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1 Transport Properties, Mechanical Behavior, Thermal and Chemical Resistance of Asymmetric Flat Sheet Membrane Prepared from PSf/PVDF Blended Membrane on Gauze Supporting Layer

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1 ABSTRACT

Asymmetric polysulfone (PSf) membrane is prepared using phase inversion method and blending with polyvinylidene fluoride (PVDF) on the gauze solid support. Casting solution composition optimization has been done to get PSf/PVDF membrane with best characteristics and permeability. The result shows that blending on PSf with PVDF polymer using phase inversion method has been very helpful in creating an asymmetric porous membrane. Increased level of PVDF in casting solution has increased the formation of asymmetry structure and corresponding flux membrane. The result from a thermal test using Differential Scanning Calorimetry (DSC)-Thermal Gravimetric Analysis (TGA) shows the resistance of the membrane to temperature 460 °C. Membrane resistance against acid looks from undetectable changes in infrared spectra after immersion process in H₂SO₄ 6–98 v/v%. While membrane color changes from white to brownish and black is detected after the immersion process in sodium hydroxide (NaOH) 0.15–80 w/v%.

Keywords: membranes; polysulfone; polyvinylidene fluoride; blending; synthesis processing

ABSTRAK

Membran asimetri polysulfone dipreparasi dengan metode inversi fasa dan blending menggunakan poliviniliden florida pada padatan pendukung kasa. Optimasi komposisi larutan cetak telah dilakukan untuk mendapatkan membran PSf/PVDF dengan karakteristik dan permeabilitas terbaik. Hasil menunjukkan bahwa blending PSf dengan polimer PVDF menggunakan metode inversi fasa telah sangat membantu menciptakan pori asimetri pada membran. Peningkatan kadar PVDF dalam larutan cetak telah meningkatkan formasi struktur asimetri dan fluks membran yang bersangkutan. Hasil uji termal menggunakan analisis Differential Scanning Calorimetry (DSC)-Thermal Gravimetric Analysis (TGA) menunjukkan ketahanan membran hingga temperatur 460 °C. Ketahanan membran terhadap asam tampak dari tidak terdeteksinya perubahan pada spektra infra merah setelah proses perendaman dalam H₂SO₄ 6–98 v/v%. Sementara itu, pe1bahan warna membran dari putih menjadi coklat dan hitam terdeteksi setelah proses perendaman dalam NaOH 0,15–80 %w/v.

Kata Kunci: membrane; polysulfone; poliviniliden florida; blending, and proses sintesis

6 INTRODUCTION

Membrane technology has many advantages including low energy consumption and high separation efficiency [1]. However, the fouling membrane has been shortening membrane lifetime and causing increased costs, therefore it has been blocking its application on a 6rge scale industry [2]. Research and development of new membrane materials is an effective way to suppress the fouling membrane. Membrane materials should be commercially available, have a thermal stability and good chemical resistance against acids and bases as

well as good mechanical strength and characteristics [3-6].

2 Microporous polymer membrane has been used extensively in many separation processes. For such purposes, 12e structure is an important factor considered in membrane manufacturing process [7]. The good porous membrane should have a high permeability and chemical resistance to the flow of feed solution. Asymmetric membrane is one of the ideal selec2n parameters to get high permeability [8].

Phase inversion process induced by immersion precipitation is used to manufacture asymmetric membranes [9-10]. When a thin layer of casting

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solution immersed in coagulation medium, the solvent in the casting solution was replaced by nonsolvent. This process has resulted in asymmetry membrane with dense top layer and porous sublayer which is composed of macrovoids, pore, and micropore [11]. Pore size and porosity of the membrane is influenced by the relative diffusion rate of solvent and nonsolvent. The formation of sublayer controlled by changing on casting solution and nonsolvent compositions as well as operational conditions such as stirring time and casting thickness [12].

