

# POLYSULFONE MEMBRANE TESTS FOR NUTRIENTS RECLAMATION OF KENAF RETTED WASTEWATER

Nabilah Huda A. H.<sup>a</sup>, Ramlah M. T.<sup>a</sup>, Aida Isma M. I.<sup>b</sup>, Siti Aisyah G.<sup>a</sup>, Nor Munirah A.<sup>a</sup>, Hasfalina Che Man<sup>c</sup>

<sup>a</sup>Faculty of Civil Engineering, Universiti Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia

<sup>b</sup>School of Engineering, Segi University, Jalan Teknologi, Kota Damansara, 47810 Petaling Jaya, Selangor, Malaysia

<sup>c</sup>Department of Biological & Agricultural Engineering, Faculty of Engineering, 43400 UPM, Selangor, Malaysia

## Article history

Received

15 July 2015

Received in revised form

2 August 2015

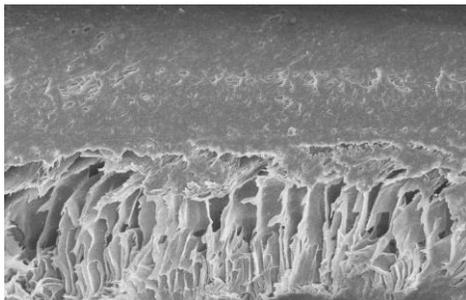
Accepted

26 August 2015

\*Corresponding author

ramla160@salam.uitm.edu.my

## Graphical abstract



## Abstract

Water retting is the main challenge faced during the processing of bast kenaf plants. This conventional method has been reported to generate much water pollution. A lab-scale experiment was performed using two different polysulfone membranes to investigate the effect of additive towards the permeability performance and nutrients reclamation efficiency of kenaf retted wastewater. From the experiment, it may be concluded that polysulfone membrane with 10wt.% of polyvinyl-pyrrolidone concentration can be used effectively to reclaimed nutrients. Tests with polysulfone membrane without additive achieved more than 75% and a polysulfone membrane with 10% additive practically reclaimed more than 85% of nutrients from kenaf retted wastewater. Nutrients analysis indicated that total nitrogen, total phosphorus and potassium could be reclaimed and can be reuse as liquid fertilizer.

**Keywords:** Kenaf, membrane, nutrient, reclamation, retted wastewater

© 2016 Penerbit UTM Press. All rights reserved

## 1.0 INTRODUCTION

Kenaf planting has been increased around the world due to its high biomass yield and the raised fiber content [1]. Kenaf (*Hibiscus cannabinus* L.) has been identified as a new commodity plant in Malaysia. The products that are manufactured from the kenaf fiber served as bio-composite materials such as ropes, nets, brushes, mats, carpets, paper, board, textiles products,

as well as automotive products. However, the rapid development of kenaf industry has caused environmental issue. The traditional water retting method for kenaf harvesting is tedious, time consuming, costly and environmentally sensitive. Apart from producing large amount of water, high levels of nutrients and non-biodegradable substances were discharge into water body triggering eutrophication in aquatic ecosystem. Water retting is a wet process by

which the bundles of cells in the outer layers of the stalk are separated from the non-fibrous matter [2]. This conventional method has been reported to generate much water pollution [3].

Retting is the main challenge faced during the processing of bast kenaf plants. Traditionally, it was carried out by immersing the stems in an open water such as river and lake. The predominant task in the pretreatment of kenaf fiber is to remove gums including pectin, hemicellulose, lignin and other impurities without damaging the cellulose fibers [4]. Microbes retting were used in the process, which require 14 to 28 days to acclimatized and degrade the pectic materials [5]. A quality of fibre is largely determined by the retting condition and duration [6]. The excessive amount of nutrient in the retted wastewater could influenced the time taken of retting process and adversely affect the quality of kenaf stalk [7].

Due to long period of retting process and large water consumption, many efforts have been focused on studying the [8]. Membrane technology offers an effective solution to the problems of treating wastewater. This paper attempts to fill in some gaps in this respect, by providing experimental data on performance of polysulfone membrane in treating and reclaiming the nutrients from the polluted kenaf ratted wastewater. Membrane separation processes with the advantages of low cost, no phase transition, high efficiency, energy saving and capability of reducing contaminant have been widely applied [9].

