

WASTEWATER TREATMENT OF NPK FERTILIZER INDUSTRY USING SEQUENCING BATCH REACTOR AND GRANULAR ACTIVATED CARBON

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ABSTRACT

The wastewater of the NPK fertilizer industry contains COD, TSS, PO₄, and Total-N pollutants that can contaminate water bodies if the treatment process is not carried out first. In this study, the NPK fertilizer industry waste treatment was carried out using sequencing batch reactors (SBR) and granular activated carbon (GAC) by adding the coagulation-flocculation process as a pre-treatment so that wastewater can meet the established quality standards. In this study, the reactors were operated at HRT 12, 24, and 48 hours using a GAC mass concentration of 0.5, 1, and 2 g/L. The results show the highest reduction of COD parameters by 67%, TSS by 82%, PO₄ by 45%, Total-N by 76%, optimal HRT by 48 hours, and an optimal GAC mass concentration of 2 g/L. The aeration rate is operated at 14 L/min, with a PAC concentration of 3 g/L being optimal.

Keywords: NPK Fertilizer Industry Waste, SBR-GAC, Coagulation-Flocculation, HRT

1. INTRODUCTION

Fertilizer is a material added to a plant or plant to satisfy the necessary nutrients so that it can produce well. According to Rao 2010, it is explained that the main nutrients required by plants are nitrogen (N), phosphorus (P), and potassium (K). NPK fertilizer is a liquid or solid artificial fertilizer in the form of rough grains containing the main elements of nitrogen, phosphorus, and potassium (BSI 2010). Despite its benefits, the chemical properties that result from fertilizer manufacturing inputs and outputs necessitate gradually reducing environmental impact.

The NPK fertilizer industrial wastewater can pollute the waters due to its high organic content, low acidity, and presence of elements such as nitrogen (N), phosphorus (P), and

potassium (K), so it needs treatment before being dumped into the river basin. The negative effects of fertilizer production on the environment require monitoring of the manufacturing, application, and disposal processes to maintain the environment. If the waste is not properly managed and is directly dumped on the water body, it will seriously disrupt the surrounding environment. Some industries that will dispose of the waste are required to treat it first to prevent environmental pollution in the surrounding environment. Many studies on the effects of fertilizer use have been conducted but little attention has been paid to the pollution caused by the production and disposal of fertilizer plant waste.

Sequencing Batch Reactor (SBR) is a wastewater treatment process unit of activated sludge process development technology that uses suspended microorganisms with an aeration and sedimentation system that is also performed on the SBR operation, where all processes are carried out sequentially in the same tank. In this paper, we consider hydraulic retention time (HRT) as one of the important parameters because it states the length of time the microbe is present in the reactor and the sequence of phase stages in SBR that occur in one cycle. The addition of granular activated carbon (GAC) activated carbon media, which can leave the dissolved organic matter in water, was used in this SBR operation to implement developments in liquid waste treatment (Putra 2016).

Based on this, the experiment was conducted on the process of treating wastewater from the NPK fertilizer industry using SBR-GAC with coagulation and flocculation as a pre-treatment. When HRT and the optimal GAC mass are determined, the removal efficiency of organic matter in wastewater can be maximized.

2. METHOD

Preparation of Tools and Materials

Tools : Reactor, Air pump, Aerator pipe and Laboratory instruments.

Materials : Fertilizer industry wastewater, GAC, and PAC.

The SBR-GAC reactor design made in this study to treat fertilizer industrial waste is as follows.

