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Foreword

It is indeed a proud moment for the University Publication Centre (UPENA) of UiTM Pulau Pinang for having realised the publication of the sixth volume of the Esteem Academic Journal UiTM Pulau Pinang. In fact, it is the undivided support and all-round commitment from all those who were directly and indirectly involved in this project that was the pivotal factor for this success.

On behalf of UPENA UiTMPP, I would like to, first and foremost, express my sincerest gratitude to Associate Professor Mohd Zaki Abdullah, Director of UiTM Pulau Pinang, Associate Professor Dr Mohamad Abdullah Hemdi, Deputy Director of Academic Affairs and Associate Professor Ir. Damanhuri Jamalludin, Deputy Director of Research, Industry Linkages, Development & Maintenance for their unwavering support and being such a driving force towards this successful endeavour.

Not to be forgotten also is the service rendered by the distinguished panel of external reviewers for their constructive comments and criticisms in ensuring that the papers published in this issue would be of the highest quality. Similarly, the panel of language editors who had worked tirelessly towards ensuring that the papers published were linguistically perfect. To both these groups, UPENA is in awe of your efforts and salutes you!

UPENA is also impressed with the nature of papers submitted for publication. While this issue comprises all engineering based articles, it covers a wide array of sub-engineering disciplines. Kudos to these writers! UPENA sincerely appreciates their efforts and hopes more of our staff will follow in their footsteps.

Finally, research and publication are integral parts of an academic's life at any institution. Apart from being an institutional requirement, it is also essential for our own continuous self-development and knowledge expansion. To this effect, UPENA hopes to play a significant role by providing the platform upon which our staff can realise their dream. So, it is our hope at UPENA UiTMPP that lecturers will take up the challenge and start to publish more vigorously from now on.

Rasaya Marimuthu
Chief Editor
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(Engineering)

Feasibility Study of Pineapple (*Ananas cosomus*) Leaf Fibres (PALFs) for Cellulosic Microfiltration Membrane

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ABSTRACT

The study was conducted to investigate the possibility of using pineapple leaf fibres (PALFs) as an alternative source of cellulose polymer for membrane fabrication. The membranes were fabricated from dope solution containing cellulose/ N-methylmorpholine-N-oxide (NMMO)/polyethylene glycol (PEG 400)/ N-propylgallate with a mass ratio of 8/ 88/ 3.5/ 0.5 by using immersion precipitation method. The permeation characteristics, structure and morphology of the membranes were investigated upon substituting the former cellulose source by the means of microfiltration rate (MFR), rejection rate (RR), Fourier Transform Infrared (FTIR) spectroscopy and Scanning

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Electron Microscopy (SEM), respectively. It was found that membranes derived from PALFs exhibited higher RR of 86.51%, but slightly lower MFR (i.e. 91.79 $mLh^{-1}m^{-2}mmHg^{-1}$) than the former membrane type under an applied pressure of 10 psi. Both of the membrane types (i.e. PALFs and hardwood) possess analogous chemical structures which have been confirmed by the existence of similar functional groups detection. Meanwhile, SEM analysis revealed that the source of cellulose had brought profound effect on the structural and morphology of resultant membranes. In general, matte and porous surfaces in a sponge-like configuration and uniform granular microporous structure were observed throughout the thickness of both membranes. The average pore size of membrane derived from PALFs exhibited to be smaller than the hardwood which in turn affected the MFR and RR performances. Hence, based on the overall results, it can be concluded that PALFs as a non-wood plant can be utilized as one of the alternative sources for cellulose polymer in preparing the cellulosic microfiltration membranes.

Keywords: *membranes, cellulosic, pineapple leaf fibres (PALFs), hardwood, immersion precipitation*

Introduction

Membrane separation technology has gained great importance in the last three decades, competing with long established technologies for water desalination, food processing and medical application (Zhang et al., 2001; Abe and Mochizuki, 2003; Rezvanpour et al., 2008; Lowe and Hossain, 2008). In chemical industries, microporous membranes play an important role in the recovery of valuable products as well as treating effluents while minimizing environmental problems. However, most of the membrane materials are prepared from petrochemical, which contribute to pollution when nonbiodegradable polymer is being abandoned after being used.

