Effect of Addition Silica Gel from Volcanic Ash of *Sinabung* Mountains to Tensile Strength on Chitosan Composite Membrane

Maulida[1], Lilis Sukeksi[1], Mara Bangun Harahap[2], Melva Ginting[1], Herlinawati Wici[1] and Ghendis Ekawati Ayu[1]

[1]Departement of Chemical Engineering, Faculty of Engineering, University of Sumatera Utara, Almamater Street,Medan, 20155, Indonesia

[2]Departement of Physics, Faculty of Mathematic and Science, University of Negeri Medan, Medan Estate, North Sumatera 20222, Indonesia

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Abstract: Silica gel is a glassy grain with a very porous shape. Silica is made synthetically from sodium silicate. In this study, volcanic ash can be used as silica, which is silica can be applied in the manufacture of composite membranes as fillers because it can increase the conductivity of composite membranes. The method used for making silica gel was extraction method and composite membrane made using phase inversion method with the composition of chitosan and silica used are 2 grams of chitosan and variations of fillers 0.6, 0.9 and 1.2 grams of silica, and the stirring time is 8,12 and 16 hours. The characteristics and analysis carried out in this study were FTIR and SEM. Based on the results of the study, the best membrane conditions were obtained from composite membrane analysis with a tensile strength of 34,118 Mpa. In the Scanning Electron Microscopy (SEM) analysis, the lowest composite membrane has a dense and homogeneous pore, while the highest composite membrane has a hollow pore and has been homogeneous.

1 INTRODUCTION

The composite membrane consists of organic polymers and inorganic fillers which can improve the performance of membranes eg, zeolites have been added into polymer membrane to increase selectivity of gas separation. Silica is one of the abundant metal oxides in volcanic ash can be utilized as the basic material of silica gel synthesis through the formation of alkali silicate precursors. Sodium silicate can be converted into silica gel by condensation process and hydrolysis using solvent, both polar and non polar. By extracting the silica in the alkaline state so that sodium silicate will form. Sodium silicate will undergo polymerization process to form silica gel on some difference of pH and solvent (Uhlmann and Kreidhl, 1980). Silica is use to reduce excessive swilling as to control the moisture content, reduce the permeability of methanol, increase mechanical stability and the conductivity of proton in PEM for fuel cell (Siniwi, 2014). The chitosan is attractive for use as a membrane base material because it has a functional group $-NH_2$ and -OH which is easy to modify. Chitosan membranes are hydrophilic, non-toxic, biodegradable, large surface area and reactive to metal ions because they have an active group $-NH_2$ and -OH. Chitosan membranes also have disadvantages of low mechanical properties indicated by the price of tensile strength (tensile strength), percent elongation (percent extension), and low modulus young. The basic ingredients of inorganic compounds are silica which aims to improve the stability of the chitosan membrane through the formation of crosslinks with silica through the formation of hydrogen bonds between the chitosan structure and the silica (Widhi Mahatmanti, 2014). The selection of chitosan as an alternative to modify the plastic cause chitosan has biodegradable characteristic (Maulida, et,al., 2018). (Neburchilov et al., 2007) reported that the addition of *nanosilika* to the membrane *nafion* (nafion / SiO_2) for DMFC can increase proton conductivity by a ratio of 0.33 to 0.38 compared to a membrane nation. Volcanic ash or volcanic sand is a falling volcanic material that is ejected into the air during an eruption. Low-energy basal eruptions (basal: dark

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frosted rocks, fine-grained clays of lava from volcanoes) produce a distinctively dark ash containing 45-55% silica which is generally rich in iron (Fe) and magnesium (Mg). Based on research of (Nakada and Yoshimoto, 2014) stated that the silica content in volcanic ash of Sinabung Mount was 58.10%. The high content of silica in volcanic ash of Mount Sinabung is an interesting subject for further investigation, especially regarding the use of volcanic ash as the base material of silica adsorbent to bind lead weight metal.

2 MATERIALS DAN METHODS

2.1 Raw Materials and Equipments

The material used chitosan was obtained from Faculty of Mathematics and Natural Sciences, silica was obtained from volcanic ash of Sinabung Mountain, NaOH, *aquadest*, acetic acid (CH_3COOH) and hydrochloric acid (HCl) were obtained from CV. Rudang Jaya The equipment used in this research were hot plate, 50 mesh sieve and 230 mesh sieve, filter paper, magnetic stirrer, funnel, measuring cup, oven and glass beaker.

2.2 Preparation of Volcanic Ash

The volcanic ash was sieved with a 230 mesh sieve to homogenize the ash size. The sifted ash was taken as much as 50 grams and soaked with HCl and filtered. Then the ash washed and dried with oven.