In membrane manufacture, PSf is known for its characteristics include a good solubility in a number of aprotic polar solvent (dimethylformamide (DMF), dimethylacetamide (DMAc), dimethyl sulfoxide (DMSO), halogen derivatives, nitrobenzene, high thermal resistance (150–170 °C), high chemical resistance almost on the entire range of pH and oxidative medium (hypochloride 5–7%, hydrogen peroxide 3–5%) as good as mechanical strength [13]. However, the use of PSf is limited because of the symmetry structure as well as a limited range of pore size and porosity. The symmetry membrane has higher mass transfer resistance than the asymmetry membrane. These conditions magnify fouling potential on the membrane surface and internal pore. The membrane lifetimes decrease will go hand in line with the increasing of fouling membrane. Some of the methods that can be done to eliminate the weakness of PSf is blending PSf polymer with another polymer material that capable of forming a dense membrane with asymmetry structure, such as PVDF [14].

Compared with many other organic polymer materials, PVDF is a material that is widely used in the manufacturing process of the microfiltration (MF), ultrafiltration (UF), and pervaporation (PV) membrane because of its excellent thermal stability and chemical resistance against acids, bases, and organic solvents [15]. In addition, PVDF membrane obtained from phase inversion process has a good mechanical strength and asymmetry structure that required for separation. Therefore, PSf membrane preparation with blending method using PVDF material will generate membrane which not only has a good thermal and chemical resistance, mechanical strength but also has higher fouling resistance.

EXPERIMENTAL SECTION

Materials

Polysulfone (PSf; $d = 1.24 \text{ g L}^{-1}$ at 25 °C; Mw 35,000; Sigma Aldrich) and polyvinylidene fluoride (PVDF; Mw 354,000; Aldrich) used as membrane material. 1-methyl-2-pyrrolidone (NMP; biotech grade; $\geq 99.7\%$; Sigma Aldrich) used as polymer solvent.

Ammonium chloride (NH_4Cl ; $\geq 99.5\%$; Riedel de Haen) used as membrane pore-forming additive, while gauze fabric (Kasa Husada Indon, 254 mesh) used as membrane supporting layer. Distilled water is used as a nonsolvent for the polymer precipitation. Meanwhile, sulfuric acid (H_2SO_4 ; $> 98\%$; Ajax Finichem Pty Ltd.) and sodium hydroxide (NaOH ; pellet; $\geq 98\%$; Sigma Aldrich) are used in order to test the membrane chemical resistance.

Instrumentation

Surface and cross-section morphology of the PSf/PVDF membranes were observed using ZEISS EVO MA 10 Scanning Electron Microscope (SEM). The mechanical strength of the membrane was analyzed using RCT-10KN-AF Toyo Seiki Stograph. The permeation performance of the PSf/PVDF membrane was carried out by dead-end membrane reactor (self-made) with distillate water as feed solution. The thermal decomposition of the membrane was analyzed by Perkin Elmer STA-6000 Differential Scanning Calorimetry (DSC)-Thermal Gravimetric Analysis (TGA). Thermogravimetric analysis under an N_2 atmosphere from 0 to 890 °C with a heating rate of $10 \text{ }^\circ\text{C min}^{-1}$. The chemical decomposition of the membrane before and after chemical resistance test was analyzed by Nicolet IS10 Thermo Scientific Fourier Transform Infra-Red (FTIR).

Procedure

Casting solution preparation

For the preparation of the membrane, as much as 2 g of PSf and 2 g of NH_4Cl are dissolved in 84 g solvent NMP. Furthermore, to ensure the formation of homogeneous casting solution done stirring using NESCO LAB MS-H280-Pro magnetic stirrer for 105 min at a temperature of 60 °C. After that, while continuing to stir using stirrer, into membrane casting solution added 12 g of PVDF. The stirring process of casting solution continued for 30 min. To get optimum casting solution composition the preparation of casting solution done by following casting solution composition that looks at Table 1.

