Engineering and Technology Journal
Journal homepage: engtechjournal.org

(Polyphenyl Sulfone-Polyether Sulfone) Blending to Performance Flat Sheet Membrane to Remove Some Heavy and Radioactive Elements from Phosphogypsum Waste

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Submitted: 01/07/2020 Accepted: 18/10/2020 Published: 25/03/2021

KEYWORDS

Ultrafiltration, Rejection, Heavy elements, Radioactive elements.

ABSTRACT

In this research, the traditional version of the phase inversion method was used to fabricate a flat sheet of a blended membrane. The method was involved using a polymer that blends polyether sulfone (PES) varied proportions (0, 3, 4 and 5 wt.%), and polyphenyl sulfone (PPSU) was 20wt%. It was found that with the addition of PES, the membrane properties increased, the best properties were with 4%wt. The ratio was chosen PES 4wt% to study the effect of time, temperature, and pressure on the rejection of heavy and radioactive elements. The increase in the porosity was with the addition of 4% PES. The rejection of heavy and radioactive elements for thUF membrane increases with increasing of the operating pressure and time. While by increasing the temperature, the rejection of heavy and radioactive elements for thUF membrane decreased. The rejection of K, Th, and Pb are higher than other elements, the order of the rejection is K>Th>Pb>U>Cd>Zn>Cu>Ni.

How to cite this article: W. T. Rashid, I. A. Alkadir , M. G. Jalhoom, K. T. Rashid, “(Polyphenyl Sulfone - Polyether Sulfone) Blending to Performance Flat Sheet Membrane to Remove Some Heavy and Radioactive Elements from Phosphogypsum Waste,” Engineering and Technology Journal, Vol. 39, Part A, No. 03, pp. 382-393, 2021.
DOI: https://doi.org/10.30684/etj.v39i3A.1762

1. INTRODUCTION

Phosphogypsum (PG) is defined as a solid state of a waste by-product that generated during phosphoric acid production from phosphate rocks using the “wet acid” process [1]. While this process is an economic process, it leads to generate a huge amount of PG (5 tons of the amounts of PG are generated when 1 ton of phosphoric acid is produced. [2]. Yet, 15% of world production of PG thought out the world can be recycled and used for different purposes, such as materials that used for the building [3,4], agricultural fertilizers, amendments for soil stabilization [5], and Portland cement[6]. The remaining (85%) of world PG production is discarded without a treatment. Usually, this by-product is put away in a large stockpile open for the weathering processes, occupying a large land area and resulting in dangerous damage to the environment, such as radioactive and chemical contaminations [7,8]. Phosphogypsum has mainly consisted of calcium sulfate dehydrates ($CaSO_4.2H_2O$). It holds several elevated levels of the impurities that present from phosphate rock usually used in the production process of the phosphoric acid. The most concern among these impurities is the radionuclides 238-U and 232-Th decay series, because of their radiotoxicity and the heavy elements which are no less dangerous than the radioactive elements. Phosphogypsum can be used in industries after the elimination of heavy and radioactive elements where it is available for use. Many methods using for the removal of heavy and radioactive elements from ore. The technologies of remediation can be summarized as solvent extraction, icon exchange, bio mediation, precipitation and coagulation, adsorption, and heterogeneous photo catalysts [9]. One of the most important of these methods is membranes. Several types of polymeric materials have been used for the preparation of different types of membranes. Each polymer has its exceptional and unique properties that established for the prepared membrane performance. Polymers like polysulfone (PSF), polyphenylsulfone (PPSU), polyether sulfone (PES), polyvinylidene fluoride, and polyether ketone) [10] are usually used for the UF membranes fabrication. The manufacturers of the PPSU described that it shows very good hydrolysis resistance, stress cracking or plasticization by a lot of solvents. In addition, it a requires lower cost (good cost effective) than other polymers, such as PES and PSF [11]. PPSU shows great chemical and thermal resistances. Furthermore, PPSU is suitable for making good material for membrane than PES and PSF. However, similar to PSF, PPSU has a hydrophobic nature. This property represents the main drawback for this type of polymer when used as a material for membrane, because it might lead to severe and extreme fouling [12]. Based on that, the characteristics of the membrane and its morphology can be enhanced and modified by blending with other hydrophilic polymers or using suitable additives. In general, membranes are classified into different types; ultrafiltration (UF), microfiltration (MF), Nano filtration (NF), and reverse osmosis (RO). The ultrafiltration membranes have designed to have smaller pores than that corresponding in the microfiltration membranes. Thus, soluble macromolecules (such as proteins), bacteria, microorganisms, and large particles can be easily rejected. The pore diameter of the ultrafiltration membranes is designed to be in the range of (1- 100) nm [13]. Generally, the main objective of membrane technology is to design and develop membranes able to provide the highest level of permeate flux and solute rejection keeping the cost of membrane production and maintenance (if applicable) as low as possible [14].