2.3 Extraction of Sodium Silica Solution

Volcanic ash was dissolved with 500 ml of 4 M NaOH and heated at 190 °C with a variation of 120 minutes. It was filtered to get the filtrate. The filtrate was tested with gravimetric (Maulida, et.al., 2017).

2.4 Process of making Silica Gel

The sodium silica solution inserted into the beaker glass. Then dripped with HCl with a variety of concentrations of 8 M to form a white gel with pH of 7. Silica gel was precipitated 24 hours and filtered with paper washed with *aquadest*. Silica gel was dried using an oven at 100 °C to remove excess acid (Maulida, et.al., 2017).

2.5 Process of Making Chitosan Composite Membranes with Silica Fillers

Composite membrane synthesis was carried out using phase inversion method. First, 2 grams of chitosan dissolved in 100 mL of 2% acetic acid at room temperature for 2 hours. Second, silica composition is 0.6, 0.9, 1.2 grams, respectively. then stirred at a temperature of 600 °C for 8.12.16 hours. The homogeneous solution is also referred to as a dope solution which has no air bubbles, then the filtered solution is then poured onto a 20 x 20 cm glass meld and dried at room temperature resulting in a dry membrane. Dry membrane soaked with 1 M NaOH for 2 hours then washed with *aquadest* to neutral pH and dried at room temperature.

3 RESULTS AND DISCUSSION

3.1 Characteristic of Fourier Transform Infra - Red (FTIR) Silica Gel

Characteristic of FTIR silica gel were carried out to identify functional groups of silica gel. The characteristics of FTIR silica gel can be see in the figure 1.

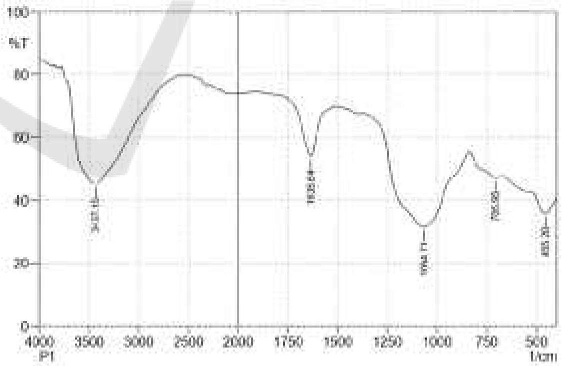

Figure 1: Characteristic of FTIR Silica Gel from Volcanic Ash of Sinabung Mountain (Maulida,et.al.,2017).

The FTIR test on silica gel obtained by the absorption peak at wave number 3437.15 cm^{-1} shown the presence of *silanol* function group derived from hydroxyl group bond Si-Si, at wave number 1635.64 cm^{-1} was indicated the presence of hydroxyl (OH) group, on the wave number 1064.71 cm^{-1} was indicated the presence of siloxane functional groups (Si-O-Si), at the wave numbers 705.95 cm^{-1} and 455. 20 cm^{-1} was indicated the presence of Si-O functional groups (Silverstein, 1981).

3.2 Characteristic of Scanning Electron Microscopy (SEM) Silica Gel from Volcanic Ash of Sinabung Mountain

Analysis of SEM was conducted to determine membrane pores.

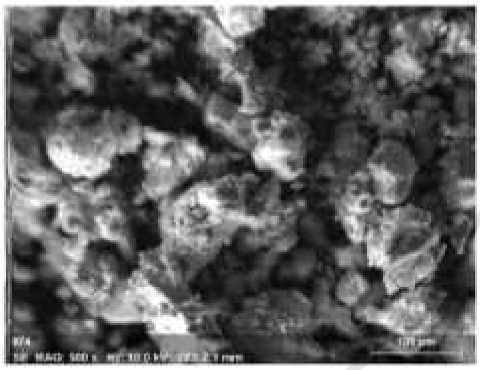

Figure 2 : Characteristic of Scanning Electron Microscopy (SEM) Silica Gel from Volcanic Ash of Sinabung Mountain.

In this study, it was cleared at the sample surfaces that there were uneven and composed of clumps, which has shown a wide variety of grains with uneven distribution on the surface. The separation between lumps is also seen quite clearly, it was micro-cracking found among clusters (Maulida, et.al.,2017).

3.3 The Effect of Silica Gel Mass Variation and Stirring Time to Tensile Strength of Composite Membrane

Characteristics of mechanical properties need to know the strength of membrane against forces outside, which can damage the membrane. The following graph shown the effect of silica variation and stirring time to tensile strength of the composite.

