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Effect of coagulation-adsorption pretreatment on ultrafiltration membrane fouling to remove organic matter on leachate

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Abstract

Fouling is something that caused the limits toward the performance of ultrafiltration membranes (UF). The presence of organic matter in leachate water becomes the main cause of membrane fouling. Pre-treated of coagulation-adsorption becomes a solution in reducing fouling and improving UF membrane performances. The objective of this study was to investigate the effect of coagulation-adsorption pre-treatment on the removal of organic matter on leachate and to analyze the most appropriate fouling model to illustrate the hybrid coagulation-adsorption and UF membrane process. The coagulation process was carried out in the optimum condition with pH: 9 and 6g/L with $Al_2(SO_4)_3.18H_2O$, then in coagulation-adsorption process using equilibrium contact time for 60 min with variation of adsorbent dose (0,4;0,5; 0,6;0,7;0,8 and 0,9g/L) and then it was filtered using UF membrane for 60 min with pressure of 1 bar. The Modified Fouling Index (MFI) is an appropriate model to illustrate the formation of fouling in the hybrid coagulation-adsorption and UF membrane processes in the leaching of organic matter in leachate.

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Introduction

Leachate is a dangerous subsection that was produced by percolation of rainwater and decomposition of waste in landfills that contained large amounts of recalcitrant organic matters (Vilar *et al.*, 2011 and Thorneby *et al.*, 2006). Filtration used low pressure ultrafiltration (UF) membrane as a single process that can replace water treatment as conventional usage for waste water and clean water (Katsoufidou *et al.*, 2005 and Huang, 2007). However, the use of UF membranes in treating leachate has an obstacle that is the presence of membrane fouling. Fouling of the membrane may cause a decrease in permeate flux, high operating costs and maintenance costs (Amin *et al.*, 2004; Yu *et al.*, 2010; Gao *et al.*, 2011). Therefore it is important to minimize the occurrence of membrane fouling so that the economic process can run well (Jayalakshmi *et al.*, 2012).

The addition of coagulant before UF membrane is a popular pre-treatment in preventing membrane fouling and is the most successful technology in increasing flux in low-pressure membranes (Jung *et al.*, 2006 and Qiao *et al.*, 2008). However, not all can be set aside by using coagulation processes especially the hydrophilic fraction of the organic material, this hydrophilic substance has a low molecular weight can also cause a blockage of membrane pores. The combination of coagulation and adsorption used an activated carbon prior to UF membrane was able to exclude better organic matter in the prevention of membrane fouling (Lee *et al.*, 2006). Since fouling is just an observable effect of a large number of interacting processes which are often difficult to describe and identify, fouling models are traditionally focused on either a single or a few individual aspects of membrane filtration. According Zondervan *et al* (2008) developed a model able to predict the effects of irreversible fouling and chemical cleaning which can be used to optimise chemical cleaning cycle sequence in a membrane filtration. There are three mechanistic models that are typically used to describe fouling such as Modified fouling index model, pore blockage model and saturated curved model.

Modified Fouling Index (MFI) model is one way to determine the potential of fouling formation. MFI is derived from filtration cake theory, usually the measurement is carried out under constant pressure.

The MFI value was taken as the slope of the linear of the flow rate (t / V) to the volume accumulation curve (V) (Schippers *et al.*,1981 and Koo *et al.*, 2013). MFI can be calculated by the following equation (Boerlage *et al.*, 2002 and Nanda *et al.*, 2010):

$$\frac{t}{V} = t\,\frac{\eta R_m}{\Delta P A} + \frac{\eta a C_b}{2\Delta P A^2} \quad (1)$$

Which are t as the filtration time, P as the transmembrane pressure, η as the viscosity of water in the feed water, Rm as the membrane resistance, α as the specific resistance in the cake layer, *Cb* as the particle concentration in the raw water and *A* as membrane surface.Membrane pore blockage (pore block) is one of the important factors that caused a large filtering resistance that can decrease the flux rate under constant pressure and increase the pressure for constant flux conditions. The pore blocking model is used to see how quickly the blockage occurs in the membrane pores. Pore blocking is described as a linear gradient of t / V to t. The pore blocking value is determined from the general gradient of the filtration equation at constant pressure using the t / V plot against time (t) (Chinu *et al.*, 2010).