Table 1. Reactor Design Details

No	Design	Unit	Score
Coagulation-Flocculation Reactor			
1	Reactor Volume		
	Volume Total	L	20
	Working Volume	L	19
	Floc Volume	L	1
	Waste Volume	L	18
2	Reactor Dimension		
	Diameter	cm	30
	Height	cm	30
3	Coagulan Concentration	g/L	3, 6
SBR-GAC Reactor			
1	Reactor Volume		
	Volume Total	L	5
	Working Volume	L	3
	Total Activated Sludge	L	1,5
	Waste Volume	L	1,5
2	Reactor Dimension		
	Diameter	cm	20
	Tinggi	cm	21
3	Retention Time	Hours	12, 24, 48
4	Adsorben Concentration	g/L	0,5 ; 1 ; 2

Source: Research, 2022

The visualizations of the SBR-GAC reactor design in the figure are as follows:

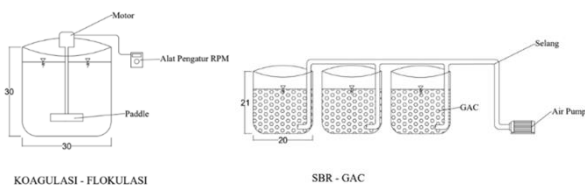


Figure 1. Reactor Illustration

Source: Research, 2022

Coagulation-Flocculation as Pre-Treatment

In the SBR-GAC process, before the wastewater is introduced into the main reactor. Wastewater first goes through the process of processing using the coagulation-flocculation

method to determine the optimal concentration of PACs, namely 3 g/L and 6 g/L (Sirianuntapiboon and Manoonpong 2001). The Jar Test method was used in this experiment, with a wastewater volume of 1 liter at each predetermined PAC concentration. This was done to determine the optimum dose before main the treactor, which is the coagulation-flocculation process with a total working volume of 20 liters.

The results of the Jar Test method show the formation of flakes in samples with a PAC concentration of 3 g/L, and at a concentration of 6 g/L the sample shows coagulants, and homogeneous wastewater is also not formed. In addition, the percentage value of TSS decreases at PAC concentrations of 3 g/L and 6 g/L respectively by 47% and 22%. This suggests that a better PAC concentration of 3 g/L was used in the coagulation-flocculation process in this study. Based on the coagulation-flocculation experiment using the Jar Test method above, it is known that the optimum PAC concentration used in the coagulation-flocculation process as a pre-treatment of fertilizer industrial waste is 3 g/L.

SBR-GAC process

Before beginning the SBR-GAC process, a seeding process was carried out to achieve a sufficient MLSS value and the formation of a biofilm layer in the reactor. Nutrition is given to microorganisms during the seeding process, and the ratio of nutrients provided by C : N : P is 100 : 5 : 1 (Hendrasarie and Fransiska, 2022).

Following that, an acclimatization process is carried out until a steady state is reached. A steady state is defined by a 50% decrease in COD values. This method aims to adapt growing microorganisms to new wastes with higher concentrations. The wastewater is gradually fed at 50%, 75%, and 100%. This is done to prevent microorganisms from being subjected to shock loading, which can result in microorganism death (Hendrasarie and Andhika, 2021).

Once the acclimatization process is complete and has resulted in a good decrease in COD value, it then enters the SBR-GAC process with 100% wastewater in operation. There are two types of reactors for SBR-GAC

operations: a reactor with wastewater that is carried out by coagulation-flocculation as a pre-treatment (CF-SBR), and a reactor with wastewater that is carried out without coagulation-flocculation as a pre-treatment (SBR). The timed division of each HRT cycle that the assessment determined is in operation for each SBR-GAC reactor is as follows.

Table 2. Timing Of Each HRT Cycle

Stage	Time Distribution For Each HRT Cycle			Description
	720 (12 hours)	1440 (24 hours)	2880 (48 hours)	
Fill	15	15	15	Aeration : On Penambahan Adsorben
React	600	1200	2400	Aeration : On
Settle	60	180	300	Aeration : Off
Draw	15	15	15	Aeration : Off
Idle	30	30	150	Aeration : Off
Total	720	1440	2880	Aeration : Off

Source: Research, 2022

3. RESULT AND DISCUSSION

Reduction of organic matter using SBR-GAC

In this SBR-GAC, NPK fertilizer industry wastewater is used. The testing of pollutant parameters is based on the Regulation of the Minister of Environment of the Republic of Indonesia Number 5 of 2014 on the Quality Standards of Waste Water for The Fertilizer Industry and/or Industry Activities and Government Regulation No. 22 of 2021 on the River Water Quality Standard (Class 1). The parameters include COD, TSS, PO₄, and Total-N in the NPK fertilizer table. The following are the results of the initial waste characteristics test shown in the table below.

