NST 2021_The Effect of Flow Rate of Adsorption Pretreatment in Removing Total Organic Carbon (TOC) Based on the SEM-EDX Test

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The Effect of Flow Rate of Adsorption Pretreatment in Removing Total Organic Carbon (TOC) Based on the SEM-EDX Test

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ABSTRACT

Adsorption is an accumulation of contact between gas or liquid molecules with a solid surface and some molecules attached to the surface. This study uses adsorption as a pretreatment of membrane processing. The adsorption process is affected several factors, including the flow rate used in the adsorption process. This study aimed to determine the effect of water flow rate on the adsorption process in removing Total organic Carbon (TOC) using Calgon and Haycarb Adsorbent. The characteristics of the wastewater used are effluent of wastewater treatment from alcohol production that remains contains Total Organic Carbon (TOC). Variation of the flow rate used is 39 mL/minute, 29 mL/minute, and 23 mL/minute. The effect of flow rate on adsorption pretreatment in removing Total Organic Carbon (TOC) on Calgon adsorbent showed that 23 mL/minute was the most effective in reducing TOC values. On the other hand, using Haycarb absorbent on 23 mL/min flow rates was less effective in removing TOC values. The Haycarb adsorbent effectiveness is shown by the morphology of the initial and final results of EDX, where the adsorbent pore of Haycarb is not filled with absorbate.

Keywords: Wastewater, Total Organic Carbon (TOC), pretreatment, adsorption, flow rate

Introduction

The production of alcoholic beverages and soft drinks produces liquid waste with a high volume and pollution level, negatively impacting the environment. The characteristics of liquid waste in the alcoholic beverage and soft drink industries vary greatly in volume, concentration, and pH, influenced by detergents and a small amount of salt. The high volume of liquid waste and the demand for clean water with good quality happen to the re-utilization of effluent from the WWTP for the alcoholic beverage and soft drink industry. One of the WWTP effluent wastewater characteristics can be seen in total organic carbon (TOC), which has 99 mg/L (Prabawaty, 1988). Therefore, a further treatment process is needed to remove several parameters in wastewater before using the water to produce alcoholic beverages and soft drinks.

Membrane technology is one of the right solutions to meet the need for quality clean water. Ultrafiltration (UF) is a type of membrane whose operation is relatively easy to remove contaminants from water (Prasetyo, 2015). The difference between the ultrafiltration membranes (UF) and other types of membranes such as microfiltration (MF), nanofiltration (NF), and reverse osmosis (RO) is the particles size can be retained. In the membrane, the pressurized liquid flows and is in contact with the membrane. The solvent and specific components then diffuse through the membrane, and the remaining particles larger than the membrane pores will be retained, while the smaller particles will pass through so that the most influential factor on membrane

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rejection is the size and shape of the particles relative to the pore size of the membrane (Wardani, 2013).

The risk of fouling on the membrane can be minimized by giving pretreatment before the membrane process (pretreatment) (Nainggolan, 2015). Various factors need to be considered in selecting a pretreatment process that can reduce contaminants and fouling, such as coagulants, adsorbents, oxidants, and temperature (Prasetyo, 2015). Adsorption is the accumulation of contact between gas or liquid molecules with a solid surface, and some of these molecules are attached to the solid surface (Ginting, 2008). The molecules that have been attached to the solid are in an unbalanced state because the cohesion force is greater than the adhesion force. The imbalance between the two forces causes the solid to attract other substances in contact with its surface. The concentration of substances attached to the solid surface was called the adsorbed phase or adsorbate, while solids that can absorb substances are called adsorbents (Pabhassaro, 2008).

Adsorption is the pretreatment option because it refers to the adhesion of fouling to the adsorbent surface. Due to the relatively high porosity, the adsorbent has a fairly large specific surface area for absorption or accumulation of absorbable impurities. The efficiency of the adsorption process as a pretreatment depends on several factors, including the adsorbent dose, type of adsorbent, adsorbent size, water characteristics, and operating mode (Arhin et al.,2016). The adsorption process treats wastewater containing heavy metals, chemicals, pesticides, and herbicides (Shanmugasundaram & Sudalaimani, 2012). Through the adsorption process, the contaminants from the wastewater are transferred to the surface of the adsorbent.

