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Ultrafiltration of Natural Organic Matter (NOM): Effect of NOM Characteristics on Membrane Rejection Mechanisms in Drinking Water Process

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ABSTRACT The main objective of this research is to study on the influence of NOM characteristics on the membrane rejection mechanisms. Polysulfone (PSF) and cellulose acetate (CA) membranes formed by a simple phase inversion technique were used in this study. The experiments were carried out through a hydrophobic 68 kDa MWCO PSF and a hydrophilic 50 kDa MWCO CA of a submerged ultrafiltration (UF) membrane reactor. The Ulu Pontian Kecil river water was fractionated into several components which were hydrophobic (HPO), transphilic (TPI) and hydrophilic (HPI) fractions using DAX-8 and XAD-4 ion exchange resins. The isolated surface water components were characterized by dissolved organic carbon (DOC), ultra violet absorbance at 254 nm (UV_{254nm}), specific ultraviolet absorbance (SUVA) or a measure of relative abundance of relative aromaticity, etc. The experimental results showed that the hydrophilic component of NOM exhibited the worst flux decline despite lesser DOC and UV rejection compared to other fractions (HPO and TPI). The electrostatic interaction has been found to play a more influential role than steric hindrance mechanism when filtering NOM fractions with a negatively charged membrane. The quantitative results of Resistance in-Series Model analysis on clean and fouled membranes elucidated that both reversible and irreversible fouling mechanisms occurred during surface water filtration.

ABSTRAK Objektif utama penyelidikan ini ialah untuk mengkaji pengaruh ciri-ciri bahan organik asli (NOM) terhadap mekanisma penyingkiran membran. Membran polysulfona (PSF) dan selulosa acetatat (CA) yang dihasilkan melalui kaedah teknik fasa balikan telah diguna dalam kajian ini. Ujikaji dijalankan dengan menggunakan membran hindar air PSF (68 kDa) dan membran CA hidrofilik (50 kDa) dalam reaktor ultraturasan (UF) membran tenggelam. Air Sungai Ulu Pontian telah diasingkan kepada beberapa komponen iaitu hindar air (HPO), tranfilik (TPI) dan hidrofilik (HPI) dengan menggunakan resin penukaran ion DAX-8 dan XAD-4. Setiap komponen yang terasing akan diukur berdasarkan kepada kandungan karbon organik terlarut (DOC), penyerapan ultra-ungu pada 254 nm (UV_{254nm}), penyerapan ultra-ungu spesifik (SUVA) atau pengukuran limpahan bagi aromatik bandingan dan sebagainya. Hasil ujikaji menunjukkan bahawa komponen hidrofilik menunjukkan kadar fluks yang paling rendah walaupun nilai penyingkirannya adalah yang paling minima berbanding komponen lain (HPO dan TPI). Interaksi elektrostatik didapati memainkan peranan utama berbanding mekanisma penahanan sterik semasa menggunakan membran bercas negatif. Analisis model rintangan bersiri ke atas membran bersih dan yang telah ditapis menunjukkan kedua-dua mekanisma kotoran boleh balik dan tidak boleh balik berlaku semasa penapisan air sungai.

(Fouling, NOM, Submerged ultrafiltration, Foulant, Surface water)

INTRODUCTION

Membrane filtration processes involving microfiltration (MF), ultrafiltration (UF), nanofiltration (NF) and reverse osmosis (RO) in potable water production have increased rapidly for the past decade and can potentially replace the conventional treatment process. Besides that, membrane filtration process does offer extra advantages over conventional treatment such as small footprint, compact module and is capable of handling wide fluctuations in feed quality. MF and UF are employed to remove microparticles and macromolecules, which generally include inorganic particles, organic colloidal (i.e. microorganism) and dissolved organic matters (DOM). DOM is ubiquitous in natural surface water and is often claimed [1 - 4] as an important factor for both the reversible and irreversible fouling in water filtration. Unfortunately the presence of free chlorine content that is used as a disinfectant in conventional treatment is found to react with this residual DOM consisting in the natural organic matter (NOM). This reaction process has been found to have the tendency to form disinfection by-products (DBPs). In this study the influence of NOM components characteristics on the fouling of a submerged ultrafiltration polysulfone membrane and fouling mechanisms were investigated. The potential foulants of NOM isolates which were detrimental to the employed submerged UF membrane were studied and identified by isolating the chosen surface water. The performance of membrane and the associated fouling mechanisms were examined based on NOM isolates rejection, flux declination, hydraulic series resistance.