Table 4. Analysis Result

No.	Parameters	Industrial Wastewater Test Results		Effluent Quality Standards	Descriptions
		mg/L	kg/ton		
1	COD	9306.1	9.31	3	Exceed
2	TSS	490	0.49	3	Fulfill
3	PO ₄	11160	11.16	-	Exceed
4	Total-N	6335	6.34	2.25	Exceed

Source: Research, 2022

The SBR-GAC reactor is operate on a predetermined HRT cycle, and COD, TSS, PO₄, and Total-N reduction are measured after passing through the entire process chain in a single cycle.

Percent reduction in each parameters

The results of the operation of this SBR-GAC reactor at the highest reduction in each parameter are: COD reduction efficiency was 3428.6 mg/L in CF-SBR reactors and 3102 mg/L in SBR reactors; TSS reduction efficiency was 90 mg/L in CF-SBR reactors and 110 mg/L in SBR reactors; PO₄ reduction efficiency was 6157 mg/L in CF-SBR reactors and 7223 mg/L in SBR reactors, and Total-N reduction efficiency was 1540 mg/L in CF-SBR reactors and 1855 mg/L in SBR reactors. Figures 2, 3, 4, and 5 show a graph of the percentage reduction in each parameter as a result of the SBR-GAC operation.

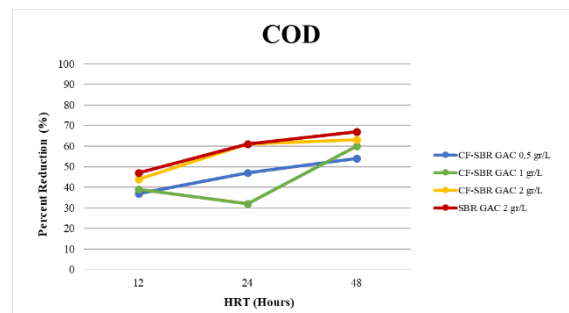


Figure 2. Correlation of HRT with percentage of COD decrease in GAC mass in each process
Source: Research, 2022

Graph of COD reduction efficiency, with the highest COD reduction efficiency of 63% in CF-SBR reactors at 48-hour HRT operations with 2 g/L GAC mass and 67% in SBR reactors at 48-hour HRT operations with 2 g/L GAC mass. The lowest COD reduction efficiency is 32% in CF-SBR reactors at 12-hour HRT operations with a GAC mass of 0.5 g/L and 47% in SBR reactors at 12-hour HRT operations with a GAC mass of 2 g/L. In both types of reactors, the optimal HRT for reduction COD parameters is 48 hours, and GAC mass 2 g/L is the most influential mass in reduction COD parameters. The presence of longer periods of aerobic conditions causes microorganisms to have longer periods of development, and in development, microorganisms require carbon sources derived from wastewater. This will lead to greater wastewater degradation, resulting in smaller levels of effluent COD, where the effect of COD decreases is greater (Handayani et al. 2009).

