

Effect of Powdered Activated Carbon Type on the Performance of an Adsorption-Microfiltration Submerged Hollow Fiber Membrane Hybrid System

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Abstract—This study highlights the importance of the type of powdered activated carbon (PAC) on the performance of an adsorption-MF hollow fiber membrane hybrid system. Submerged UF hollow fiber membrane with pore size of 0.4 μm was used along with the PAC in a single reaction tank to treat the wastewater. This procedure is different from the commonly adopted practice of having adsorption with PAC as a pretreatment step to membrane operation. This system is helpful in containing the PAC within one system, allows certain variation in feed characteristics and improves the efficiency of the hybrid system. The overall performance of the membrane hybrid system was evaluated based on TOC removal, UV_{254} absorbance, turbidity removal and flux development. It is important to select a right PAC type to be used in the hybrid system in order to minimize the PAC dose and to enhance the performance. The adsorption capacity of the activated carbon had a greater influence on the overall performance of the hybrid system than other physical properties of the PACs and must be given greater consideration when selecting the PAC for the membrane hybrid system. Scanning electron micrograph (SEM) analysis of the cake deposit over the membrane helps to support the reason for moderate retention of organics.

Key words: Microfiltration, Submerged Hollow Fiber Membrane, Hybrid System, Activated Carbon

INTRODUCTION

Due to rapid industrialization and urbanization, there has been an increased amount of pollution in the water environment that has greatly limited water resources. Better water management by reusing wastewater could be an ideal solution to water shortage and will play a significant role in achieving a sustainable development by reducing the demand for fresh water. With the growing need for water reuse, membrane separation is an emerging process that has recently gained greater attention compared to the conventional treatment because of its simplicity of operation, requirement of less space, ability to produce consistently high quality of treated water and to meet the stringent water quality standards. The use of low-pressure membrane processes such as ultrafiltration (UF) and microfiltration (MF) has received greater attention lately compared to reverse osmosis and nanofiltration, which demand a considerably high operational cost because of high-energy requirements. Moreover, recent advances in membrane technology have led to the reduction in the cost of the membrane, and studies indicate that the low pressure membrane process appears to be a cost effective option [Gere, 1997; Wiesner, 1994].

Although UF and MF can effectively remove particulates and pathogens present in wastewater, they are less effective due to their large pore size in the removal of organic matter that contributes to disinfection by-product (DBP) formation. Moreover, it is now evident that these organic materials are largely responsible for membrane fouling, which reduces the membrane performance capability and causes irrecoverable loss in permeate flux, thereby resulting in

more frequent replacement of the membrane [Crozes, 1993; Kaiya et al., 1996; Nystrom et al., 1996]. However, conventional treatment technologies like coagulation and adsorption have been combined with MF/UF membrane processes in an effort to remove organic material and improve membrane performance [Adham et al., 1991; Turcaud, 1990; Vickers et al., 1995; Jacangelo, 1995; Kim et al., 1996; Abdessemed et al., 2000; Jung and Kang, 2003]. The main idea in having a pretreatment operation before the membrane process is to make the organic material that would otherwise pass through the large pores of the membrane to somehow (form aggregates during coagulation or get lodged on the larger adsorbent surfaces) associate with the particulate phase and thereby be physically sieved out on the membrane surface. Evaluating the performance of the above-mentioned processes, powdered activated carbon (PAC)-membrane system looks more promising for the removal of organic compounds [Laine et al., 2000]. The PAC-MF system combines the organic adsorption capability of PAC and effective particle removal ability of MF membrane.

However, conflicting results have emerged over the years on the efficiency of the PAC-porous membrane systems with researchers reporting 5-90% organic removal [Anselme and Charles, 1990; Adham et al., 1991; Lin et al., 1999; Tomaszewska and Mozia, 2002]. These conflicting observations were attributed to the differences in water chemistry, type of PAC, type and concentration of organic material and membrane characteristics. Studies have been carried out to investigate the influence of membrane characteristics (type of membrane material, molecular weight cutoff (MWCO) value of membrane) [Laine et al., 1989; Crozes et al., 1993; Yiantsios and Karabelas, 2001] and the type and concentration of organic material [Lin et al., 1999, 2001; Kim et al., 1996; Jung and Kang, 2003]. However, the effect of PAC type on the performance of PAC-MF

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system still needs to be investigated in detail.

The objective of this study is to evaluate the performance of an adsorption-membrane hybrid system based on the type of adsorbent PAC. In order to achieve this goal, a systematic laboratory scale experimental analysis was carried out by first studying the adsorption capacity of three types of activated carbon, namely, wood based (WB), coal based (CB) and coconut based (HA) PACs followed by the investigation into their influence on the performance of the PAC-MF membrane hybrid system. The activated carbons chosen have different surface characteristics. In the hybrid MF membrane system, a hollow fiber membrane module was immersed in the influent tank containing the wastewater to be treated. One striking peculiarity of this study is that unlike in other studies where adsorption is generally carried out as a pretreatment, the PAC is directly added to the wastewater in the influent tank. Adsorption and membrane separation take place simultaneously in one single influent tank. Some of the main advantages of this system are (1) simple operation using a single step process, (2) better physical removal of organic matter, (3) absolute containment of PAC within the reaction tank, (4) the presence of high concentrations of PAC in the tank allows any slight variation in the feed water quality, and (5) minimized membrane fouling.

EXPERIMENTAL

1. Wastewater Characteristics

The wastewater used for the study was secondary treated wastewater obtained from an apartment complex in Gwangju city, South Korea. The characteristics of the wastewater are given in Table 1. The collected wastewater was stored in the refrigerator at 4 °C in order to minimize any biological activity. The wastewater was brought to room temperature just before the start of the experiment.

2. Properties of Activated Carbon Used

The characteristics of three PACs used in the study are given Table 2. Detailed information on the characterization techniques of the activated carbon can be obtained elsewhere [Bansal et al., 1988; McKay, 1996]. Knowledge of the particle size distribution of the PACs used is very important especially when it is used in conjunction with porous membranes, as there can be wide range of size distribution of the carbon particles. The particle size distribution of each of the PACs used is shown in Fig. 1.