2. EXPERIMENTAL WORK

I: Materials

Phosphogypsum samples that have been used in this work from Iraq (Al-Qaim fertilizers complex at Al-Anbar government)., the feed should be achieved firstly by dissolving the needed quantity of phosphogypsum (0.64 g\1L) in deionized water with stirring speed 500 rpm, contact time 60 min., and 25 °C. The solution was filtered with a 0.40 µm membrane filter. The solution from the tank of feed has been pumped by various pressures (2,4 and 6 bars), where changing pressures during the gradual closing of the valve of reject water (it must not be closed totally). These installed after pumps and before entering the membrane chamber (cell). The reading of feed stream pressure gauged for getting the required pressure for all operations. After leach solution preparation depending on the needed properties and regulation of the pressure, the flow rate and other parameters as stated, the system operated for 2 min at least to reach a steady state. However, in the meantime, the permeate solution returned to the feed tank to maintain the concentration of the solution. After that permeate and feed solution has been obtained in a flask for testing heavy and radioactive metals amount for calculating the rejection of membranes and determining the rate of permeate flow for calculating the flux of membrane.

II: Preparation of PES–PPSU Flat Sheet Ultrafiltration Membranes

A casting solution containing polymer polyphenyl sulfone (PPSU), solvent Dimethylacetamide (DMAc) (which helps dissolve polymers) and polyether sulfone (PES) will be prepared. Were dried at 40 °C to remove trapped moisture in a vacuum oven. Then 20 wt. % of PPSU with various percentages of PES (wt.%) were mixed with DMAc solvent in a conical flask with stirring rate around 250 rpm and at 40 °C for 24 hr. The homogeneous solution will be casting on a glassy smooth plate after that casting knife will be used for spreading the solution with thickness at~100 µm. The glass plates with the membrane film will be fast transferred to the water bath for the remainder of the process of phase-inversion at room temperature. The membranes will be immersed in a water bath for 24 hr. Membranes formed will be left for air-dried at 25 ºC to allow the removal of remaining solvent. Table I, lists the composition of PPSU/ PES /DMAC casting solutions. The prepared membranes were firstly frozen using liquid nitrogen, and then fractured to obtain the cross-sectional image. Figure 1, shows the process of the preparation of membrane by phase inversion. Figure2, exhibited the photograph images of the ultrafiltration system and Figure 3, shows the flat sheet produce. The morphology of the membrane was examined by scanning electron microscope (SEM).

TABLE I: Formulation of membranes casting

Membrane Code	PPSU wt.%	PES wt.%	DMAc wt.%
A1	20	0	80
A2	20	3	77
A3	20	4	76
A4	20	5	75

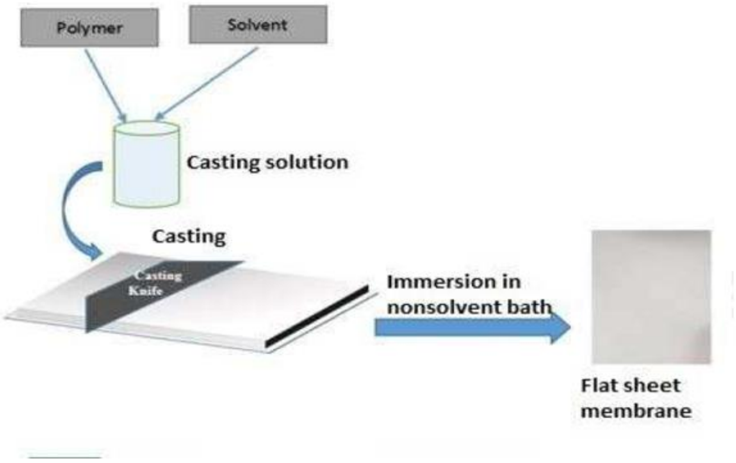

Figure 1: Phase inversion steps (solution formation, casting, and immersion in a no solvent bath) [15].