APPROACH AND METHODS

Fabrication of UF membrane

The polysulfone polymer was supplied by Amoco Performance Product Inc. The additive polymer of PVP3000 was bought from Fluka Milwaukee while the solvent (DMEC) was purchased from Merck Darmstadt Germany. The PVP3000, solvent and PSF were sequentially mixed in a container until a homogenous mixture was achieved at a constant temperature of 60 °C. The second dope was made of cellulose acetate polymer base, acetone and formamide solvents. The PSF and CA polymer were chosen as membrane materials due to their wide commercial application, commercial availability and favorable rejection-flux decline characteristics. The formulated dope was then spun by using a forced convective dry/wet spinning technique under pressurized nitrogen gas. The membrane characteristic is shown in Table 1.

Table 1. Characteristics of the experimental membranes

PARAMETER	MRUTM55	MRUTM66
Membrane type	Hollow fiber	Hollow fiber
Membrane material	Polysulfone (PSF)	Cellulose Acetate
Contact angle	56°	20°
MWCO	68 kDA	50 kDA
Surface property	Hydrophobic	Hydrophilic
Pure water specific flux ($Lm^{-2}h^{-1}bar^{-1}$)	43 ± 5	85 ± 7

Table 2. Surface water characteristics

SOURCE	SUNGAI ULU PONTIAN, JOHOR
Coordinate	N1° 35.541', E103° 30.704'
DOC (mg/L)	6.8
UV_{254} (cm^{-1})	0.088
SUVA (L/mg.m)	1.29
Conductivity (µs/cm)	37

Table 3. DOC concentration of Sungai Ulu Pontian fractions (based on DOC and mass balance technique)

FRACTION		DOC (mg/L)	DOC (%)
Hydrophobic	(HPO)	2.38	35
Transphilic	(TPI)	1.7	25
Hydrophilic	(HPI)	2.6	38

Water quality

Soft surface water rich in NOM (Table 2) was taken from the Ulu Pontian River in the state of Johor, Malaysia. The raw water was filtered by a 0.45 μm filter and fractionated into three fractions: hydrophobic (HPO) which is Supelite DAX-8 (Sigma-Aldrich) adsorbable, transphilic (TPI) which is Amberlite XAD-4 adsorbable and hydrophilic (HPI) which passes through the DAX-8 and XAD-4 resins without any adsorption (Table 3). The HPO and TPI components were collected after eluting the resins with 0.1 NaOH. The pH of the fractionated NOMs (HPI, TPI and HPI) was adjusted to the approximate value of raw water (pH = 7.03) since fractionation procedure resulted in variation on this water quality parameter. In general, NOM in river water consists of thousands of particulate and soluble components which could act as detrimental foulants and may impose significant reduction in membrane permeability. However, NOM can be classified, grouped and fractionated according to their molecular size, surface charge and hydrophobicity. In this study the river water of Ulu Pontian was fractionated into HPO, TPI and HPI fractions using ion-exchange process with macro-porous resins of DAX-8 and XAD-4. Each of the NOM fractions would be different in terms of molecular weight, molecular structure, molecular shape and its intrinsic charge. The charge of the NOM fractions is derived from its ionizable functional groups as shown in Figure 1 and Figure 2. Each of this NOM fraction is expected to carry identical charge quantity (mV) as it existed either in its natural presence (real river water condition) or after being fractionated with the ion exchange resin. The ionizable functional group, aromatic structure of the HPO and TPI fractions and the aliphatic linear molecular structure or the neutral character of the HPI fraction are strongly believed to assist in better explaining the cause and effect of membrane behaviors (rejection of NOM, fouling mechanisms and flux decline) during surface water-membrane interactions.

