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PERFORMANCE STUDIES OF POLYSULFONE-BASED MEMBRANE: EFFECT OF SILICA MORPHOLOGY

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Abstract.
In the present work, polysulfone (PSf) ultrafiltration membranes were prepared by solution casting. The effects of rice husk silica (RHS) on the surface properties of the PSf/Polyethylene glycol (PEG) membrane were observed and investigated. Characterizations were conducted to determine the membrane cross-section area and RHS distribution. The structure of RHS and morphology of membrane were analyzed by using X-ray diffractometer (XRD) and scanning electron microscope (SEM). XRD pattern showed that the amorphous silica was produced from rice husk ash (RHA). The analysis of SEM indicates that the addition of RHS obviously changed the microstructure of the membrane especially at top layer and sub layer.

Introduction

Polysulfone (PSf) membrane has been widely used in fabrication ultrafiltration membrane due its low cost, superior film ability, good mechanical and anti-compaction properties and strong chemical and thermal stabilities [1]. However, the hydrophobic nature of PSf gives major problem in water filtration that is fouling. The fouling mechanism is described as pore blocking, solute aggregation or adsorption phenomena. Membrane fouling is characterized in general as a reduction of permeate flux through membrane, as a result of the increased of flow resistant due to pore blocking, concentration polarization and cake formation. The effect of this fouling mechanism on flux decrease depend of factors such as membrane pore size [2] , solute loading and size distribution [3].

Membrane surface modification is one method that can be used to increase hydrophobicity and minimize the fouling problem. The common modification method that used to improve the performance is addition of additive into membrane formulation. The addition of small amount of additive into casting solution can lead to significant changes to the characteristic and performance of membrane [4, 5, 6]. It is reported that additives can affect the final membrane characteristic either by changing the solvent capacity or by changing phase separation kinetic and thermodynamic mechanism [7, 8]. The additive have tendency to enlarge macrovoids formation which then improve the interconnectivity of the pores and resulting in higher porosities in the top layer and sub layer [9]. Furthermore, the addition of additive into solution may contribute to the changeable properties of membrane in term of pore distribution, physical properties and mechanical characteristics [10]. According to previous studies, by using fumed silica as an additive**,** membrane conductivity and mechanical properties was improved [10]. Other co-workers that used graphite, silica, zeolite, metal oxide nano particles or carbon nanotube also proved that the membrane properties i.e permeability and fouling resistance increased [5, 11, 12,13].

In this work, the effect of addition RHS into polysulfone membrane were investigated. RHS was considered to be in an amorphous phase. According to Mishra *et al*, amorphous silica content in the rice husk are approximately 92 – 97 wt% [14]. Beside that it also offers a biodegradable properties and green technology to membrane separation process.

Preparation of RHS

RHS was prepared by burning rice husk in a furnace at 600 ^{0}C for one hour. The process of RHS burning and cooling takes about 24 hours [15]. After that, the RHS was sieved until the size is less than 25μm. The RHS was used as an additive for all formulation.

Membrane preparation

PSf membranes were prepared by using the phase inversion method. Casting solution were prepared by dissolving PSf in N-methyl-2-pyrrolidone (NMP) and stirred for 4 h. Then, PEG and RHS additive at different concentration (0%, 0.5%, 1.0%, 1.5%, and 2%) was subsequently added with continuous stirring and heating at 60 °C until the solution was completely homogeneous. Subsequently, the casting solution sonicated for 1 h to release the bubbles. The solution was then cast by using flat sheet membrane casting system and immersed in a coagulation bath containing distilled water. Finally, the flat sheet membranes were dried for 24 h.

Membrane Analysis

The XRD analysis was carried out by using XRD Bruker D8 advance with a 40kV scaled copper tube as source and a graphite crystal as monochromator. 150 mg of RHS was used with diffraction angles of 2θ, 20-90°. Membrane morphology was examined using a scanning electron microscope (SEM). Membranes were immersed in liquid nitrogen, fractured carefully then coated with a thin gold prior to SEM analysis.

Results and Discussion

X-ray diffraction (XRD)

XRD pattern as shown in Figure 1 were obtained from 2θ range of 20˚ to 90˚ at 40 kV and 20 mA. Figure 1 shows a typical pattern of amorphous silica with higher broad peak observed at about 2θ=25 ˚. This broad peak indicates that silica generated from rice husk ash (RHA) at 600 ^{0}C is in amorphous phase.