The adsorption process in the industry usually uses activated charcoal and ion exchange resins for water purification (Shanmugasundaram & Sudalaimani, 2012). From the results of previous studies, adsorption pretreatment on UF can increase flux (Rahma et al., 2018). In addition, as well as from the research results of Monnot (2016) that pretreatment adsorption can improve performance by reducing the resistance to UF fouling.

Based on the molecular interactions between the adsorbent and the adsorbate, adsorption can be divided into two types, namely:

1. Physical Adsorption
   Physical adsorption is where the adsorbate is attached to the surface only through the Van Der Waals attraction (Kumar & Singh, 2017). Physical adsorption occurs when the attractive force between the adsorbent and the adsorbate is weak, which can cause the adsorbate to move from one surface to another (Pabhassaro, 2008).
   Physical adsorption is a reversible event. If one condition changes in a state of equilibrium, some of the adsorbates will be released and form a new equilibrium (Pabhassaro, 2008).
   In physical adsorption, there will be the formation of a multilayer layer on the surface of the adsorbent because physical adsorption occurs without the need for activation energy (Pabhassaro, 2008).
2. Chemical Adsorption
   Chemical adsorption occurs due to the formation of covalent and ionic bonds between the adsorbate molecules and the adsorbent surface, which can cause the formation of a monolayer layer. This chemical adsorption-desorption event requires energy.

In general, there are three types of adsorbents commonly used, namely:

1. Silica Gel
   In the desorption of contaminants, silica gel requires a low temperature. The desorption ability of silica gel will increase with increasing temperature. Silica gel is made from chemically bonded silica which contains approximately 5% water. In general, the maximum working temperature of silica gel is 200°C. If the temperature is more than 200°C, the adsorption process on silica gel will decrease (Ginting, 2008).

2. Activated Carbon
   Activated carbon is a porous solid that has changed its physical and chemical properties due to activation by heating at high temperatures (Arif et al., 2015). There are two kinds of activated carbon, namely powder and granular (Ginting, 2008). Powdered activated carbon is usually smaller than 0.18 mm, used for drinking water treatment, decolorization, sugar refiner, and citric acid purification. Meanwhile, activated carbon is in granular form with 0.2 – 5 mm used for water treatment, wastewater treatment, groundwater treatment, and deodorizing. Activated carbon contains carbon, hydrogen, and oxygen, chemically bonded to various functional groups such as carbonyl, carboxyl, phenol, lactone, quinone, and ether groups. Functional groups are formed during the activation process by interacting free radicals on the carbon surface with atoms such as oxygen and nitrogen. This functional group makes the activated carbon surface chemically reactive and affects its adsorption properties.
3. Zeolite
   Zeolite is a hollow mineral produced from hydrothermal processes in alkaline igneous rocks. In addition, dehydrated zeolite crystals are selective adsorbents and have high adsorption effectiveness (Ginting, 2008).

The research results from Monnot et al. (2016) prove that the flow rate affects the removal of natural organic matter in the adsorption process. From this research, the flow rate of 2.4 liters/hour was proven to have 78% natural organic matter compared to another more significant flow rate. Therefore, a minor flow rate variation was chosen in this study than in the previous study from Monnot et al. (2016). The flow rate used in this study is 39 mL/minute, 29 mL/minute, and 23 mL/minute. This study was conducted to determine the effect of various flow rates on TOC removal efficiency.

Research Method

In this study, adsorption combination processing using samples from the outlet channel of WWTP PT. Multi Bintang Indonesia, Tbk. The main parameter used in this study is Total Organic Carbon (TOC). This study was conducted to determine the best flow rate variation in the adsorption process using Calgon and Haycarb Adsorbent to improve as pretreatment process's performance before the membrane process. The reactor system is illustrated in Figure 1.