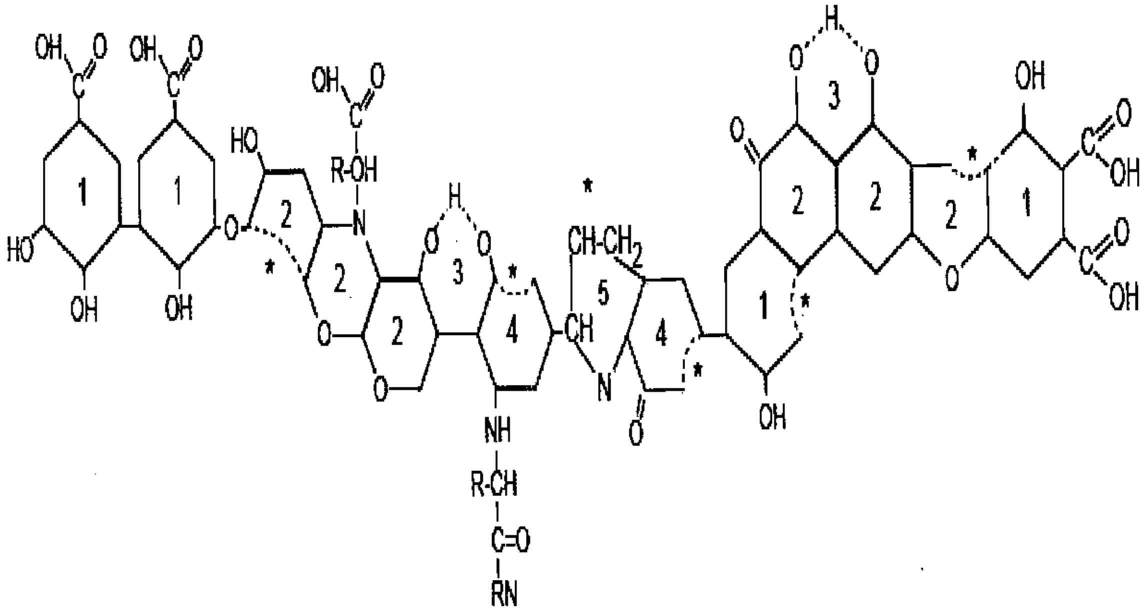


Figure 1. Schematic of humic acid model structure

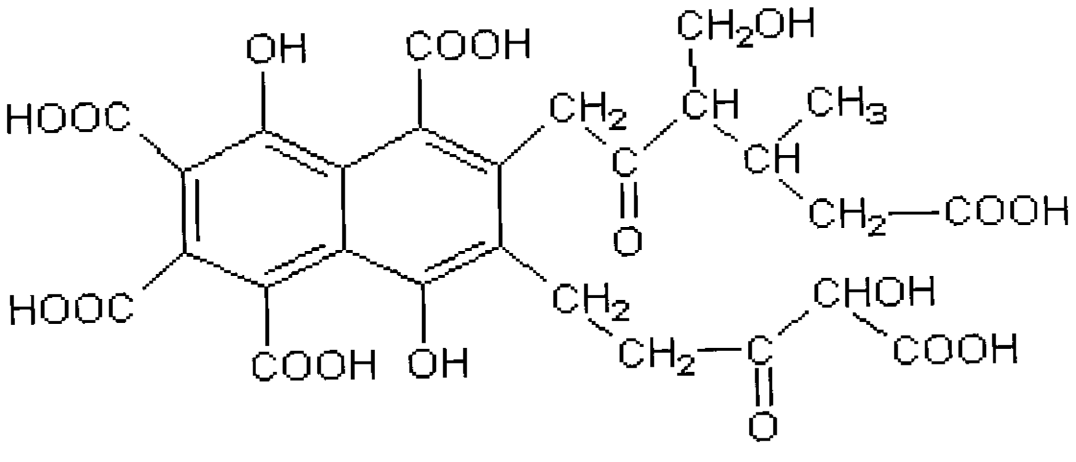


Figure 2. Schematic of fulvic acid model structure

Determination of intrinsic membrane resistance (Rm)

The intrinsic membrane resistance (Rm) was measured using pure water flux (J_{pwf}) at several transmembrane pressures (TMP) according to Darcy's law (Equation 1), where J is the permeate flux ($m^3. m^{-2}. s^{-1}$), ΔP is the transmembrane pressure (Pa), μ is the dynamic viscosity (Pa. s) and Rm is the intrinsic membrane resistance (m^{-1}). The membrane was filtered with deionized water water (DI) until permeation reached a steady value. The pure water flux was conducted under steady-state flow and was determined as in Equation 2 where the J_{pwf} is the pure water flux, V is the permeate volume (L), A is the membrane area (m^2) and ΔT is the sampling time. The Rm value was then determined from the slope of linear relationship of water flux (J) versus ΔP.

$$J = \frac{\Delta P}{\mu Rm}$$ **Equation 1**

$$J_{pwf} = \frac{V}{A.\Delta T}$$ **Equation 2**

Membrane filtration operation and determination of hydraulic resistances (Rcp, Rc and Ra)