In this work, Polyvinyl-pyrrolidone (PVP) was used as an additive to prepared Polysulfone (PSF) membrane. The uses of additive in membrane fabrication provides a lot of advantages, where additive helps in produces more porous membrane structure, larger pore sizes, increase hydrophilicity on hydrophobic polymer and increase the permeability performance. In this study, the effects of additive on membrane morphology and permeability performance were investigated. Meanwhile, membrane ability towards reclamation of nutrients was evaluated.

## 2.0 EXPERIMENTAL

### 2.1 Materials and Wastewater

The contents of membrane dope solution; polysulfone (PS,  $M_w \sim 22,000$  Da), Polyvinyl-pyrrolidone (PVP,  $M_w \sim 58,000$  Da) and N,N-Dimethylacetamide (DMAC) were purchased from ACROS Organic. All other used reagents were analytically grades. Approximately 1000 g of freshly harvested kenaf stalks was soaked in 2000 mL of river water at the planting area for 14 days. The samples were collected directly from the retted container as the retting process completed.

### 2.2 Membrane Fabrication

For PS membrane preparation, firstly PSF (15 wt.%) was added to DMAC solvent (85wt.%) and was stirred until it

was completely dissolved. The solution was stirred at 60°C for more than 8 hours in order to form a homogeneous dope solution. Before casting, the dope solutions were kept in a bottle at room temperature for 24 h until no bubbles were observed. Then, the casting solutions of PS membrane were spread with a casting knife gap setting of 150  $\mu\text{m}$  on a glass plate. The dope solution was then formed a thin layer sheet. Then, the glass plates were immediately immersed in a coagulation water bath ( $\text{pH} = 6.8$ ,  $T = 25^\circ\text{C}$ ) to obtain polymer precipitation. The changes of cast film from transparent liquid to a white flat sheet can be clearly seen. After 30 min, the membrane was removed from the water bath and washed thoroughly with deionized water and stored in deionized water including 100  $\text{mgL}^{-1}$  of glycerol at 4°C for two weeks. The membrane preparation was repeated by adding additives PVP (10wt%). Membranes with and without additives produced were coded with P0 (without additives) and P10 (with additives), respectively.

### 2.3 Membrane Morphology

The top surface morphology of membranes was observed using field emission scanning electron microscope (FESEM) technique under Carl Zeiss SMT – Nanotechnology System Division (SUPRATM 40VP) at standard high vacuum conditions. The outer surfaces of membrane samples were coated with gold.

### 2.4 Membrane Performances and Nutrients Reclamation

Membrane performances were characterized based on permeability against pressure and time, and also nutrients reclamation. The permeability test against pressure and time were conducted in sequence. During the permeability characterization against pressure, the membrane was initially filtrated with ultrapure water (UPW) at 1 bar for half an hour until the water permeability was stable before it being tests. Then, the permeability of ultrapure was obtained from the volume of the permeated water under a pressure 1 bar and 5 bar with the constant temperature applied. After about 1 hour permeability test, by changing the feed water from ultrapure water (UPW) to tap water (TW) and by using the same membrane, the permeability test against time was continued under the pressure of 5 bars and each value recorded was based on 10 minute of permeate collected. Tested were continued for another 3 hours.

Meanwhile, for nutrients reclamation, kenaf retted wastewater was used as feed solutions for reclamation of potassium (K) in this study. Then, the feed solutions are allowed to pass through the membrane at Five (5) bars and permeated water of the PFS membrane experiments were collected for total nitrogen (TN), total phosphorus (TP), and potassium (K) analysis. Each of samples was tested for three (3) times to get accurate results. The nutrients reclamation,  $R_n$  could be calculated as,

$$R_n = (1 - C_a/C_b) \times 100\% \tag{1}$$

where,  $C_a$  (mg/l) is the concentration of the nutrient in the wastewater after the membrane separation and  $C_b$  is the concentration of the nutrient in the wastewater before the membrane separation. Meanwhile, the water permeability,  $J_w$  was obtained from the volume of permeated water and was determined by using the following equation:

$$J_w = V/A\Delta t \tag{2}$$

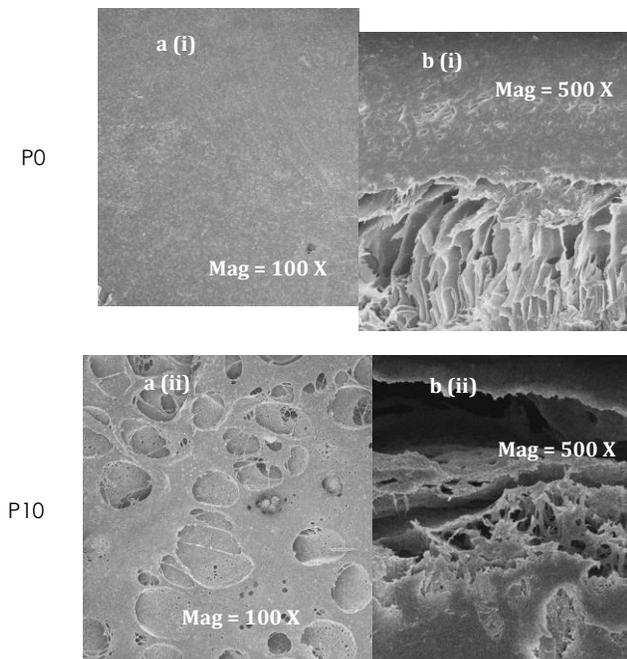
where,  $V$  (L) is volume of water permeated,  $A$  ( $m^2$ ) is the effective membrane area, and  $\Delta t$  (h) is the operation time.

### 3.0 RESULTS AND DISCUSSION

#### 3.1 Effects of Additive on Membrane Morphology

The surface layer and cross section morphologies for both fabricated membranes were captured in Figure 1. The figure shown, the membrane with 10wt.% PVP concentration has the biggest average pore size compared to the membrane without PVP. This proved that from previous study, PVP is usually used as a pore forming agent (Zhang *et al.*, 2011). This result agrees well with Wang, Li, & Teo, (1999), where they found that large molecular weight Polyethylene-glycol (PEG) or Polyvinyl-pyrrolidone (PVP) additives tends to form large pore size and thicker skin layer containing bigger pore. Both membranes produce a lot of pore with average same size and it separated well in membrane surface.

Memb rane	Morphology	
	Surface Layer	Cross Section

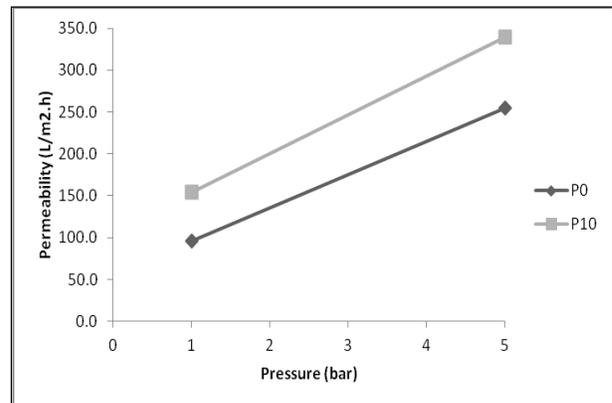


**Figure 1** SEM image of the (i) surface layer and (ii) cross section for fabricated PSF membranes

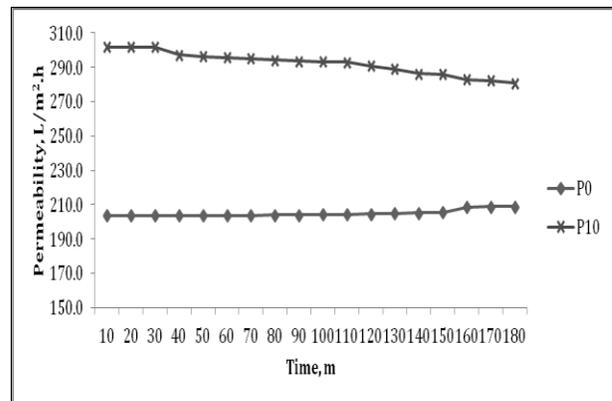
#### 3.2 Effects of Additive on Membrane Performance

Figure 2 illustrated the effect of PVP additives on the membrane permeability against pressure. Figure shown, the permeability for both fabricated membrane P0 and P10 were increased when pressure increased. According to Ramlah, (2006), state that increasing pressure will increase the volume of solute passing the pore on the skin but does not increase the pore size of the skin layer.

Based on the experimental results, the permeability of P0 was increased from 95.9 L/m<sup>2</sup>.h to 254.6 L/m<sup>2</sup>.h, while, the permeability of P10 was increased from 153.8 L/m<sup>2</sup>.h to 339.5 L/m<sup>2</sup>.h at 10wt.% PVP concentration. The addition of PVP additive into the dope solution increased the water permeability. Additives can lead to better permeability in membrane performance (Aminudin *et al.*, 2013).