Table 1. Composition of PSf/PVDF solutions

Membrane	PSf	PVDF	NMP	NH_4Cl
M1	14 wt%	0 wt%	84 wt%	2 wt%
M2	0 wt%	14 wt%	84 wt%	2 wt%
M3	8 wt%	6 wt%	84 wt%	2 wt%
M4	7 wt%	7 wt%	84 wt%	2 wt%
M5	6 wt%	8 wt%	84 wt%	2 wt%
M6	3 wt%	11 wt%	84 wt%	2 wt%

Membrane preparation

The PSf/PVDF membrane which is prepared using phase inversion method are generally used for the preparation of various types of asymmetry membrane. The solution is cast on a glass sheet which coated with gauze fabric supporting layer using casting knife method at temperature 80 °C and thickness 800 µm. Subsequently, membrane transferred in the coagulation medium for immersion precipitation process on temperature 60 °C. The nonsolvent used is distilled water. Throughout the rest of the solvent and pore forming agent is removed by immersing in distilled water for 24 h. After casting, the membrane is removed from the glass sheet.

Membrane characterization

The membrane cross section prepared by breaking at the temperature of liquid nitrogen. The entire membrane is coated with a thin layer of gold before it is tested using Zeiss EVO MA10 Scanning electron microscope (SEM). The result from SEM shows surface, cross section, and pore sizes of PSf/PVDF membrane.

In order to know the permeation performance of PSf/PVDF membrane, the permeability is tested using dead-end filtration system with pure water as feed solution. The PSf/PVDF membrane permeability is calculated using Eq. 1:

$$J = \frac{V}{AT} \quad (1)$$

where V is the volume of permeate in a liter, A is the

membrane surface area (m²), and T is the permeation time (h). Minimum three membrane samples were tested and the permeability tabulation result showed the average value.

Meanwhile, the PSf/PVDF membrane mechanical characterization is conducted by measuring the mechanical strength of membrane using RCT-10KN-AF Toyo Seiki Strogaph. The mechanical strength of PSf/PVDF membrane evaluated using Young's modulus that is calculated based on the ratio between load and stroke as seems on Eq. 2:

$$E = \frac{\text{stress}}{\text{strain}} \quad (2)$$

where stress is the magnitude of forces that acting in the unit area of the material in (Nm⁻²), and strain is the ratio of value added material to the initial length. Minimum three membrane samples were tested and the mechanical strength tabulation result showed the average value.

Membrane chemical resistance test

The chemical resistance test of the PSf/PVDF membrane is carried out by immersing the membrane cutting in sulfuric acid (H₂SO₄) solution 98% (v/v) and sodium hydroxide (NaOH) 80% (w/v) for 24 h. Further, to know the changes that occur after immersing treatment of membrane in acid and base solution has been carried out an analysis of the PSf/PVDF membrane functional groups using Nicolet IS10 Thermo Scientific Fourier Transform Infra-Red (FTIR).