Figure 2 : Photograph image for the ultrafiltration system

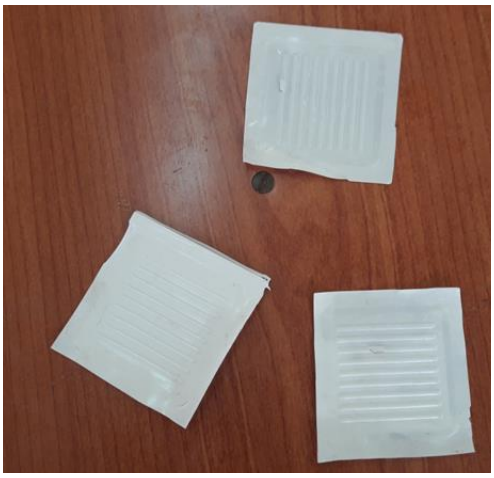

Figure 3: Flat sheet membrane

3. MEMBRANE CHARACTERISTICS

I: SEM Analysis

This test has been achieved at the University of Technology in the Department of Production Engineering and Metallurgy, to study the morphology of the membranes, and determine the distribution of the porosity of the membranes that were manufactured. SEM has been employed for observing the top surface and cross-section properties of membranes. The membrane cross-section has been achieved using liquid nitrogen (cut of membranes).

II: Measurement of porosity

The membrane porosity is defined as ratio of volume of voids to the total volume of the porous membrane. It is simply symbolized by (ε). This property can be calculated by Eqn. (1) as [16].

$$\varepsilon = \frac{\rho m}{\rho p}\ldots\ldots\ldots\ldots\ldots(1)$$

Membrane density was measured using the following Equation:

$$\rho_m = \frac{m}{l*w*t} \text{……….. (2)}$$

Where ρ_m is the membrane density measured by (g/cm^3), ρ_p is the density of polymer measured by (g/cm^3), w is the membrane width measured by (cm), m is the membrane weight measured by (g), l is the length of the membrane measured by (cm), and t is the thickness of membrane measured by (cm). The densities of the PPSU and PES were 1400 kg/m^3 and 1370 kg/m^3, respectively.

III: Rejection Percent

The rejection R (%) of the dissolved heavy and radioactive element concentration was calculated by equation (3) [17]:

$$R (\%) = (1- C_p \backslash C_f) \times 100 \text{….(3)}$$

Where C_f and C_p are the heavy and radioactive element concentrations (mg\L) of the feed and permeate respectively.

4. RESULTS AND DISCUSSION

I: Scanning electron microscopy (SEM)

The membrane morphology the performance of the membrane’s application particularly for the process of filtration [18]. A lot of studies focused on the modification of membrane morphology by using many methods. The prominent purpose that is related to modification has been enhancing the filtration performance and keeping the membrane lifetime through controlling the fouling problem [19]. One of the most important determining factors in manufacturing the membrane is the addition of an alternative polymer in the casting solution for improving the structure of the membrane and its performance. The concentration of PES influence on the PPSU membrane structural morphology was investigated at 0, 3, 4 and 5 wt.% of PES concentrations and at 20 wt.% PPSU concentration, for all fabricated membranes for the two sides regarding each one of the membranes (bottom and top surfaces), also for membrane’s cross-section. Images regarding the membrane’s top surface have been indicated in Figures (3a, b, c & d). It is indicated from Figure (3a) that the surface regarding membrane of the (PPSU 20%) has been dense, also it has few pore density, while it might be specified from Figures (3b, c & d) for membranes that are prepared from the (PPSU% and PES%) has showed a high density of pores blending with PPSU resulted in increased porosity. More pores are available on the surface at the concentration (20% PPSU-4% PES), also pores are small and less regular with concentrations (5%) PES, such behavior is because of the impact demixing, where the process has been slow with the increase in concentration PES that results in increasing polymer solution viscosity, hence get smaller pores and less dense. Figure3 showing the images of prepared membranes’ cross-section. Might be indicated from Figure3A, for a membrane that has been made from (PPSU 20%) and has a few finger-like structures has been created and the dominant structural was the sponge-like structure because of the dope solution’s high viscosity. Figures (3b, c, & d) shows a lot of finger-like structure in the membrane especially at concentrations (PPSU 20%-PES 4%), due to the difference of dope solution’s viscosity on the basis of percentage of the blending. The finger-like structure is preferred in the membrane structure and gives a high flux. Adding different concentrations of PES (3 and 4 wt. %) leads to an increase in the affinity produced between water and polymer mixture solution that will result in the accelerated exchange rate between water and solvent, thus creating a finger-like structure. Furthermore, increasing the PES concentration (5 wt%) in the dope solution leads to an increase in the solubility value between solvent and polymer mixture, also delayed outflow related to the solvent from PES/ PPSU mixture solution to water has been a result of the strong interaction between solvent and water [20]. Furthermore, the thickest top layer has been indicated in (20%PPSU) membrane because of the delay demixing in the case when phase separation, whereas the thin top layer created in other membranes which has an excellent affinity to non-solvent.