Table 1. Characteristics of secondary treated domestic wastewater

Wastewater parameters	Unit	Average values
pH		7.8
TOC	(mg/L)	4.6
Turbidity	(NTU)	0.8
Suspended solids	(mg/L)	14.2
Conductivity	(μ S/cm)	611
BOD	(mg/L)	12.4
COD	(mg/L)	21.2
T-N	(mg/L)	18.24
T-P	(mg/L)	1.21
Elements present		
Ca	(mg/L)	64.77
Na	(mg/L)	55.32
K	(mg/L)	46.21
Mg	(mg/L)	4.015
P	(mg/L)	1.2
Cu	(mg/L)	0.024

3. Characteristics of Membrane

The membrane used in the study is a hollow fiber microfiltration membrane. The membrane was obtained from The Korea Express Co., Korea. The physical and chemical properties of the membrane are given in Table 3. The new membrane was soaked in organic free deionized water for 30 min in order to hydrate the membrane and to remove any impurities or coatings on the membrane surface. Then it was soaked in mild acid solution for 1 day in order to avoid cross contamination from any trace amount of organic material present on the membrane.

4. Membrane Hybrid System

The term "hybrid system" refers to coupling of two treatment operations. For instance, in this study, adsorption unit operation was combined with MF membrane process. The schematic of the membrane hybrid system is shown in Fig. 2. The membrane hybrid system was operated as a continuous stirred tank reactor (CSTR). Kim and Chang, 1994 studied the experimental and theoretical concepts of the CSTR type membrane system in detail. The wastewater was continuously fed from the feed tank so as to maintain a constant

Table 2. Characteristics of Powdered Activated Carbons (PACs)

Specifications	Wood based PAC (WB)	Coal based PAC (CB)	Coconut based PAC (HA)
Type	Wood based	Coal based	Coconut based
Product code	MD3545WB power	MD3545CB powder	MD3545CO powder
Bulk density (kg/m ³)	290-390	100-300	350-500
Surface area (m ² /g)	882	915.2	1198.6
Mean pore dia (A)	30.61	24.2	30.41
Micropore vol (cc/g)	0.34	0.192	0.067
Mean diameter (mm)	19.72	10.9	34.15
Nominal size	80% finer than 75 micron	55-65% finer than 45 micron	75% finer than 75 micron
Iodine number (mg/g min)	900	800	900
Ash content	6% Max	5% Max	3% Max
Moisture content	5% Max	8% Max	10% Max

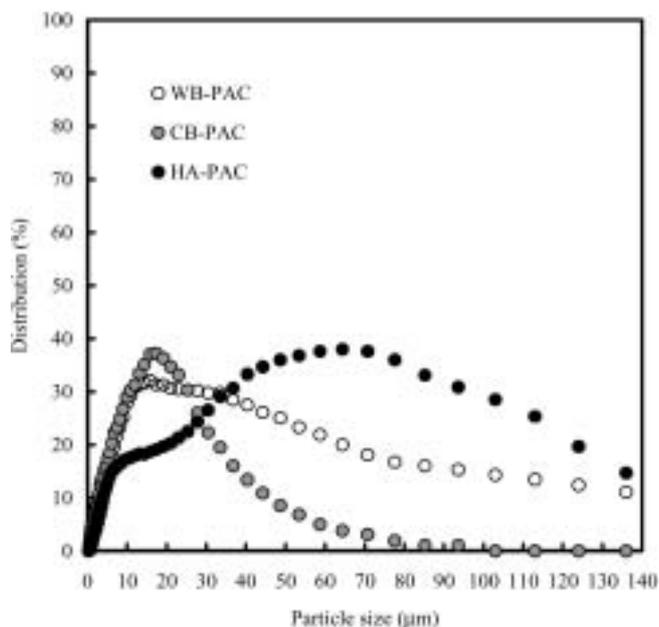


Fig. 1. Particle size distribution of three types of PACs.

Table 3. Characteristics of the MF hollow fiber membrane

Properties	Unit	Description/Value
Material		Polyethylene
Type		Hydrophilic
Total surface area (10 modules 0.2 m length each)	m ²	0.0034
Pore size	µm	0.4
Internal diameter	mm	0.36
External diameter	mm	0.54

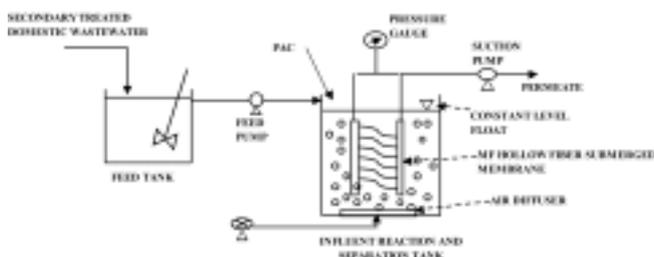


Fig. 2. Schematic of MF membrane hybrid system.

level in the influent tank. Specific amount of PAC dose was added once at the beginning of the experimental run. A thorough mixing was provided in order to keep the PAC always in suspension and to facilitate the adsorption reaction. The permeate was not recirculated.

The organic content was determined by measuring the Total organic carbon (TOC) amount by using a TOC analyzer (Dhorman, Pheonix 2000) and the absorbance at 254 nm using UV Spectrophotometer (Varian DMS 100S). TOC and UV₂₅₄ are regarded as surrogate parameters to measure the organic content in wastewater [Edzwald et al., 1985]. Turbidity was measured with the turbidity meter (HACH 2100P). The permeate volume was monitored in order

to calculate the permeate flux. TOC, UV₂₅₄ and turbidity were measured constantly both in the influent tank as well as in the permeate. Before being collected for TOC and UV₂₅₄, the samples were filtered through 150 mm filter paper in order to eliminate the PAC particles and not the particulate organics in the samples, so as to avoid the error in sample estimation due to the presence of PAC particles. All samples were analyzed as per the standard procedures [APHA, 1998].