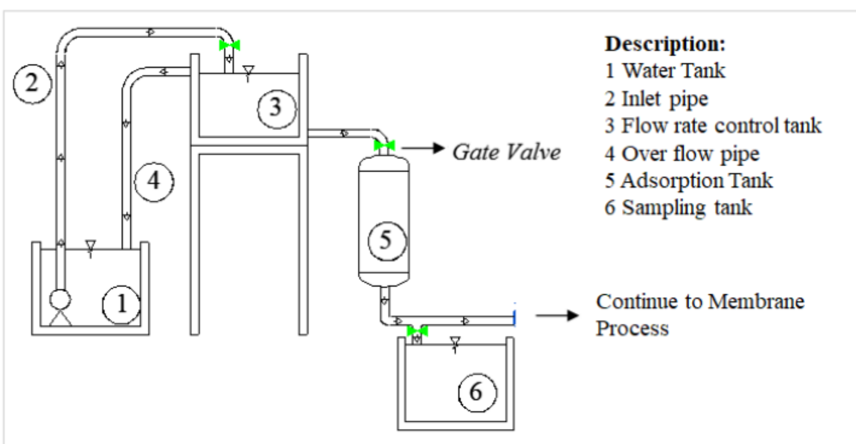


Figure 1. Reactor system

This study analyzes TOC (SNI 06-6989.28-2005) and analyses the morphology, topography, and elemental mapping using SEM EDX (FEI type Inspect-S50). SEM is a tool capable of photographing a surface with magnification from 20 to 100,000 times. The working principle of SEM is that high-energy electrons shoot the sample's surface with kinetic energy between 1–25 kV, the electrons that directly hit the sample are called primary electrons, while the electrons reflected from the sample are called secondary electrons. Lower energy secondary electrons are released from the atoms on the sample's surface and will determine the shape of the sample. The image formation in SEM comes from the electron beam that is reflected on the sample surface. The difference in wavelength from this lighting source results in different levels of resolution that can be achieved. SEM-EDX can also be shown the elemental content in the sample by looking at its surface structure, the elemental content that can be detected ranges from Beryllium to Uranium.

Result and Discussion

The type of wastewater that is used as a sample was from PT.Multi Bintang Indonesia Tbk. Samples were taken from the outlet channel of the wastewater treatment plant, and then the samples were stored in jerry cans for further processing into clean water by processing with adsorption as pretreatment. The wastewater contains a Total Organic Carbon (TOC) of 343 mg/L. The wastewater sample used in this study had a TOC value of 343 mg/L, then after the adsorption pretreatment process with Calgon adsorbents and Haycarb adsorbents at various flow rates resulted in a decrease in the TOC value. This decrease in TOC value indicates that the adsorption pretreatment can absorb organic matter well. The decrease in TOC value on Calgon adsorbent continued from 0 minutes to 5 hours sampling time for all flow rate variations. However, the TOC value increased for all flow rate variations when the adsorption water mixture. The increase in TOC value was due to the adsorption water in the tank, experiencing mixing from the initial adsorption results for up to 5 hours. In contrast with the Calgon adsorbent, the increased value of TOC in the Haycarb adsorbent so when the time is sampling 5 hours. Indicating each adsorbent has time saturation, each of which may affect adsorption capacity. It is that time sampling 5 hours is when the adsorbent Haycarb already started to enter the phase of saturation, thus causing the rise in the value of TOC. Correlation of flow rates to adsorption pretreatment in removing Total Organic Carbon (TOC) on Calgon adsorbent illustrated in Figure 2 below.