Recently, renewable resources have attracted a lot of attention due to its great importance for a sustainable development and also environmental conservation. It is known that cellulose possesses a remarkable hydrophilic property which enables it to be biodegradable. These have been widely applied to the various applications in membrane separation technology such as microfiltration, ultrafiltration and dialysis (Abe and Mochizuki, 2003; Li et al., 2006; Radiman et al., 2008; Rezvanpour et al., 2008). However, until present, cellulose and its derivatives are derived from hardwood plants. The major drawbacks of this source are the production cost and process which are too costly, as well as the utilization of toxic and environmental-unfriendly intermediates

during the pulping process. The needs to investigate on alternative and cost-effective membrane materials are very crucial in overcoming these concerns. Therefore, non-wood plants have been seen as the most promising source to substitute it.

Non-wood sources are commonly associated with lignocellulosic agricultural byproducts and/or wastes. These sources are renewable, copious and cheap from the annual cultivation activities (Xiao et al., 2001; Reddy and Yang, 2005). However, the literature on utilization of cellulose and/ or cellulose derivatives derived from non-wood plant as polymeric material for membrane applications is still scanty (Basta et al., 2003; Radiman et al., 2008). Until present, no research has been published on the utilization of pineapple (*Ananas cosomus*) leaf fibres (PALFs) as one of alternative sources of cellulose polymer for membrane fabrication. Thus, the objective of this research is to study the possibility of using PALFs as an alternative source of cellulose in preparing cellulosic microfiltration membranes.

Experimental

Materials

The polymer used in this work was divided into two types in accordance to its' sources as depicts in Table 1. Cellulose derived from hardwood plant was used as referential material. The PALFs were extracted mechanically from raw pineapple leaves and preceded for further treatment with 5 wt% NaOH solution for an hour at 30°C. The polymers were dried at 80°C for 24 hours until constant weight prior to be used.

Table 1: Types of Cellulose Polymer and Its' Properties

Type	Source	Cellulose composition (%)	Remarks
A	Non-wood (PALFs)	± 94.58	Results obtained from the solubility extraction analysis
B	Hardwood	± 98	Purchased from Acrōs Organics

N-methylmorpholine-*N*-oxide monohydrate (NMMO·H_2O), in white crystal form with a melting point approximately 72°C and 13.3 wt% of water, was selected as solvent. *N*-propylgallate was used as an antioxidant for stabilizing the degree of polymerization of cellulose during the

dissolution process (Zhang et al., 2001). Polyethylene glycol with an average molecular weight of 400 Dalton (PEG 400) was used as the pore former. Dextran T-2000 was purchased from Sigma for permeation tests. All chemical reagents used in this work were analytical grades and purchased from Acrōs Organics.

Preparation of Cellulose Membrane

Initially, 88 grams of NMMO·H_2O was heated at 90°C until completely dissolved in round flask. PEG 400 and *N*-propylgallate were then added and dissolved by stirring. After the mixture became transparent, 8 g of dried cellulose was added into the solution and stirred vigorously under nitrogen for 6 hours until homogeneous dope solution was obtained. Finally, the dope solution was poured into clean bottles and degassed by ultrasonic bath at 90°C to eliminate air bubble formation before proceeding to casting process.

The dope solution was then casted on a glass plate using a casting knife with a clearance of 200 μm to form a solution film. Then, the casted solution was immersed gently into coagulation bath of water at 25°C for 2 hours without any agitation. The membrane was gradually formed during the immersion precipitation process. After coagulation, the membrane was taken out of the coagulant and washed with distilled water for 24 hours to leach out the residual solvent. The resultant membrane was soaked in 50 wt% glycerin aqueous solution and finally air-dried at ambient temperature.