5. Membrane Cleaning

At the end of each experiment the membrane was backwashed for 10 min by using the permeate obtained, which removed the PAC cake formed on the membrane. The membrane was then physically cleaned by using clean distilled water to remove any further carbon particles on the surface of the membrane. Then the membrane was cleaned chemically with mild acid solution until the original permeate volume was regained and the adsorbed organic materials in the membrane were removed.

6. Microscopic Imaging

In order to view the surface of the new membrane and the cake deposit after the experiment, a scanning electron microscope (SEM) (Jeol, JSM-5400, Japan) was used.

RESULTS AND DISCUSSION

1. Adsorption Studies

The adsorption abilities of the three PACs were evaluated from the equilibrium and kinetic studies. Fig. 3 shows the results of adsorption isotherm experiments. The Freundlich isotherm equation [Tien, 1994] was used to describe the equilibrium data. The nature of the fit indicates an unfavorable type. Based on the results from

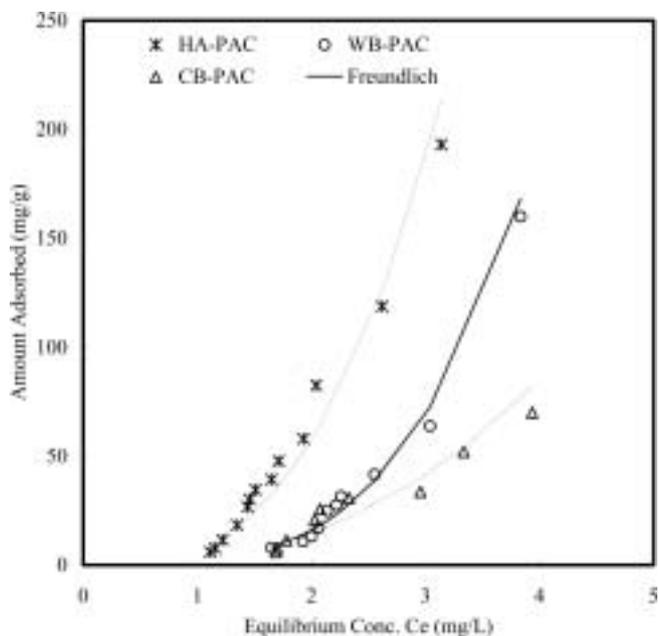


Fig. 3. Adsorption isotherm for the three PACs (equilibrium time 4 days, temperature 25 °C, average initial TOC concentration 4.6 mg/L, isotherm constants - WB PAC: $k=1.3097$, $n=0.2769$, Error %=13.61; CB PAC: $k=2.606$, $n=0.3973$, Error %=25; HA PAC: $k=7.577$, $n=0.3429$, Error %=25.22).

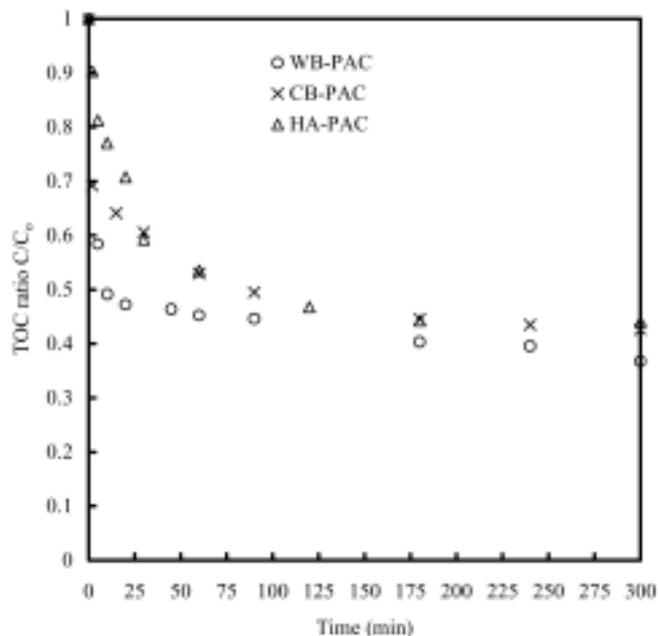


Fig. 4. TOC removal performance at different time (PAC dose 75 mg/L, mixing speed 100 rpm, average influent organic concentration 4.6 mg/L).

the four-day study period, it was evident that HA performed comparatively better among the three. The optimum PAC dose was found to be 75 mg/L with TOC removal of 63%, 51% and 49% for HA, WB and CB respectively. It can be referred to as the optimum dose because further addition of PAC amount had very little improvement in the organic removal [Thiruvengkatchari et al., 2004]. However, this optimum PAC dose does not apply to the CSTR membrane system, where several factors are taken into consideration in determining the optimum dose of PAC.

The results of a batch kinetic test for TOC adsorption from wastewater using the three PACs are shown in Fig. 4. The results indicate that WB had a faster rate of kinetics than HA and CB. It is expected that particles with smaller particle size would have faster kinetics, and so HA having the largest particle size among the three PACs had the slowest rate of kinetics. However, CB having the smallest average particle diameter had slower rate of kinetics than WB. This can be attributed to the influence of several other surface properties of the activated carbon. All the three PACs adsorbed almost 90% of their total capacity within 1 hour.

2. Membrane Hybrid System

Before analyzing the effect of PAC type on the membrane hybrid system performance, it is important to examine the membrane performance without PAC. When wastewater alone was fed into the system without the PAC, it was evident that the fouling of the membrane was severe by reducing almost 80% of the initial permeate flux within one hour of operation. As expected, there was almost negligible amount of organic removal (about 2-3%). This finding was in line with studies by other researchers that organic substances are not being removed effectively and these substances significantly facilitate flux decline [Laine, 1989; Crozes, 1993]. However, addition of PAC decreased the rate of permeate flux decline, as also reported by other researchers [Jacangelo, 1995; Konieczny

and Klomfas, 2002]. In all these studies by having PAC as a pre-treatment before membrane operation, it is generally agreed that the reason for reduced fouling is due to the adsorption of organic matter, which is believed to be the cause of membrane fouling, by the activated carbon.

