

CHARACTERIZATION ON SILICA FROM WASTE SUGARCANE BAGASSE FOR MEMBRANE FABRICATION

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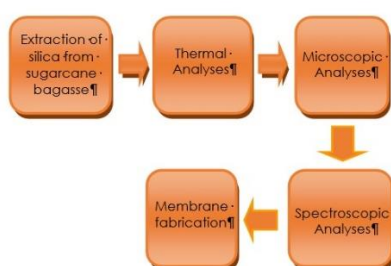
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Graphical abstract



Abstract

The used of additive enhance the surface of the membrane layer and can be choose from various material. Examples of commonly used additive in membrane are Polyethylene glycol, silica oxide, cellulose acetate and Polyvinylpyrrolidone. Silica oxide was proven have ability to minimize the fouling problem hence increase hydrophobic properties of membrane. Silica also can be extracted from rice husk ash, sugarcane bagasse, sorghum vularae seed and kenaf by precipitation method, biodigestion and sol-gel process. Silica extraction from sugarcane bagasse was chosen as the organic additive for membrane formation. In order to investigate the suitability of the material, several characterization test have been conducted. There are thermal, microscopic and spectroscopic analyses. Thermal gravimetric analysis was performed on sugarcane bagasse to determine the amount of silica that can be extracted from it. Results of TGA on sugarcane bagasse show that the peak temperature at 315.70 °C are defined as crystalline melt. After the melt transition, the baseline takings to a slightly lower position than the pre-melt baseline. The post-melt baseline changes slope as the sample begins decomposition while TGA extrapolated onset temperature of 241.56 °C as this sample decomposes. The analysis of microscopic shows that the addition of silica from sugarcane bagasse changed the surface structure of the membrane especially at top layer and sub layer. Sugarcane bagasse show bands for carboxylate (COO-) and hydroxyl (OH-) groups. The availability of negatively charged groups at the surface of sugarcane bagasse shows potential to be used as additive in membrane fabrication.

Keywords: Membrane, microscopic, silica, sugarcane bagasse, thermal

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1.0 INTRODUCTION

Sugarcane was harvested from one of the plantation in Malaysia. Sugarcane bagasse is the fiber source that remains after sugarcane are crush to extract their juice. Sugarcane bagasse were considered as one of the most crucial commercially fiber sources and proven to be a better option than wood fibers in producing textiles, paper, pressed wood materials and few more products [1]. Silica that can be extracted from sugarcane was found to be suitable

as the additive for membrane fabrication. In 19 century, silica was found in the plant and was used to produce a constant material. Since then, silica is generally accepted as polymer compound that are sustainable. The use of sugarcane bagasse helps the industries in term of environmental ecosystem [1]. Sugarcane bagasse can replace the usage of wood in paper industry and also helping in reducing the damage of tropical rainforest.

A study on research done related to membrane technology explains that membranes were

fabricated using additive from various materials but none of it using silica from sugarcane bagasse. Idris, Kee, & Ahmed, (2008) [2] used MSG as additive for membrane formulation reported that the suitable increment of monosodium glutamates in dope solution of dialysis membrane has improved its performance. Bowen, Doneva, & Yin, (2002) [3] find that Sulfonated Poly(ether ether ketone) (SPEEK), also give effective function in membrane surface condition, however, Kim & Lee, (1998) [4] who found that the increase of Polyethylene glycol (PEG) ratio will result in casting solution becomes thermodynamically less stable. Membranes are proven to have ability to treat wastewater effectively; however, there is weakness on membrane where its hydrophobic absorption may cause fouling [5]. Therefore, this research produced new membrane formulation and fabrication using new material that is sustainable and low cost. Percentage of the silica extracting from sugarcane bagasse was formulate to fabricate the membrane. This study also investigates the characteristics of the silica from waste sugarcane bagasse such as thermal, microscopic and spectroscopic analyses.

