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Manufacture of PVDF-Kitosan Composite Membrane and its Utilization in Batik Industrial Wastewater Treatment.

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ABSTRACT

Generally, the purpose of this research is to improve the hydrophilic properties of polyvinylidene fluoride membranes (PVDF) by blending technology using a hydrophilic polymer, chitosan. More specifically, this research aimed to determine the physical characteristics (morphology and size of porous) of PVDF membrane blended chitosan and the influence of chitosan concentration and stirring temperature of a membrane casting solution against mechanical strength and the performance of the membrane in treatment process of batik indsutrial wastewater treatment. In this research, the preparation of PVDF-chitosan composite membrane is carried out using the phase inversion method with immersion -precipitation techniques. Furthermore, to know the surface and cross-section morphology, and also pore size of PVDF-chitosan composite membrane, conducted the analysis using scanning electron microscopy (SEM) and surface area analyzer (SAA) instruments. Results of an analysis showed that the PVDF-chitosan composite membrane surface derived from a casting solution with low levels PVDF and higher stirring temperatures, will have pore with broad area, pore volume and pore size are more tenuous compared with PVDF-chitosan composite membrane surface resulting from the casting solution with high levels of PVDF and lower stirring temperature. In the meantime, to find out the mechanical strength of the PVDF-Chitosan composite membrane, was using autograf instruments. Mechanical strength measuring results, indicating that the mechanical strength of PVDF-Chitosan composite membrane are in the range of 476,02 N/m^2 – 1,963.35 N/m^2. The mechanical strength of the PVDF-Chitosan composite membrane will increase, with increasing levels of PVDF in casting solution and decreasing levels of temperature stirring was applied. As for knowing the permeability of PVDF-Chitosan composite membrane in the treatment of batik industrial wastewater, has conducted the analysis using "Dead end" membrane reactor. The permeability measurement results, show that the PVDF-Chitosan composite membrane have flux on the range 1,244.66 L/m^2.hour-2,990.45 L/m^2.hour. The permeability of the PVDF-Chitosan composite membrane will increase, with increasing levels of PVDF in casting solution and temperature stirring was applied.

Keywords: Membrane, PVDF, Chitosan, Phase inversion, Imersion-precipitation

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INTRODUCTION

PVDF is a polymer material that is sufficiently important in the preparation of the membrane due to its chemical resistance. PVDF is resistant to almost all organic and inorganic acids and can be used in a broad pH range. PVDF is also stable in an aromatic hydrocarbons, alcohols and solvents, tetrahydrofuran and halogenated solvent. In addition, PVDF are also resistant to oxidation environment including ozone, commonly used in sterilization of water. PVDF is a semi-crystalline polymer with a very low glass temperature (-40 °C), which makes it quite versatile and suitable for membrane applications with operating temperatures in the range of -50 and 140 °C, which is only a few degrees below the melting temperature [1]. However, because the PVDF membrane is hydrophobic, it is to be utilized in the scale of wide application, especially for liquid waste processing needs, hydrophilic PVDF membrane properties should be improved, one of them with the coating process using a hydrophilic polymer, such as Chitosan [2].

Chitosan is a hydrophilic polymer that is highly frowned upon to hydrophobic membrane surface modification. Chitosan has been identified as hydrophilic material, non-toxic, biodegradable, antibacterial, and biocompatible bonding agent. Chitosan is widely used as a modifier of the hydrophobic membrane to enhance the hydrophilic properties of concerned membrane [3]. Only a few studies that report the modification process of PVDF membrane [4]. This is likely due to the highly hydrophobic nature of PVDF membrane, which makes it difficult to be modified with hydrophilic polymers.

Several techniques have been examined for modification of polymer membrane, which is through the method of coating (coating), mixing (blending) and grafting of chemical species at the surface membrane (surface grafting). Of these three methods, blending method is a method which is being continuously developed through this time because this method a new polymer material can be generated with the desired properties permanently when compared with using the method of coating and grafting, because based on the research of Yan et al. note that modifications made with polymer coating and method of grafting on the surface is only temporary and the layers apart bit by bit from the repeated use of the membrane. It is certainly very harmful because it causes the membrane to be very limited time of use [5].

The process of modification by means of blending chitosan on PVDF membrane casting solution, will be able to solve two problems at once, where by doing that process, the structure of chitosan being more robust and resistant to high operational pressures and because of chitosan is hydrophilic material, then blending with this material will improve the hydrophilic properties of membrane through the formation of a composite material with PVDF which mechanically stronger and is hydrophobic. Improved hydrophilic properties of PVDF membrane is indispensable, given the application on this research is for wastewater treatment from batik industry. Meanwhile, the ability of PVDF membrane blended chitosan on batik industrial wastewater treatment is affected by the polyelectricity properties of chitosan.

