Effect of solvent concentration on performance of polysulfone membrane for filtration and separation

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Abstract. This study was conducted to investigate the effect of solvent concentration on the performance of polysulfone membrane via airbrush spray method. The solvent concentration was varied from 73% to 80% in dope solution. The study also investigated airbrush processing parameter such as spray time and distance at different solvent concentration. The prepared membrane was characterized in respect to its morphology and the performance of the membrane were evaluated via gas permeability performance. This study found that the membrane fiber size was reduced as solvent concentration increases. When time increased the diameter of fiber also increased. The distance also affected the fiber size, when the distance increased the diameter of fiber became smaller. 80% of solvent concentration has better filtration and separation ability compared to other solvent due to its porosity and morphology. From the gas permeability cell testing it shows that the permeability is increasing as the solvent concentration decrease.

1. Introduction
Air pollution is one of the major problems facing the world today due to a substantial population growth which generates more waste products through industries, transportation and households[1],[2]. Recently, it is known that biological and chemical contaminants in air or water [3]. The field of membrane separation technology is presently in a state of rapid growth and innovation. Membrane process is a method for separation process that being used widely in air purification and filtration industries [4]. In membrane technology, there are huge benefits such as the continuous separation process, low in energy consumption, small area required and do not involve any chemical for air filtration. There are two generations of membrane process if we see it from an economic point of view. The first generation focused on the water separation process and while for the second generation is more focused on liquid-gas separation process. In this study, gas separation process is a main point for the membrane development. Therefore, some advantages can be achieved from membrane development such as an excellent combination of selectivity and productivity, not requiring a phase change for solute or the carrier solvent and no need for regeneration of solid or liquid Sorbent [5]–[8].

Nowadays, phase inversion is a most popular technique for the membrane preparation [9]. Preparation of membrane is called as asymmetric and symmetric membrane. Another technique for membrane fabrication is coating, stretching, track-etching and template leaching. Chemical structure
has a strong influence on the physical and mechanical properties of thin films [10]–[12]. Varieties of membrane morphologies can be obtained from different fabrication technique [13]–[15]. In this process, the polymer is transformed from liquid or soluble state into a solid state. Phase inversion method was employed to transform a polymer in control of manner from a liquid dispersion to solid film [16].

Electrospinning technique is one of improvement technique is used in membrane fabrication. This technique is used the technology of electrostatic fiber formation which is utilizes electrical forces to produce polymer fibers with diameters ranging from 2 mm to several micrometers using polymer solutions of both natural and synthetic polymers. The process of electrospinning is gained much more attention nowadays with excellent membrane characteristics such as smaller pores and higher surface area. Electrospinning process are successfully applied in various fields, such as nanocatalysis, tissue engineering scaffolds, protective clothing, filtration, biomedical, pharmaceutical, optical electronics, healthcare, biotechnology, defense and security, and environmental engineering [17]. However, applications of electrospinning filters were more expensive than other traditional methods [18]. Pervious researcher by Brooker et al., (2010) discovered that the potential of preparation of membrane using air brush spray. Air brush spray technique is one excellent technique in fiber production for membrane preparation [19].

In this study, membrane preparation via spray method will be used. The influence of solvent concentration in a membrane preparation will be investigated. Different parameter will be considered in membrane fabrication using by air brush techniques. The parameter will be focused on the concentration of solvent, variety of time and spray distance in this technique. Therefore, the effect of solvent concentration on morphology and performance of the membrane will be characterized.

2. Experimental

2.1. Materials and procedure
In this research, different concentration of solvent was used to produce the membrane from 73% to 80%. The materials that had been used such as Polysulfone (PSF) as polymer and N-Methyl-2-pyrrolidone (NMP) as solvent. The membrane is produce using air brush spray technique. By using this technique there are other two parameters that need to be considered. There are times of spray and the distance of spray that will need to be highlighted.

The time and distance of spray need to be considered while making this membrane if the airbrush spray method want to be used. This is because these two parameters will have a big influence to the membrane characterization.

2.2. Preparation of membrane
In this study, we want to investigate the effect of solvent concentration toward the performance of Polysulfone membrane. The parameters of solvent concentration used were 73% to 80%. The membrane was made by using air brush spray technique. By using this technique, the time and distance of spraying need to be considered.