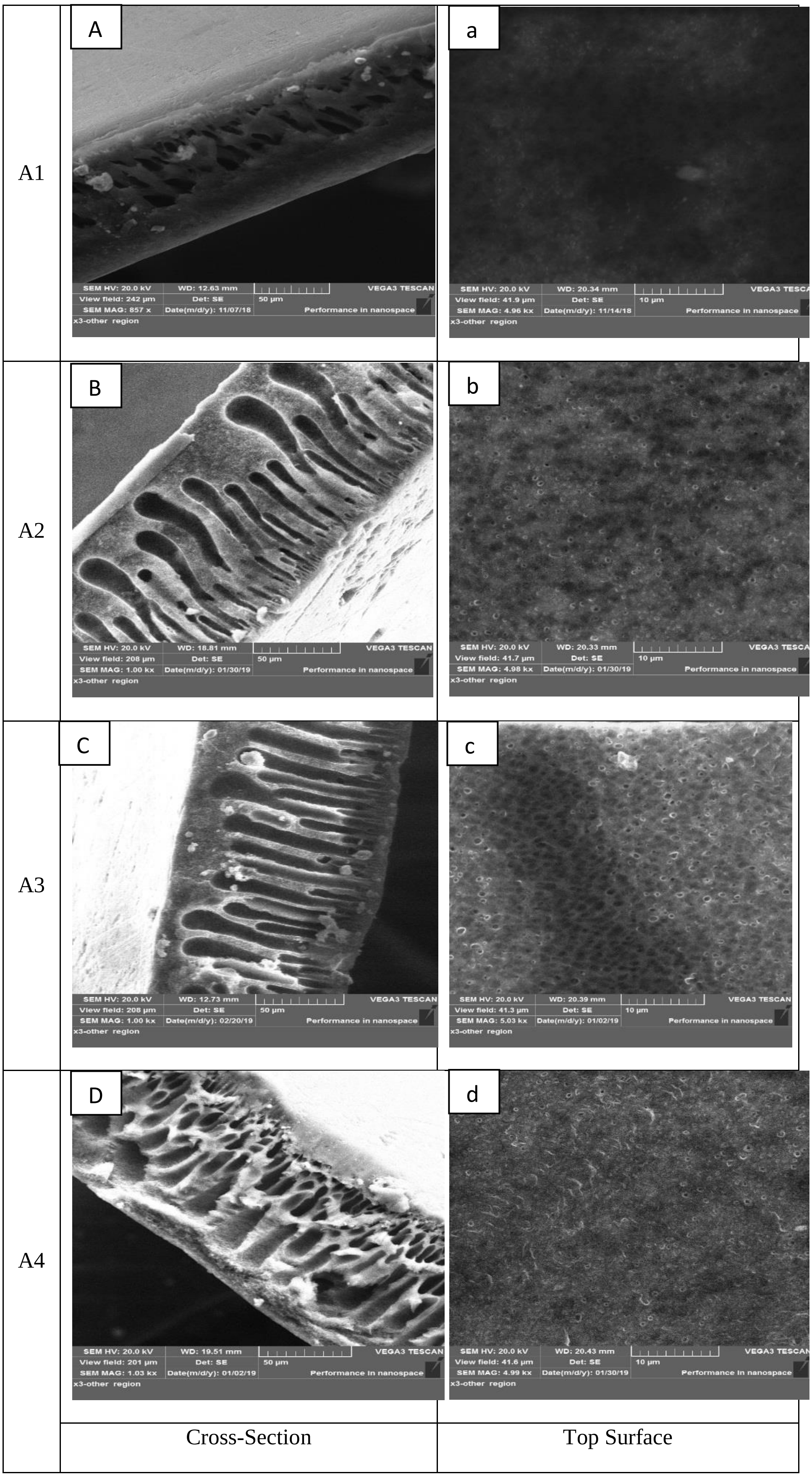

Figure 3: SEM images of PPSU and PPSU PES membranes at various PES contents

II: Membrane porosity

Membrane porosity is significantly affected by the type and the number of additives during the membrane preparation process [21]. The membranes' porosity that was made from neat PPSU and prepared from different concentrations of PES is depicted in Figure4. It is clear that the porosity of the membrane has increased with increasing the PES concentration of the polymer with the highest porosity by the 4%PES+20%PPUS. The membrane that made from (PES 3% & PPSU 20%) has a porosity equal to (61.31%) and it is increased to (72.3%) when the concentration has been increased to (PES 4% & PPSU 20%) and then decreasing to (51.12%) when the concentration is equal to (PES 5% & PPSU 20%). The least value of the porosity is provided by the membrane made from neat (PPSU 20%), which is equal to (21.22%). In case when increasing the PES concentration from 3% to 4%, the porosity value is increased, this might be due to increasing the number of finger-like shapes that are caused by the impact of blending regarding PES polymer with PPSU. where, adding different concentrations of PES (3, 4 wt%) leads to enhancing the affinity between water and polymer blend solution, which will result in the accelerated exchange rate between water and solvent, thus creating a finger-like structure. The macro-void and finger-like shapes in the blend membrane resulted in increasing the porosity, which was increasing with adding PES as finger-like structures related to membranes enhanced the pore interconnectivity. More increment in PES (5%) concentrations had not much impact because of agglomerations, which led to slowdown regarding the rate of solvent and non-solvent (water) demixing via phase inversion operation, which resulted in lower porosity in addition to minimizing the mean pore radius in the membrane[22].