Ultrafiltration (UF) submerged reactor was used throughout this study and the experimental set-up is schematically illustrated in Figure 3. Initial flux of pure water flux (J_{pwf1}) was introduced to the membrane at TMP of 140 mmHg until the pure water flux was stabilized. The DI water was then replaced with a NOM fractional source at the same TMP of J_{pwf1}. The operational flux and flux decline were regularly measured throughout the experiment. Samplings for the UV_{254} and DOC rejection were periodically taken untill permeation reaches steady state conditions. The system was then replaced with DI water to remove the (CP) layer and the second J_{pwf2} was measured. The system was back flushed thoroughly for 10 minutes [2] with DI water to remove all the reversible adsorption layers before the third J_{pwf3} was measured and the membrane was then taken out from the reactor. The foulants were extracted using 0.1 N NaOH for three hours followed by 0.1N HCl for another three hours solutions respectively [3]. The fourth J_{pwf4} was then measured to complete the Resistance in series model (Equation 3). The assumption on this model is made whereby the osmotic pressure is subsumed into the concentration polarization resistance. The whole procedure in measuring the hydraulic resistances is represented by Figure 4.

$$J = \frac{\Delta P}{\mu(Rm + Rcp + Rc + Ra)}$$ **Equation 3**

where Rm is membrane intrinsic hydraulic resistance, Rcp is concentration polarization resistance, Rc is cake resistance and Ra is adsorption resistance.