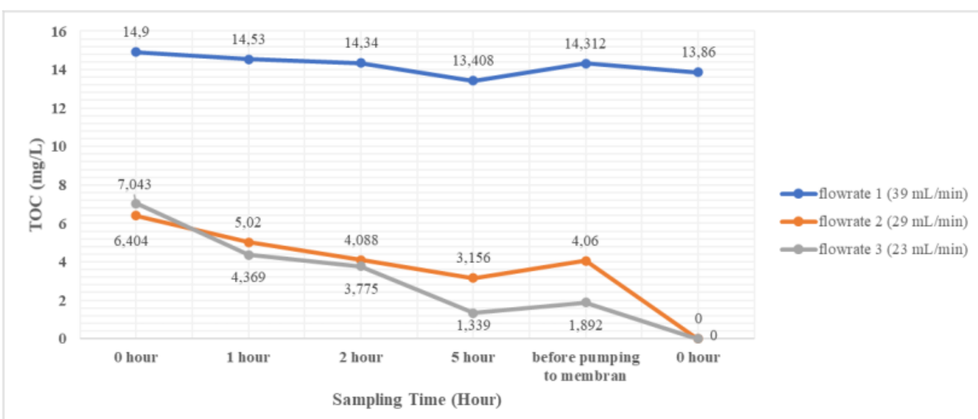
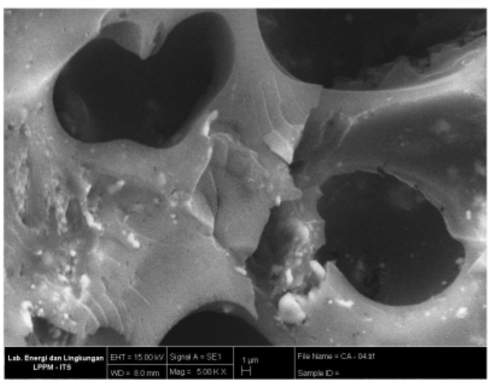
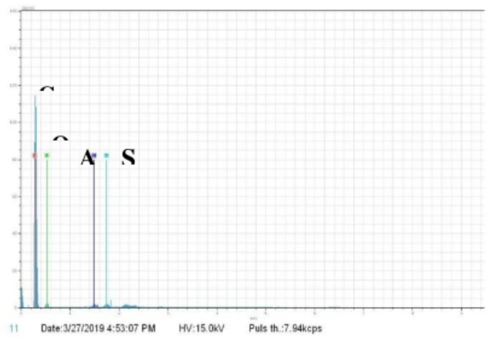


Figure 2. Correlation of flow rates to adsorption pretreatment in removing Total Organic Carbon (TOC) on Calgon adsorbent

The effect of flow rate on adsorption pretreatment in removing Total Organic Carbon (TOC) on Calgon adsorbent is shown in the graph in Figure 2 above. The graph shows that flow rate 3, equal to 23 mL/minute, is the most effective in decreasing the TOC value. The decrease in TOC value is because the smaller the flow rate, the greater the residence time in the adsorption process. The longer the residence time, the higher the ability of the adsorbent to adsorb the adsorbate. Flow rate 2 (debit 2) has a higher removal rate at 2 hours process but then flow rate 3 (debit 3) got better removal rate at 5 hours process. Flow rate 1 (Debit 1), as in Figure 2, shows that has less effective for adsorption in removing Total Organic Carbon (TOC) than the other two types of flow rate.

The SEM-EDX test of Calgon adsorbent strengthened this condition at the lowest flow rate. It can be seen in Figure 3, where the images are a morphology early surface adsorbent Calgon before passing through wastewater to the process of adsorption. The image was taken at 5000X magnification. It can be seen that the pores of the adsorbent are still open, with an average pore diameter of 21.5 m.





El	AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error [%]
C	6	K-series	81.38	81.38	85.71	24.9
O	8	K-series	17.33	17.33	13.70	5.8
Si	14	K-series	0.69	0.69	0.31	0.1
Al	13	K-series	0.60	0.60	0.28	0.1
		Total:	100.00	100.00	100.00	

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Figure 3. Initial morphology of Calgon adsorbent with a magnification of 5000X

Figure 4. The results of observations of EDX on an adsorbent Calgon before bypassed water waste

When adsorbent Calgon has been bypassed by water waste to the process of adsorption, directly absorbate will stick to the pores of the adsorbent. Quantitative analysis was also done on the Calgon adsorbent that has not been passed by wastewater. The results of the observations can be seen in Figure 4 at the bottom of this. Based on Figure 4 above, note that Al and Si are found on the adsorbent Calgon before bypassed water waste to the process of adsorption with percentages respectively amounted to 0.28% and 0.31%. They were initially open to be closed and filled with adsorbents following Figure 5 below.