$$\frac{t}{V} = S_{pb}\cdot t + b \quad (2)$$

Where t is the filtration time, V is the filtration volume, S_{pb} is the pore compression with the critical time-the pore compression index (1/L) and b are constants. The saturated curve model is used to observe the saturation level of each feed water. The saturated curve model is described as a linear gradient from 1/V to 1/t. The slope and intersection curve of the inverse filtrate (1/V) with time inverse (1/t) is used to calculate the maximum accumulated filtrate and the resolution of filtration (Kf) to compare the fouling potential level of the dissolved organic material. According to the saturated curve model, the higher values of V_{max} dan K_f show the lower fouling potential of dissolved organic matter (Chuang, 2009; Fachrozi, 2013).

$$\frac{1}{V} = \frac{1}{V_{max}} + \frac{K_f}{V_{max}} \frac{1}{t} \quad (3)$$

Where t is the filtration time, V is the filtration volume and Vmax is the maximum filtration volume. This study was to investigate the effect of coagulation-adsorption pre-treatment on the removal of organic matter on leachate and to analyze the most appropriate fouling model to illustrate the hybrid coagulation-adsorption and UF membrane process.

Materials and methods

Material

The materials used in this study were leachate, aquadest, $Al_2(SO_4)_3.18H_2O$ pro-analytical (Merck) coagulant, Pro-Analyl (Merck) powder activated carbon, polysulfone (PSf), KCl (Merck), NN-imetylacetamide / DMAc (Merck), Polyethylene glycol 600 (Merck), NaOH dan H_2SO_4 0.1, the materials for the analysis of leachate organic matter include $KMnO_4$. Tools used are dead-end ultrafiltration cells, Jar Test Mascotte Flocculator FC-6, Daihan Scientific Digital Reciprocating Shaker SHR-2D hot plate, analytical balance, Compressor, UV visible Spectrophotometer Spectroquant Merck Pharo 300, Scanning Electron Miscroscope Jeol JSM 6360LA, pH meter, measuring cup, erlenmeyer, beaker glass, measuring flask, and burette.

Methods

Leachate Characteristics

The Lecheate samples that had been used in this study were taken from Cahaya Kencana landfill sites, Banjar District, South Kalimantan. Samples are stored and homogenized in a sealed container. Characterization of leachate was done by measuring parameters of pH, BOD, COD, TSS, DOC, UV_{254} absorbance, $KMnO_4$ organic substances.

Coagulation-Adsorption Hybrid Process and UF Membrane

The membranes used in this study were Ultrafiltration (UF) membranes of polysulfone polymer (PSf) that was modified with poly ethylene glycol (PEG) 600.

Composition and method refers to Tutriyanti's (2017) research by using inversion phase technique with impregnation of 0,5% (w/w) KCL concentration. The coagulation pretreatment experiment with a variation of pH (7; 7,5; 8; 8,5; 9; 9,5) to obtain the optimum pH that was followed by varying the coagulant dose (4,5; 5; 5,5; 6; 6, 5;7g/L) at optimum pH to obtain the optimum dose. Coagulation process performed at a speed of 100 rpm for 1 minute, and then, continued with a speed of 40 rpm and sedimentation, each for 30 minutes.

After coagulation process was finished, then the adsorption process will be done by variation of contact time (15, 30, 45, 60, 90, 150, 240 and 360min) to obtain the equilibrium contact time by varying the dose of activated carbon (0.4; , 5; 0,6; 0,7; 0,8; 0,9g/L). The adsorption process is stirred at a rate of 150 rpm.

The processed water is filtered by using vacuum filter to analyze the organic material content. The stage of coagulation-adsorption hybrid process and UF-PSf membrane is done by filtering processed water with UF-PSf membrane for 60 minutes, and every 5 minutes the permeate volume is calculated, using *dead-end* flow.