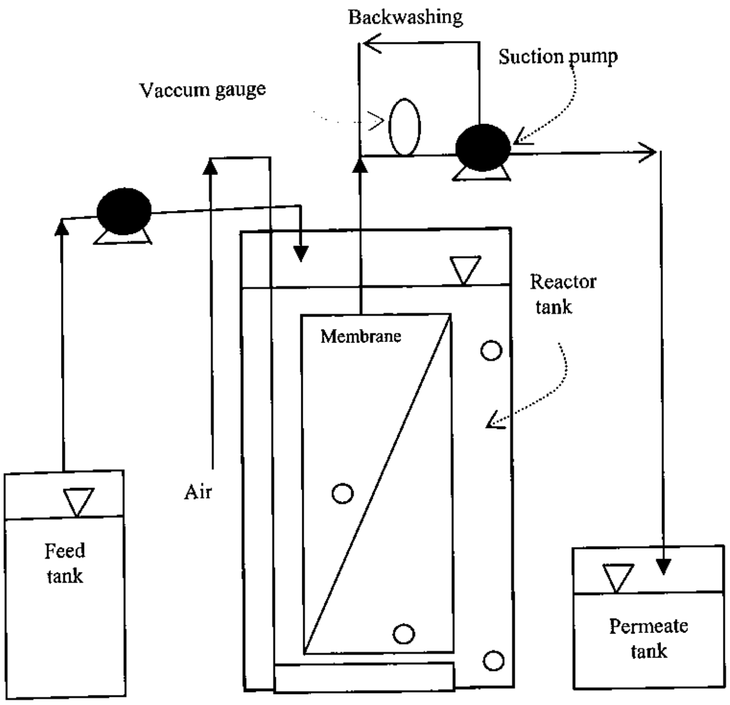


Figure 3. Schematic diagram of submerged UF reactor

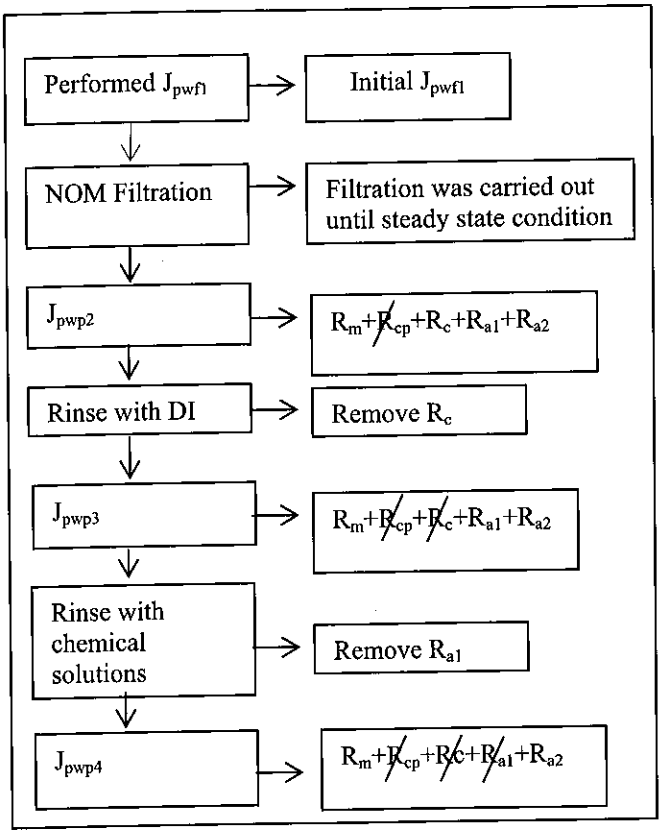
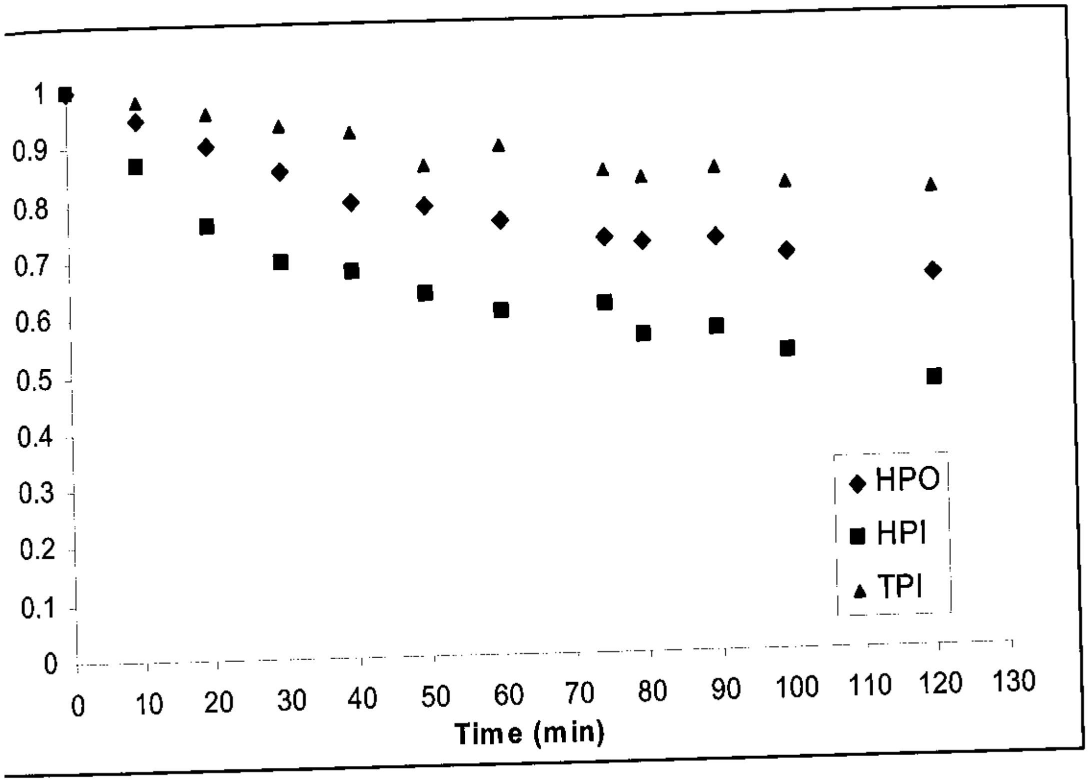