EXPERIMENTAL

Material

Materials research include: PVDF (MW 534 000, Sigma-Aldrich), NMP (anhydrous 99.5%, Sigma-Aldrich), NH_4Cl (≥ 99.5%, Sigma-Aldrich), chitosan (low molecular weight, Aldrich), acetic acid (≥ 99.7%, Sigma-Aldrich), deionized water, ethanol (C_2H_5OH) (≥ 99.9%, Sigma-Aldrich). In the meantime, to conduct performance tests (flux and rejeksi) PVDF-Chitosan composite membrane, used batik industrial wastewater obtained from batik SMEC Sidoarjo, East Java, Indonesia.

Instrument

Research equipment includes: a watch glass, petri dish (d = 9 cm), spatulas, beaker glass, measuring cups, glass plate and magnetic stirrer. Becide that, in this experiment also used Fourier Transform Infra Red (FTIR), autograph, "Dead end" membrane reactor, and spectrophotometer (Shimadzu UV-2401-PC) instrument.

Preparation of PVDF-Chitosan Composite Membrane

PVDF dissolved in NMP at a temperature of 40 ºC. Casting solution composition was varied in the range (PVDF/NMP/NH$_4$Cl) (% w/w) 12/84/2-16/84/0. NH$_4$Cl is used as a pore-forming additive (porogen). Furthermore, 2 %(w/w) chitosan solution 2% (w/v) in acetic acid is added to the PVDF membrane casting solution and then stirred using magnetic stirrer for 15 minutes. Polymer solution that formed subsequently casted on a petri dish and then soaked in a coagulation bath that contains non-solvent (distilled water) at a temperature of 40 ºC to regardless of casting.

The solid membrane that resulting, further removed from the coagulation bath and rinsed with running water to remove residual solvent and NH$_4$Cl from the membrane. The membrane is then dried and stored in glass sheets given load to prevent membrane shrinkage. The same procedure also applies to the preparation of PVDF-Chitosan composite membrane with the Chitosan solution addition: 1.5; 1; and 0.5% (w/w).

Depreciation of PVDF-Chitosan Composite Membrane

To determine the level of membrane shrinkage during the phase inversion process, membrane diameter measurements were taken before and after the phase inversion process. Membrane measurements performed on 4 (four) sides, includes: (a) the vertical, (b) the horizontal and (c) two sides of the diagonal.

Characterization of PVDF-Chitosan Composite Membrane

To determine the physical characteristics (surface morphology and pore size) and mechanical strength of the membrane (resistance to stress and strain) also permeability, conducted an analysis of the PVDF-chitosan composite membrane, with the following information: (a) Imaging surface morphology and cross section of the membrane by scanning electron microscopy (SEM), (b) Measurement of membrane pore size with a Surface Area Analyzer (SAA), (c) Measurement of membrane mechanical strength with autograph; and (d) measurement of membrane permeability (flux) with a "dead-end" membrane reactor.

Permeability of PVDF-Chitosan Composite Membrane

The procedure is follows. At the bottom of the "Dead-end" reactor membrane, ie at the laying of the membrane, placed filter paper that was cut in the size of the membrane (± 6 cm). Membrane then placed on the top of filter paper. After that, the "dead-end" membrane reactor strung back and 100 mL aquadestilata applied thereto as feed solution. At the same time, pressure of 1 atm of compressor flowed into the "Dead-end" membrane reactor. Obtained permeate is collected and recorded the time it takes until all the feed solution filtered out. Furthermore, based on the data of feed solution volume, membrane area and the time it takes until all the feed solution filtered out, the flux was calculated.

The measurement of PVDF-Chitosan composite membrane permeability on batik industrial wastewater treatment done with the same procedure with membrane permeability test above, except that the original feed solution aquadestilata replaced by the wastewater.

RESULT AND DISCUSSION

Preparation of PVDF-Chitosan Composite Membrane

In this study, a PVDF-chitosan composite membrane prepared by phase inversion method using immersion-precipitation technique. The making process of the PVDF-chitosan composite membrane begins with making a casting solution of PVDF blended chitosan, which is mixture of PVDF material and chitosan solution in the NMP solvent. In the preparation of PVDF blended chitosan membrane, PVDF dissolving in NMP, which is one of the best solvents of PVDF. Solubility of PVDF in the NMP will largely determine the characteristics and performance of the resulting membrane. Through the selection of the appropriate solvent, high mobility of polymer rings can be maintained, since the mobility is influenced directly by the interaction between polymer with solvent and polymer with polymers. To increase the PVDF blended chitosan membrane porosity that produced, on the casting solution added porogen NH$_4$Cl. To get solid PVDF-chitosan membrane,

done the immersion-precipitation process of membrane casting solution in coagulation bath containing non solvent. The exchange process of solvent from polymer solution with non-solvent from a coagulation bath produces phase separation. The occurrence of phase inversion process will lead to the solidification of the casting solution, and then will be produced solid   PVDF-chitosan membranes.