Membrane Morphology

Figure 2 shows the SEM image of different membranes cross section prepared with different percentage of rice husk silica as an additive. The SEM images indicate that membrane is having an asymmetric structure consisting of a dense top surface layer (skin layer, air side), a porous sublayer (support layer) and a small portion of sponge-like bottom surface layer (glass side). The skin layer acts as a separation layer and the support layer provides the mechanical strength. The sub layer seems to have finger like cavities beneath the top surface layer and large voids near the bottom surface layer. This result was similar to the previous work by Yanglei su *et al* and C.Bath e*t al* [16, 17]. As can be seen in figure 2 (a), pure PSf membrane showed dense top surface and short finger-like with weak interconnection structure. By adding RHS to the casting solution, it may obviously noticed that the membrane microstructure was changed in term of finger-like, interconnectivity and pore size especially at top and bottom layers.

As reported before small addition of additive into casting solution has generated major changes to the membrane characteristic and performance [4]. From the result, it was noticed that by increasing the RHS content, the finger-like macrovoids at the sublayers or middles layer become longer, while irregular sponge macrovoids become less in sizes at the bottom layer. It was also demonstrated that the sponges structures at bottom layer tends to be a finger- like structure with bigger size. This straight or longer finger-like structure formed at the bottom give better permeation compared to short finger-like structure [7]. In fact, by increasing the RHS content, the pore at the top layer membrane become smaller. The addition of silica as quoted by Jian Huang *et al.* reduced the pore size formation at the top separation layer and this lead to the better rejection mechanism [18].