Permeability Characteristics Measurement

Membrane performances were investigated with respect to the microfiltration rate (MFR) for pure water and the rejection rate (RR) for dextran. The permeability characteristic measurement involved the use of dead-end permeation cell in which the membrane was placed on a sintered metal plate at pressure of 10 psi. Circular membrane discs with an effective permeation area of 18.1 cm^2 were used. Experiments were carried out at ambient temperature. The microfiltration rate (MFR) through the wet membrane was calculated by using the following equation:

$$MFR = \frac{V}{A \times t \times P} \quad (1)$$

where MFR is the permeability which expressed as $mLh^{-1}m^{-2}mmHg^{-1}$; V is the filtrate volume (mL); A is the membrane effective surface areas (m^2); t is the time taken (h) and P is the operation pressure (mmHg).

The selectivity of membrane performance was determined by the rejection rate (RR), which is calculated using the following equation:

$$RR = \left(1 - \frac{C_p}{C_f}\right) \times 100\% \quad (2)$$

where C_p and C_f are the dextran's concentration in permeate and feed, respectively.

Fourier Transform Infrared (FTIR) Spectroscopic Analysis

FTIR is a very useful tool to detect the existence of the functional groups in a membrane. Measurements were carried out using Nicolet™ 6700 FTIR spectrometer. The spectra were measured with an average of 64 scans and a resolution of 4 cm^{-1}. Then, the spectra were analyzed in transmittance mode in a wave number ranging from 4000-600 cm^{-1} by using commercialized available software.

Scanning Electron Microscopy (SEM)

SEM (Carl Zeiss–FESEM LEO SUPRA 50VP) was used to investigate the structure and morphologies of the membranes. The membranes were mounted on the sample studs using double-sided adhesive tape. The sample holder was then placed and evacuated in a sputter-coater with gold at a working voltage of 20 kV. The purpose of membrane coating with a thin layer of gold is to facilitate the transport of electrons from the electron beam that were not reflected or transformed to secondary electrons.

Results and Discussion

Permeability Characteristics

Microfiltration rate (MFR) and rejection rate (RR) have been identified as key factors in determining membranes performances. It was found that at the same cellulose concentration, the MFR of cellulose membrane

derived from PALFs was much lower than hardwood. The MFR values of cellulose membranes derived from PALFs and hardwood were at 91.79 ± 5.42 $mLh^{-1}m^{-2}mmHg^{-1}$ and 103.68 ± 4.28 $mLh^{-1}m^{-2}mmHg^{-1}$, respectively. This behavior can be attributed by the structure of respective membranes types. Whereas, it is known that high surface porosity of resultant membranes could lead to the enhancement of permeation flux (Radiman et al., 2008). This finding was corroborated by the SEM results, whereby, the cellulose membranes derived from PALFs possessed lesser surface porosity than the hardwood.

However, it was also noted that cellulose membranes derived from PALFs possessed higher RR than hardwood. The RR values for cellulose membranes derived from PALFs and hardwood were at 86.51 ± 0.73% and 73.83 ± 2.11%, respectively. The determination of RR is a key factor in permselectivity aspect. The difference of RR between these two sources is approximately 12.68%. Hence, it can be deduced that the cellulose membranes derived from PALFs showed better performance than the hardwood for both aforementioned factors due to significant differences in RR compared to MFR. From the above discussions, it is clear that source of cellulose has a profound effect on MFR and RR properties of cellulose membranes.

FTIR Analysis

Figure 1 shows the FTIR spectra of cellulose membranes for each respective type (i.e. A: non-wood; B: hardwood). In general, both spectrums depict similar existence of functional groups except for the strong transmittance at wave number of 1732 cm^{-1} for Spectrum A. This feature was attributed by aromatic C=C due to the presence of 4.32% lignin in the extracted PALFs. Table 2 summarizes the significant transmittance bands assigned to corresponding functional groups presence for both membranes. These results are in harmony with those reported by Moran et al. (2008), Ruan et al. (2004) and Carrillo et al. (2004) for cellulose films and membranes.