This suboptimal COD reduction is caused by the 48-hour stationary phase, in which cell division slows due to medium fatigue and bacterial cells secrete secondary compounds or metabolites that can poison the medium, causing several bacteria to die and grow. The mortality of new cells is offset by the death of old cells (Handayani et al. 2009)

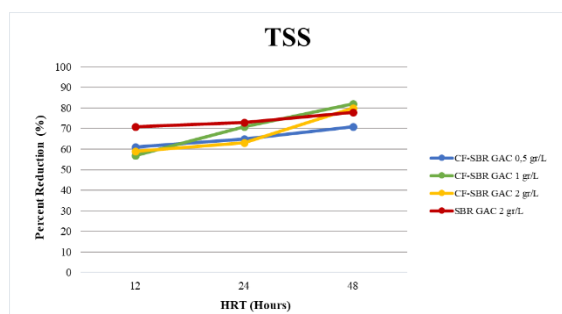


Figure 3. Correlation of HRT with percentage of TSS decrease in GAC mass in each process
Source: Research, 2022

Graph of TSS reduction efficiency, with the highest TSS reduction efficiency of 82% in CF-SBR reactors at 48-hour HRT operations with 1 g/L GAC mass and 78% in SBR reactors at 48-hour HRT operations with 2 g/L GAC mass. The lowest TSS reduction efficiency is 57% in CF-SBR reactors at 12-hour HRT operations with a GAC mass of 1 g/L and 71% in SBR reactors at 12-hour HRT operations with a GAC mass of 2 g/L. The optimal HRT for reducing TSS parameters is 48 hours, and GAC mass 2 g/L is the most influential mass in reducing TSS parameters in both types of reactors. The increase in TSS reduction efficiency is due to the long settle time division, so the solid settles well.

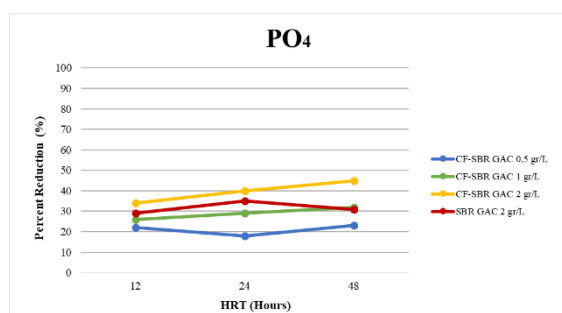


Figure 4. Correlation of HRT with percentage of PO₄ decrease in GAC mass in each process
Source: Research, 2022

Graph of PO₄ reduction efficiency, with the highest PO₄ reduction efficiency of 45% in CF-SBR reactors at 48-hour HRT operations with 1 g/L GAC mass and 35% in SBR reactors at 24-hour HRT operations with 2 g/L GAC mass. The lowest PO₄ reduction efficiency is 12% in CF-SBR reactors at 12-hour HRT operations with a GAC mass of 0.5 g/L and 29% in SBR reactors at 12-hour HRT operations with a GAC mass of 2 g/L. In both types of reactors, the optimal HRT for reduction PO₄ parameters is 48 hours, and GAC mass 2 g/L is the most influential mass in reduction PO₄ parameters. The fluctuating phosphate removal efficiency values may occur in various biological processing, according to Shatasivan (Haque 2017). Because phosphate removal using biological processing often shows fluctuating results without knowing the exact reason, and there has been an intense study to date.

According to (Yusuf et al., 2021) and (I. Said, 2017), the decrease was caused by the anoxic reaction phase during which the denitrification process occurred, resulting in the release of the PO₄ compound. Phosphate compounds present in wastewater and phosphorus discharged by microorganism biomasses are reabsorbed by biomasses in the aerobic reaction phase. In anoxic conditions, there is an increase in phosphate concentration resulting from the release of phosphorus from polyphosphate, which can induce precipitation as well as microbial activity in biofilms and active sludge. In the aeration process, the pH drops, which dissolves the phosphate, and then at the end of the aeration process, there is an increase in the pH, which causes phosphate precipitation and dissolves the phosphate into the slurry.