The performance of the membrane hybrid system based on TOC and turbidity removals and in terms of permeate flux for various HA-PAC doses is shown in Figs. 5, 6 and 7 respectively. There was a

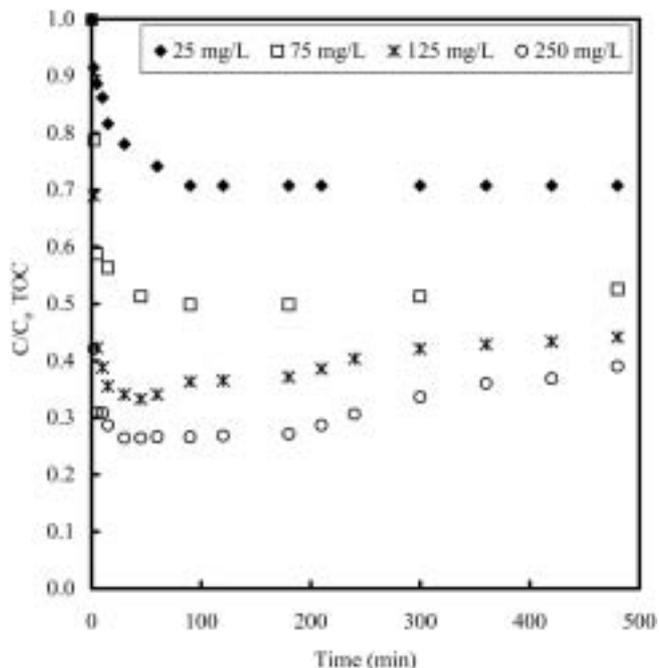


Fig. 5. Overall TOC removal performance of the hybrid system using HA-PAC.

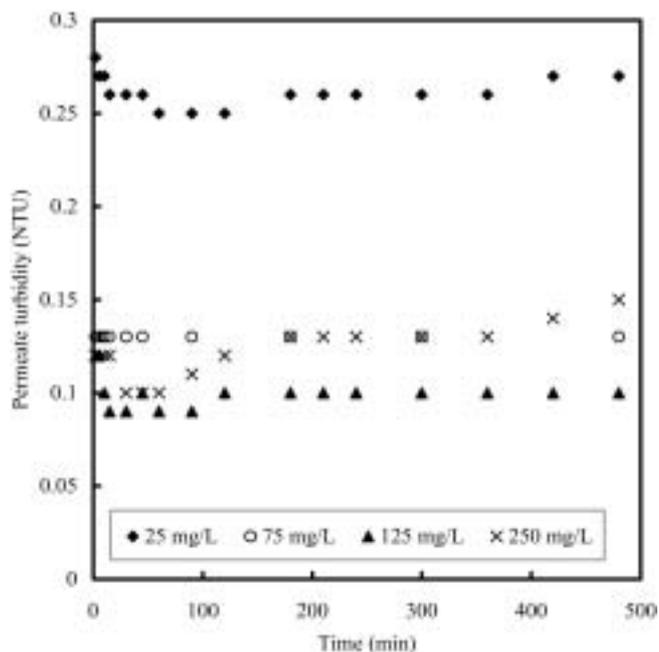


Fig. 6. Performance of the hybrid system based on turbidity removal for HA-PAC.

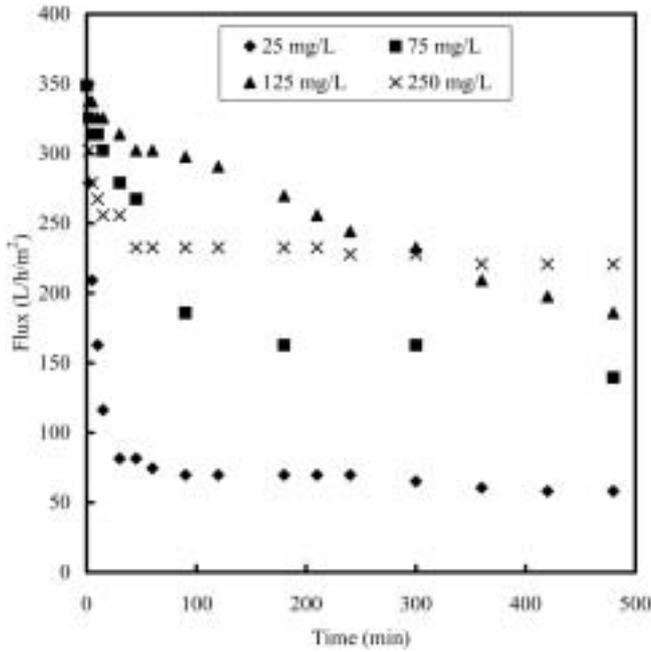


Fig. 7. Flux development pattern for the hybrid system using HA-PAC.

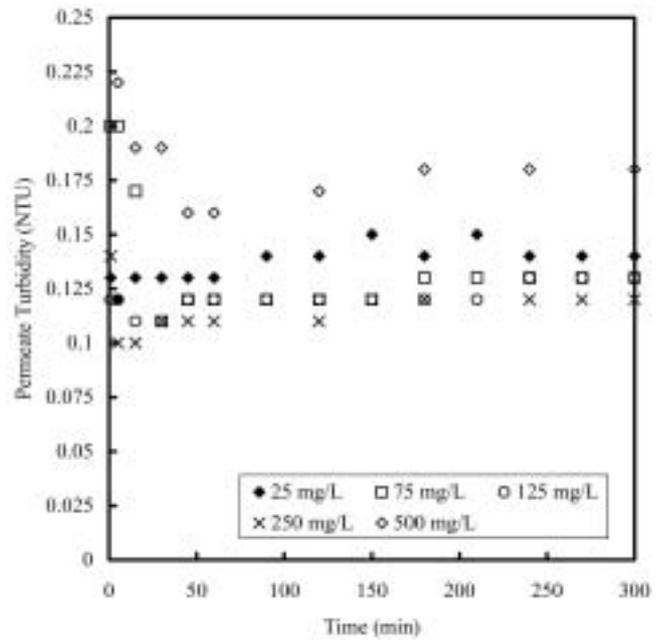


Fig. 9. Performance of the hybrid system based on turbidity removal for WB-PAC.