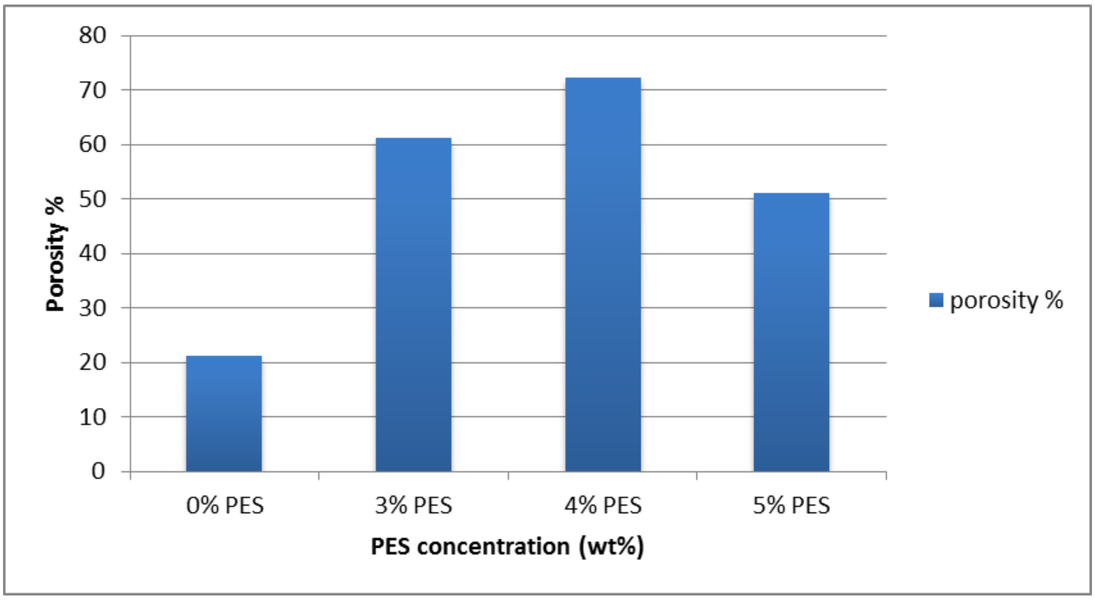

Figure 4: Effect of PES polymer concentration on the porosity of the membranes

III: Effect of PES Concentration on Rejection

Figure5, presents the membranes rejection showed by PPSU and PPSU\PES membranes for Cu, Zn, Pb, Cd, U, Ni, K, and Th elements .The operating pressure was 6 bar, time 1horse and temperature 25ºC. PES blend into PPSU resulted in an increase in retention of heavy and radiation elements. Furthermore, it can be inferred that the increased efficiency of the rejection with an increase of the addition ratio PES and then decreased at 5% PES. This result may be due to the higher immobility of PES in the membrane matrix in terms of its increased content in the dope solution. Such results have been due to the high immobility regarding PES polymers in the PPSU matrix despite their water solubility. Even though that the PPUS matrix has not been chemically cross-linked with the PES, there are strong interactions in the matrix and adequate for maintaining the matrix in the physically cross-linked state, which is immobilizing the PES chains, the led to membrane structures with high porosity with decreased size of pore and that has been the cause behind the elevated rejection with increasing PES content in PPUS membrane [23]. From Figure5, the maximum rejection was for PPSU+4 % PES membrane was 68.2, 75.3, 82.5, 79.3, 81.9, 64.7, 84.3 and 83.1% for Cu, Zn, Pb, Cd, U, Ni, K, and Th respectively . While notice less rejection was for 0% PES (PPSU only) was 25, 34.5, 50, 45, 48, 28.8, 59.34and 53% for, Ca,Fe,Pb,Ni,Cu and K respectively . The rejection of K, Th, and Pb are higher than other elements, the order of the rejection is K>Th>Pb>U>Cd>Zn>Cu>Ni. The result is showed that the ultrafiltration process is efficient in rejected the elements that occurred based on the size exclusion principle were observed increased rejected with increased metal ions radii [13].

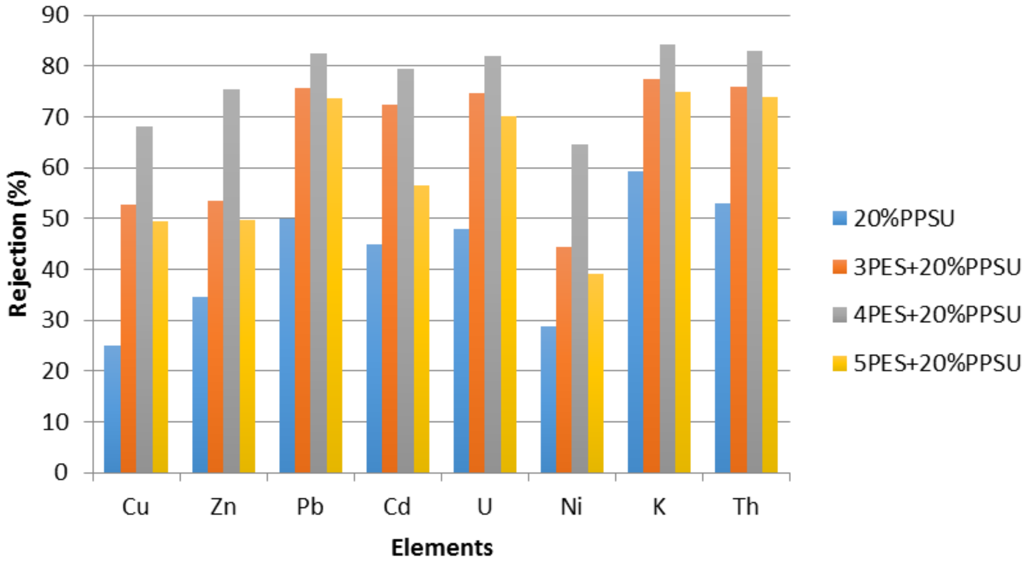