2.0 EXPERIMENTAL

2.1 Materials

Material use in this study included sugarcane bagasse, N, N- Dimethylacetamide (DMAc), Polysulfone (PSF), Polyvinylpyrrolidone (PVP), Sodium chloride (NaCl), Sodium Hydroxide (NaOH), Hydrochloric Acid (HCl), and Sulfuric Acid (H₂SO₄).

2.2 Extraction of Silica

Sugarcane bagasse samples were undergo refluxing process with HCl for 4 h and washed frequently using deionised water to make it acid free. After that the sample was dissolved in NaOH by stirring continuously for 10 h on a magnetic stirrer [6]. Then concentrated H₂SO₄ was added to adjust pH in the range of 7.5-8.5. The precipitated silica was washed repeatedly with warm deionised water until the filtrate became completely alkali free. The deionised water was used time after time for washing process. The silica was dried at 50 °C for 48 h in the oven [6].

2.3 Membrane Fabrication

After that, sugarcane bagasse silica was formulated with Psf, PVP and DMAc to fabricate membrane. Membrane formation was started with the titration process. Titration process is a process where Psf was added into DMAc and stirred continuously until the polymer is dissolved with the solvent. PVP and silica were added and the mixture was stirred with a control temperature of 60 °C until homogeneous. The

percentage of silica added vary from 1% to 6%. The mixture of those materials is called dope of membrane. The dope was left for 24 hour to ensure the air that trapped in that mixture during the stirring process bubble out. The pneumatically controlled flat sheet membrane casting unit (Model: TR31-A) was used to fabricate the membrane, wet-dry immersed membrane process and also for flux permeate and salt rejection tests.

2.4 Characterization

2.4.1 Thermo Gravimetric Analysis (TGA)

The sugarcane bagasse was tested for thermal analysis at Nano-SciTech Centre, Universiti Teknologi Mara (UiTM) to estimate the amount of sugarcane bagasse needed for extracting certain amount of silica. Thermal Gravimetric Analyzer (Pyris Diamond TGA, Perkin-Elmer) was used to characterize the thermal stability of the sugarcane bagasse samples. Approximately 10 mg of each sample was heated from 30 °C to 750 °C at a heating rate of 10 °C/min. All of the measurements were performed under a nitrogen atmosphere with a gas flow of 50 ml/min in order to prevent any thermoxidative degradation.

2.4.2 Microscopic Analyses

Field Emission scanning electron microscopy (FESEM) photographs of silica surfaces from sugarcane bagasse were captured. In this case, the samples were coated with gold using the sputtering technique.

2.4.3 Fourier Transforms Infrared (FTIR) Spectroscopy

The FTIR spectra of the sugarcane silica from sugarcane bagasse were recorded on an instrument (Shimadzu FTIR 8400) in the range of 400–4000 cm⁻¹ with a resolution of 4 cm⁻¹.

3.0 RESULTS AND DISCUSSION

3.1 Membrane Efficiencies

Membranes were tested on permeate flux using pure water to verify the characteristic of the membrane. The effect of silica as an additive on the pure water flux and salt rejection performance is illustrated in Figure 1. From the figure, the flux increases when the silica content increases up to 3%. The pure water flux of 1% silica is 25.68 Lm⁻²h⁻¹, 2% silica is 27.60 Lm⁻²h⁻¹ and 3% silica is 39.45 Lm⁻²h⁻¹. This increasing behaviour might due to the silica 3% is more hydrophilicity compare to the other percentages of silica. However, the pure water flux decreases to 38.28 Lm⁻²h⁻¹, 35.47 Lm⁻²h⁻¹, and 35.31 Lm⁻²h⁻¹ when the silica increases to 4%, 5% and 6% respectively. Similar trend reported by Ahmad *et al.* (2007) shows the 2% of silica was the optimum for water permeability [7]. This

is due to the interactions between contaminants and membrane surface were reduced by silica particles of the membrane surface. At 3% of silica, hydrophilicity of the membrane increases and consequently it attracts water molecules into the composite membrane. Moreover, hydrophilicity properties may also facilitates water penetration through the membrane thus enhances the flux. Nevertheless, higher concentration of silica will cause the pure water flux to decrease immediately. As the consequences, the hydrophilicity of membrane also decreases where the pure water flux suddenly drops at the 4% of silica. At 5% silica, the flux rate keeps on dropping and remains constant at 6% of silica. Thus, based on this trend, the optimum flux rate can be considered at membrane which contains 3% of silica.