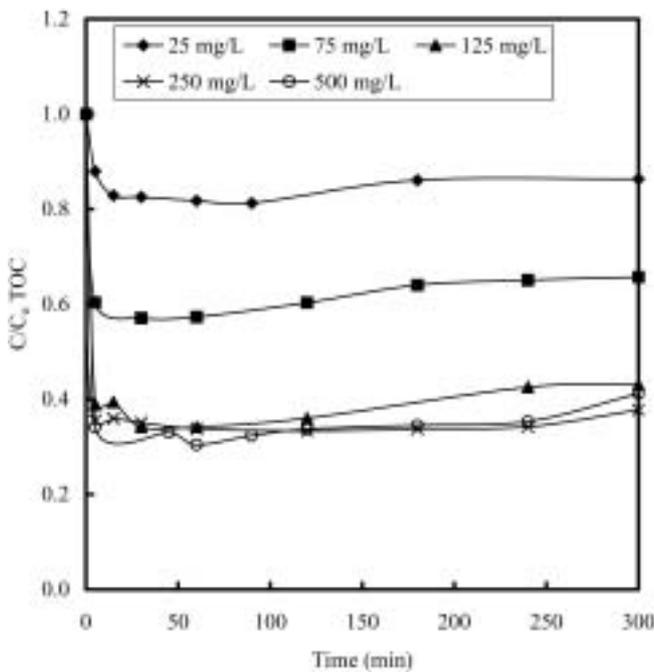


Fig. 8. Overall TOC removal performance of the hybrid system for WB-PAC.

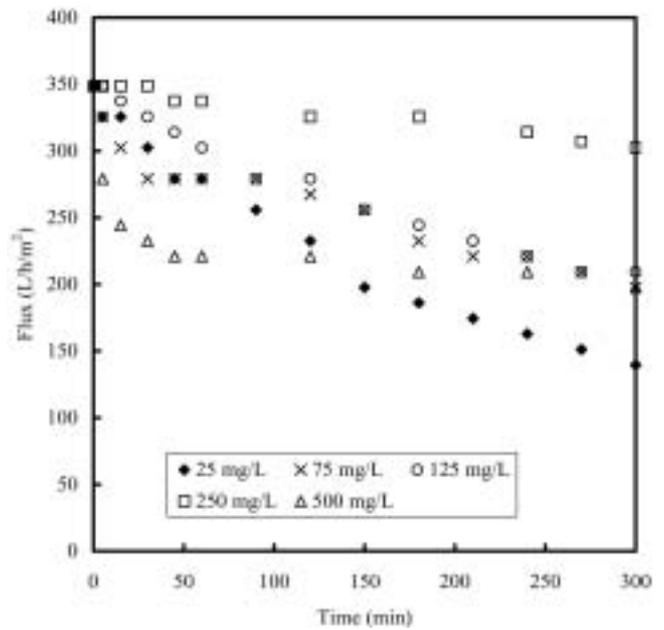


Fig. 10. Flux development pattern for the hybrid system using WB-PAC.

significant improvement in the performance of the membrane hybrid system in terms of TOC removal with the increase in PAC amount. Maximum TOC removal of up to 73% was achieved with 250 mg/L of HA-PAC. However, the permeate flux was higher with 125 mg/L PAC dose compared to 250 mg/L. A similar effect was seen with the turbidity removal. The permeate turbidity increased with the increase in PAC amount from 125 mg/L to 250 mg/L. It appears that 125 mg/L of HA-PAC was the optimum amount for the mem-

brane hybrid system.

Similarly, the performance of the membrane hybrid system based on TOC and turbidity removal and in terms of permeate flux for various WB-PAC doses is shown in Figs. 8, 9 and 10, respectively. There was a significant improvement in the performance of the membrane hybrid system in terms of TOC removal with the increase in PAC amount until 125 mg/L and further increase in dose had very little effect. But, maximum permeate flux was achieved at 250 mg/L WB-PAC dose. Further increase in PAC amount had a negative

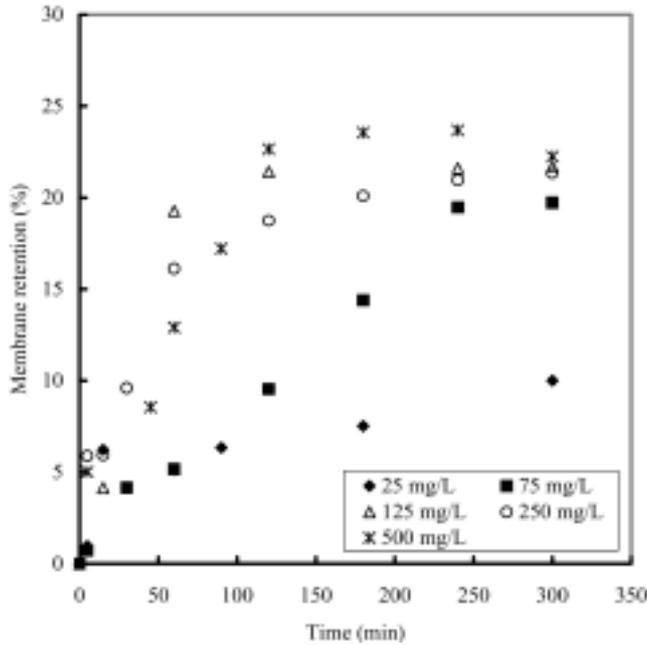


Fig. 11. Membrane TOC retention ability in the hybrid system with WB-PAC.

