Development and Preparation of Microfiltration Polyethersulphone Membrane for Catalytic Membrane Application

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ABSTRACT

The use of catalytic membrane reactor, as promising technology is recommended for carrying out heterogeneous reactions. If reactants are forced to flow through membrane pores, which are impregnated with convenient catalyst, intensive contact provides high catalytic activity with negligible diffusive resistance. Polyethersulphone (PES) is widely used for preparation of microfiltration (MF), ultrafiltration (UF) membranes and it has characteristics such as wide temperature limits, wide pH tolerances, fairly good chlorine resistance, wide range of pore sizes available for UF and MF applications ranging from 10 Å to 0.2 µm. It is also characterized by good chemical resistance to aliphatic hydrocarbons, alcohols, and acids leading to consider PES as a good candidate for application on the membrane reactors. The best conditions for PES membrane preparation to be suitable for grafting process were 8:2.5 PES/PEG ratio, and 2 g / tween-20/10 g PES/PEG at 6 h mixing time. Grafting of sulfonic group on Polyethersulphone membranes was developed to catalyze esterification reactions. The grafting process comprises; initiation, polymerization and termination steps. The developed membrane was also tested in esterification reaction of acetic acid and ethanol; the reaction conversion has reached 82.6% after 10 sec which was similar to the reaction conversion provided by grafted purchased membrane.

Key words: Catalytic membrane; Membrane preparation; grafting process; Esterification reaction.

Introduction

The subject of membrane reactors (MRs) has attracted much interest over the last decade (Ozdemir, et al., 2006). Membrane research across the world is still concerned with the development of new techniques and the comprehension of the phenomena in membrane formation. This is due to the difficulties to obtain membranes with the desired properties. Different methods of polymer membrane preparation have been covered in several reviews (Nunes, and Peinemann, 2006). Most membrane technologists prefer the use of polyethersulphone (PES) due to its lower hydrophobicity when compared to other polymers usually used for membrane preparation (Solvay, 2004). One of the important impediments for further commercial development of MRs are the membrane themselves, which still need optimization (Ozdemir, et al., 2006).

There are several ways to prepare porous polymeric membranes, such as sintering, track etching and phase separation processes (Van de Witte et al., 1996). The majority of polymer membranes used for micro and ultrafiltration of liquids are prepared by the process of immersion precipitation (Akhakul et al., 2002), in which a film of concentrated polymer solution is casted on a suitable substrate and subsequently immersed in a non-solvent bath where the replacement of solvent by coagulant and the precipitation of polymer take place.

However, the classical procedure is not adaptable enough to produce all the desirable membrane structures and properties. Modifications of the basic procedure are usually needed, including the addition of suitable additives to the casting solution or the coagulation bath, introducing additional steps, such as evaporation (Mosqued-Jimenez et al., 2004) or annealing (Bumsuk and co-authors, 2005) and coupling chemical reaction with phase separation process [Yang & Liu, 2003]. Changing the composition in the casting solution or the coagulation bath has been one of the convenient and efficient methods to prepare membranes with optimal structure and special properties (Arthanareeswaran et al., 2010). The additives may be inorganic salts (Shinde et al., 1999), polymer (Liu et al., 2003), mineral fillers (Ebert et al., 2004) and even nonsolvents for membrane materials (Lai et al., 1996). To sum up, the role of these additives is to suppress the formation of macrovoids, enhance pore formation and improve pore interconnectivity and/or hydrophilicity.

Polyethersulphone (PES) is widely used for preparation of microfiltration (MF), ultrafiltration (UF), and gas separation (GS) membranes. It has been suggested as a polymeric material for preparation of phase inversion membranes. Because it has favorable characteristics such as wide temperature limits, wide pH tolerances, fairly good chlorine resistance of pore sizes available for UF and MF [Urbanczyk co-authors, 2010].
A novel micro porous membrane has been developed using the polymer polyethersulphone. The sulphone groups tend to make the polymer stiff with a high glass transition temperature and together with the ring structures tend to make the polymer chemically resistant and relatively hydrophobic. The ether linkages, derived from its precursor, make the polymer more flexible and processable. These groups also make the polymer less hydrophobic and more amenable to further treatment to manufacture hydrophilic membranes. Without these functional groups, it would be more difficult to produce a hydrophilic micro porous membrane without complicated processing [James and co-workers, 2002].