Figure 4. Steps in Resistance-In-Series procedure for Rcp, Rc and Ra measurement

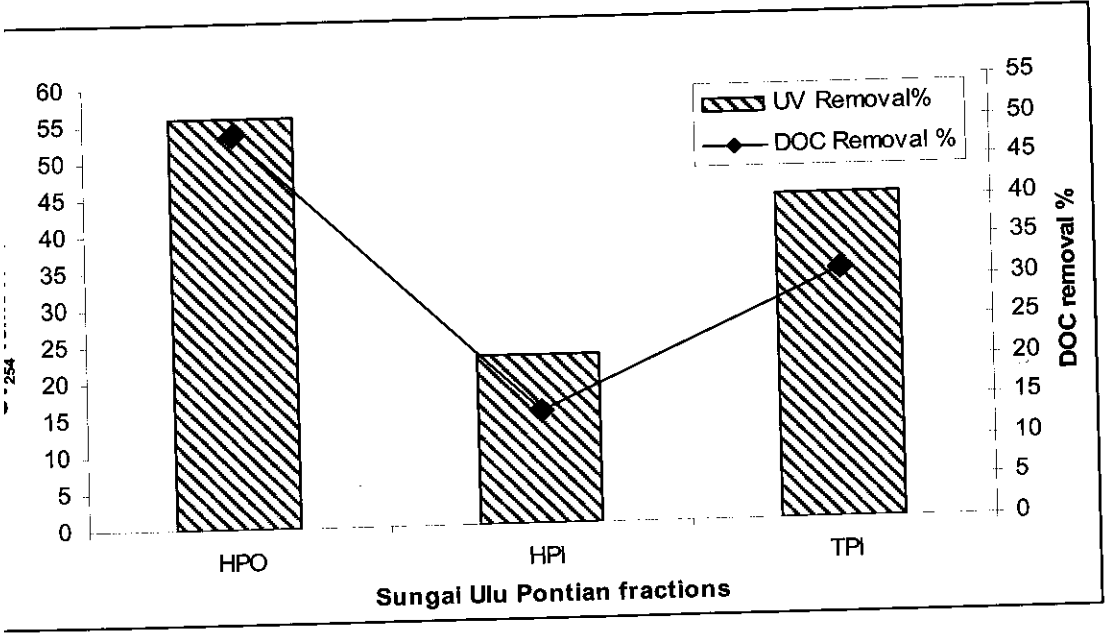
RESULTS AND DISCUSSION

Influence of NOM fractions on fouling characteristics

The Ulu Pontian River exhibited a relatively low hydrophobic fraction concentration and therefore can be classified as hydrophilic surface water type (Table 3). Surface water with SUVA less than 3 $Lm^{-1}mg^{-1}$ is classified as low-humic water [4]. The normalized permeate fluxes for the three fractions are shown in Figure 5. All three fractions showed an obvious flux decline at the initial period of filtration. The fouling that occurred in these cases were mostly attributed to the dissolved NOM. The HPI fraction was found to have the most significant fouling potential compared to other fractions and this finding could be taken as the most outstanding phenomenon observed in this study. The detrimental effect of HPI fraction to membrane permeability was even more pronounced as it showed greater flux decline and higher fouling rate than HPO and TPI components. This phenomenon could be explained due to higher content of DOC in the HPI fraction (2.6 mg/L) than other NOM sources such as HPO (2.38 mg/L) and TPI (1.7 mg/L). However, this finding was contradictory with the results obtained by [5 - 6]. In their studies, the HPI components showed less fouling effect than the humic fraction. Nevertheless the result presented here showed that the HPI exhibited the worst flux decline by approximately 54% of the initial flux after a 120 minutes operation. On the other hand, both HPO and TPI flux declines were about 35% and 20% in spite of having the same filtration duration. Thus, it is worth noting that the HPI fraction was the prime foulant in NOM and was mainly responsible for fouling and major flux decline in surface water filtration. This could be reasoned from the low UV absorbing compound and high molecular weight component such as polysaccharides or the polysaccharide types contained by the HPI fraction [2]. This finding was well supported by studies carried out by Jarusutthirak [7] and Lin *et al.* [8] where they also found that the hydrophilic components cause more fouling potential than humic substances. Although the flux results showed the HPI fraction is the most detrimental foulant and exerted the worst flux decline but its DOC removal was the lowest (23%) among the fractions. In this scenario, the rule of thumb where the highest flux decline would perform the best rejection did not occur. In fact it seems that the flux declination is not linearly parallel with the DOC removal and in this case, it worsened as the UV_{254} removal was also the least. In this case, apparently the HPI fraction is not only has the most significant fouling potential but also would result in the worst membrane fouling (flux decline) and the lowest NOM removal (DOC and UV_{254} rejections). On the other hand, the HPO fraction was found to be well rejected than the fractions in terms of DOC and UV_{254} (Figure 6). Surprisingly, the humic compounds such as HPO and TPI exhibited better NOM removal compared to their non-humic counterpart (HPI fraction) despite possessing smaller DOC concentrations (Table 3) than that of the HPI fraction. As a matter of fact, the HPO fraction with the lowest DOC concentration (2.38 mg/L) performed the greatest NOM rejection followed by the TPI and HPI fractions, respectively. This phenomenon is presumed to occur due to the electrostatic interaction between HPO fraction and the PSF membrane. The HPO fraction contains mainly humic acid and fulvic acid that are anionic polyelectrolytes with negatively charged of carboxylic ($COOH^-$), methoxyl carbonyls (C=O) and phenolic (OH^-). Besides that, the PSF membrane was also widely reported by other researchers to have high negative charge density especially at higher pH as was employed in this experiment. Therefore less flux decline or higher HPO fraction was rejected by PSF membrane with regard to the charge repulsion between negative functional groups of HPO and negative surface charge of PSF membrane, respectively. Besides that, the results of Figure 4 also showed that UV_{254} removal were always higher than DOC rejection for all the three fractions. This could be again due to the tendency or preference of polysulfone membrane to repulse the high aromatic compound such as the HPO fraction compared to the other fractions and it was shown high by the percentage of UV removal, meanwhile the DOC is not a specific measurement of certain compound but a total carbon concentration comprised by the three fractions.



re 5. Flux profile of Sungai Ulu Pontian fractions by MRUTM55



gure 6. UV$_{254}$ and DOC removal (%) of hydrophobic, transphilic and hydrophilic fractions by UTM55 membrane