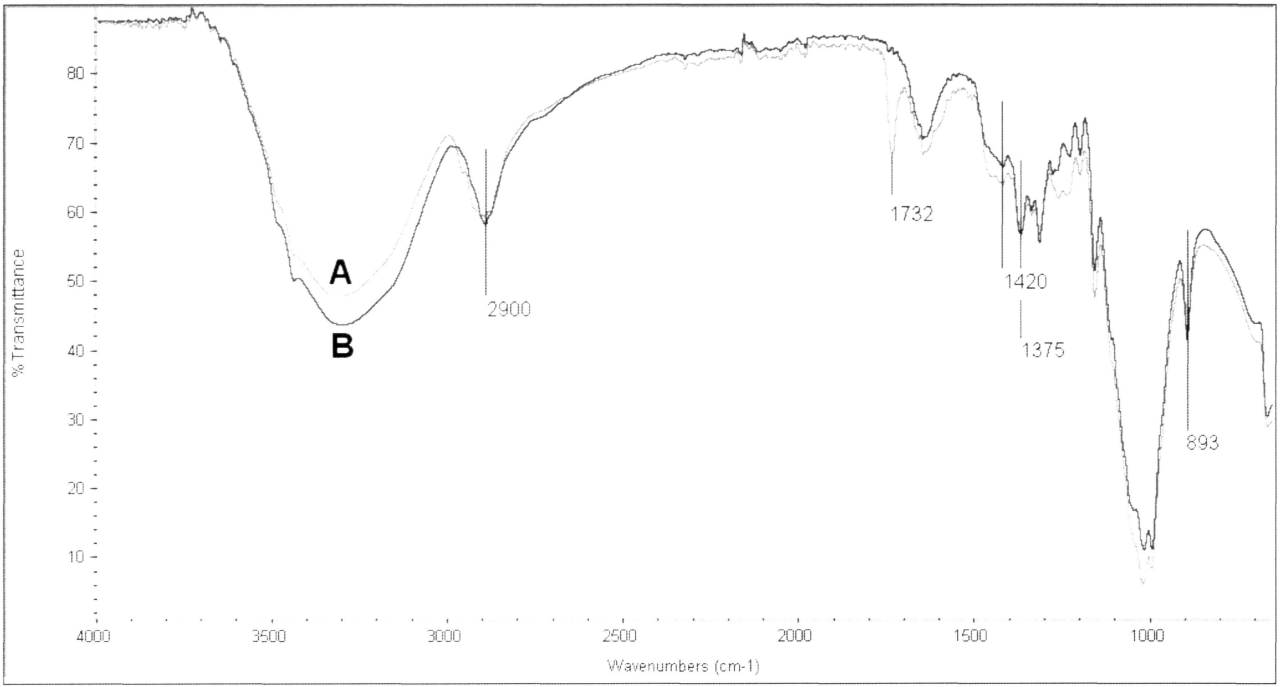

Figure 1: FTIR Spectra of Optimized Cellulose Microfiltration Membranes Derived from (A) Non-Wood Plant, i.e PALFs and (B) Hardwood Plant at Cellulose Concentration of 8 wt%

Table 2: Transmittance Band Assignments for Cellulose Membranes

Wave Number (cm^{-1})	Assignment	Component
3000 - 3500	-OH stretching intramolecular hydrogen bonds	Cellulose
2900	CH stretching	Cellulose
1732	Aromatic C=C skeletal vibration	Lignin
1640 - 1650	OH of water absorbed from cellulose	Cellulose
1420	CH_2 symmetric bending	Cellulose
1350 - 1380	CH bending	Cellulose
1300 - 1315	CH_2 wagging	Cellulose
1150 - 1160	C-O-C asymmetric streching	Cellulose
1010 - 1020	C-O streching	Cellulose
893 - 895	Group C_1 frequency	Cellulose

Scanning Electron Microscopy

Scanning electron microscopy was carried out in order to elucidate the structural and morphological changes of membranes derived from different types of cellulose sources (i.e. non-wood and hardwood plant). The surface morphologies for both types of membranes were revealed by SEM as depicted in Figure 2. Matte and porous surfaces in sponge-like configuration were exhibited at the surface of both membranes. The lighter and darker areas signify the solid portions and pores or voids of the membranes, respectively.