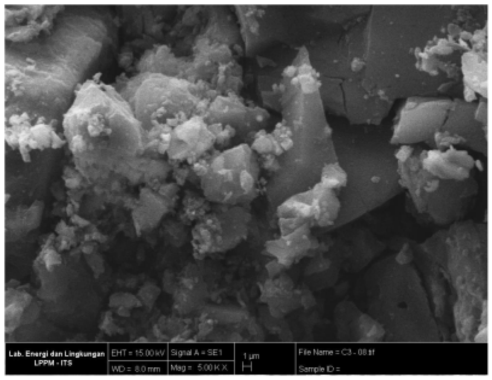


Figure 5. Final morphology of Calgon adsorbent with a magnification of 5000X

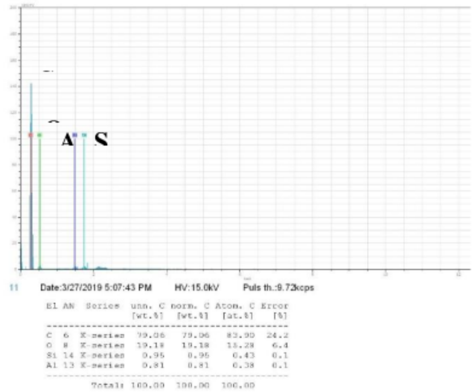


Figure 6. The results of observations of EDX on an adsorbent Calgon after bypassed water waste

Although it has been known that porous adsorbent Calgon has been charged after bypassing water waste in the process of adsorption, we need to do more tests to know which elements fill the adsorbent. Tests were carried out to quantify using EDX. The examiner's results can be seen in Figure 6.

Based on Figure 6, note that Al and Si are also found on the adsorbent Calgon who has passed the water waste that is then dried. Following the observation results, note that the content of Al and Si, respectively, amounted to 0.38% and 0.43%. Values are experiencing an increase compared with the adsorbent Calgon before bypassing water waste. These results indicate that the pores on the Calgon adsorbent are filled with organic matter and Al and Si elements. On the Haycarb, adsorbent flow rate also affects the adsorption process in removing Total Organic Carbon (TOC). Correlation of flow rate to adsorption pretreatment in removing Total Organic Carbon (TOC) on Haycarb adsorbent illustrated in Figure 7 below. As seen in Figure 7 below, all three various flow rates types fluctuate after 2 hours of the process.

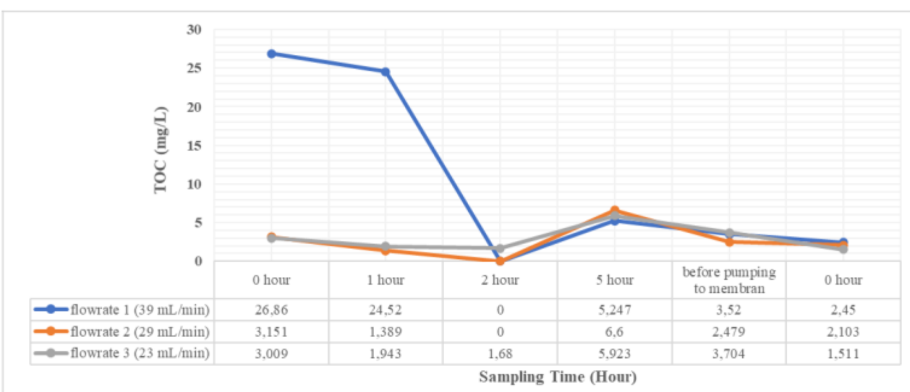


Figure 7. Correlation of flow rate to adsorption pretreatment in removing Total Organic Carbon (TOC) on Haycarb adsorben