Dissolving the polymer material will always be accompanied by a free energy (ΔG) change. Despite the dissolving polymer material led to the change of entropy are great, but the spontaneous will be largely determined by the amount of mixing heat (ΔH) reaction. One of the equations that can be used to evaluate the magnitude of the ΔH is Hilderbrand equation (equation 5.1).

$$\Delta H_m = V_m . [(\Delta E_1 / V_1)^{\frac{1}{2}} - (\Delta E_2 / V_2)]^2 . \emptyset_1 . \emptyset_2 \qquad \textbf{(1)}$$

Whereas,   ΔHm   = total heat of mixing
Vm   = total molar volume of mixing
ΔE_1 = vaporization enthalpy of components 1
ΔE_2 = vaporization enthalpy of components 2
V_1 = molar volume of components 1
V_2 = molar volume of components 2
$\emptyset_1$ = volume fraction of components 1
$\emptyset_2$ = volume fraction of components 2

From equation 1, it seems clear that ΔHm will always be positive and when the value is greater, the value of the Gibbs free energy (ΔG) that accompanying the polymer dissolving reaction will be even greater and cause a reaction becomes increasingly not spontaneous. This condition then induces the phase inversion process, so that the dense membranes can be obtained. Application of temperature stirring above room temperature at this research, will cause an increase of non solvent temperature and induce the occurrence of phase inversion more quickly.

The phase separation process of five PVDF-chitosan membrane composition created on this research takes time-varying, which is when the higher stirring temperature of the casting solution was applied, the longer time will be required for the occurrence of phase inversion to generate the solid PVDF-chitosan membrane. Based on the results of the study, noted that the stirring process of the casting solution with temperature 40 °C; 50 ºC and 60 °C, phase separation occurred respectively in 78 seconds; 73 seconds; and 67 seconds. Not only stirring temperature that plays a role in the phase separation process, the composition of the casting solution used also affects the time it takes for the occurrence of the phase inversion. On the casting solution composition (PVDF/chitosan/NMP/NH$_4$Cl) (%w/w) 12/2/84/2; 13/1.5/84/1.5; 14/1/84/1; 15/5/84/0.5; and 16/0/84/0 with stirring process temperature 40 ºC, respectively obtained phase separation time 71; 73; 78; 79; and 89 seconds.

Depreciation of PVDF-Chitosan Composite Membrane

Establishment of solids PVDF-chitosan composite membrane can also be described using the concept of Hansen solubility parameter and Hilderbrand equations, where the flow of heat from a casting solution into a non solvent has led to thermodynamic disruption on chemical equilibrium of PVDF and chitosan dissolving reaction in NMP. The amount of heat energy that is channeled into a non solvent will largely determine the rate of phase inversion occurs, which will automatically also determine the physical quality of the PVDF-chitosan composite membrane formed. More faster phase inversion rate that occurs in membrane casting solution, the greater depreciation levels of membrane will be happen. This is strengthened by the presence of the membrane depreciation level data obtained in this research.

Based on the depreciation rate measurements results of the PVDF-chitosan composite membrane, it can be noted that the composite membrane generated from casting solution with stirring temperature 60 ºC has the biggest depreciation, i.e. 21,78%. While on deployment of the stirring temperature 50 °C, the PVDF-chitosan composite membrane generated has depreciation of 20,22%. Meanwhile, at the application stirring temperature 40 °C, the   PVDF-chitosan composite membrane generated has rates depreciation of 19,11%. Depreciation rate of the PVDF-chitosan composite membrane is continues to decline or wane in line with the reduced heat flow into a non solvent. Thus, it can be said that more smaller heat flow is going into a non