Figure 5: Effect of PES concentration on rejection at (6 bar, 1h and 25ºC)

IV: Effect of Temperature and Pressure on Rejection

Figure5 showed the effect of addition (PES) on membrane performance. Where noted the best result can be obtained by adding 4%PES+PPUS. Thus, selected for studying the impact of temperature, time and pressure on rejection.

A: Effect of operating pressure rejection of the heavy and radioactive elements.

Figure 6 clears the rejection of heavy and radiation elements at pressures 2,4 and 6 bar at contact time 60 minutes and temperature 25°C. It was evident that the percentage rejection of heavy and radiation elements increases with the increasing of the pressures which was only 60.4, 62, 79, 75, 78, 59, 82.8 and 80% at 2 bar was improved with increasing the pressure whose rejection percentage was found to be 68.2, 75.3, 82.5, 79.3, 81.9, 64.7, 84.3 and 83.8% at 6 bar for Cu, Zn, Pb, Cd, U, Ni, K and Th respectively. It has been indicated that there is an increase in rejection as the operating pressure is increased, similar results were found by [24]. At low operating pressure, fouling is prospected to occur on the surface of the membrane as some suspended solids did not get the driving force required for pushing them into the retentate side. Fouling indicates membrane resistance increasing throughout a process. It is the phenomenon that normally occurred due to the material's deposition and adsorption on the membrane. Previous studies demonstrated that the solute flux highly related to the gradient of the concentration within the membrane. Therefore, when increasing the pressure of the transmembrane, the solute goes through the membrane due to the faster rate of solute pushing achieved by the water that in turn assists the transportation of the solute. Consequently, the rejection has been increased with pressure increasing. The present work results were in agreement with the work achieved by [25].