In order to obtain membranes with special properties, additional additives can be dissolved in the casting solution (Barth et al., 2000). The role of these additives is to create a spongy membrane structure but prevents the formation of macrovoids, enhances pore formation, improves pore interconnectivity and/or introduces hydrophilicity (Rahman et al., 2008). Generally, hydrophilic structures are obtained by the addition polyethylene glycols (PEG) (Anildris et al., 2009). Other frequently used additives are: glycerol, alcohols, dialcohols, water, polyvinylpyrrolidone (PVP), polyethylene oxide (PEO), LiCl and ZnCl₂ (Kesting, 1981; Munari et al., 1988; Kraus et al., 1979).

Addition of surfactant additives to the casting solutions can influence morphology and performance of the membranes [Munari and co-authors, 1988]. Some researchers studied the effects of surfactant additives with hydrophilic properties on the membrane morphology and performance. Rahman et al. (2008) studied the effects of Tetronic-1307 as a surfactant additive on morphology and performance of PES porous hollow-fiber membranes and found that increasing the Tetronic 1307 content increases hydrophilicity of the membranes.

Alsari et al. (2001) used sodium dodecyl sulphate surfactant additive as gelation media in formation of PES membranes. Rahimpour and co-workers (2007) investigate the effects of Triton X-100 as a nonionic surfactant additive. There are few previously published articles regarding addition of Tween series surfactants as hydrophilic additives for improvement of permeation and anti fouling properties of the PES membranes.

Flows through catalytic membrane reactors are use catalytic porous membranes where the reaction mixture is convectively passed through membrane pores. The polymeric porous membrane doesn't perform any separating task but is used as micro structured catalyst support. However, the concept of catalytic membrane reactor is to allow a high catalytic activity due to an intensive contact between reactants and catalyst with a narrow residence time distribution (El-Zanati et al., 2011; Westermann et al., 2009).

To modify the properties of polymeric membrane according to membrane catalysis, grafting process for membrane can be applied. The functionalization of polymer grafts is accomplished by cationic polymerization of styrene and substituted styrene monomers. Sulfonic acid groups (SO₃⁻) immobilized in the pores of the microfiltration membrane act as initiator for cationic polymerization. However, completion of grafting process the membrane becomes catalytic membrane (Bhattacharya & Misra, 2004).

Estrification reactions are industrially carried out under batch conditions in homogeneous liquid phase. Strong acids, such as sulphuric acid, hydrochloric acid or ortho-phosphoric acid, are usually used as catalysts (Castanheiro et al., 2006). However, these strong mineral acids are corrosive and are usually separated by neutralization after reaction. The use of solid acid catalysts for these reactions is preferable as they can be easily separated from the bulk of the reaction (Castanheiro et al., 2006; Iachon et al., 2003).

In this work, Tween-20 as a hydrophilic surfactant additive was selected to blend with PES in the membrane preparation process. The effects of addition of Tween-20 as a nonionic surfactant and PEG as a pore former polymeric additive in the casting solution on morphology and fouling-resistant ability of the PES membranes were investigated.

After, preparation and development of PES membrane to reach the optimal preparation conditions, grafting of sulfonic group to membrane backbone was studied and the esterification reaction using ethanol and acetic acid was carried out on the modified membrane to test its stability and efficiency as a catalyzing membrane.

2. Experimental:

2.1. Materials:

Polyethersulphone (PES Ultrason E6020P with MW ¼ 58,000 Da) supplied by BASF Germany was used as polymer for preparation of the membrane casting solution. This polymer absorbs moisture very rapidly. Therefore, the polymer was dried for more than 12 hr at 100–105°C prior to the process. N, N-dimethylformamide (DMF) was used as solvent provided by ADWIC, El-Nasr Pharmaceutical Chemicals Co., Egypt. Polyethylene glycol (PEG, reagent grade, MW = 400 Da) supplied by Sigma – Aldrich, Belgium were used as a pore former polymeric additive in the casting solution. Tween-20 (Polyoxyethylene sorbitan monolaurate, HLB =16.7) supplied by Sisco, India was used as a nonionic surfactant additive in the casting solution. Distilled water was used as the main non-solvent in the coagulation bath. Concentrated sulfuric acid (99 wt %), methanol, and 0.1N KOH, styrene monomer (99.8%) and toluene were used in grafting step. The used reactants for the esterification experiments were pure ethanol and glacial acetic acid.
2.2. Preparation of the membranes:

Asymmetric flat sheet PES membranes were prepared by the phase inversion method. To the homogeneous solution of PES in DMF, PEG 400 as polymeric additive and Tween-20 as surfactant additive were added and mixed by stirring at room temperature. The variations of PES/PEG ratios were done to investigate the impact of PES/PEG on membrane porosity.