In view of these results, the whole coagulation process can be described as follows: When the dope solution is in contacts with the coagulant, a counter-diffusion and chemical neutralization process occurs between solvent in the dope solution and nonsolvent in the coagulant. The removal of solvent from dope solution and penetration of nonsolvent into the dope solution have resulted in cellulose dissolubility which leads to the formation of fluid gel that gradually being converted into solid state after precipitation and regeneration. Therefore, the coagulation mechanism contributes to phase separation, culminates in a cellulose rich phase and cellulose-poor phase. The weave penetration of nonsolvent in the cellulose rich phase contributes to the formation of mesh structure in sponge-like configuration. These findings are in accordance to Zhang et al. (2001; 2005) and Ruan et al. (2004).

By referring to Figure 2 (B, D), it can be seen that the average pore size distributions of membrane derived from non-wood plant was smaller

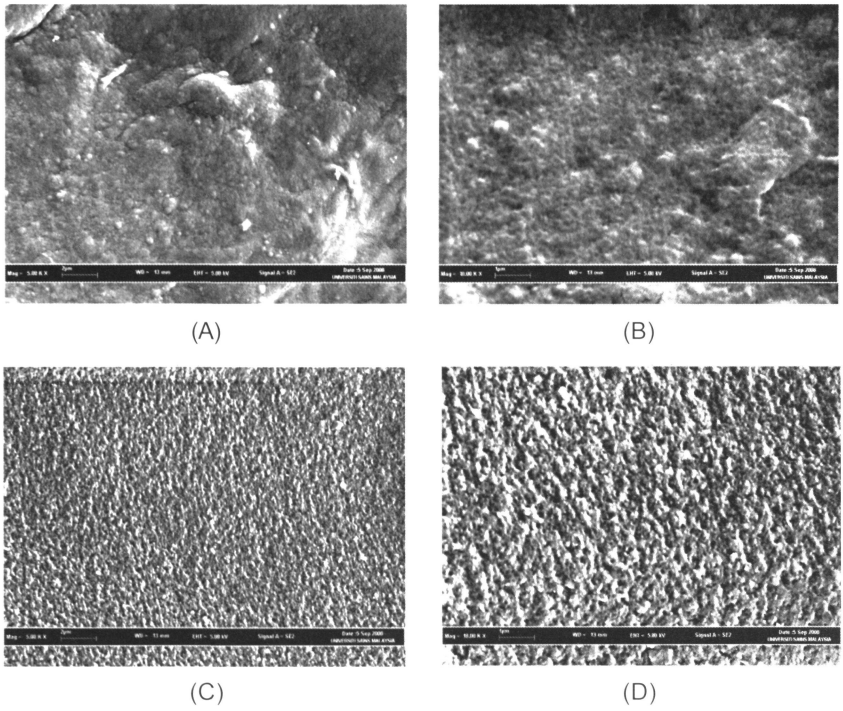

Figure 2: SEM Surface Micrographs of Optimized Cellulose Microfiltration Membranes Derived from Non-Wood Plant, i.e. PALFs (A, B) and Optimized Cellulose Microfiltration Membranes Derived from Hardwood Plant (C, D) having Cellulose Concentration of 8 wt%
(Magnification power = 5 K x (A, C) and 10 K x (B, D))

than the hardwood plant. The difference in the average pore size had led to slight reduction in MFR but increased in RR values as has been discussed previously. Therefore, it can be concluded that type of cellulose sources, can greatly affect the membrane structure and morphology.

Conclusion

This study has demonstrated that PALFs can be utilized as an alternative cellulose polymer in preparing cellulosic microfiltration membranes. The dextran's RR was 86.51% and 73.83% for membranes derived from PALFs and hardwood, respectively. Both of the membranes possess analogous chemical structures which have been confirmed by the

existence of similar functional group detections. The SEM analysis elucidated that the source of cellulose has profound effect on the structure and morphology of resultant membranes. In general, matte and porous surfaces in sponge-like configurations are observed throughout the surfaces of both membranes. The average pore size of membrane derived from PALFs is exhibited to be smaller than hardwood. These findings signify the potentiality in substituting hardwood to non-wood plant as the prominent source of cellulose polymer in near future.

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