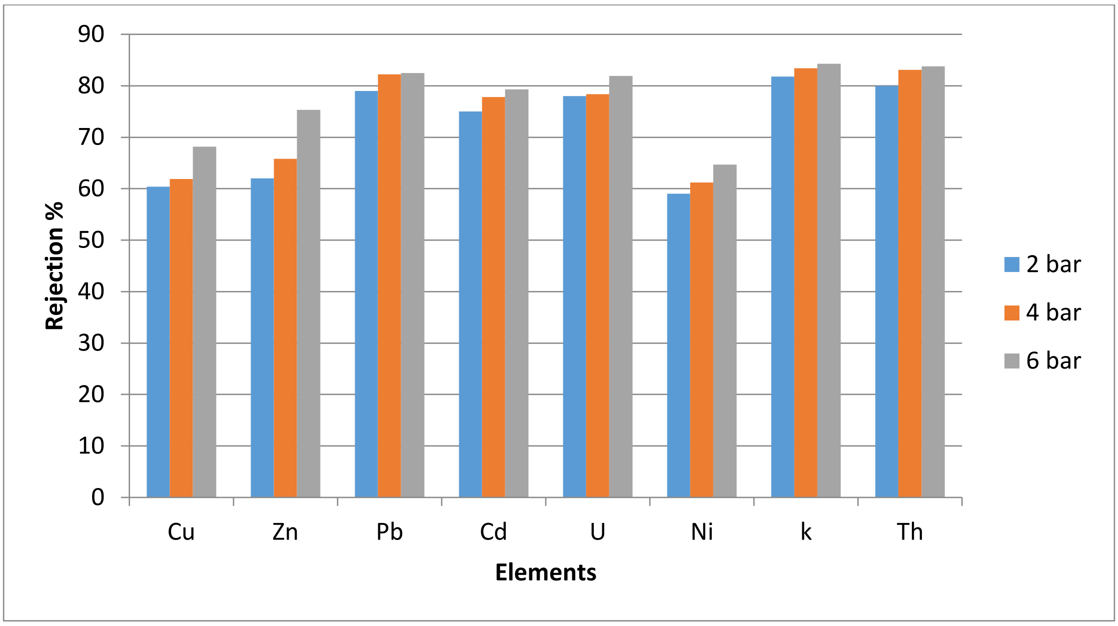

Figure 6: Effect of pressure on the rejection of 4%PES+PPUS membrane at (1h and 25ºC)

B: Effect of temperatures on rejection for heavy and radioactive elements

Table II, shows the effect of the temperature on the rejection of heavy and radioactive elements. At constant conditions like the pressure of 6 bars and operating time 1 hour. Increasing the temperature of the feed solution lead to decrease rejection of heavy and radioactive elements .The temperature has an impact on both the solution and membrane properties. With temperature variations there are changes in the structural parameters of the membrane, as previous studies have proven, for instance, increasing temperature leads to decrease membrane thickness and increase pore radius [26]. The results are showing that the elements' rejection has been decreasing as the temperature increased; comparable results are indicated by [26]. Solvent and solute transport often increasing with increasing the temperature; thus, as the temperature is increased, there will be a decrease in the rejection ratio [27].

TABLE II: Effect of temperature on the rejection of 4%PES+PPUS at (6 bar,1h)

Elements	**Temperature (°C)**		
	25 °C	**35 °C**	**45 °C**
Cu	68.2	62.5	62.2
Zn	75.3	64.4	62
Pb	82.5	78.8	74.8
Cd	79.33	75	70.9
U	81.9	77.1	73
Ni	64.7	62.4	62.1
K	84.3	80.8	78.5
Th	83.8	79.8	76.8

C: Effect of time on rejection for heavy and radioactive elements

Figure7 clears the impact of time on percentage rejection of heavy and radioactive elements, using 60, 90, and 120 min, with pressure 6 bars and temperature 25 °C. It can be seen that the rejection of heavy and radioactive elements with 60 min, 68.2, 75.3, 82.5, 79.3, 81.9, 64.7, 84.3 and83.8% for Cu, Zn, Pb, Cd, U, Ni, K, and Th respectively. Where using 90 min, gives rejection percentages of was69.4, 75.8, 83.8, 79.9, 82.3, 66, 85 and 85.8 for Cu, Zn, Pb, Cd, U, Ni, K, and Th respectively.