The stirring speed was studied at 400, 800 and 1200 ± 20 rpm. When necessary, an ultrasonic bath was employed to help freeing up of the air bubbles entrapped in the polymeric solution. The prepared homogeneous solutions were casted using a film applicator to 250 lm clearance gap on a glass plate substrate. Composition of the casting solutions is shown in Table I.

The glass plate carrying the PES/PEG-DMF film was moved to distilled water coagulation bath at room temperature, while having the glass plate slide in at approximately 30 degrees. The immersing time was adjusted to obtain uniform opaque white colour. The membrane will spontaneously release the plate and kept for 24 hr to ensure complete removal of PEG from the membranes aiming at pore formation. Finally, the membranes were dried by placing between two sheets of filter paper for 24 hr at room temperature.

### Table 1: Preparation conditions of PES/PEG membranes

<table>
<thead>
<tr>
<th>Solvent (ml/10g PES/PEG)</th>
<th>60</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stirring time (hr)</td>
<td>2</td>
</tr>
<tr>
<td></td>
<td>4</td>
</tr>
<tr>
<td></td>
<td>6</td>
</tr>
<tr>
<td></td>
<td>8</td>
</tr>
<tr>
<td>PES/PEG ratio (wt/wt)</td>
<td>8:1.5</td>
</tr>
<tr>
<td></td>
<td>8:2</td>
</tr>
<tr>
<td></td>
<td>8:2.5</td>
</tr>
<tr>
<td></td>
<td>8:3</td>
</tr>
<tr>
<td>Tween 20 dose (g/10g PES/PEG)</td>
<td>1.5</td>
</tr>
<tr>
<td></td>
<td>2</td>
</tr>
<tr>
<td></td>
<td>2.5</td>
</tr>
<tr>
<td></td>
<td>3</td>
</tr>
<tr>
<td>Stirring speed (rpm)</td>
<td>400 ± 20</td>
</tr>
<tr>
<td></td>
<td>800 ± 20</td>
</tr>
<tr>
<td></td>
<td>1200 ± 20</td>
</tr>
</tbody>
</table>

2.3. Characterization of the membranes:

2.3.1. Scanning Electron Microscopy (SEM):

Samples of prepared and grafted membranes were coated with gold to provide electrical conductivity. The micrographs were taken on a JEOL 5410 scanning electron microscope (SEM) operating at 20 kV.

2.3.2. Determination of mean pore size, surface porosity and membrane roughness:

The membranes properties; pore diameter, porosity, bulk and apparent density determined after interfacial polymerization using Pore Sizer, Micromeritics model 9320, USA, at the Center of Research and Development of Metals, Egypt.

2.4. Grafting process:

Grafting the polyethersulphone (PES) membranes was developed in three steps. The first step involves formation of the initiator by permeating 1N H2SO4 solution through PES membrane at 60 °C under a pressure drop of about 2 bars for 1 hour. The feed tank was pressurized with nitrogen to prevent contamination. Permeate was collected under atmospheric pressure. The second step is polymerization of styrene, where 12% volume of styrene in toluene was used for 10 minutes at a constant pressure drop of 2 bar and at 25 °C. The third step is to activate the graft through permeation of 1 N H2SO4 through the membrane for 1 hr at a constant pressure drop of 2 bar and at 25 °C. The membrane was then rinsed with water and dried in air. Figure (1) illustrates setup of grafting and esterification setup comprises membrane reactor, the feeding tank, and a nitrogen cylinder for pressure control. The membrane reactor consists of a stainless steel membrane holding the grafted PES membrane (Shah, Goodwin & Ritchie, 2005; Shah, 2003).
2.5. Estrification reaction:

The estrification of ethanol and acetic acid with molar ratio 10:1 was investigated at 25°C using developed casted membrane PES membrane of 47 mm diameter. Experiments were accomplished using two different grafted membranes under the conditions indicated in table (2).