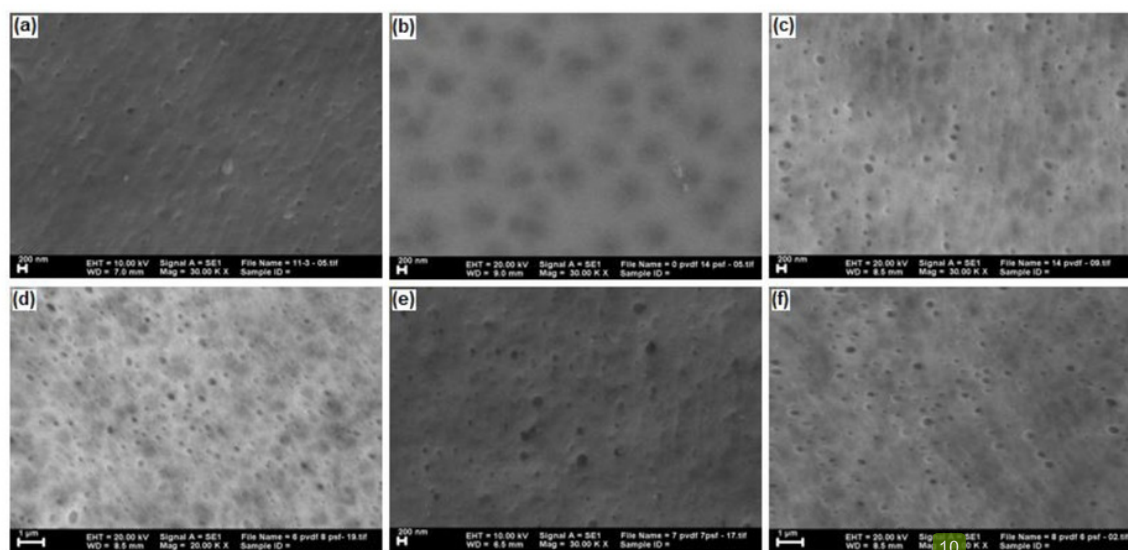


Fig 1. SEM images of PSf/PVDF membranes surface with four casting solution compositions. (a) M3; (b) M4; (c) M5; (d) M6; (e) M1; and (f) M2

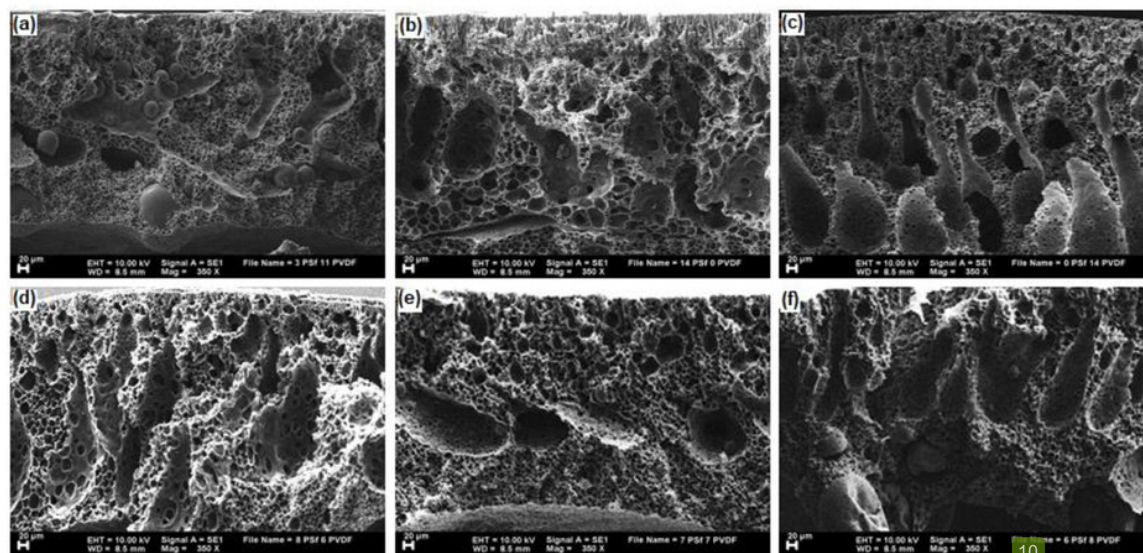


Fig 2. SEM images of PSf/PVDF membranes cross section with four casting solution compositions. (a) M3; (b) M4; (c) M5; (d) M6; (e) M1; and (f) M2

Membrane thermal stability test

The thermal stability test of the PSf/PVDF membrane is carried out through evaluation of mass changes using Perkin Elmer STA-6000 Differential Scanning Calorimetry (DSC)-Thermal Gravimetric Analysis (TGA).

RESULT AND DISCUSSION

Membrane Characterization

Membrane cross-section analysis

The result of PSf/PVDF membrane surface and cross-section morphology analysis with various casting solution compositions seems at Fig. 1–2. Fig. 1 shows SEM micrographs of PSf/PVDF, PSf, and PVDF membrane surface morphology and Fig. 2 shows SEM micrographs of PSf/PVDF, PSf, and PVDF membrane cross-section morphology. From Fig. 2 seems obvious asymmetry structure in membrane M3, M4, M5, and M6. The cross-section morphology data in Fig. 2 shows that compared with membrane M4, M5, and M6, membrane M3 has highest porous layer thickness with biggest pore size. This shows that the addition of an increased level of PVDF on PSf polymer solution has led to decrease the thickness and the pore size of the porous layer on the membrane. This is because the PVDF polymer tends to fill the membrane framework which is dominated by the PSf.