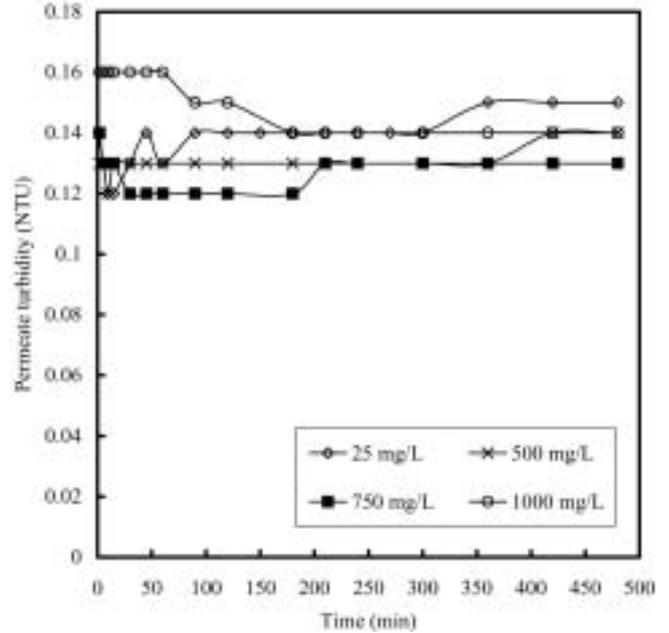


Fig. 13. Performance of the hybrid system based on turbidity removal for CB-PAC.

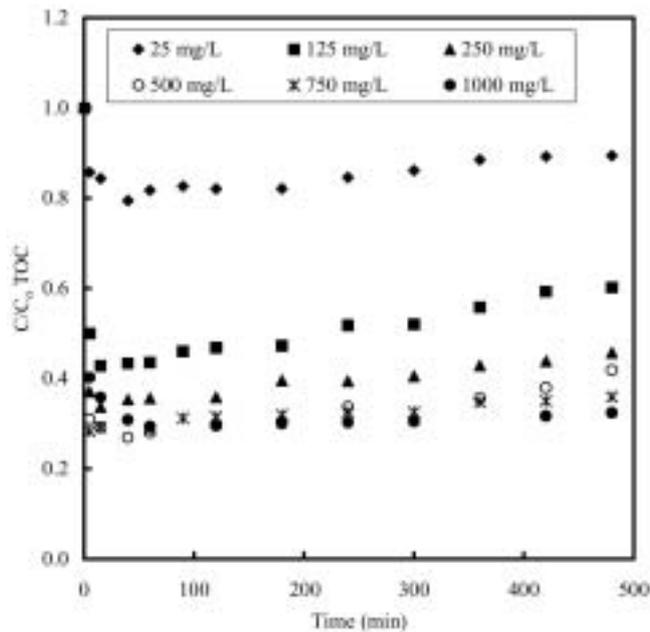


Fig. 12. Overall TOC removal performance of the hybrid system for CB-PAC.

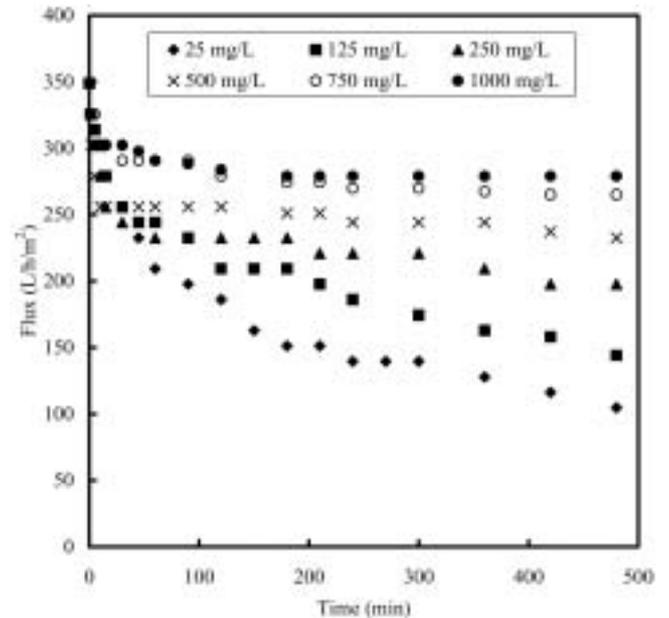


Fig. 14. Flux development pattern for the hybrid system using CB-PAC.

effect as flux declined and the permeate turbidity increased. The maximum membrane TOC retention efficiency was found to about 21%, as shown in Fig. 11. Although the retention was slightly higher for 500 mg/L PAC, considering the overall efficiency of the hybrid system based on TOC, turbidity and permeate flux, 250 mg/L of WB-PAC was found to be the optimum amount.

In the case of CB-PAC, it was evident that the optimum dose for the hybrid system based on the TOC removal, permeate flux pattern and permeate turbidity was 750 mg/L as shown in the Figs.

12, 13 and 14, respectively. There was no appreciable increase in permeate flux and TOC removal when the PAC concentration was increased from 750 mg/L to 1,000 mg/L. However, the effect on permeate turbidity was clearly noticeable as the turbidity increased at that point. The maximum TOC retention efficiency by the membrane was also maximum for 750 mg/L PAC dose, reaching up to about 20%, as shown in the Fig. 15.