solvent, the lower level of PVDF-chitosan composite membrane depreciation will be happens. A complete overview of the depreciation rate data of   PVDF-chitosan composite membrane looks on the figure 1.
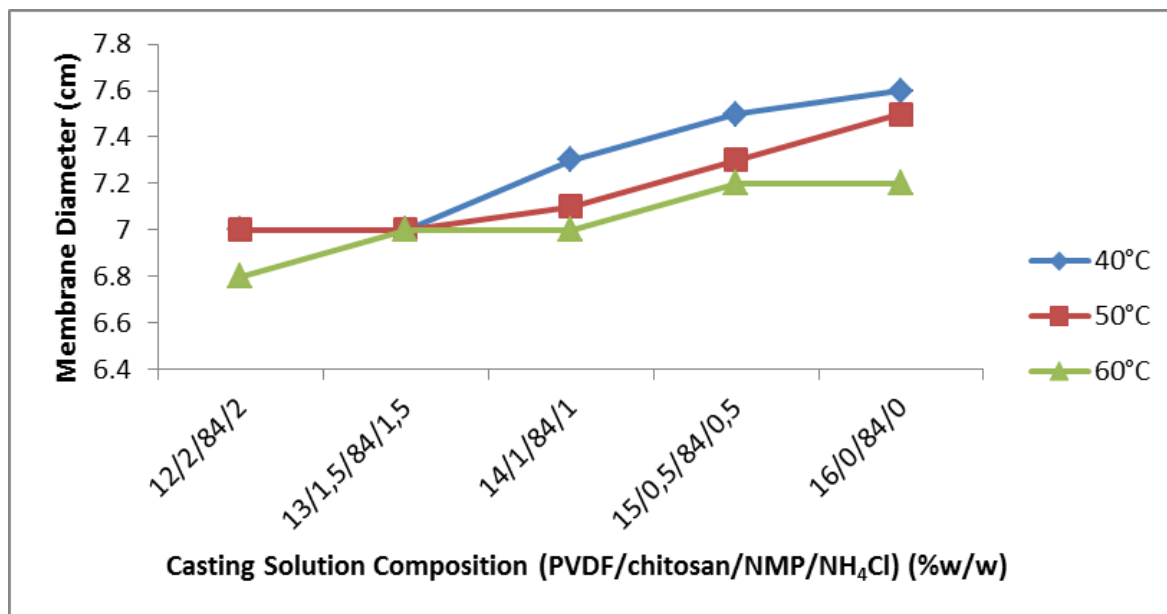

Figure 1:   Graph of PVDF-chitosan composite membran shrinkage rate data with variation of stirring temperature

Characterization of PVDF-Chitosan Composite Membrane

Surface and cross-section morphology analysis of PVDF-chitosan composite membrane

To know the effect of chitosan blending on structure morphology and pore size of PVDF dan chitosan membrane membrane, at this research stage has done an analysis of the surface and cross-section morphology of the PVDF-chitosan composite membrane using SEM, as well as pore size analysis using SAA.

Based on surface morphology analysis which is obtained, it is known that the casting solution composition affects the surface morphology of PVDF-chitosan composite membrane. This is evidenced by the appearance of more denser pore size on the membrane with casting solution composition PVDF/chitosan/NMP/NH$_4$Cl (%w/w) 15%PVDF/0.5% chitosan/84% NMP/0.5% NH$_4$Cl (figure 2a) compared with PVDF-chitosan composite membrane with casting solution composition of 12% PVDF/2% chitosan/84% NMP/2% NH$_4$Cl (figure 2b), which are both produced from the membrane preparation process using a mixing temperature of 40 °C. The same condition is also observed in the PVDF-chitosan composite membrane with casting solution composition PVDF/chitosan/NMP/NH$_4$Cl (%w/w) 15% PVDF/0.5% chitosan/84% NMP/0.5% NH$_4$Cl (figure 2c) compared with PVDF-chitosan composite membrane with a casting solution composition 12% PVDF/2% chitosan/84% NMP/2% NH$_4$Cl (figure 2d), which are both produced from the membrane preparation process using a mixing temperature of 60 °C .