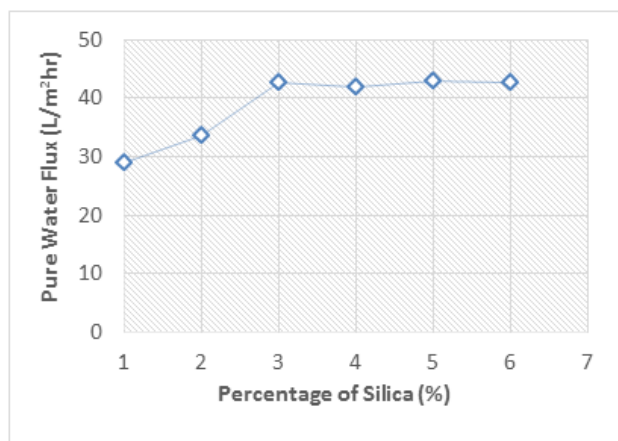


Figure 1 Pure water flux for different percentage of silica.

Figure 2 describes the effect of different percentage of silica in terms of percentage of salt rejection. The results indicated that at the first 3% of silica, the salt rejection were slightly similar approximately 75.33 % rejection. Then, the salt rejection shows gradually decreased from 75.35 % to 71.18 % with addition of 4% to 6% silica. According to the results, the 3% silica shows the highest NaCl rejection of 75.35% due to good hydrophilic properties.

The high hydrophilicity membrane may reduce interaction between hydrophobic contaminants and the membrane surface; hence, the rejection properties were improved effectively [8]. This is clearly show by the rejection properties when silica is added as additive in membrane fabrication. However, with more addition of silica result to low NaCl rejection. This may be due to crystalline effect of the excess silica that has low compatibility when mixed with polymer [9]. Therefore, the small particles can be a small defect and this surely will affect the rejection value.

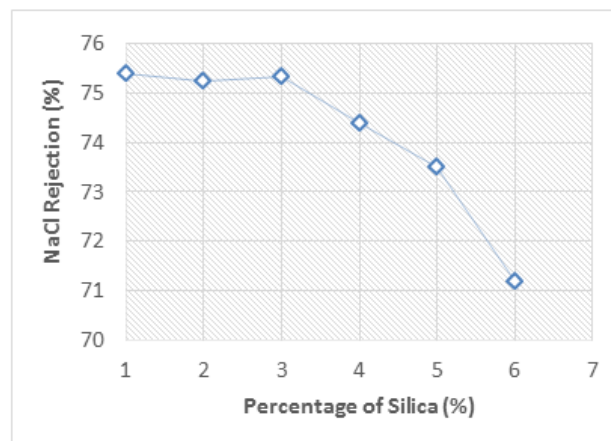


Figure 2 Salt rejection for different percentage of silica

3.2 TGA Analyses

In this research, TGA was used to analyse the thermal behaviour of sugarcane bagasse. TGA result of sugarcane bagasse is as shown in Figure 3. Base on the TGA curves, indicates that the sugarcane bagasse have about 539.93 °C maximum heating temperature but the inflection point for the sample was 315.70°C with residue of 6.0056 mg. Initial weight of sample was 10 mg, and the weight loss was about 39.944%. Results of TGA in Figure 3, show that the thermal curve is displayed from left to right which is in the descending curve indicates that weight loss of the sugarcane bagasse silica occurred. Based on the curve, several diffusion controlled reactions were occurs and the silica was also observe to be melting during the TGA process. It is quite interesting to note that, the inflection point for bagasse at 315 °C have similar trend with study of Arup Mandal and Debabrata Chakrabarty (2011) at temperature 311°C. They also indicate that the untreated bagasse were undergo degradation at temperature of 273 °C and the rate of degradation reaching its peak at 363°C [1].