**Figure 2** Permeability against pressure



**Figure 3** Permeability against time

Meanwhile, the effect of PVP additives on the membrane permeability against time is plotted in Figure 3. It can be clearly seen that, even membrane P10 obtain the highest permeability, however it permeability performance was slight declined from 291.0 L/m<sup>2</sup>.h at minute of 10 to 280.9 L/m<sup>2</sup>.h at minute of 180. This result occurred due to bigger pore size form on

the membrane surface caused the elements, which is available in tap water was trapped inside. In long working period, the permeability of the membrane can be decreased.

Referring to Figure 3, the permeability of P0 membrane, which is designed without additives was increased from 205.3 L/m<sup>2</sup>.h to 208.9 L/m<sup>2</sup>.h during minutes of 150 to minutes of 180. It is due to durability of this membrane against pressure during long period of time is relatively low. During experiment, some cracks were observed on membrane which caused leaking. Even though, the permeability changes were not so obvious, but, when it is apply on wastewater, the reduction of treatment percentage rate will occurred. Moreover, P0 membrane is less suitable for wastewater treatment at high pressure in long working period. Therefore, additional of additive in dope composition is favorable to increase the membrane resistance against pressure and increase the filtration duration.

### 3.3 Effects of Additive on Nutrients Reclamation

The effect of PVP additive on nutrients reclamation was presented in Figure 4. Based on the bar chart, both fabricated membranes were successfully reclaimed the nutrient from kenaf retted wastewater. The nutrients reclamation obtained from this study are listed in Table 1.

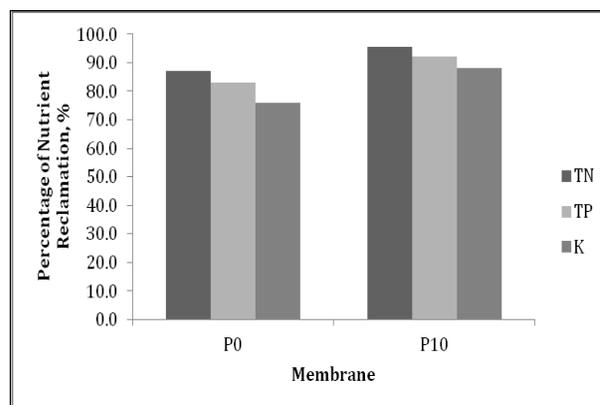


Figure 4 Percentage of nutrients reclamation

However, the P10 membrane with consist of PSF/DMAC/PVP at 10wt.% of additive resulted the highest nutrients reclamation at 95.6% (TN), 92.1% (TP), and 88.1% (K). This shows that, additional of additive not only improved the membrane permeability but also nutrients reclamation ability. According to Ismail & Hassan, (2007), the additive concentration in the polymer solution has a significant influence on membranes separation performance and structural properties.

Table 1 Nutrient reclamations obtained using fabricated PSF membranes

Nutrient	Concentration of Reclamation (mg/L)		Percentage Reclamation (%)	
	P0	P10	P0	P10
TN	3.2	1.1	87.2	95.6
TP	2.4	1.1	83.0	92.1
K	90.0	44.3	76.0	88.2

## 4.0 CONCLUSION

The fabricated PSF membranes in this study have been successfully used for nutrient reclamation from kenaf retted wastewater. Both membranes were capable to reclaim nutrients content for more than 75%. Results showed that the best membrane with 10wt.% PVP concentration was the effectively in reclamation the total nitrogen (TN), total phosphorus (TP), and potassium (K) by 95.6%, 92.1, and 88.2% respectively which is more higher compared to the P0 membrane. This proved that, additional of additive in membrane dope composition was helped in membrane performance. From this study, this treatment process can reduce the volume of the wastewater discharged by over half, besides, reclamation of nutrients content which can be reuse as liquid fertilizer for plant growth. Even though P10 membrane is shows the best performance in term of nutrients reclamation, but its working ability period is low. Therefore, further study on

membrane working performance period was needed toward increase the membrane working performance ability.

## Acknowledgement

The authors would like to express greatest appreciation to the SEGi University and the Faculty of Civil Engineering of Universiti Teknologi MARA for providing necessary facilities for this research. Not to forget, Mr. Hazri Othman for his assistance during laboratory work.