Looking at the performance of the hybrid system for the three PACs under study, it is interesting to note that PAC type does play an important role and the amount of PAC required to achieve the

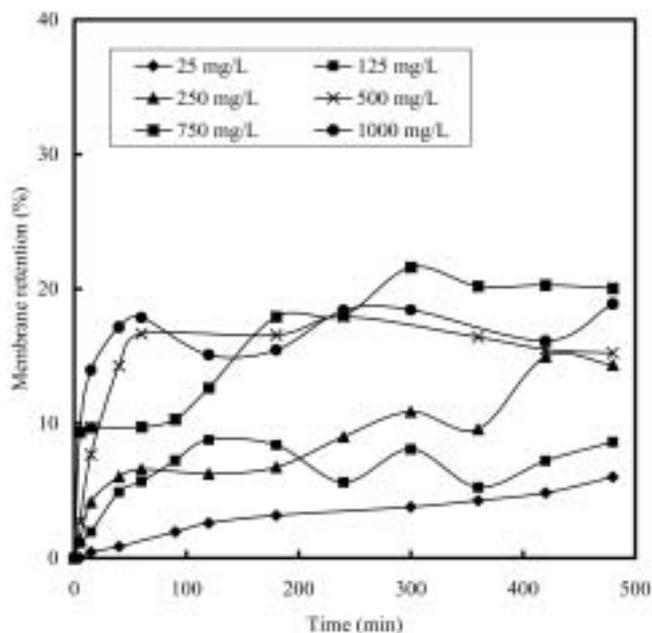


Fig. 15. Membrane TOC retention ability in the hybrid system with CB-PAC.

optimum performance varies. Keeping all other conditions like the membrane material and characteristics and the influent organic type and concentration the same, the characteristics of the activated carbon does seem to have an effect on the membrane hybrid system performance. It was found that lower PAC amounts were less effective in TOC removal; however, further increase in PAC dose after a certain amount was also found to be less useful. As the maximum adsorption capacity of carbon was reached, the performance of TOC removal by the membrane hybrid system also showed little gains.

It can be seen from the permeate flux patterns that increasing the PAC dose beyond the optimum concentration, proved to be either less effective or unfavorable in the improvement of flux. During the initial stages of membrane operation, there was a severe decline in the flux, indicating a rapid pore blocking by the organic material. The gradual reduction in permeate volume could possibly be due to the deposition of PAC particles onto the membrane. Considering the rapid flux decline in the absence of PAC and the high rate of adsorption kinetics, it is envisaged that during the initial period of the hybrid system the organic carbon is competitively adsorbed both by membrane and onto the activated carbon. However, at an optimum PAC dose, initial rapid decline in the permeate flux was reduced, which indicates that the organic matter is not allowed to reach the membrane surface possibly due to some specific interaction like the charge effect between PAC particle and the organic carbon. Schafer [1999] found that, under certain conditions, organics imparted a stabilizing effect on the adsorbent hematite particles, which resulted in the reduction of fouling and improved their removal by the membrane.

At the same time, there is a rapid build up of deposition of PAC particles onto the membrane during the initial stages, followed by slower pace of particle deposition and finally reaching a steady state where the deposition and detachment of particles is established. De-

Table 4. Performance analysis of the membrane hybrid system for three PAC types (Influent organic conc. 4.6 mg/L), Initial turbidity 0.8 NTU, Initial UV_{254} Absorbance 0.1)

Parameters	HA-PAC	WB-PAC	CB-PAC
PAC amount introduced (mg/L)	125	250	750
(μm)	34.15	19.72	10.9
Amount deposited on membrane after 8 hrs expet. run (mg/L)	70	196	694
Overall TOC removal after 5 hrs operation (%)	69.55	56.8	47.95
Removal in terms of UV_{254} absorbance (%)	70	68	65
Membrane TOC retention (%)	18.45	21.7	8
Flux decline (%)	33.3	40	50
Turbidity removal (%)	87.5	83.7	80

position of organic material on the membrane could alter the charge on the membrane and in turn the deposition of further organic material and also PAC particles. Moreover, as the wastewater containing the organic material is constantly supplied into the reaction tank, the surface of the activated carbon is increasingly coated with the organic material and would have an effect on the deposition onto the membrane. Visual observation of the membrane during the experiment reveals that the membrane surface is not uniformly coated with the PAC particles. Table 4 illustrates the amount of PAC particles deposited on the membrane and their effect on the performance of the membrane hybrid system. The properties of the cake deposit on the membrane surface also play a vital role in determining the filtration ability of the membrane [Iritani and Mukai, 1997]. As the influent PAC dose, hydrodynamic conditions, membrane characteristics, influent type and organic concentration are the same, the difference in PAC amount deposited on the membrane can only be attributed to the effect of surface properties of the PACs. The PAC particle size does seem to have an effect in deciding the amount of particle deposited on the membrane. CB having the lowest average particle size was deposited more on the membrane. However, HA having greater particle size than WB and also having greater bulk density than WB (Table 2), was found to have the least amount of particles (70 mg) deposited on the membrane compared to WB (196 mg). CB-PAC had the maximum ratio of amount of particle deposited to the amount of PAC introduced, followed by WB and HA. It is possible that there is an underlying effect of surface charge of the reacting species, which determines the variation in deposited amount of PACs. Fig. 16 shows the SEM micrograph of the surface of clean membrane and the surface with the three types of PAC deposited on them after the experiments were carried out for 8 hours. Based on optical observation, a porous cake layer of 0.1-0.5 mm was formed on the membrane surface. From the SEM images, voids or pores of the deposited cake appear to be in the range of about 2-7 μm , which is much greater than the membrane pore size of 0.4 μm and is expected to have little effect in retaining the organic material by the process of sieving. It was evident with the fact that even with maximum amount of particle deposited (92 mg) in the case of CB-PAC, the membrane retention for TOC was the least compared to all the three kinds of PACs. Also, the average size of the all three PAC particles is too big to block the membrane pores and cause