Table 2: The membranes grafted conditions

<table>
<thead>
<tr>
<th>Process</th>
<th>Membrane I</th>
<th>Membrane II</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initiation</td>
<td>Concentration (gmol/l H2SO4)</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>Temperature (°C)</td>
<td>60</td>
</tr>
<tr>
<td></td>
<td>Time, h</td>
<td>1</td>
</tr>
<tr>
<td>Polymerization</td>
<td>Styrene (%)</td>
<td>12</td>
</tr>
<tr>
<td></td>
<td>Temperature (°C)</td>
<td>25</td>
</tr>
<tr>
<td></td>
<td>Time, min</td>
<td>10</td>
</tr>
<tr>
<td>Activation</td>
<td>Concentration (gmol/l H2SO4)</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>Temperature(°C)</td>
<td>25</td>
</tr>
<tr>
<td></td>
<td>Time(hr)</td>
<td>1</td>
</tr>
</tbody>
</table>

Also, estrification reaction of ethanol and acetic acid was performed using grafted ready-made membrane (I) and grafted casted membrane (II) and it was repeated, to assure the reproducibility. The estrification reaction was done at the conditions; 10:1 MR ethanol to acetic acid, 25°C and 2 bars.

Results and Discussion

3.1. Effect of Stirring time:

Microscopic studies using SEM imaging were carried out to reveal qualitative information regarding morphology of the prepared membranes. Figure 2 displays the typical asymmetric spongy structure in the sublayer for the PES membrane which prepared in absence of PEG or Tween-20. SEM images of the prepared PES/PEG membranes using stirring speed 400±20 rpm, PES/PEG ratio 8:2, Tween – 20 dose 1ml /10gr of PES and PEG and different stirring times are presented in Figures 3, 4, 5, 6.

The effect of stirring duration on morphology of membranes was studied. Figure 3 represents the SEM images of the sample prepared by mechanical stirring of PES, PEG and Tween 20 for 2 hours and kept in ultrasonic bath for an hour. It is clear that the pores are few and not dispersed homogenously, where as increasing the stirring duration (4, 6 and 8 hour) caused an improvement in the number of pores and its homogeneity as shown in Figures 4, 5 and 6.
Figure 7 illustrates the dependence of PES/PEG membrane porosity upon stirring duration. The Increase in the percentage porosity is directly related to the mechanical stirring time of prepared membrane. It was evident that percentage porosity increased sharply upon prolongation of the stirring time up to about 6 hours and thereafter levelled off. Results revealed that percentage porosity were 76.38% to 87.41% and 88.01% using of mechanical stirring duration of at 2, 6 and 7 hours respectively.

Also Average pore diameter of PES/PEG membrane is related to the mechanical stirring duration. Figure 8 shows the relation between stirring time and the average pore diameter of the prepared membrane. The average pore diameter was 0.1395 μm, 0.0831 μm and 0.0873 μm at stirring time 2, 6 and 7 hours respectively. Upon correlation between Figures 7 and 8, it can be concluded that average pore diameter of the prepared PES/PEG
membrane is inversely proportional to the data of percentage porosity. The average pore diameters were found to be in accordance with those shown in Figure 8, whereas the average pore diameters was inversely proportional to percentage porosity.

3.2. Effect of PES/PEG ratio on membrane porosity:

The effect of PEG dose as pore former in the membrane structure were examined by SEM. Variation of the amount of PEG in the membrane structure changes the number of pores. Figures 6, 9, 10 and 11 represent the SEM images of a membrane prepared with PES/PEG ratio of 8:1.5, 8:2, 8:2.5 and 8:3 wt/wt, respectively. Obviously, there is a relationship between PES/PEG ratio and porosity of the membrane. The number of pores increases with decreasing ratio between PES and PEG. Also pore size were affected by ratio between PES and PEG, where increasing the ratio caused a decrement in the pore size. This is may be due to the effect of the non-ionic surfactant dose on the dispersion and surface tension of the PEG droplets in the casting solution. Decreasing PES/PEG ratio means higher amount of PEG, hence the pore size increased due to ineffectiveness of the surfactant for dispersing PEG droplets. The opposite is holds true when increasing PES/PEG ratio, the particles of the mixture become smaller, subsequently, and smaller pores are formed. Moreover, SEM images of the prepared membrane indicate that the distribution of pores influenced by PES/PEG ratio used for preparation. Among the images investigated, the use of PES/PEG ratio of 8:2.5 led to the formation of highly distributed pores in the membrane sample under investigation. Thus, it can be concluded that within the range of ratios studied, best ratio for membrane preparation is 8:2.5.