In addition to casting solution variable, stirring temperature variables also affect the surface morphology of PVDF-chitosan composite membrane is formed, which detected from the appearance of more denser or small pore on composite membranes with casting solution composition of 15% PVDF/0.5% chitosan/84% NMP/0.5% NH$_4$Cl using stirring temperature of 40 °C compared with PVDF-chitosan composite membrane resulting from casting solution with a composition of 15% PVDF/0.5% chitosan/84% NMP/0.5% NH$_4$Cl using stirring temperature 60 °C. Overview learn more about the surface morphology of PVDF-chitosan composite membrane shown in figure 2.
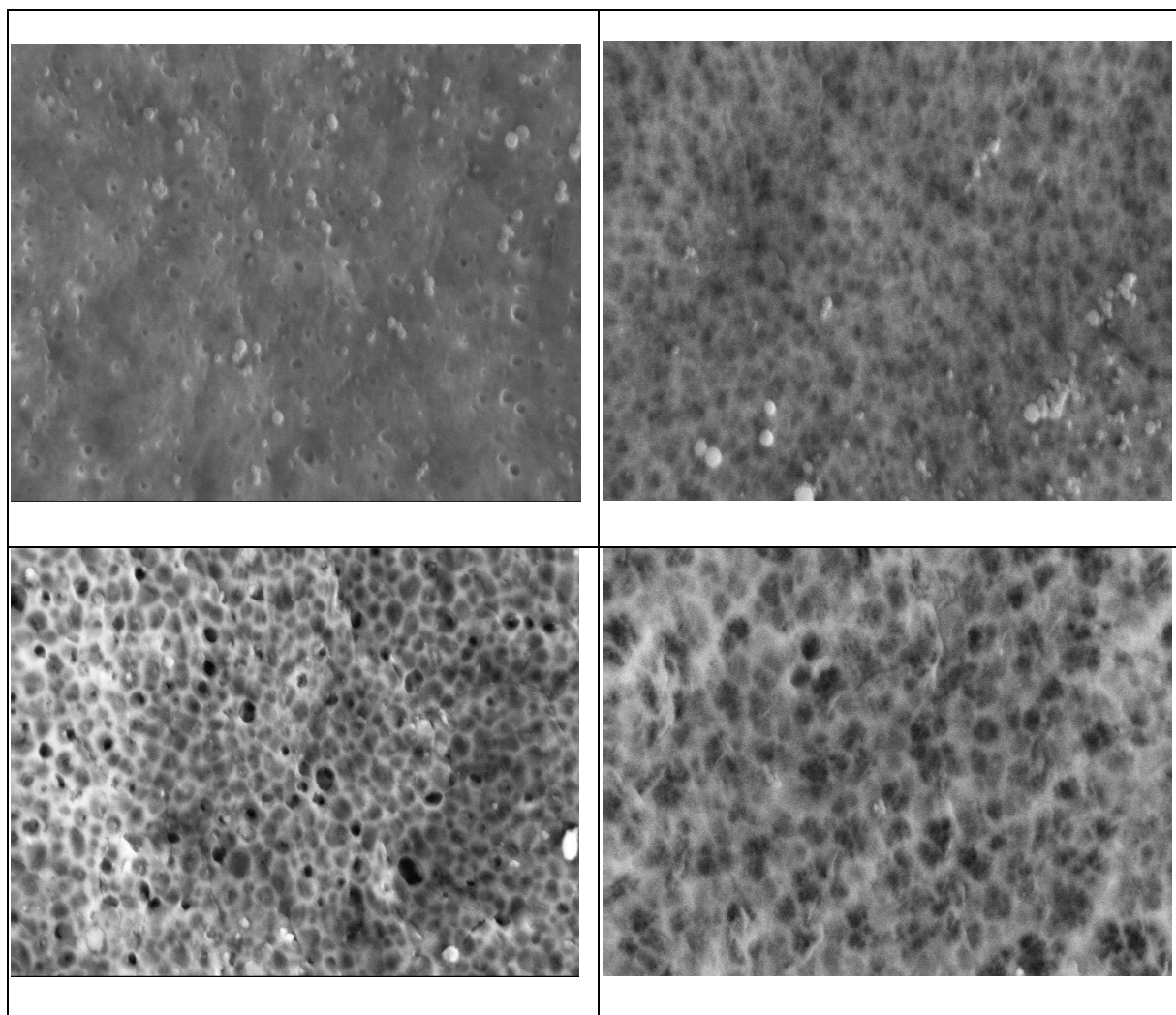
Figure 2: Surface morphology of PVDF-chitosan composite membrane with casting solution composition : (1) 15% PVDF/0,5% chitosan/84% NMP/0,5% NH$_4$Cl; and (2) 12% PVDF/2% chitosan/84% NMP/2% NH$_4$Cl (stirring temperature 40 °C); (3) 15% PVDF/0,5% chitosan/84% NMP/0,5% NH$_4$Cl; and (4) 12% PVDF/2% chitosan/84% NMP/2% NH$_4$Cl (stirring temperature 60 °C) (magnification 5000x)