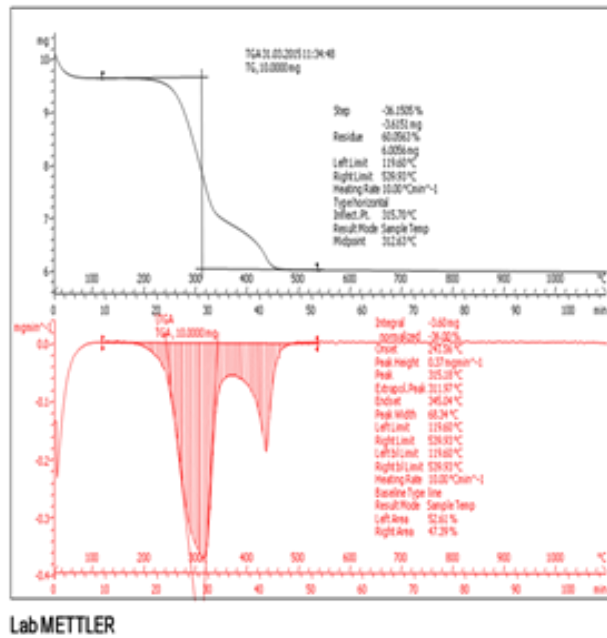


Figure 3 TGA for sugarcane bagasse

3.2 Microscopic Analyses

The image of silica from waste sugarcane bagasse analyses using FESEM was shown in Figure 4. The surface of silica showed rough surface which value to the high surface area lead to easy reaction with solution. EDX profile of silica from sugarcane bagasse contained the elements of Silica, Oxygen, Carbon and Magnesium, Potassium and Gold as shown in Figure 5. Both Si and O peaks resemble as percentage of silica which is 20.28%. The dominant signals originate from gold (Au) (65.0%) is due to gold coating.

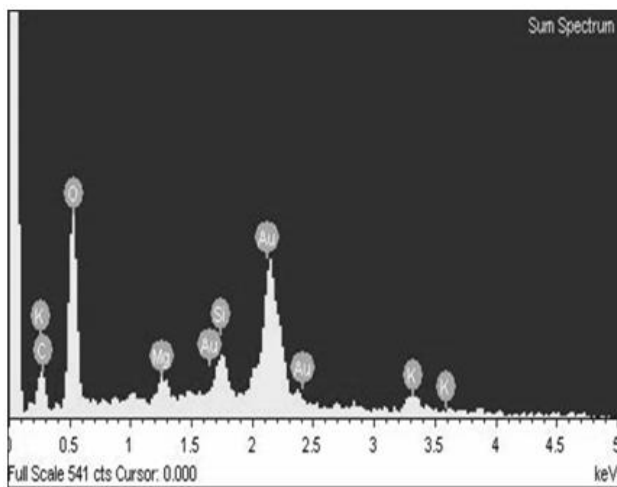


Figure 4 FESEM image of silica from sugarcane bagasse.