Flat sheet PSF membranes were prepared by airbrush spray method. The polymer needs to be dry in the oven 24 hour at a temperature of 60°C before used. The mechanical stirrer was used to mix the polymer and solvent until they were completely dissolved. After that the dope solutions need to be keeping in room temperature about 24 hours to make sure there are no air bubbles. The dope solution, then are sprayed used an air brush spray technique with different time of sprayed and distance of spray tip to the collector. In this study different solvent concentration, time and distance was studied. Finally, membranes need to dry at room temperature for 1 to 2 days before inspection of membrane characterization and performance of the membrane can be carried out.
2.3. Membrane Evaluation
After the production of membrane, the membrane characterization need to be conducted to check on the morphology and performance of the membrane. Understanding the characteristic of membrane not only will help to select a suitable membrane for specific application, but it also can guide the design of membrane with desired properties. Scanning Electron Microscopic (SEM) was used for investigating the morphology of membrane and for membrane performance test the gas permeation cell was used to check the ability of the membrane to filter the nitrogen gas. In this study, the most importance characterization that needs to analyze is the string diameter of the Nanofiber that has been produced. This string diameter was examined by using a Scanning Electron Microscope (SEM). Table 1 shows the identification of all samples that are produced.

| Parameter          | 73%    | 75%    | 77%    | 80%    |
|--------------------|--------|--------|--------|--------|
| Constant distance  |        |        |        |        |
| 25 cm              | S73M2  | S75M2  | S77M2  | S80M2  |
| 4 min              | S73M4  | S75M4  | S77M4  | S80M4  |
| 6 min              | S73M6  | S75M6  | S77M6  | S80M6  |
| 8 min              | S73M8  | S75M8  | S77M8  | S80M8  |
| 10 min             | S73M10 | S75M10 | S77M10 | S80M10 |
| Constant time      |        |        |        |        |
| 10 min             | S73C15 | S75C15 | S77C15 | S80C15 |
| 15 cm              | S73C20 | S75C20 | S77C20 | S80C20 |
| 20 cm              | S73C30 | S75C30 | S77C35 | S80C30 |
| 30 cm              | S73C35 | S75C35 | S77C35 | S80C35 |
| 35 cm              | S73C35 | S75C35 | S77C35 | S80C35 |

Table 1: Sample identification

3. Result and Discussion

3.1. Scanning Electron Microscope (SEM)
SEM is used to produce the sample images by scanning with a focused electron beam. The sample images are produced by the secondary electrons emitted by the atoms excited by the electron beam [20]. In this study, SEM analysis the data had been calculated to take the average of string diameter. Table 2 shows the different time taken at a constant distance of 25 cm. Table 3 for different distance taken at constant time of 10 minutes.

| Sample | Time (min) | 2   | 4   | 6   | 8   | 10  |
|--------|------------|-----|-----|-----|-----|-----|
| S73    | 0.97       | 1.1 | 1.28| 1.35| 1.48|
| S75    | 1.05       | 1.25| 1.33| 1.48| 1.51|
| S77    | 1.04       | 1.28| 1.39| 1.47| 1.76|
| S80    | 0.99       | 1.31| 1.32| 1.46| 2.00|
Table 3: Average diameter of fiber at different distance

| Sample | Distance (cm) | 15  | 20  | 25  | 30  | 35  |
|--------|---------------|-----|-----|-----|-----|-----|
| S73    | 2.86          | 2.67| 2.11| 1.71| 1.58|     |
| S75    | 2.42          | 1.57| 1.51| 1.69| 1.40|     |
| S77    | 2.35          | 1.99| 1.76| 1.67| 1.22|     |
| S80    | 2.29          | 2.22| 2.00| 1.66| 1.44|     |

Fig. 1 shows that the diameter of the string is increased as spray time increases. The air brush spray was continued and the increment of string size might be due to the tendency of string to merge with each other. The observation of solvent effect to diameter air brush spray shows that as the concentration of solvent was increased, the diameter of fiber was decreased. This behaviour might be due to the polymer solution tending to aggregate as solution concentration increases. According to Huan et al., (2015) reported that the high solvent concentration leads to the low viscosity of the solution. Hence, as viscosity was reduced, the solvent molecule tends to make entangled molecular chain which resulted reduction of aggregation [21],[22]. This behaviour lead to the formation of the smaller size of the fiber as solvent concentration increases [23].
Fig. 2 shows the average diameter of nano-spider web at different collector distance at constant time (10 minutes). This result show that as collector distance was increased, the diameter of fiber was decreased. This result is expected due to short collector distance was not provided sufficient time for the polymer to solidify. Refers to Chowdhury et al., (2012) revealed that the longer collector distance allowed more time for polymer to fully stretch and solidify which result low diameter of fiber [24].