Analysis Techniques

Parameters that had been analyzed were DOC, UV_{254} absorbance, organic substance (KMnO4) and pH. DOC is measured using TOC analyzer (Shimadzu TOC-L). UV_{254} absorbance was measured using UV visible Spectrophotometer Spectroquant Merck Pharo 300. Membrane characterization was done by calculating membrane permeability value by measuring aquades flux with variation of operating pressure (1; 1,5; 2; 2,5; 3 bar), morphological analysis of membrane was done by using Scanning electron microscope (SEM) Jeol JSM 6360LA on the surface and cross-sectional view with 2500 x magnification.

Furthermore, membrane fouling modeling was analyzed using MFI model, Pore Blocking and Saturation Curve on each pretreatment of treated water.

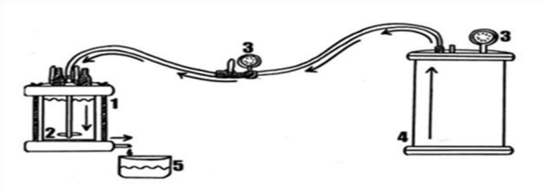

Fig. 1. Dead-end membrane filtration test system.

Results and discussion

Leacheate Samples Characterization

The results of leachate characterization analysis was taken at Cahaya Kencana Landfill site are presented in Table 1. The UV_{254} absorbance indicates the presence of organic matter content in leachate. The organic matter was detected by the UV_{254} absorbance, then it will be known if it is a reactive or aromatic organic material that has a double ring structure. Based on the result of UV_{254} absorbance, BOD, COD, and DOC analysis in table 1, it can be seen that leachate contains organic matter in high concentration. Low value of BOD_5/COD ratio was 0,395. This low ratio value indicates that the organic material contained in leachate is difficult to be biologically degraded. A lower comparative Fig. indicates a highly volatile organic material (Priambodho, 2005).

Table 1. Leachate Characterization.

No	Parameter	Satuan	I	II	Rata-rata
1	UV_{254}	1/cm	0,523	0,529	0,526
2	pH	-	7,38	7,41	7,395
3	DOC	mg/L	1.194,8	1.140,8	1.167,8
4	TSS	mg/L	206,0	221,0	213,5
5	BOD_5	mg/L	855,89	1.303,1	1079,495
6	COD	mg/L	2.165,4	3.296,9	2731,15
7	Organic Subtance $KMnO_4$	mg $KMnO_4$/L	450,3	450,3	450,3

Membrane Characteristics

UF Membrane Permeability

The permeability test on the UF membrane is done by looking at the fluxes, according to Mulder (1996) the flux is the amount of permeate volume that pass through the unit area of the membrane within a certain time with the force of thrust (pressure). The largest membrane flux values were found at 3 bar pressure which was 38,10 L / m². Hour.bar and the smallest flux value was at 1 bar pressure ie 6,46L/m². hour. bar. The value of the membrane flux is directly proportional to the operating pressure which means the greater pressure applied to the membrane, the flux that was gained also greater than before. The permeability number in this study is 15,865L/m². hour.bar refers to the literature (Mulder, 1996) it is in the range between 10 50L/m².hour. bar.

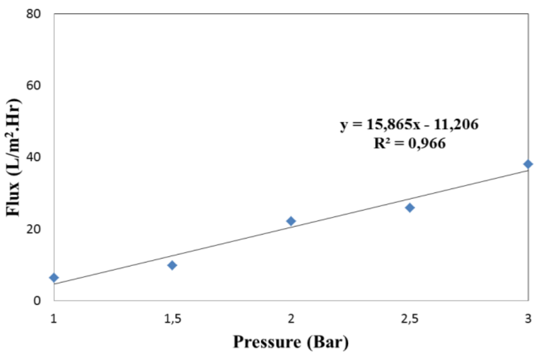

Fig. 2. Membrane Permeability.

Membrane Morphology Analysis

As we can see, the surface and cross section of the morphological results of the membrane are shown in Fig. s 3 and 4 (a and (b).

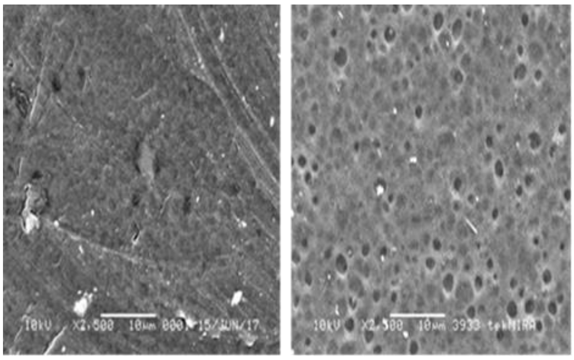

Fig. 3. Membrane surface with 2500 x magnificent (a) before filtration; (b) after filtration.