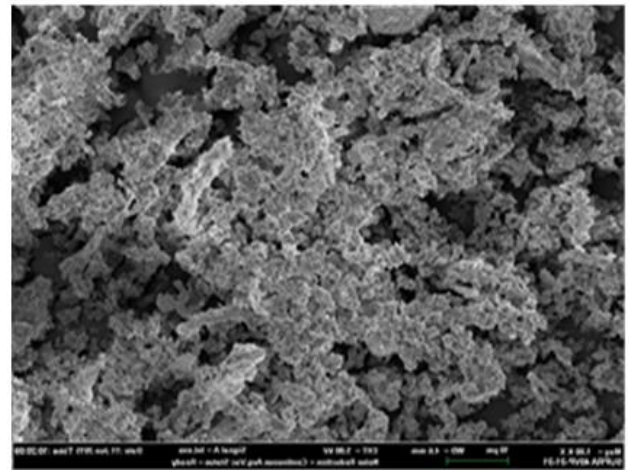


Figure 5 EDX profile of sugarcane bagasse

3.3 Fourier Transforms Infrared (FTIR) Spectroscopy

The surface functional groups of waste sugarcane bagasse were determined. Table 1 shows the bond and functional groups that associated with certain specific bands (cm^{-1}) for silica. The bands presence in Figure 6 was range from 404-3929 cm^{-1} . Sugarcane bagasse show bands for carboxylate (COO^-) and hydroxyl (OH^-) groups. The availability of negatively charged groups at the surface of sugarcane bagasse shows potential to be used as additive in membrane fabrication. Addition of this additive will improve hydrophilicity of the membrane pores. The band of 3221-3500 cm^{-1} are assigned to O-H stretching associated with alcohol group. The bands 2100-2245 cm^{-1} represent $\text{C}\equiv\text{C}$ stretch in alkynes group. The peak of 1475 cm^{-1} indicate as C-H bend in alkanes. The bands from 1050 cm^{-1} to 1250 cm^{-1} represent carboxylic acids group. The bands of 800-891 cm^{-1} specify for C-N stretching associated with aliphatic amines. The appearance peak at 515-690 cm^{-1} is a characteristic of C-Br in alkyl halides. Previous study by Livia *et al.* 2011, indicated peak 1235 cm^{-1} as the carboxylic acids in plane OH bending and C-O stretching [10].

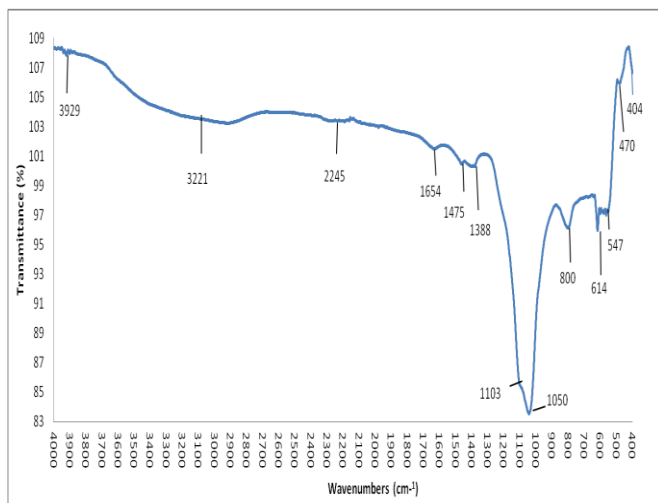


Figure 6 Bands for sugarcane bagasse

Table 1 Functional group of sugarcane bagasse

Bands (cm ⁻¹)	Functional groups
515-690	C- Br in alkyl halides
800-891	C-N stretch in aliphatic amines
1050-1250	C-O stretch in alcohol or Carboxylic acids
1475-1450	C-H bend in alkanes
2100-2245	-C≡C- stretch in alkynes
3221-3500	O-H stretch in alcohol

4.0 CONCLUSION

In conclusion, the thermal, microscopic and spectroscopic analyses on silica from sugarcane bagasse were successfully investigated. The shape of the TGA curve indicates that from 10 mg of sample weight, it decreased to about 6.0056 mg (39.944%.% of residue) which have right limit temperature of 539.93 °C and inflected point of 315.70°C. Microscopic analyses using FESEM shows that sugarcane bagasse contained approximately 20.28% silica and oxygen. The Fourier transform infrared spectrometric analyses indicate that the addition of sugarcane bagasse will improve hydrophilicity of the membrane pores due to availability of negatively charged groups such as carboxylate (COO⁻) and hydroxyl (OH⁻) groups. From the results and analyses, the membrane performance with addition of 3% silica give the best salt rejection of 75.35 % and 42.65 L/m²hr water flux.

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