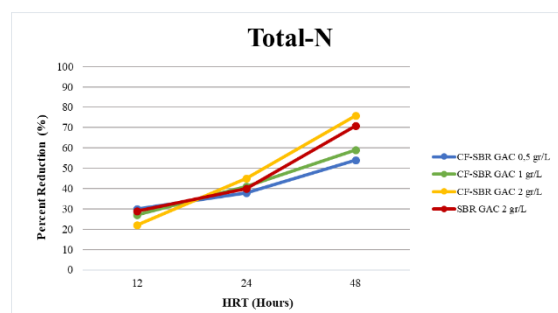


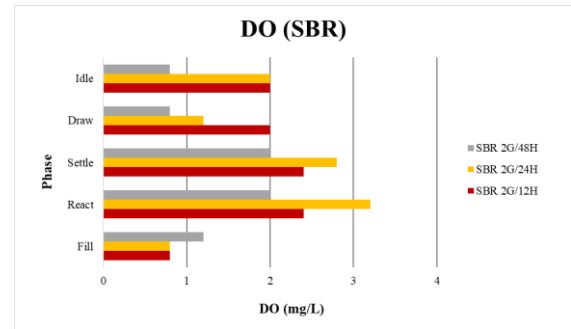
Figure 5. Correlation of HRT with percentage of Total-N decrease in GAC mass in each process
Source: Research, 2022

Graph of Total-N reduction efficiency, with the highest Total-N reduction efficiency of 76% in CF-SBR reactors at 48-hour HRT operations with 2 g/L GAC mass and 71% in SBR reactors at 48-hour HRT operations with 2 g/L GAC mass. The lowest Total-N reduction efficiency is 22% in CF-SBR reactors at 12-hour HRT operations with a GAC mass of 2 g/L and 29% in SBR reactors at 12-hour HRT operations with a GAC mass of 2 g/L. The optimal HRT for reducing Total-N parameters is 48 hours, and GAC mass 2 g/L is the most influential mass in reducing Total-N parameters in both types of reactors. According to (Brown et al., 2005) (Ikhwan 2020), Total-N consists of amounts of ammonia and organic nitrogen plus forms of oxidized nitrogen (nitrite and nitrate). Nitrate is the result of the nitrification process, which converts ammonia to nitrate. Dissolved ammonia exists as both ammonia molecules (NH_3) and ammonium ions (NH_4^+) in equilibrium.

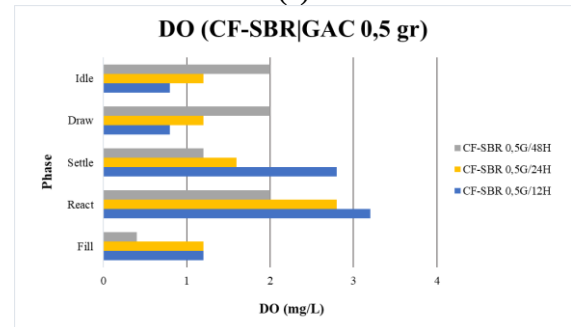
The characteristic of DO, pH, Temperature, and adsorbent

DO is one of the main components of the metabolism process of aquatic microorganisms and one of the main control parameters in achieving nutrient release. It can be a good indicator of biological activity, one of which is nitrification. Improper DO control may result in inadequate parameter reductions (USEPA 2010).

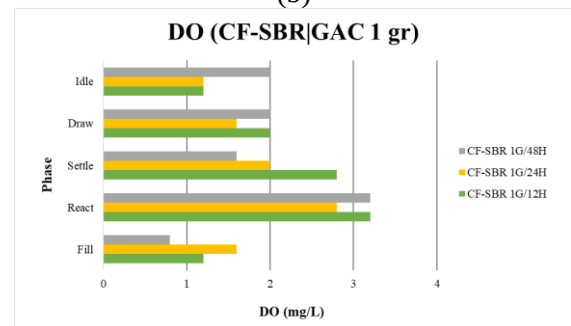
The growth of nitrified bacteria can only function under aerobic conditions. The concentration of dissolved oxygen can have a significant effect on the growth rate of nitrification. In general, nitrification can occur at a higher dissolved oxygen concentration of 2.0 mg/L. Soluble oxygen must be monitored during the anoxic phase so that it does not exceed 0.2 mg/L (NEIWPPCC, 2005). Dissolved oxygen (DO) treatment of synthetic wastewater and, industrial and non-industrial waste uses conditions ranging from 2-8 mg/L (aerobic), 0.2-1.0 mg/L (anoxic), 0.5-1.0 mg/L (anoxic), and 1.6-5 mg/L (anoxic) in (Ikhwan, 2020). Based on the operation of this SBR-GAC reactor, the DO chart generated at each phase is presented in Figure 6 below.