The result of the SEM image in Fig. 3 (a) shows the membranes that had been used in this study that has a dense pore, in Fig. 3 (b) the morphological structure of the upper membrane that had been filtered with the treated water. It looks more dirty than the initial membrane, because of the blockage of *foulant* or solid particles that can not be filtered by the membrane.

Fig. 4 (a) on this cross section, we can see the pore structure and the pore distribution of the membrane that was made. In the cross-section transverse the asymmetric membrane consists of two layers, in top layer is thin and dense and the bottom layer serves as a buffer and provide resistance to the membrane. This is because the exchange process of the solvent at the bottom of the membrane is much slower than the top of the membrane, It is blocked by the layers above it so that the resulting pore structure is larger, while Fig. 4 (b) shows the morphological structure of the side membrane after being used to filter processed water. The membranes that had been used have a greater thickness than the original membrane. The thicker membrane was exist due to the deposition of particles on the membrane or foulant surfaces that can not be filtered by the membranes and accumulate to form the cake layer.

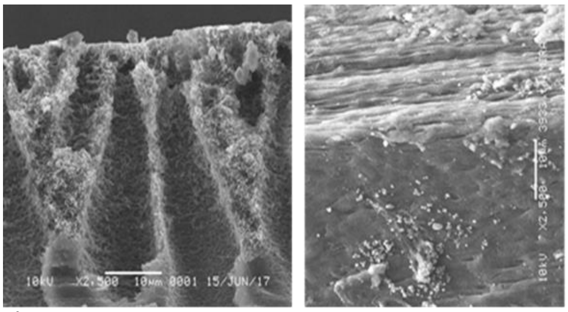

Fig. 4. Membrane cross section with 2500 x magnificent (a) before filtration; (b) after filtration

Coagulation-Adsorption Hybrid and UF Membrane Process

Determination of pH and Optimum Coagulation Dosage

On the research for coagulation process in determining optimum pH, variations are 7; 7,5; 8; 8,5; 9; 9,5. The pH adjustment was prepared with the addition of H_2SO_4 or NaOH and then $Al_2(SO4)_3$.$18H_2O$ coagulant was added with 6g/L dosage and leachate sample then stirred using flocculator at 100 rpm for 1 minute, 40 rpm speed for 30 min. In pH variation from 7 to 9,5, it can be seen the highest organic matter removal is pH 9 equal to 68,12% with UV_{254} absorbance while for parameter of KMnO4 at pH 9 got provision equal to 34,50%. However, for pH 9,5 was decreased the removal of organic matterl well indicated by UV_{254} absorbance and $KMnO_4$. The optimum pH of pH 9 was selected considering compared to the other removal at pH 9, the highest organic matter removal was obtained.

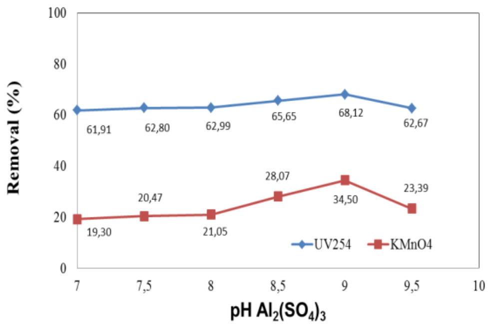

Fig. 5. pH ($Al_2(SO4)_3$.$18H_2O$ dose = 6g/L).

After the optimum pH was obtained, optimization dose was set up by adjusting the leachate pH to become optimum (pH 9). Then, 500 mL Leachate is inserted into 1 L beaker glass and coagulant of $Al_2(SO_4)_3$.$18H_2O$ with variation dose of 4,5g/L; 5g/L; 5,5g/L; 6g/L; 6,5g/L; 7g/L for each beaker glass, and stirred by flocculator at 100 rpm for 1 minute, and at 40 rpm for 30min.