Fig. 2 shows that the PSf/PVDF membrane is manufactured from phase inversion process forming the characteristic structure of macrovoid. The solvent-non solvent system produced by a combination of NMP-water has a high mutual affinity which means that fast liquid-liquid demixing process will occur during the phase transition. According to SEM image of PSf and PVDF membranes in Fig. 2e and 2f, it is obviously seems that the addition of PVDF polymer in PSf polymer solution has formed membrane with lower thickness and pore size of porous layer, and instead, the addition of PSf polymer in PVDF polymer solution has led to increasing the thickness and pore size of porous layer on PSf/PVDF membrane.

The molecular structure of the more long chain on PSf compared with PVDF has caused the forming of more porous surface and internal pore sizes with a higher thickness of the porous layer on PSf membrane. In line with this, the much higher molecular weight of PVDF used has created smaller pore sizes with a lower thickness of the porous layer on PSf/PVDF membrane with higher PVDF content.

Membrane mechanical strength analysis

The lower thickness of a porous layer on cross-section structure of PSf/PVDF membrane produced from casting solution with higher PVDF content has impacted on the lower mechanical strength of the concerned membrane. In Table 2 looks mechanical

Table 2. Mechanical strength of PSf/PVDF membrane

Membrane	PSf	PVDF	NMP	NH ₄ Cl	Young's Modulus (Nm ⁻²)
M1	14 wt%	0 wt%	84 wt%	2 wt%	1523.8
M2	0 wt%	14 wt%	84 wt%	2 wt%	647.2
M3	8 wt%	6 wt%	84 wt%	2 wt%	991.4
M4	7 wt%	7 wt%	84 wt%	2 wt%	962.1
M5	6 wt%	8 wt%	84 wt%	2 wt%	938.3
M6	3 wt%	11 wt%	84 wt%	2 wt%	662.3

strength data of PSf/PVDF, PSf, and also PVDF membrane used as a control. The lower thickness of porous layer in PSf/PVDF membrane as a result of higher PVDF polymer addition has decreased the ability of membrane to maintain its pore sizes when applied high operating pressure (for example, from 991.4 Nm⁻² for membrane M3 to 962.1 Nm⁻² for membrane M4; 938.3 Nm⁻² for membrane M5 to 662.3 Nm⁻² for membrane M6). The use of gauze as a solid supporting layer of the PSf/PVDF membrane has created membrane layer with higher mechanical strength than PSf/PVDF membrane that prepared without supporting layer [16].

Membrane Performance

Filtration of pure water

PSf/PVDF membrane permeability test carried out by pure water dead-end filtration. The permeability test resulted seems in Fig. 3.

Fig. 3 shows pure water permeability of PSf, PSf/PVDF, and PVDF membrane which precipitated using water nonsolvent. The added of PVDF to PSf polymer solution has increased the pure water permeability of PSf membrane. It is certainly not in line with the prediction that permeability will increase as a result of the increased in membrane pore size. In this state, the hydrophilic properties which increase with increasing of polar atomic -CH and -CF existence that have filled the membrane frame dominated by PSf has increased the pure water permeability of the membrane. Further, the domination of PVDF polymer in the casting solution has significantly increased the hydrophilic properties and prevent the occurrence of an increased porous layer in the membrane. This condition has resulted in an increase in the permeability of the membrane to pure water. Thus it can be said that the pure water flux of the PSf/PVDF membrane is determined by the thickness of the porous layer and hydrophilic properties of the membrane.