The similar trend was observed for effect of solvent when the collector distance was varied. Table 4.5 shows that as the solvent concentration was increased with collector distance, the fiber diameter was decreased as well. This result was proved that the different parameters was influenced the membrane morphology [25], [26].
Fig. 2: Different solvent concentration at various distances

Fig. 3 shows the morphology of fiber filter at different parameters with respect to the solvent effect on fiber formation. Fig.3 (1st column) shows the effect of solvent concentration. The result shows that as the concentration of the solvent was increased, the amount of fiber was decreased. The amount of fiber was inversely proportional to the solvent concentration. The higher the solvent concentration was the less the amount of fiber. This is due to the viscosity of the solution [27]–[29]. According to Huan et al. (2015), it is confirmed that the viscosity is the factor that leads to the fiber production [23]. As the viscosity was increased, the amount of fiber was increased significantly.

Fig. 3 (2nd column) shows the effect of time on the amount of fiber produced. Time duration was started from 2 minutes to 10 minutes. Figure 4.3 shows that the amount of fiber was decreased as the time was increased. The amount of fiber might lose as the time increased [30]–[33]. From this result, the spraying process was taken much more time to solidified and the fibers was merged with each other. Hussin et al., (2014) found that the solution needs more time to solidify and it can be fastened...
by exposing to the heat [14]. This will lead to the losses of the amount of fiber as the time increase because it needs to solidify its self in room temperature to maintain its properties.

Fig. 3 (3rd column) shows the amount of fiber in distance effect. The distance was varied from 15 cm, 20 cm, 25 cm, 30 cm and 35 cm. Figure 4.3 shows that the amount of fiber was increased as the distance was increased. This situation was happened due to the short deposition time that will makes the fiber tends to emerge with each other. Subbiah et al. (2005), proved that the collection distance was affected the deposition time simultaneously and this resulted on the evaporation rate of the solvent and instability interval [34]. From that, the amount of fiber was disappeared with the longer time duration to solidify.
Effect of solvent concentration

80% solvent concentration

77% solvent concentration

75% solvent concentration

73% solvent concentration

Effect of time

10 minutes

8 minutes

6 minutes

4 minutes

Effect of distance

15 cm

20 cm

25 cm

30 cm

Figure 3: SEM image on amount of fiber of several chosen sample

3.2. Permeability

The nitrogen gas was used in this process as the main element for gas filtration. The total of nitrogen gas was measured and investigated to pass through the membrane by some duration of time. There are only four samples were prepared and suitable for the gas permeability test. Table 4 shows the gas permeation at constant pressure for 1 bar.
| Sample  | Gas permeation (GPU) |
|---------|----------------------|
| S80M10  | 52.8                 |
| S77M10  | 63.5                 |
| S75M10  | 88.3                 |
| S73M10  | 146.5                |

Fig. 4 shows the gas permeation cell results on a different sample parameter. Fig. 4 was demonstrated that the permeability was increased as the solvent concentration was decreased. This might be due to the porosity of the membrane. From previous test, the pore size was affected by the solvent concentration of the membrane. Thus, smaller pore size will make membrane filtration performance became better [35]. However, the higher solvent concentration can cause the pores size reduction.

4. Conclusion
The flat sheet Polysulfone (PSf) membrane was successfully prepared by using air brush spraying technique. The concentration of solvent was used from 73 % to 80 % for membrane preparation. Three parameters were investigated which as solvent concentration, time duration of spray toward the membrane and the distance of spraying. In this study, these parameters were influenced the membrane morphology. Based on the testing was successfully conducted, the different type of the membrane morphology was formed. The different concentration of solvent was produced the different membrane viscosity. The membrane viscosity was affected the membrane morphology. 80 % of solvent concentration was affected the reduction of membrane viscosity. The performance of membrane became higher by filtration or separation. The gas permeation cell result shows 80 % of the concentration solvent was recorded at 52.8 (GPU). The sample of 80 % concentration solvent shows the less membrane viscosity. From that, this sample was produced the less porosity of membrane and the membrane performance by filtration or separation become better. The other two parameter of time duration and distance were affected the membrane morphology in diameter of the string. When the time duration was increased, the diameter of the string also increased while the distance was increased but the diameter of the string became smaller. From that, the effect of time shows that as the time was increased the diameter of fiber also increased. For the 10 minutes shows the highest amount of fiber that lead to produce a good membrane structure for membrane performance.
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