At a dose of 4,5g/L to 6g/L that reached the highest removal level of UV_{254} absorbance about 68.12% and 34,50 % for organic substances of KMnO4. From 6g/L to 7g/L was decreased for both indications. That was happened due to the restabilization of the loading on the addition of $Al_2(SO_4)_3$ coagulant (Saputra, 2014). According to Pratiwi (2017) different levels of organic matterl removal are shown by the UV_{254} absorbance and $KMnO_4$ because of the different fractions. According to Mahmud *et al* (2013) organic matter fraction is dominantly set aside in the form of hydrophobic organic material with large molecular weight than hydrophilic.

In addition, according to Suslova *et al* (2014) $KMnO_4$ is able to oxidize various types of organic components whether it has a large molecular weight or a small molecular weight.

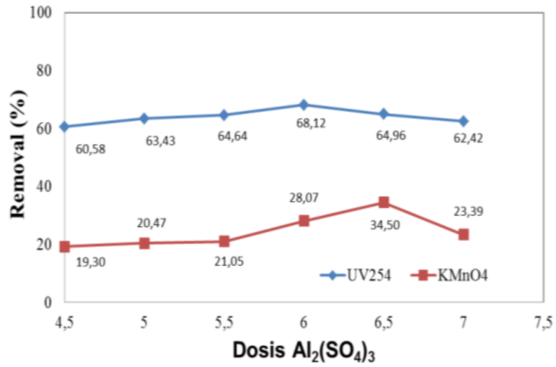

Fig. 6. Dosage $Al_2(SO_4)_3.18H_2O$ (pH 9).

Determination Time of The Equilibrium In Coagulation-Adsorption Process

Varying the contact time of 15; 30; 45; 60; 90; 150; 240; 360 minutes at optimum pH conditions and optimum coagulant dose to optimize the contact time. The pH condition and optimum coagulant dose based on variation of pH and dose on coagulation process is at pH 9 with dose of 6g/L. the adsorbent dose that had been used was 0,4g/L at each contact time and stirred at a rate of 150 rpm. The adsorption process with contact time variation reached equilibrium time at 60 minutes. At 60 minutes contact time, the UV_{254} absorbance removal is 96,13% and for parameter of KMnO4 is 91,35%.

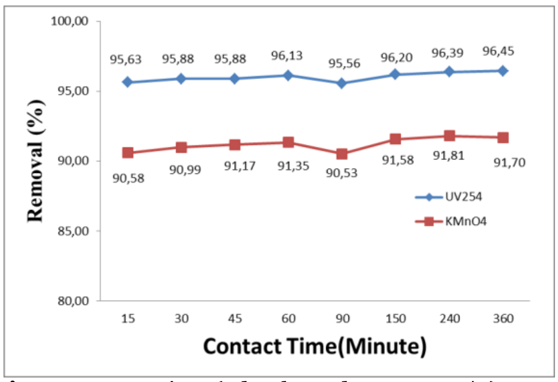

Fig. 7. Contact time (adsorbent dose = 0,4 g/L).

Along with increasing doses of activated carbon then larger the number of the UV_{254} absorbance and $KMnO_4$. Based on Fig. 7, the highest removal level was at a dose of 0,9g/L with a percentage of UV_{254} absorbance and $KMnO_4$ of 99,18% and 94,80% while the lowest removal was at a dose of 0,4g/L is only 96,13% for UV_{254} absorbance and 91,40% for $KMnO_4$. As the dose of adsorbent increased, the number of the removal was also increased the highest removal level at the dose of 0,4g/L to 0,5g/L of for UV_{254} absorbance is 97,91% and 92.69% of $KMnO_4$, for a dose of 0,6g/L to 0,9g/L the incremental rate of the inclusion tended to be smaller.

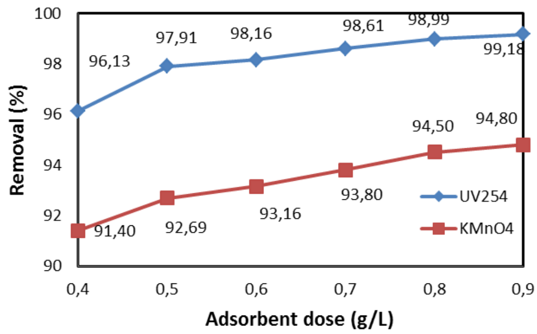

Fig. 8. Organic subtance removal at variation on adsorbent dosage (contact time = 60 mit).