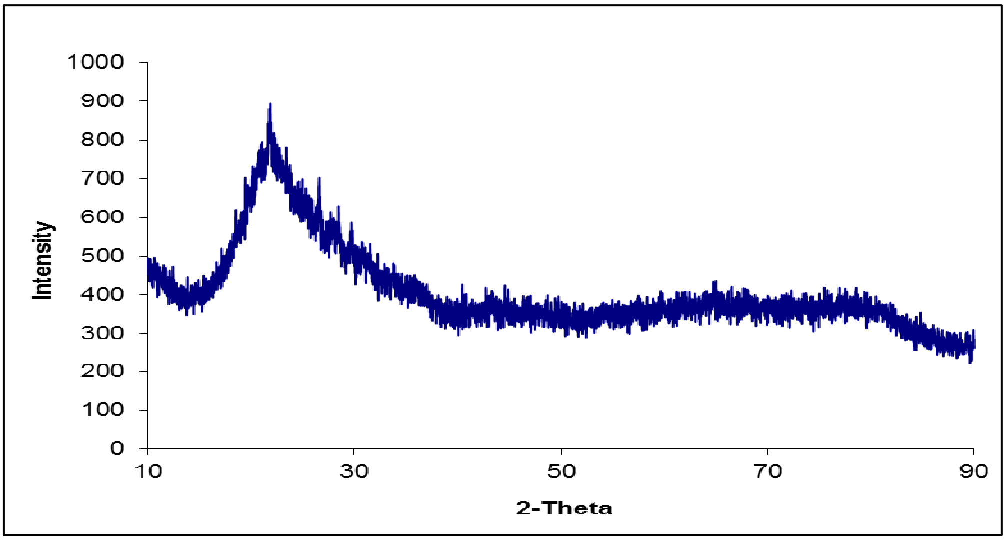
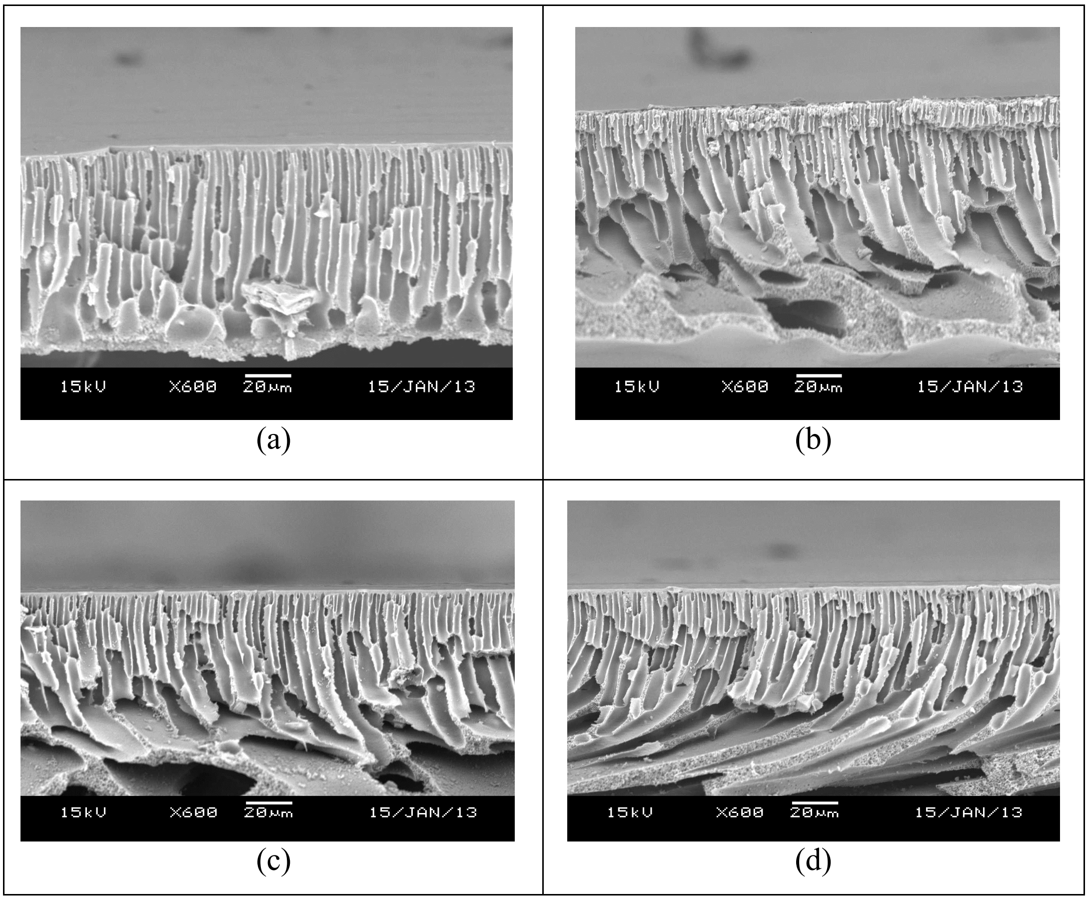
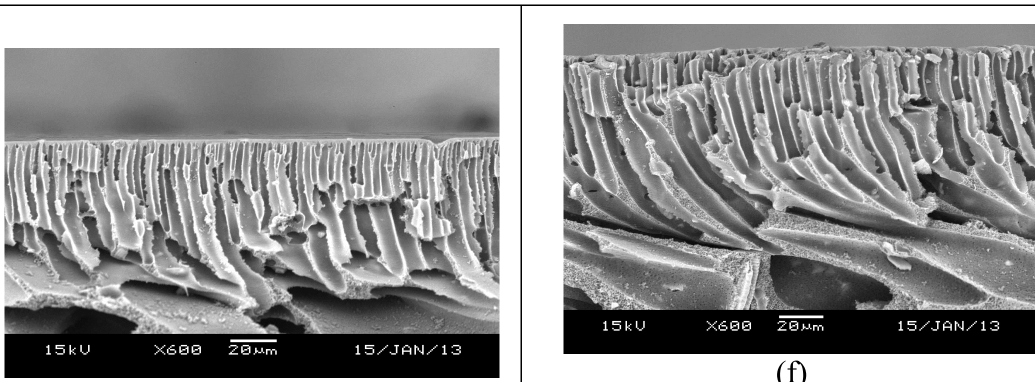


Figure 1



(a) (b) (c) (d)



(e) (f)

Figure 2. SEM cross-section of PSf membrane with different weight percentages of rice husk silica (RHS). a) 0% RHS b) 1% RHS c) 2% RHS d) 3% RHS e) 4% RHS f) 5% RHS.

Conclusion

Fused RHS with PSf/PEG membrane was successful prepared by phase inversion method. In this work, the effect of rice husk silica as an additive on polysulfone membrane structure was investigated. The SEM results indicated that the addition of the RHS has enlarged the pore size in the sub layer creating the finger-like macrovoids which then resulted in longer sub layer. In addition, RHS has improved the interconnectivity of pores between top and bottom layer and reduces the pore sizes formation at the top separation layer [18].

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