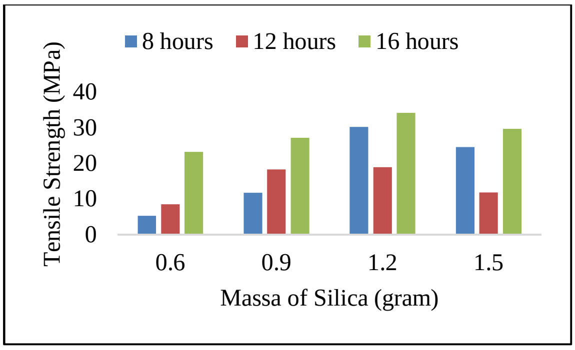

Figure 3 : The Effect of Silica Gel Mass Variation and Stirring Time to Tensile Strength Of Composite Membrane.

Based on figure 3, the mass of silica and the length of stirring time increase; the value of tensile strength increases too. The results of this study was indicated that at 8 hours of stirring time, the tensile strength of membrane was 7,793 MPa on silica variation of 0.6 gram, then increased to 11,735 MPa at variation of 1.2 gram silica. At 12 hours of stirring time, tensile strength was 18.260 MPa at 0.6 gram, then increased to 23,140 MPa at variation of 1.2 grams of silica. In the 16 hour stirring time, the membrane flux value of 27.168 MPa at 0.6 grams, then increased to 34.118 MPa in a variation of 1.2 grams of silica.

From Figure 4, we can see the effect of adding silica filler to the tensile strength of composite membrane. The increased amount of silica can cause the tensile strength of the membrane increased too. Tensile strength is the maximum resistance that can be retained by the material when given a force before the material is broken. The more silica added to the membrane, the more interaction between the silica and the chitosan. This will lead to stronger bonds and intermolecular forces in the membrane. The addition of silica will cause crosslinks between silica and chitosan by hydrogen bond (Karlina, 2016). The tensile strength of this membrane has increased as the percentage of silica increased. The more silica added to the membrane, the more interaction between the silica and the chitosan. This will lead to stronger bonds and intermolecular forces in the membrane. The addition of silica will lead to crosslinking between silica and chitosan by hydrogen bonding. The increase in tensile strength in the addition of 0.6 grams of silica to 1.2 grams of silica followed by a drop in tensile strength at the next point of 1.5 grams of silica. The occurrence of a drop in the value of tensile strength is likely due to the excess amount of silica that causes during the drying process of the cracked composite membrane. This indicates that the increase in concentration does not guarantee an increase in the value of tensile strength.

Generally, the results show the greater filler to the membrane and stirring time. They give higher tensile strength results. Previous research on the manufacture of composite membranes from chitosan showed similar results where the tensile strength value obtained was greater by increasing the addition of silica, it is. due to its dense structure which causes the distance between the molecules in the membrane to be denser so as to have a large tensile strength (Thermo, 2001).

3.4 Characteristic of Scanning Electron Microscopy (SEM) Chitosan Composite Membrane with Silica Gel

An analysis of SEM was performed to determine membrane pores. The test was performed at 0.6 gram silica fused chitosan membrane with 8 hours of stirring time and composite membrane with silica addition of 1.2 grams of silica with a stirring time of 16 hours. Because the composite membrane with 0.6 gram of silica filler has a low flux value, while 1.2 grams of silica has a high flux value.

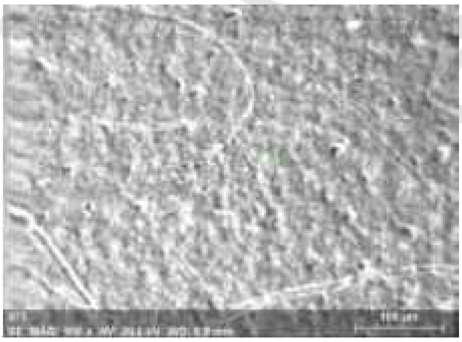

Figure 4: Characteristic of Scanning Electron Microscopy (SEM) Chitosan Composite Membrane with Silica Gel.

Figure 4 shows the result analysis SEM of the membrane product with the addition of 1.2 grams of silica and 16 hours of stirring time has a relatively more dense morphology, which indicated that silica was evenly mixed in the resulting membrane. The addition of silica causes a dense chitosan membrane to be hollow because the negative charge of the chitosan OH-reacts with the silica so that it will attract and form a small cavity (Siniwi, 2014). Based on Figure 4 it can be seen that the pore size of the membrane composites with 1.2 g silica added with 16 hours of stirring time was homogeneous. It’s due to the addition of silica which made the membrane structure hollowed with longer stirring time, so the membrane becomes more homogeneous and can be applied for filtration.

4 CONCLUSIONS

The addition of silica too much and string of time too long causes the composite membrane to overlap until homogeneous so that the tensile strength increases, acetic acid is a good solvent use in dissolving chitosan and silica, addition of the filler on making composite membrane cause the greater pore in membrane.

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