While with 120 min, the rejection was high when compared with 60 and 90 min, where the rejection of heavy and radioactive elements was 70.1, 76.4, 84.2, 80, 82.9, 67.9, 86.1 and 86% for Cu, Zn, Pb, Cd, U, Ni, K, and Th respectively. As time is increased, the rejection efficiency increases due to the fouling. There are two fouling effects on the rejection. Firstly, increasing in rejection is owing to the decrease in the size of the pores membrane. Secondly, the improvement in the membrane's separation behaviour arises from the deposit. This improvement led to an increase or decrease in rejection [28].

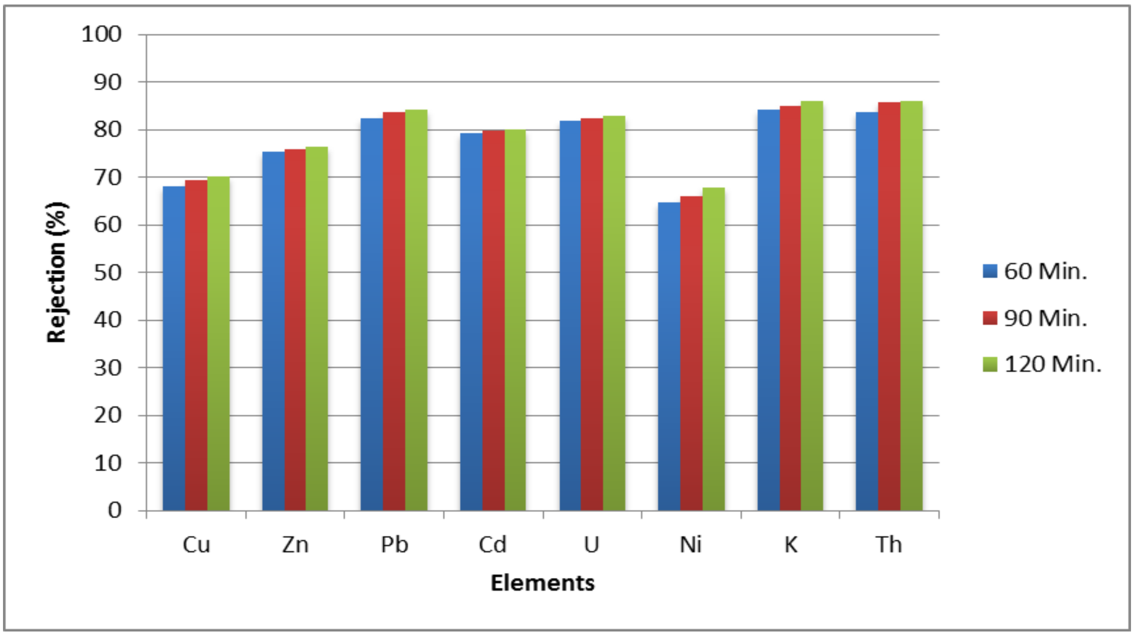

Figure 7: Effect of time on the rejection of 4%PES+PPUS membrane at (6 bar and 25ºC)

5. CONCLUSIONS

1- Polymer blending can improve the morphological structure of membranes. Adding PES equal to 4% led to improve the membrane properties such as the mean size of the pore, hydrophilicity of the membrane and narrower distribution of pore size.

2-The rejection of heavy and radioactive elements for the UF membrane increases with the increase of operating pressure.

3- By increasing the temperature, the rejection of heavy and radioactive elements for the UF membrane decreased.

4-By increasing the operating time, the rejection of heavy and radioactive elements for the UF membrane increased.

5- The rejection of K, Th, and Pb are higher than other elements, the order of the rejection is K>Th>Pb>U>Cd>Zn>Cu>Ni.

ACKNOWLEDGEMENTS:

The authors are grateful to the management and department of production and metallurgy engineering, university of technology, for assisting and supporting this work.

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