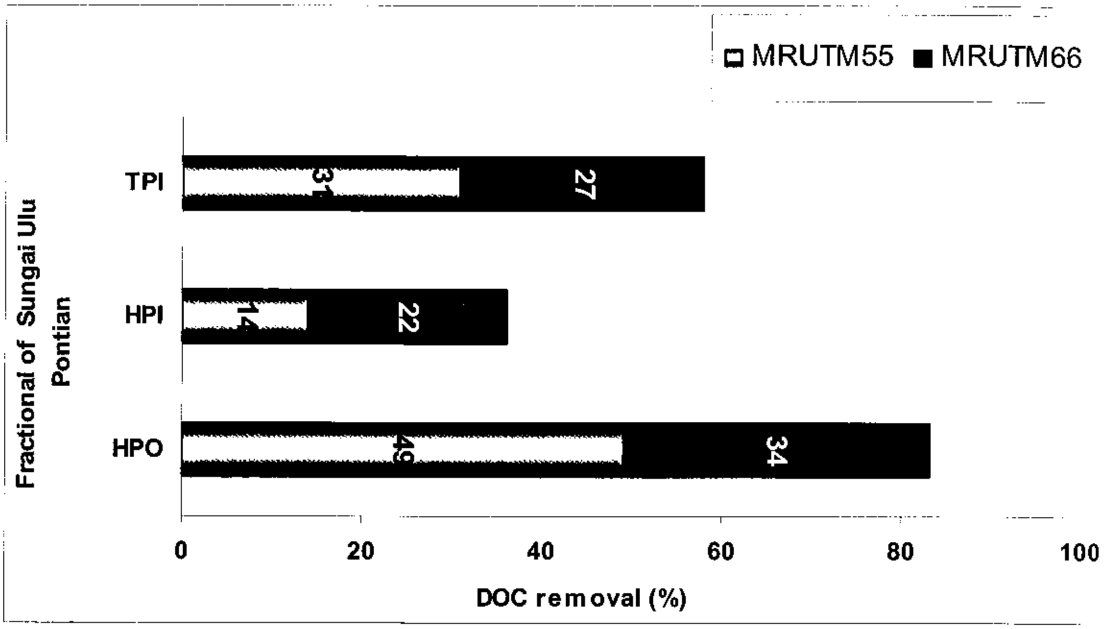


Figure 7. Comparison of DOC removal (%) between MRUTM55 and MRUTM66 on Sungai Ulu Pontian components

The effect of membrane characteristics on NOM rejection was further studied with a MRUTM66 membrane. This membrane has lower MWCO (58 kDA), more hydrophilic and less negative charge [11] compared to MRUTM55 membrane. From Figure 7 it was clearly shown that the MRUTM66 resulted in lesser DOC (%) rejection despite its lower MWCO than MRUTM55 membrane. This scenario is again presumably related to the shape, charge and size effects of NOM components towards the rejection mechanism of MRUTM66 membrane. In general the MRUTM66 increases the DOC rejection (%) of HPI fraction but decreases for both the HPO and TPI fractions when compared with MRUTM55. The HPI fraction removal (in term of % DOC rejection) by MRUTM55 was about 14% but increases to 22% when introduced to MRUTM66. However, overall, the DOC removal was still dominated by HPO fraction followed by TPI and HPI fractions. Better removal in HPI fraction could be explained by the lower MWCO (50 kDA) employed by the MRUTM66 membrane whereas higher rejection of HPO and TPI than HPI component was due to higher intrinsic molecular weight (MW) possessed by both the fractions. Both the HPO and TPI fractions had been widely reported to have greater MW [11] or higher relative molecular mass [8] than HPI fraction by previous researchers. Furthermore the HPO fraction and TPI fraction have more complex molecules structures (aromatic shape) than HPI fraction (aliphatic structure) that made them easier to be excluded by the MRUTM66 membrane. During filtration with MRUTM66 most of the HPO fraction was successfully passing through the pores as the membrane was negatively less in charge and the electrostatic interaction between the functional groups of HPO and MRUTM66 (cellulose acetate) was no longer the rejection dominant mechanism. Thus it is worth to note here that the rejection mechanism imposed by the MRUTM66 was a steric exclusion hindrance while the MRUTM55 was an electrostatic repulsion mechanism. Based on the DOC removal (%) exhibited by all the NOM fractions of the Sungai Ulu Pontian, the order of rejection mechanisms is shown in Table 4.

Table 4. Dominant rejection mechanism on NOM fraction with different membrane (from most dominant to less dominant)

MEMBRANE CHARACTERISTICS
i) Negative Charged and hydrophobic membrane Electrostatic interaction > Hydrophobicity > Steric exclusion
ii) Less negatively charged and hydrophilic membrane Steric exclusion > Electrostatic interaction; Hydrophobicity