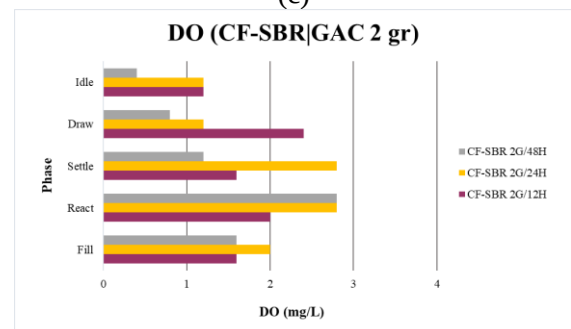
(a)



(b)



(c)



(d)

Figure 5. The DO chart in each phase (a) of the SBR-GAC without the Coagulation-Flocculation process as a pre-treatment, (b), (c), (d) DO in the SBR-GAC with the Coagulation-Flocculation process as a pre-treatment of each variation in the mass of the GAC

Source: Research, 2022

The DO chart at each phase shows that the presence of lower DO and longer retention

times at the time of the batch will affect the growth of microorganisms in the reactor. Dissolved oxygen (DO) is a major control parameter in achieving nutrient depletion. In the study of (Azhdarpoor, Mohammadi, and Dehghani 2014) the DO value of the aerobic cycle was approximately 2.5-3.5 mg/L and that of the anoxic cycle was approximately 0-0.2 mg/L, and all experiments were conducted at room temperature.

Table 3. pH and Temperature in SBR-GAC

pH			
Process	12 Hours	24 Hours	48 Hours
CF-SBR GAC 0,5 g/L	4.13	4.24	4.48
CF-SBR GAC 1 g/L	4.13	4.24	4,48
CF-SBR GAC 2 g/L	4.11	4.51	4,43
SBR GAC 2 g/L	6.63	6.86	7.66
Temperature			
Process	12 Hours	24 Hours	48 Hours
CF-SBR GAC 0,5 g/L	28.2	27.5	29.3
CF-SBR GAC 1 g/L	28.5	28.4	29.5
CF-SBR GAC 2 g/L	28.3	27.3	29.5
SBR GAC 2 g/L	28.8	28	29.8

Source: Research, 2022

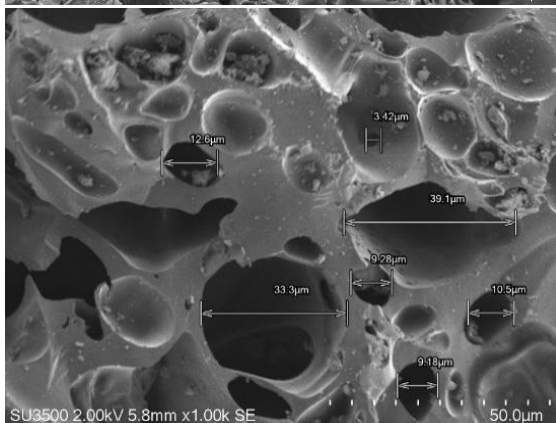
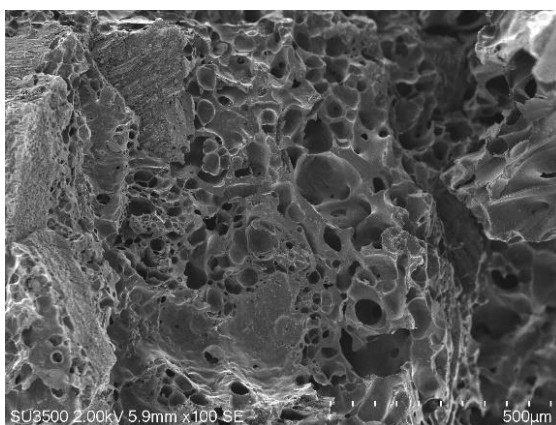
Based on Table 3, it can be seen that the pH and temperature of fertilizer industrial waste in influenza and effluent have changed. The pH of the solution in the CF-SBR reactor decreases as the PAC content of the coagulation-flocculation process affects the pH of the water to be fed into the SBR-GAC reactor. The addition of a PAC coagulant affects pH in wastewater, the more concentration of coagulants given, the lower the pH value (Yustinawati 2014).