It is in line with the judging result from the Fig. 2a and 2b in which the thickness of the porous layer on the membrane M3 and M4 look similar with more porous surface morphology than membrane M5 and M6. From Fig. 2c and 2d, it can be seen that membrane M5 and M6 have a denser structure as a result of the increasing quantity of PVDF particles which decomposes within the

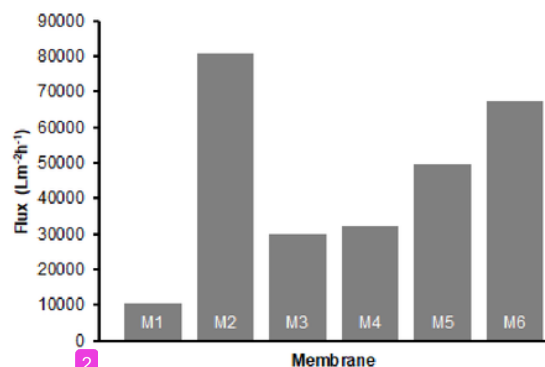


Fig 3. Pure water flux of PSf/PVDF blend membranes with different casting solution compositions

PSf polymer framework. This phenomenon can be seen explicitly through comparison with Fig. 2e and 2f which shows the cross-section morphology of the PSf and PVDF membranes. Compared with the Fig. 2a, 2b, 2c, and 2d, Fig. 2e shows that membrane M1 has a porous layer with a higher thickness and lower dense pore as well as limited porosity compared to membrane M2 that seems in the Fig. 2f. The combination of higher hydrophilic properties and lower thickness of membrane porous layer then resulted in a difference in the pure water flux of the PSf/PVDF membrane.

Thermal Stability and Chemical Resistance of the Membrane

Membrane thermal stability

Thermogravimetric analysis results show that PSf and PVDF were decomposed in some steps as shown in Fig. 4. The decomposition is indicated by the weight lost during the temperature increases. The first and second mass reduction which are detected at around 100–200 °C corresponds to the releasing of water as vapor [17] and 20–260 °C for the releasing of NMP that adsorbed on the surface of PSf/PVDF membrane. The third mass reduction occurs at about 340–360 °C. It is predicted that the third mass reduction is caused by the decomposition of PVDF. The result is consistent with those reported in [18] that PVDF was decomposed

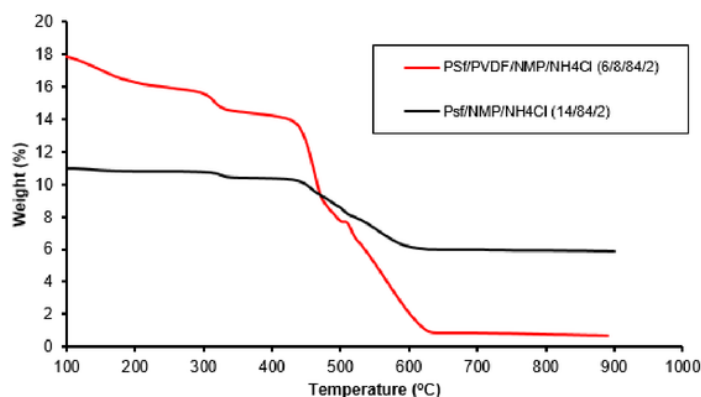
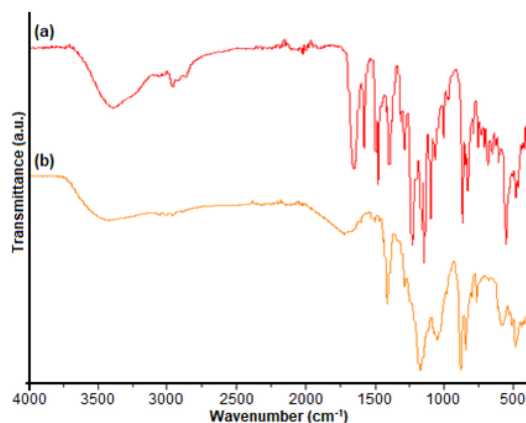


Fig 4. Thermogram TGA of M5 and M1

Fig 5. Infrared spectra of M5. (a) before; and (b) after immersion in H₂SO₄

at about 375 °C. While the fourth mass reduction at about 460 °C that was caused by the decomposition of PSf. These data indicate that its lower thermal stability of PSf/PVDF than PSf membrane.