For the next characterization of PVDF-chitosan composite membrane surface morphology, at this stage also has done the membrane pore size analysis using SAA instruments. Based on the SAA analysis on PVDF-chitosan composite membrane resulting from casting solution composition of 13% PVDF/1.5% chitosan/84% NMP/1.5% NH$_4$Cl using stirring temperature of 60 °C is known that the membrane concerned have: (1) surface area 22.186 m^2/g; (2) The pore volume 0.099 cc/g; and (3) pore size19.121 Å. While   PVDF-chitosan composite membrane resulting from casting solution composition of 15% PVDF/0.5% chitosan/84% NMP/0.5% NH$_4$Cl using stirring temperature of 40 °C is known that the membrane in question has: (1) The surface area 12.927 m^2/g; (2) The pore volume 0.058 cc/g; and (3) The pore size 15.344 Å. Thus, it can be concluded that the greater the level of PVDF in casting solution and the lower stirring temperature is applied in the manufacture of PVDF-chitosan composite membrane, will cause the formation of membrane surface area, pore volume and pore size, which is denser. Vice versa, the smaller the PVDF content in casting solution and the higher stirring temperatures that are applied in the manufacture of PVDF-chitosan composite membrane, will cause the formation of membrane surface area, pore volume and pore size is more tenuous.

Mechanical strength analysis of the PVDF-chitosan composite membrane

Furthermore the surface morphology, to find out more about the characteristics of the PVDF-chitosan composite membrane are primarily related to the concerned of the membrane's ability in maintaining the pore size, at this stage has been done membrane mechanical strength test using autograf. The measuring results of

PVDF-chitosan composite membrane mechanical strength look at figure 3. Based on that data, it appears that blending chitosan in materials PVDF have lowered the PVDF membrane mechanical strength, which had acquired the average of mechanical strength 1697,89 N/m^2; 1404,728 N/m^2; and 1200,95 N/m^2, respectively to PVDF-chitosan composite membrane are produced from membrane preparations using stirring temperature 40 °C; 50 °C; and 60 °C. The results of each was 25,77%; 38,59%; and 47.50% lower than mechanical strength of PVDF membrane unmodified chitosan on the same temperature stirring.

In addition, based on such data may also be aware that with the increasing levels of chitosan that blended in PVDF materials, the lower mechanical strength of PVDF-chitosan composite membrane was obtained. For example, from the preparation of PVDF-chitosan composite membrane as stirring results using the temperature of 40 ºC, retrieved the young's modulus value 1,963.35; 1,877.35; 1,875.67; 1,454.02 and 1,319.06 N/m^2, respectively for the casting solution composition (PVDF/chitosan/NMP/NH$_4$Cl)% (w/w) 16/0/0/84; 15/5/84/0.5; 14/1/84/1; 13/1.5/84/1.5; and 12/2/84/2. In the meantime, for the PVDF-chitosan composite membrane resulting from the membrane preparation process using stirring temperature 50 ºC, the young's modulus values obtained were lower, which amounted to 1,800.76; 1,328.25; 1,307.19; 1,304.23 and 1,283.21 N/m^2, respectively for the casting solution composition (PVDF/chitosan/NMP/NH$_4$Cl)% (w/w) 16/0/0/84; 15/5/84/0.5; 14/1/84/1; 13/1.5/84/1.5; and 12/2/84/2. The higher chitosan composition that using as a blending agent of PVDF, the more chitosan polymer particles that decomposized between PVDF polymer particles. The interaction between chitosan and PVDF formed through hydrogen bonding between the hydrogen atom on the amine groups with fluorine atom on PVDF molecule, have contributed to the decline in bond energy possessed by PVDF material, which will have an impact on decreasing the membrane ability in maintaining the pore size of (young's modulus), when applied a driving force with particular magnitudes.
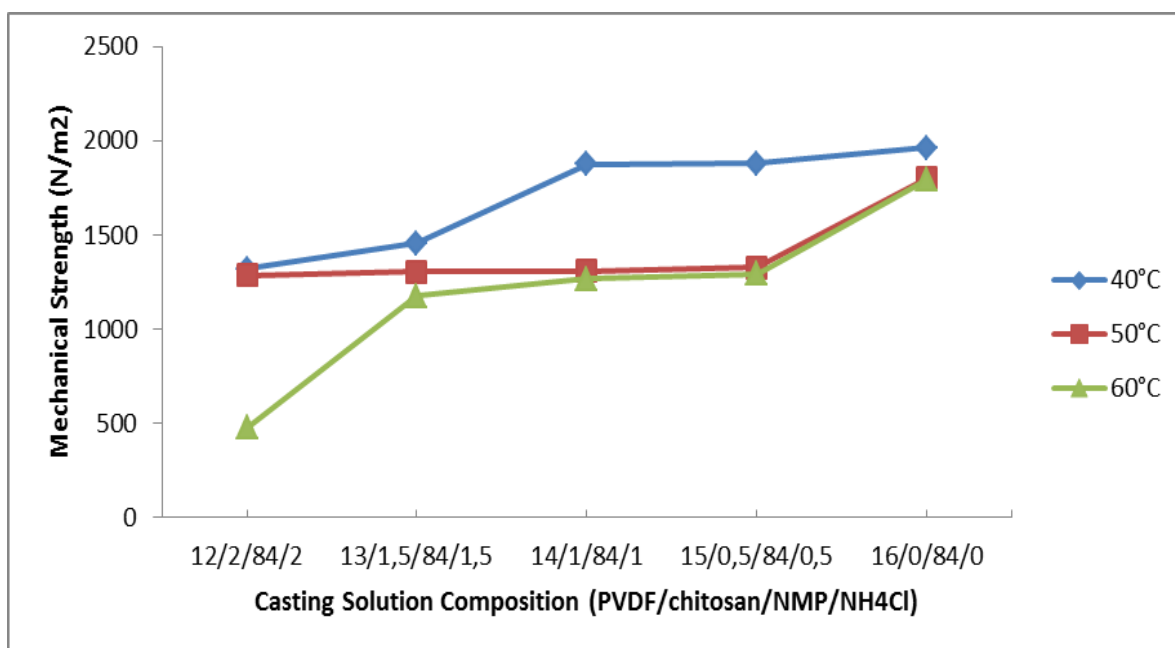