Effect of NOM fractions on Resistance in Series

The flux decline by NOM fractions were further quantitatively studied through resistance in series model (Equation 1). The explanation of flux decline caused by Sungai Ulu Pontian fractions was made by correlating their flux decline with the incurred fouling mechanisms and hydraulic resistances (Table 5). There seems to appear a relationship between the high fouling potential of HPI fraction and its high irreversible resistance (Ra). The HPI fraction has the highest Ra value (30.9%) and exhibited the worst flux decline in among the NOM fractions. Thus this severe flux decline was mostly attributed to the adsorption fouling mechanism of non-humic fraction (HPI) occurred during the filtration. This adsorption fouling mechanism can be described by saturation and precipitation processes of dissolved organic in the membrane pores; in this case it can be compounds derived from polysaccharide (neutral) and amino sugar [11]. This HPI fraction was expected to have the capability of passing through the membrane pores (HPI has an aliphatic structure) and not being able to be excluded by the steric hindrance mechanism as it was smaller than membrane MWCO (68 kDa). The normal molecular mass of HPI fraction found in river waters were reported [7, 9] to be lower than 10 kDa. In addition, as the polysaccharide does not have any ionizable functional group would definitely help them to easily overcome the electrostatic repulsion by the membrane. The precipitated HPI compounds would then adsorb onto the pores, constrict, reduce the pore sizes and to the extent of blocking it. This initial adsorption enhanced further mass accumulation of HPI that cover the pores and later lead to cake layer formation. This statement is well supported from the result of Table 5 that showed HPI fraction to have high cake resistance (12.5%). Meanwhile, the HPO fraction which has an aromatic structure showed the concentration polarization (CP) as the primary fouling mechanism to its flux declination (Table 6). This is supported by the highest Rcp (22%) possessed by HPO component compared to other fractions. Moreover there seems to be an underlying rejection mechanism that governs the preferential rejection of the HPO fraction by the PSF membrane that consequently led to high Rcp.

Table 5. Percentage of fouling resistance to the total resistance (R_T) by NOM fractional components of Sungai Ulu Pontian

FRACTION	Rm (%)	Rcp (%)	Rc (%)	Ra (%)
HPO	59	22	3	16
HPI	55	1.08	12.5	30.9
TPI	65	5.34	19	7

Table 6. Dominance fouling mechanism in NOM fractions as a function of hydraulic fouling resistance

FRACTION	PRIMARY FOULING MECHANISM
HPO	Concentration Polarization
HPI	Adsorption
TPI	Cake formation

This result also indicates that the hydrophobicity interaction between HPO fraction and PSF membrane were found to be less significant in affecting the adsorptive fouling although previous studies [2] claimed that intrinsically hydrophobic membranes tend to adsorb NOM and foul more than hydrophilic membranes. This phenomenon could be well explained from the high humic content and negative charge (due to carboxylic and phenolic moieties) of HPO fraction that resulted in a significant rejection by the negative charge of polysulfone membrane. Therefore it can be hypothesized that there are two dominant factors which are responsible in determining the HPO adsorption and type of fouling mechanism occurring. The factors are adsorptive behaviour (due to hydrophobicity property) and the electrostatic repulsion interaction (due to charge similarity with the membrane). HPO fraction possessed negative charge functional groups that prevent it from adhering or adsorbing onto the negatively charged membrane surface and therefore impacted in high Rcp. However in this experiment the HPO fraction did exhibit a significant adsorption fouling mechanism with adsorption resistance (Ra) 16% higher than TPI fraction. The Ra of HPO fraction showed higher value than TPI fraction (7%) despite its electrostatic interaction with the membrane. This scenario was believed to happen when the hydrophobic interactions was able to overcome the electrostatic repulsion incurred by the pressure gradient of suction process. Thus for the HPO fraction, the adsorption mechanism could be taken as the secondary influential fouling mechanism after the concentration polarization. In general it could be summarized that the fouling potential would be higher for the negative charge membrane when it is exposed to hydrophilic components compared to highly negative charge materials (HPO fraction) although they possessed high adsorptive tendency. The fouling mechanisms of the NOM fractions based on the hydraulic resistance can be disclosed as follows:

HPO Concentration > Adsorption > Cake formation

HPI Adsorption > Cake formation > Concentration Polarization

TPI Cake formation > Adsorption > Concentration Polarization

The responsible foulant that affects membrane performance was the HPI fraction which was found to perform the worst flux decline during membrane filtration. Furthermore the adsorption fouling can be taken as the primary fouling mechanism that governs the membrane flux decline as the HPI fraction had the highest Ra. Therefore in future application, NOM hydrophobicity or aromaticity (feed source characteristic) and charge interaction (membrane property) can be employed as a quantitative predictor for NOM rejection and flux declination of membrane filtration of surface water.

CONCLUSIONS

Based on the results of this experiment several conclusions can be drawn out as follows:

1) Each of the Ulu Pontian River NOM fractions caused substantial flux decline and the order of fouling potential based on flux decline was HPI > HPO > TPI
2) HPI and HPO fractions performed strong irreversible fouling due to their neutral property and hydrophobicity interaction with the membrane.
3) Charge interaction is a more influential factor than hydrophobicity and steric exclusion mechanisms in determining the rejection, fouling mechanisms and the extent of flux decline.

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