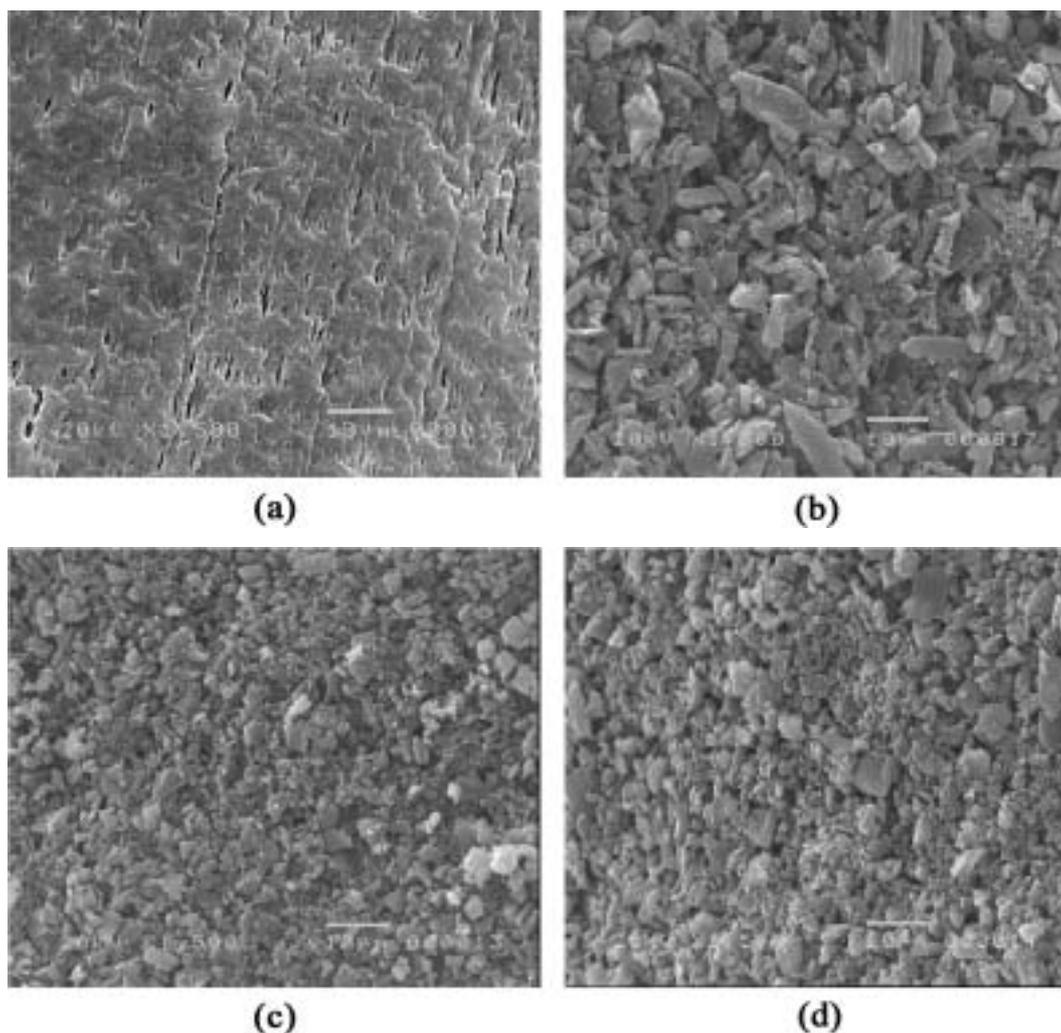


Fig. 16. SEM micrographs for the membrane hybrid system showing the surface of clean membrane and after particle deposition after 8 hrs experimental run (Clockwise from top left: (a) surface of clean membrane (b) HA-PAC deposited on membrane (c) WB-PAC deposited on the membrane (d) CB-PAC deposited on membrane)).

reduction in permeate flux. So, the influence on the overall TOC removal performance of the hybrid system appears to be the effect of adsorption capacity of the PACs.

This finding highlights the importance of PAC type on the performance of the hybrid system. This study will be of much significance when adopting PAC-membrane hybrid system and help in deciding the PAC type for the system. It shows that while choosing the PAC for adsorption hybrid system, it is important to select the PAC having maximum adsorption capacity, which outweighs their influence due to other physical characteristics on the membrane performance. Further studies are warranted to explain the effect due to the surface charge of the reacting species and its effect on the particle deposition on the membrane. More experiments are needed to understand the nature of the cake deposited on the membrane and their effect on TOC removal and flux patterns, especially under long-term experiments.

CONCLUSIONS

Effect of PAC type on the performance of the membrane hybrid

system was clearly demonstrated by using three kinds of PACs having different characteristics. The adsorption abilities of the PACs were determined by using preliminary adsorption studies. It was evident that HA-PAC (coconut based) performed best followed by WB-PAC (wood based) and CB-PAC (coal based). Adsorption equilibrium experiments conducted for a duration of 4 days indicated that 75 mg/L of HA, WB and CB PACs achieved a maximum TOC removal efficiency of 64%, 52% and 50%, respectively. Further increase in PAC amount was found to have very little effect on organic removal. The average initial TOC concentration of the wastewater was 4.6 mg/L. The equilibrium results were found to fit well with Freundlich isotherm equation. Almost 90% of total TOC removal was achieved within one hour of operation.

Addition of PAC significantly lowered the rate of flux decline. TOC removal performance of the membrane hybrid system was in line with the adsorption capacity pattern of the PACs. The optimum performance of the hybrid system was achieved at specific PAC amounts for the three PACs. HA required the least amount of 125 mg/L followed by WB at 250 mg/L and CB at 750 mg/L. The maximum TOC removal of the hybrid system at these PAC con-

centrations was 67%, 69% and 71% for HA, WB and CB, respectively. With respect to the organic removal based on UV_{254} absorbance at these optimum PAC doses, removal efficiencies of 67%, 70% and 82% for HA, WB and CB PACs were achieved, respectively. The initial absorbance for the influent wastewater was 0.1. At the optimum PAC dose amounts the reduction of permeate flux after 8 hours of membrane run time was 46.7%, 26.6% and 24.9% for HA, WB and CB PACs, respectively. Almost 80-90% of turbidity removal was achieved with the initial wastewater turbidity of 0.8 NTU. It appears that the porous cake formed with a thickness of about 0.1-0.5 mm on the membrane was not helpful in retaining the organic material. The membrane generally had a TOC rejection of about 20-30%.

This study highlights the importance of PAC type in the adsorption membrane hybrid system and would help researchers and treatment plant operators make a right choice in selecting the PAC to be used in the hybrid process. While selecting the PAC for the hybrid system, importance must be given to the adsorption capacity of the PAC.

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