The Analysis of Membrane Fouling Modeling

The Analysis of Membrane fouling Modeling using MFI is one of the ways to determine membrane fouling potential by calculating the mechanism of cake filtration formation on the membrane surface (Rachman, 2011). The potential for fouling by using MFI is done by plotting t / V on Volume (Chinu *et al*, 2010). The value of MFI is directly proportional to the formation of fouling. The larger the MFI gradient, the fouling is also greater, and vice versa (Rahma, 2017). Leachate that had been through the process of adsorption coagulation with adsorbent dose 0,9g/L has the smallest MFI value that is equal to 1.832.337,675 compared with other feed water. The small value of MFI on the pretreatment of coagulation-adsorption with a dose of 0.9g/L indicates that this pretreatment has the lowest potential in fouling (cake formation) formation on UF membrane while for the largest MFI value is in leachate without any treatment of 93.087. 536,310 which indicates with no-treatment leachate has the greatest potential in the formation of membrane fouling. Then, the MFI value is used as the data source in making the modeling curves as seen in Fig. 9.

Tabel 2. MFI Value.

Pretreatment		MFI	R^2
Without Pretreatment	Leachate	93.087.536,310	0,984
Coagulation	Coagulation	8.196.604,544	0,968
Coagulation-adsorptio with adsorbent dose (g/L)	0,4 g/L	5.855.799,232	0,999
	0,5 g/L	3.339.338,461	0,993
	0,6 g/L	2.430.539,071	0,994
	0,7 g/L	2.439.480,961	0,975
	0,8 g/L	3.104.445,469	0,963
	0,9 g/L	1.832.337,675	0,993

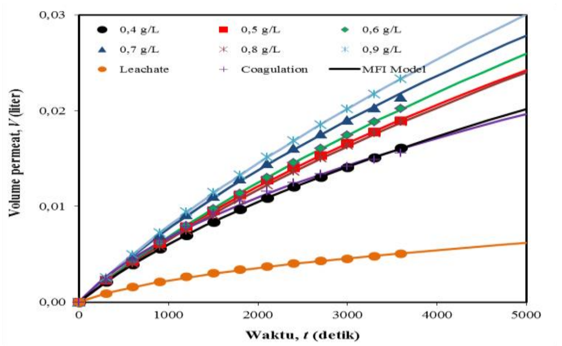

Fig. 9. MFI model from each pretreatment.

Pore blocking is one of the events that caused the fouling of UF membranes. Fouling due to pore blocking is caused by the organic material in the leachate water that filled the membrane pores to cover it and result in the limited permeate produced. According to Herwati (2013) the pore blocking model is used to see how quickly the blockage occurs in membrane pores. Pore blocking itself is described as a gradient t / V against t. The pore blocking value is directly proportional to the pore blockage rate of the membrane.

Tabel 3. Pore Blocking Value.

Pretreatment		Spb	R^2
Without Pretreatment	Leachate	114,391	0,997
Coagulation	Coagulation	32,730	0,999
Koagulasi-Adsorpsi dengan dosis adsorben (g/L)	0,4 g/L	24,361	0,992
	0,5 g/L	16,819	0,994
	0,6 g/L	13,096	0,984
	0,7 g/L	14,166	0,996
	0,8 g/L	15,498	0,946
	0,9 g/L	11,442	0,997

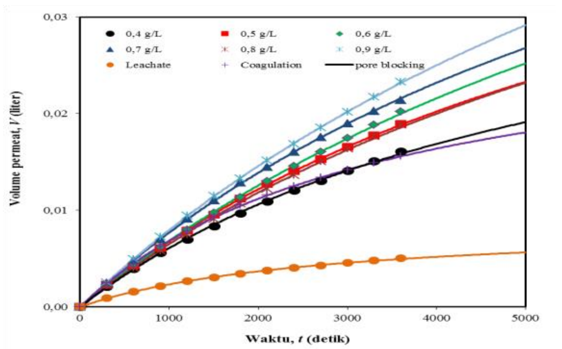

Fig. 10. Pore blocking model from each pretreatment.