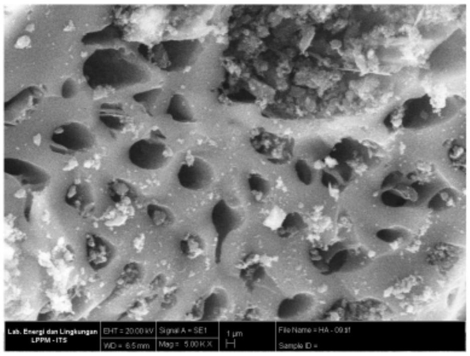
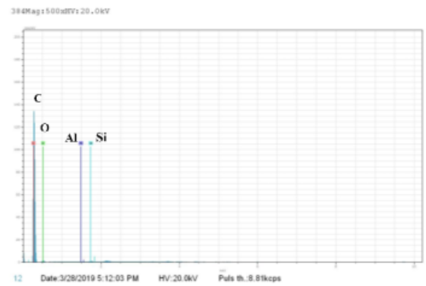


Figure 8. Initial morphology of Haycarb adsorbent with 5000X magnification



El	AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error [%]
C	6	K-series	87.43	87.43	90.31	26.7
O	8	K-series	12.39	12.39	9.61	4.4
Si	14	K-series	0.18	0.18	0.08	0.0
Al	13	K-series	0.00	0.00	0.00	0.0
		Total:	100.00	100.00	100.00	

Figure 9. The results of observations of EDX on an adsorbent Calgon has not passed water waste

The effect of flow rate on the adsorption pretreatment in removing Total Organic Carbon (TOC) on the Haycarb adsorbent is shown in the graph in Figure 7 above. Graphs show that flow rate 3 (debit 3), at 23 mL/min, is less effective in decreasing the value of TOC. This condition can be proven by the initial and final morphology of the Haycarb adsorbent, along with the EDX graph. Based on Figure 8, the adsorbate does not occupy the pores in the adsorbent Haycarb perfectly. It indicates the cause of the value of TOC suffered fluctuating.

Quantitative analysis was also shown through the EDX test on the Haycarb adsorbent before passing through the wastewater. The results of the observations can be seen in Figure 9 above this. Based on Figure 9 above, it is known that only Si was found in the Haycarb adsorbent before the wastewater passed for the adsorption process with a percentage of 0.08%. When the Haycarb adsorbent has passed by wastewater for the adsorption process, it is seen that not all the adsorbent pores are filled with an adsorbate. It can be seen in Figure 10 that are still no pores filled.

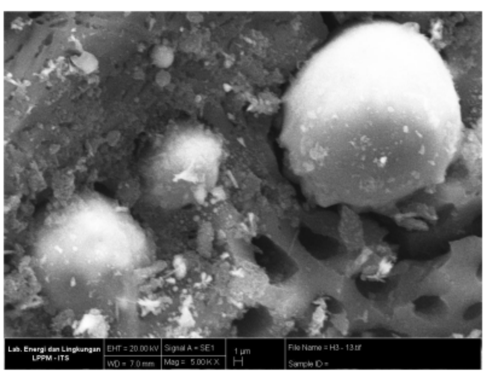
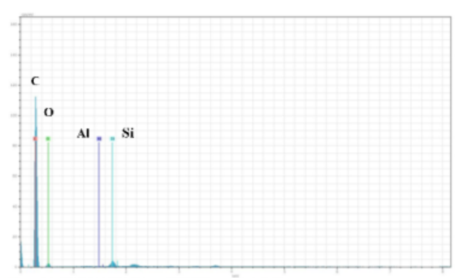


Figure 10. Final morphology of Haycarb adsorbent with 5000x magnification



El	AN	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error [%]
C	6	K-series	79.56	79.57	84.17	24.4
O	8	K-series	19.27	19.27	15.30	6.6
Si	14	K-series	1.17	1.17	0.53	0.1
Al	13	K-series	0.00	0.00	0.00	0.0
		Total:	100.00	100.00	100.00	

Figure 11. The results of observations of EDX on an adsorbent Haycarb after bypassed water waste

Condition of the adsorbent pores that are not filled perfectly, indicating that the TOC attached to the porous adsorbent is not much, just the elements Si are much attached to the porous adsorbent. It is proven back with the percentage of Si which experienced an increase from prior bypassed water waste, which became 0.53% following Figure 11 above.