Figure 3: Graph of the PVDF-chitosan composite membrane mechanical strength data

Permeability of PVDF-Chitosan Composite Membrane

Permeability can be defined as the speed of a species to penetrate the membrane.. Permeability is strongly influenced by the number of pores, pore size, pressure-operated and membrane thickness. In measuring the performance of the membrane, the permeability is expressed in flux, the amount of permeate volume that passes through a unit area of the membrane in a given time.
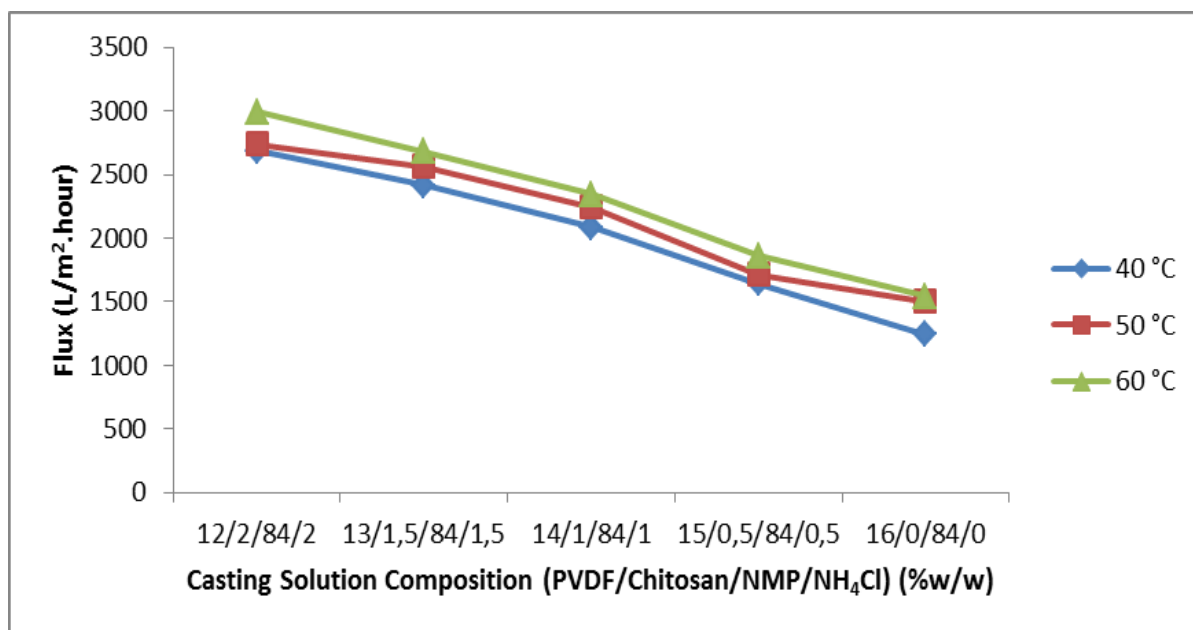
Figure 4: Graph of the PVDF-chitosan composite membrane permeability data

Chitosan concentration in casting solution greatly affects the flux membranes. In this study, the casting solution composition of the PVDF-chitosan composite membrane was made is in the range (PVDF/chitosan/NMP/NH$_4$Cl) (%w/w) 12:2:84:2-16:0:84:0. The relation between chitosan levels in a casting solution and the membrane flux can be seen in figure 4. From figure 4, it also appears that increased levels of chitosan in the casting solution, which is followed by increased levels of ammonium chloride, will result in an increased flux of waste. This is due to the increased levels of chitosan and NH$_4$Cl in the casting solution will result in the formation of polymer configurations which distributed more tenuous compared with PVDF particle configuration in the PVDF membrane casting solution without chitosan addition. The lower density of the polymer particle configuration, has led the driving force that necessary to be able to pass certain particles penetrate the membrane becomes smaller and because it, the feed solution speed which passes the membrane be higher.