Fig. 7: The effect of stirring time on the percentage porosity of PES/PEG membrane

Fig. 8: The effect of stirring time on the average pore diameter of PES/PEG membrane.

Fig. 9: SEM images of PES/PEG membrane prepared with ratio of 8:1.5

Fig. 10: SEM images of PES/PEG membrane prepared with ratio of 8:2.5
3.3. Effect of Tween 20 dose on pores distribution:

Tween 20, in particular, is a non-ionic surfactant, it is often added to prevent and/or minimize aggregation during mixing (Ziani et al., 2008). Tween 20 enhances the intermolecular attractive forces at the surface of PEG and possesses the ability to increase its surface tension. The action of various concentrations of the non-ionic surface active agent (Tween 20) on pores of the PES/PEG membrane was examined. The mixture of PES/PEG consists of two phases: continuous phase (PES soluble in DMF) and droplet phase (PEG) as shown in Figure 12.

In this work, Tween-20 as a hydrophilic surfactant additive was selected to blend with PES/PEG mixture to improve the porosity and pores distribution. The effects of Tween-20 on porosity of the PES/PEG membranes were investigated. SEM images of the prepared PES/PEG membranes using different concentrations of Tween-20 as surfactant additive (1.5, 2, 2.5 and 3 g/10g PES/PEG) are presented in Figures 10, 13, 14, and 15. As expected, the presence of Tween 20 was the most important factor affecting distribution and pores size of the membrane. Tween-20 increases significantly the surface tension of PEG and subsequently the size and distribution of the droplets by which the pores are formed. Data revealed that the distribution of membrane pores were found to be homogeneous by increasing the Tween-20 dose from 1 to 2 and 2.5 g/10g PES/PEG of the casting solution as shown in Figures 13, 16 and 17. It is evident from figures 16 and 17 that there are no significant difference in pores size and their distribution. Further increase in the concentration of Tween-20 in the casting solution to 3 g/10g PES/PEG enhances the pores distribution but the pores became smaller as shown in Figure 18. So that it is logical to choose Tween-20 dose 2, g/10g PES/PEG as the optimum one. These results were in accordance with those of the report published by Amirilargani et al., (2010).

3.4. Effect of stirring speed:

A study of the effect of variation in stirring speed employed in membrane preparation on the porosity as well as average pore size of the product is undertaken. The mixture was stirred constantly at three stirring speeds of 400, 800 and 1200 rpm.

Data of porosity and average pore size of the so-obtained membrane and their dependence on the stirring speed are displayed in table 3. Results indicated that increasing the stirring speed from 400 to 800 rpm caused
remarkable increase in porosity value from 70.21 % to 83.12 %, whereas further increase in stirring speed to 1200 rpm caused a drop in porosity to be 76.85 %. This can be attributed to formation of higher number of droplets leading to an increase in average pore diameter from 0.1463nm to 0.1617 nm at stirring speeds of 400 rpm and 800 rpm respectively. When stirring is conducted at 1200 rpm, relatively higher average pore size evaluated as 0.3072 was observed. This can be explained in terms of increased number of droplets having weak boundary layer and adjacent to each other, and therefore, when PEG is dissolving in the coagulation bath, two or more droplets are combined to form one droplet with longer diameter. Moreover, it has been reported that in membrane preparation the speed of the continuous phase flow and the adsorption kinetics of the surfactant, which is added in the continuous phase, have a significant effect on the size and size distribution of the droplets (Dong Hoon et al., 2011).

Table 3: Effect of the use of different stirring speed on Porosity and Average pore diameter average pore diameter of the prepared PES/PEG membrane.

<table>
<thead>
<tr>
<th>Stirring speed (rpm)</th>
<th>Porosity (%)</th>
<th>Average pore diameter (nm)</th>
</tr>
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<tbody>
<tr>
<td>400</td>
<td>70.21</td>
<td>0.1617</td>
</tr>
<tr>
<td>800</td>
<td>83.12</td>
<td>0.1463</td>
</tr>
<tr>
<td>1200</td>
<td>76.85</td>
<td>0.3072</td>
</tr>
</tbody>
</table>

Figures 16, 17 and 18 show the effect of stirring speed on the morphology of the prepared membranes. The size and distribution of pores are evidenced by the previous explanation.