Conclusion

This study uses flow rate variation to show the Total Organic Carbon (TOC) removal effectiveness on the pretreatment process that utilizes adsorbent. Variation of the flow rate used is 39 mL/minute, 29 mL/minute, and 23 mL/minute and using two types of adsorbent that Calgon and Haycarb. Flow rate affects the removal of Total Organic carbon in the pretreatment process that utilizes adsorbent both Calgon and Haycarb. As in Calgon adsorbent, the lower flow rate refers to flow rate 3 (debit 3) as 23 mL/minute, which was more effective in Total Organic Carbon (TOC) removal than two other types of flow rate.

The effect of flow rate on the adsorption pretreatment in removing Total Organic Carbon (TOC) on the Haycarb adsorbent is shown that flow rate 1 (debit 1), at 39 mL/min, is more effective in the removal of Total Organic Carbon (TOC). Based on research conducted (Monnot et al. 2016), adsorption can minimize the occurrence of fouling, so that based on this research, the other research related to adsorption as a pretreatment.

Acknowledgment

The author would like to thank PT. Multi Bintang Indonesia, Tbk that allowed us to use the outlet channel of WWTP as the sample in this study. The Environmental Engineering study program, Faculty of Engineering, University of Pembangunan Nasional "Veteran" Jawa Timur, Indonesia, supports this article to happen.

References

Arif, A. R., Saleh, A., & Saokani, J. (2015). Adsorpsi karbon aktif dari tempurung kluwak (*Pangium edule*) terhadap penurunan fenol. *Al-Kimia, 3*(1), 34-47.

Arhin, S. G., Banadda, N., Komakech, A. J., Kabenge, I., & Wanyama, J. (2016). Membrane fouling control in low-pressure membranes: A Review on pretreatment techniques for fouling abatement. *Environmental Engineering Research, 21*(2), 109-120.

Ginting, F. D. (2008). *Pengujian alat pendingin*. Jakarta: Universitas Indonesia.

Kumar, L., & Singh, S. (2017). *Column study for chloride removal from waste water by a low-cost adsorbent (Bio adsorbent).*

Monnot, M., Laborie, S., & Cabassud, C. (2016). Granular activated carbon filtration plus ultrafiltration as a pretreatment to seawater desalination lines: Impact on water quality And UF Fouling. *Desalination, 383,* 1-11

Nainggolan, R. P. (2015). *Pengendalian fouling pada sistem pengolahan air berbasis membran.*

Pabhassaro, D. (2008). *Pengembangan model adsorpsi tekanan tinggi terhadap gas metana untuk memprediksi potensi coalbed methane Indonesia sebagai sumber energi baru.* Jakarta: UI Press.

Prabawaty, D. H. (1988). *Optimasi proses biologis pada pengolahan air buangan industri bir menggunakan RBC (Rotating Biological Contractors) di PT. Multi Bintang indonesia, Surabaya.*

Prasetyo, H. A. (2015). Pengendalian fouling pada operasi membran Ultrafiltrasi (UF) untuk produksi air minum. *Chemical Engineering, 1*(2), 1-5.

Rahma, A., Mahmud, M., & Abdi, C. (2018). Pengaruh pra-perlakuan adsorpsi karbon aktif terhadap fouling membran Ultrafiltrasi Polisulfon (Uf-Psf) Pada penyisihan Bahan Organik Alami (BOA) air gambut. *Jernih: Jurnal Tugas Akhir Mahasiswa Program Studi Teknik Lingkungan, 1(02*), 1-5.

Shanmugasundaram, M. M., & Sudalaimani, D. K. (2012). A study on natural adsorbents for the removal of chloride ion in water. *International Journal of Engineering Research & Technology (IJERT), 1*(5), 1-5.

Wardani, A. K. (2013). *Pengaruh aditif pada pembuatan membran ultrafiltrasi berbasis polisulfon untuk pemurnian air gambut*. Bandung: Institut Teknologi Bandung.

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