Accordingly, the application of higher stirring temperature on membrane casting solution also has led to the formation of more tenuous polymer particles configuration on the solid PVDF-chitosan composite membrane. An increase in temperature of casting solutions, has led to greater heat flow into the non-solvent H$_2$O in the coagulation bath. The existence of the heat flow has caused the mutual difusity rate of non-solvent on the casting solution to be fast, which automatically also speeds up the precipitation rate of the membrane casting solution which prepared by phase inversion with immersion-precipitation technique.

The mutual diffusity rate acceleration between non-solvent and solvent in the casting solution can be occur because of the availability of a larger energy for termination of interactions that occur both between PVDF with PVDF, or between PVDF with NMP and NH$_4$Cl. Membrane resulting from precipitation events are relatively fast, generally has a morphological structure which is a mixture of sponge- pore and pore finger type. While the membrane resulting from precipitation events are relatively slow, generally have morphological structure with uniform pore sponge structure in the cross-sectional.

In figure 4, appear the wastewater flux data of each PVDF-chitosan composite membranes with different a casting solution composition and stirring temperature, resulting from the coagulation/precipitation process using 100% H$_2$O as a non-solvent. In casting solution composition (PVDF/chitosan/NMP/NH$_4$Cl) (% w/w) 16: 0: 84: 0 resulting from the mixing process using a temperature of 40 °C, obtained flux at 1244.66 L/m^2.h. The wastewater flux values begin to rise when in the casting solution was added chitosan. Where in the chitosan addition of 0.5% (w/w) to casting solution, has obtained the membrane flux value of 1640.71 L/m^2.h. Further increase in flux occurs when the levels of chitosan in casting solution enhanced up to 1% (w/w) - 2% (w/w), each of which produces a flux value of 2088.12 L/m^2.h and 2417.69 L/m^2.h. In line with that, the

highest flux value, which amounted to 2687.23 L/m^2.h obtained when the chitosan concentration increased up to 2% (w/w).

The higher flux values of membranes obtained when the PVDF-chitosan composite membranes which used in separation, homogenized by stirring temperature of 50 °C. An increase in flux compared with the flux generated by the PVDF-chitosan composite membrane resulting from the mixing process of casting solution at temperature of 40 °C is due to the application of the higher stirring temperature on the casting solution has led to greater disruption of the thermodynamic equilibrium of the reaction. This disorder is due to the availability of heat energy that is greater in the non-solvent, which can facilitate the termination of the interactions that occur between PVDF with NMP and NH_4Cl. Based on the data in figure 4, flux values obtained for the PVDF-chitosan composite membrane resulting from the mixing process using a temperature of 50 °C, each of which is equal to 1503.87 L/m^2.h; 1716.9 L/m^2.h; 2243.11 L/m^2.h; 2563.78 L/m^2.h and 2740.15 L/m^2.h to casting solution composition (PVDF/chitosan/NMP/NH_4Cl) (% w/w) 16/0/84/0; 15/0.5/84/0.5; 14/1/84/1; 13/1.5/84/1.5; and 12/2/84/2. In line with this, the highest flux value of membranes obtained from casting solution homogenized using a stirring temperature of 60 °C.

CONCLUSION

Based on the research results that have been obtained, it can be concluded, including (1) The surface of the PVDF-chitosan composite membrane resulting from casting solution with lower PVDF levels and the higher stirring temperature, will have a pore area, pore volume and pore size that is less dense than the surface of the PVDF-chitosan composite membrane that resulting from casting solution with PVDF levels are higher and the lower the temperature stirring; (2) The casting solution composition and stirring temperature effect on the mechanical strength of the PVDF-chitosan composite membrane. The higher levels of PVDF in casting solution and the lower stirring temperatures used in the preparation of the membrane, the higher mechanical strength PVDF-chitosan composite membrane produced. And vice versa; and (3) The casting solution composition and the stirring temperature effect on the permeability of PVDF-chitosan composite membrane. The higher levels of PVDF in casting solution and the lower stirring temperatures used in the preparation of the membrane, the lower permeability of the PVDF-chitosan composite membrane against wastewater samples which resulting. And vice versa.

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