3.5. Grafting process:

Grafting process was carried out on purchased membrane and developed casted membrane for comparison, using 1N H₂SO₄ at 60°C at 1hr initiation step of grafting, 12% styrene (vol.) in toluene for 10 min at polymerization step of membranes and 1N H₂SO₄ at 25°C for activation step of grafted membranes. Figure 19 indicates
the appearance of the membrane after grafting process for purchased membrane at the same magnification power, the membrane showed a rubbery appearance and the electron scan of membrane indicates reduction in pore numbers due to the occurring of more blocking, where the fully extended graft chains would mandate increasing in pore size. Figure 20 indicates the totally grafted casted PES membrane where the reduction in pore numbers also is watched, the scanning was conducted at two magnification power (a) 2500 and (b) 5000.

**Fig. 16:** SEM images of PES/PEG membrane prepared with ratio of 8:2.5 and Tween 20 dose 2.5 g/10g polymers for stirring time 6 hr and stirring speed 400 rpm

**Fig. 17:** SEM images of PES/PEG membrane prepared with ratio of 8:2.5 and Tween 20 dose 2.5 g/10 g polymers for stirring time 6 hr and stirring speed 800 rpm

**Fig. 18:** SEM images of PES/PEG membrane prepared with ratio of 8:2.5 and Tween-20 dose 2.5 g/10 g polymers for stirring time 6 hr and stirring speed 1200 rpm

**Fig. 19:** Electron scan on commercially available membrane after grafting step
3.6. Application of catalyzed membranes using esterification reaction:

Comparative study of esterification reaction catalyzed by the grafted membranes using ethanol and acetic acid with molar ratio 10:1 was investigated at 25°C using developed prepared PES membrane of 47 mm diameter. Experiments were accomplished using two different grafted membranes at conditions of table (2). The conversion is approached to 87.35% using developed grafted membrane I, while the conversion was 75.6% using the grafted membrane II as shown in Figure 21, where the enhanced performance is referred to the improvement of grafting process.

Also, esterification reaction using ethanol and acetic acid was performed using grafted purchased membrane (I) and grafted developed casted membrane (II), to assure that the reproducibility of performance of membrane (II), and the experiment was duplicated to confirm the results. The esterification reaction was carried out at conditions of: 10:1 MR ethanol to acetic acid, 25°C and 2 bars. The reaction conversion using purchased membrane (I) was 87.35% after 10 sec, while it was 82.6 % using developed casted and grafted membrane (II), (Figure 22).

Fig. 21: Comparison between two different grafted membrane I and II, 25°C
Fig. 22: Estriﬁcation reaction using grafted ready-made membrane (I) and grafted casted membrane (II).

Conclusion:

Development of new polymeric membrane materials is one of the most important elements in the quest for advances and improvements in membrane technology. Asymmetric flat sheet PES membranes were prepared by the phase inversion method. The homogeneous solution of PES in DMF, PEG 400 as polymeric additive and Tween-20 as surfactant additive were added and mixed by stirring at room temperature. The optimal conditions of prepared membrane to be suitable for grafting process were 8:2.5 PES/PEG ratio and 2 g /10 g Tween-20 at 6 hr mixing time. Grafting of sulfonic group on polyethersulphone membranes was developed to catalyze estriﬁcation reactions. Grafting process was applied on purchased membrane and developed casted membrane for comparison, using 1N H2SO4 at 60ºC at 1hr initiation step of grafting, 12% styrene (vol.) in toluene for 10 min at polymerization step of membranes and 1N H2SO4 at 25ºC for activation step. The membrane showed a rubbery appearance and the electron scanning of membrane indicates reduction in pore numbers due to the occurring of more blocking, where the fully extended graft chains would mandate increasing in pore size. Comparative study of estriﬁcation reaction catalyzed by the grafted membranes using ethanol and acetic acid with molar ratio 10:1 was investigated at 25ºC using developed prepared PES/PEG membrane of 47 mm diameter, where the reaction conversion has reached to 82.6% after 10 sec which was matched with previously obtained data of grafted commercially available membrane.

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