## References

- [1] Hossain, D., Musa, M. H., Talib, J., Jol, H. 2010. Effects of Nitrogen, Phosphorus and Potassium Levels on Kenaf (*Hibiscus cannabinus* L.) Growth and Photosynthesis under

- Nutrient Solution. *Journal of Agricultural Science*. 2(2): 49-57.
- [2] Zhang, T. 2003. Improvement of Kenaf Yarn for Apparel Application. Master of Science in Human Ecology thesis. Beijing University of Chemical Technology, Beijing, China. 4.
- [3] Lu, S. S., Huang, X. B., Chen, J. H. 1999. Study On Anaerobic Microbe Retting Of Kenaf In China. *J.China Text. Univ*. 25(6): 107-110, 114.
- [4] Hongquin Yu and Chongwen Yu. 2010. Influence Of Various Retting Methods On Properties Of Kenaf Fiber. *Journal of Textile Institute*. 101(5): 452-456.
- [5] Zawani, Z., Chuah-Abdullah, L., Ahmadun, F.-R., & Abdan, K. 2013. Acclimatization Process of Microorganisms from Activated Sludge in Kenaf-Retting Wastewater. *Developments in Sustainable Chemical and Bioprocess Technology*. 59-64.
- [6] Paridah, M.T., Khalina A. 2009. Effects Of Soda Retting On The Tensile Strength Of Kenaf Bast Fibers. Project Kenaf EPU. 21.
- [7] Webber, C. L. I., & Bledsoe, V. K. 2002. Kenaf Yield Components and Plant Composition. In J. Janick & A. Whipkey (Eds.). *Trends In New Crops And New Uses*. 1st ed. Alexandria, VA: ASHS Press. 348-357.
- [8] S. H. Choi, J. W. Chung, R. D. Priestley, S.-Y. Kwak. 2012. Functionalization Of Poly- Sulfone Hollow Fiber Membranes With Amphiphilic B-Cyclodextrin And Their Applications For The Removal Of Endocrine Disrupting Plasticizer. *J. Membr. Sci.* 409-410, 75-81.
- [9] Aminudin, N. N., Basri, H., Harun, Z., Yunos, M. Z., & Sean, G. P. 2013. Comparative Study on Effect of PEG and PVP as Additives on Polysulfone. *Jurnal*. 4(64): 47-51.
- [10] Aroon, M. a., Ismail, A. F., Montazer-Rahmati, M. M., & Matsuura, T. 2010. Morphology And Permeation Properties Of Polysulfone Membranes For Gas Separation: Effects Of Non-Solvent Additives And Co-Solvent. *Separation and Purification Technology*. 72(2): 194-202.
- [11] Ismail, A. F., & Hassan, A. R. 2007. Effect Of Additive Contents On The Performances And Structural Properties Of Asymmetric Polyethersulfone (PES) Nanofiltration Membranes. *Separation and Purification Technology*. 55(1): 98-109.
- [12] Lin, S. H., & Lin, C. S. 1998. Reclamation of Wastewater Effluent From A Chemical Fiber Plant. *Desalination*. 120(3): 185-195.
- [13] Poff, N. L., & Richter, B. D. 2012. Water Resources and Sustainable Aquatic Ecosystems: A Vision for 2050. In W. M. Grayman, D. P. Loucks, & L. Saito (Eds.). Reston, Virginia: American Society of Civil Engineers. 175-186.
- [14] Ramlah, M. T. 2006. Development Of Hollow Fiber Ultrafiltration Membrane Reactor For Palm Oil Mill Effluent (POME) Membrane Reactor Tertiary Treatment. Universiti Teknologi Malaysia.
- [15] Walter, M. F. 2012. Toward a Sustainable Water Future: Visions for 2050. In W. M. Grayman, D. P. Loucks, & L. Saito (Eds.). *Toward a Sustainable Water Future: Visions for 2050*. Reston, Virginia: American Society of Civil Engineers. 147-156.
- [16] Wang, D., Li, K., & Teo, W. 1999. Preparation and Characterization Of Polyvinylidene Fluoride (PVDF) Hollow Fiber Membranes. *Journal of Membrane Science*. 163(2): 211-220.
- [17] Winarto, B. W., & Hartono, J. 1995. *Proses Pengolahan Kenaf Menjadi Serat* Malang, Indonesia. 126-137.
- [18] Zhang, Z., An, Q., Liu, T., Zhou, Y., Qian, J., & Gao, C. 2011. Fabrication Of Polysulfone Ultrafiltration Membranes Of A Density Gradient Cross Section With Good Anti-Pressure Stability And Relatively High Water Flux. *Desalination*. 269(1-3): 239-248.
- [19] Sim, C. H., Yusoff, M. K., Shutes, B., Ho, S. C., & Mansor, M. 2008. Nutrient Removal In A Pilot And Full Scale Constructed Wetland, Putrajaya City, Malaysia. *Journal of Environmental Management*. 88(2): 307-17.