Membrane chemical resistance

The functional group test result using FTIR (see Fig. 5) has shown the better resistance of PVDF in sulfuric acid compared with PSf. This seen from the disappearance of the characteristic peak on the infrared spectra of PSf/PVDF membrane that has been immersed in sulfuric acid 98% (v/v) for 24 h. The characteristic peak intended are in the wavenumber of 1643 cm⁻¹ (C=C alkene); 1643; 1584; 1504; and 1487 cm⁻¹ (C=C aromatic); also 1149 cm⁻¹ (C-O ether). This is evident from the infrared spectra of PSf/PVDF before and after immersion process in sulfuric acid solution in Fig. 5.

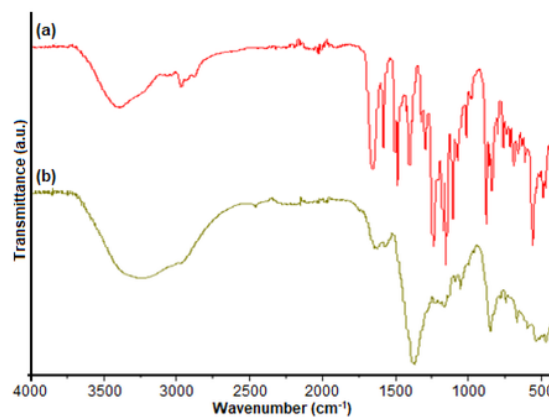


Fig 6. Infrared spectra of M5. (a) before; and (b) after immersion in NaOH

The chemical resistance result of PSf/PVDF membrane against alkaline environment shows that PSf/PVDF membrane has not resistance to alkaline environment despite very dilute concentration. This looks from the detection of physical changes on the PSf/PVDF membrane which gradually become black after immersion process in sodium hydroxide. The black color formed more concentrated with the increasing concentration of sodium hydroxide are used.

Fig. 6 shows the occurrence of significant changes in the peak intensity of the functional group -HF at the wavenumber 1105 cm⁻¹ and the disappearance of the peak at the wavenumber 1014 and 1080 cm⁻¹. This condition indicates that the treatment of the alkaline environment has made dehydrofluorination on the membrane, i.e. losing functional group -HF. Thus it can be said that the color changes of PSf/PVDF membrane gradually from white

to black after alkaline treatment is because of the dehydrofluorination of PVDF compound in the composite structure. These conditions indicate that although it is known to have good chemical resistance, the results of this study show that PSf/PVDF membrane has good thermal resistance in the base environment (NaOH) to concentrations below 80%(w/v). This fact corresponds to that reported by a number of researchers assert that there has been a color change from white to brown and finally black after immersing process in an alkaline solution for a few hours [18-21]. The formation of PVDF film colors become darker after treatment in aqueous hot alkaline shows have been onset the formation of C=C bonds caused by dehydrofluorination on the PVDF polymer chain.

Infrared spectra data in Fig. 6 shows that the lower chemical resistance of PSf/PVDF on the alkaline environment is more affected by the weakness of PVDF in alkaline solution. This seems from the PSf characteristic peak that more detected compared with PVDF in the infrared spectra of PSf/PVDF membrane. The dominance of the PVDF influence in determining the chemical resistance of PSf/PVDF membrane influenced directly by the domination of the PVDF polymer in the casting solution, i.e. 6:8 wt%.

CONCLUSION

The effect of casting solution composition against membrane characteristics: the PVDF polymer addition has resulted in an increase in the permeability as a result of hydrophilic nature increases in line with the decrease in the thickness of the porous layer. This PSf/PVDF membrane has smaller pore size with lower mechanical strength compared to PSf membrane which prepared with the same conditions. The TGA test analysis results show the lower thermal stability of the PSf/PVDF membrane than PSf membrane. The FTIR test analysis results show the higher and lower chemical resistance of PSf/PVDF membrane than PSf membrane, each in the acid and alkaline environment.

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