An important factor to consider in the performance of the nitrification process is pH. Nitrification requires alkalinity for the oxidation process, meaning that the value of alkalinity decreases. The decrease in alkalinity results in a decrease in pH as well, thus affecting the performance of nitrification (NEIWPCC, 2005). The temperature of each reactor is quite stable, and changes in temperature values fluctuate in each reactor. Bacterial activity is best at $25^{\circ}\text{C} \pm 35^{\circ}\text{C}$, but at 50°C , aerobics and nitrification cease (Metcalf & Eddy, 2003).

Activated carbon is widely used as an adsorbent to absorb toxic liquids, toxic gases, odors, water purifiers, drinking water filters, and so on (Abu Akhmad B, Diah Susanti 2007). The pore size and surface area of the adsorbent are important characteristics of the adsorbent.

The pore size is related to the surface area, the smaller the pore size of the adsorbent, the higher the surface area. So the number of adsorbed molecules will increase. In addition, the purity of the adsorbent is also a major characteristic, and in which a more pure adsorbent is preferred because of its good adsorption ability. SEM analysis aims to determine the surface structure of the adsorbent, the results of SEM analysis are presented in the following figure:

(a)



(b)

Figure 6. Results of SEM GAC analysis at magnitudes (a) 100 times, and (b) 1000 times
Source: Research, 2022

The characteristic results of the GAC morphological structure were determined on SEM tests, with magnitudes ranging from 50 times to 1000 times. In SEM testing of GAC before 1000 magnification processing, a GAC surface pore size of 3.42–39.1 μm was obtained, as shown in Figure 6. Activated carbon has very large pores, so it can absorb anything that passes through it. Active charcoal absorption occurs due to the presence of large, micro-sized pores (Yustinawati 2014). The pores on the surface of the active carbon are formed by the heating and

dissipation of oxide gas so that it can be used to absorb and bind other substances. Large pores affect the absorption power of adsorbable toxins.

4. CONCLUSION

Based on the data from the research results and data processing, the following conclusions were reached:

- a. The optimal concentration of Poly Aluminum Chloride (PAC) in the coagulation-flocculation process as a pre-treatment used in the industrial waste treatment of NPK fertilizer is 3 g/L.
- b. The optimal Hydraulic Retention Time (HRT) required and the influence of Granular Activated Carbon (GAC) mass on Sequencing Batch Reactor (SBR) in NPK fertilizer industrial waste is an HRT of 48 hours with a GAC mass of 2 g/L, a TSS parameter reduction of 82% and a Total-N of 76% that meets the effluent quality standard and a COD parameter reduction of 67% and a PO_4 of 45% that still exceeds the effluent quality standard.
- c. DO, pH, Temperature, and Adsorbents Characteristics of Sequencing Batch Reactor (SBR)-Granular Activated Carbon (GAC) in NPK fertilizer industrial waste using Scanning Electron Microscope (SEM) has a DO of 3.2 mg/L-2 mg/L at the react phase, a pH of 6.7 (without coagulation-flocculation) and 4 (with coagulation-flocculation), and an average temperature of 29°C, in the GAC adsorbent obtained pore yield on the 3.42 μm - 39.1 μm diameter surface.

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