- Different cross sectional structure was observed for all PPSU hollow fiber membranes most of them similar to the sponge structure.
- The mean roughness and mean pore size for inner and outer surfaces of hollow fiber membranes were seen to be decreased with increasing the extrusion pressure at two different PPSU concentrations.
- The lead ion rejection was extensively improved from 19 to 78 % with the altitude of extrusion pressure from 2.5 to 3 bar at 25 wt.% PPSU concentration.

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