The pore blocking values of each feed water vary from one another, leachate without treatment has the greatest pore blocking value that indicates the organic material is potentially great to give UF membrane pore blockage. For feed water that was through coagulation-adsorption process with dose 0,9g/L has the smallest pore blocking value that is equal to 11,442, it shows that pretreatment coagulation-adsorption with dose of adsorbent is equal to 0,9g/L and has the smallest potency in causing pore blocking of the membrane. We can make a model based on this Pore blocking. The modeling of the pore blocking curve can be made from the pore blocking value for each feed water.

Another thing that could potentially lead to fouling is the occurrence of saturation levels in the membrane. The saturation level of the membrane represents the rate of cake formation in the membrane's upper layer, can caused a kind of accumulation of previous pore blocking, and resulting faster fouling formation, and impeded performance on UF membranes (Fachrozi, 2013). The formation of the cake in membrane saturation is characterized by the decreasement of flux value over the time of operation that was caused by fouling. Saturation curve modeling or saturation curve can be made on the basis of the saturation curve values. The large values' number of Vmax and Kf in Table 4 indicated that these pretreatments have little potential for membrane saturation levels compared to other pretreatments. Pretreatment of coagulation-adsorption with a dose of 0,9 g / L in this study has the highest Vmax and Kf values, so this pretreatment is really potential to cause the lowest membrane saturation if we compared that with other pretreatments. The modeling curve for *saturation curve* can be seen in Fig. 11. It shows that the rate of cake filtration formation produced by feed water through pretreatment of coagulation-adsorption at a dose of 0.9g/L is smaller than other treatments. This shows that in the coagulation-adsorption process with the adsorbent adsorbent 0,9g/L, the smallest membrane saturation is really potential among other pretreatments. This membrane saturation level modeling curve shows the potential of each pretreatment in the event of saturation level in the UF membrane.

Table 4. Saturated Curve Value.

Pretreatment		Vmax	Kf	R^2
Without Pretreatment	Leachate	0,008	40,102	0,999
Coagulation	Coagulation	0,030	55,722	1,000
Koagulasi-Adsorpsi dengan dosis adsorben (g/L)	0,4 g/L	0,037	83,024	1,000
	0,5 g/L	0,053	112,547	1,000
	0,6 g/L	0,063	134,067	1,000
	0,7 g/L	0,076	149,745	1,000
	0,8 g/L	0,045	96,476	0,998
	0,9 g/L	0,091	174,321	1,000

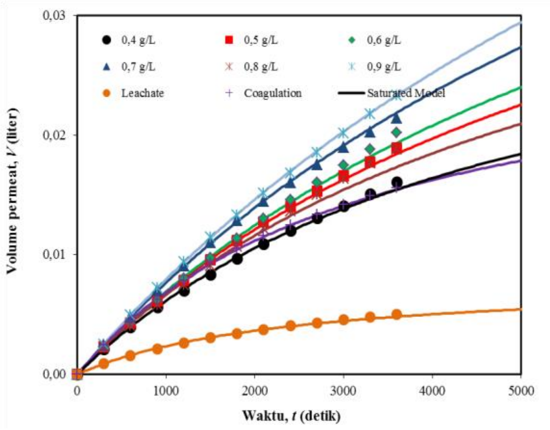

Fig. 11. Saturated curve model from each pretreatment.

Conclusions

This research was conducted to investigate effect of coagulation-adsorption pre-treatment on the removal of organic matter on leachate and to analyze the most appropriate fouling model to illustrate the hybrid coagulation-adsorption and UF membrane process.

The coagulation-adsorption pretreatment process can remove the organic matter in leachate 99,18% for UV_{254} absorbance and 94,80% for KMnO4 organic substances. All of the modeling that had been used in hybrid coagulation-adsorption and UF membrane processes are capable to represent membrane fouling but the most appropriate fouling modeling represents the fouling potential in the coagulation-adsorption hybrid process and UF is MFI (Modified Fouling Index) modeling at 0,4g/L of adsorbent dose with R2 = 0,999.

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