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Reduction of Ammonia Nitrogen and Chemical Oxygen Demand of Fertilizer Industry Liquid Waste by Coconut Shell Activated Carbon in Batch and Continuous Systems

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ABSTRACT

The fertilizer industry laboratory produces urea and ammonia nitrogen waste that can harm living things in the surrounding water bodies. Urea, nitrogen, and ammonia can be reduced by adsorption using activated carbon. This research reduced urea nitrogen and ammonia through activated carbon adsorption with a batch and continuous system. Percentage indicator of urea and ammonia nitrogen removal through Ammonia Nitrogen (NH_3-N) and Chemical Oxygen Demand (COD) NH_3-N and COD analysis was determined. This study aimed to obtain: 1) the percentage of NH_3-N and COD reduction in stem batch; 2) the percentage of NH_3-N and COD reduction in the continuous system; 3) the Freundlich and Langmuir isotherm adsorption equation against NH_3-N wastewater. They are testing the adsorption power of activated carbon in a batch system using variable levels of activated carbon: 40 g/L, 55 g/L, 70 g/L, 85 g/L, and 100 g/L and testing the adsorption power of activated carbon in a continuous system using the variable frequency of wastewater in contact with activated carbon filter cartridges, namely 2, 3, 4, 5, and 6 times. The results showed: 1) in the batch system NH_3-N reduction of 98.26–98.82% and COD reduction of 92.53–97.05%; 2) in continuous system reduction of NH_3-N of 86.05–88.07% and COD reduction of 93.91–97.05%; 3) Freundlich isotherm adsorption equation yields constant R^2 0.9464, n 0.4482, K_F 0.0616 mg/g; while Langmuir's isotherm adsorption equation yields constant R^2 0.8684, b -0.1046 L/mg, and q_m 7.9872 mg/g.

Keywords: activated carbon, waste, fertilizer, coconut shell.

INTRODUCTION

Indonesia is an agrarian country where agriculture is booming throughout the archipelago. This thriving farm requires much fertilizer to optimize its yields. Some industries that produce fertilizers in Indonesia include PT Petrokimia Gresik, PT Pupuk Kujang Cikampek, PT Pupuk Kalimantan Timur, PT Pupuk Iskandar Muda, and PT Pupuk Sriwijaya Palembang. Production of urea fertilizer in Indonesia in 2021 reached 6.5 million tons per year. The quality of fertilizers on the market is controlled by analyzing packaging samples on the market and for production purposes. Laboratory operations in the fertilizer industry produce nitrogen waste from urea and its derivatives. Waste with the ammonia content that exceeds quality standards causes ecosystem inequality in water bodies.

Several treatment methods reduce the nitrogen content of urea and ammonia, and organic substances contained in wastewater, including filtration, adsorption, exchange ions, and wetland use. Sulfur-based wetlands and traditional artificial wetlands reduced total nitrogen and nitrates in wastewater by 69% and 82% (Wang et al., 2022). The removal of various types of nitrogen in wastewater can also be done using microorganisms of *the Klebsiella oxytoca* (EN-B2) strain, namely ammonium 51%, nitrite 32%, and nitrate 47% (He et al., 2023). In addition, ZrO_2 (DWS500@ZrO_2) sludge can also remove nitrates from contaminated water up to 31 mg/g at pH of 2 and temperature of 500 °C (Quang et al., 2022). Nitrogen removal can also be done by neutralization and coagulation methods (Nurhayati, 2018). The studies that have been carried out can reduce the content of nitrogen and organic substances in wastewater but require considerable operational costs and quite complicated methods. Research is still needed by applying the adsorption methods that are cheap and easy to operate.

Adsorbers that can be used to adsorb pollutants include activated carbon (Chansa et al., 2021; Kosim et al., 2015; Kozyatnyk et al., 2021; Kusdarini et al., 2022; Kusdarini &; Budianto, 2022; Mayyas &; Sahajwalla, 2019; Pérez-Rodríguez et al., 2021; Yagub et al., 2014; You et al., 2020). The research related to activated carbon adsorption power resulted in findings of activated carbon adsorption capacity; however, the adsorption process has not been maximized, because the process uses a batch system and even causes an increase in TSS. The research on reducing the levels of dissolved nitrogen compounds in waste by maximizing contact between wastewater and activated carbon, namely using a continuous system and circulating (repeated contact) between activated carbon and wastewater, may solve the problem experienced.

MATERIALS AND METHODS

Materials and tools

The materials used in this study are liquid waste from the fertilizer industry laboratory containing urea and its derivatives, coconut shell activated carbon, concentrated sulfuric acid (H_2SO_4), iron (II) sulfate ($FeSO_4$); potassium dichromate ($K_2Cr_2O_7$) 0,25 N; silver sulfuric acid (AgSO4), ferro ammonium sulfate (FAS) 0.1 N, fenantrolin monohydrate, sodium azide (NaN_3), mercury (II) sulfate ($HgSO_4$), manganese sulfate monohydrate ($MnSO_4.H_2O$), potassium hydroxide (KOH) or sodium hydroxide (NaOH), zinc sulfate monohydrate ($ZnSO_4.H_2O$), ethylene diamine tetraacetate acid (EDTA), Nessler reagent and aquadest. The tools used in this study were *cartridge* activated carbon, beaker glass, measure glass, analytical balance, pH meter, set of reflux tools, *magnetic stirrer*, and oven.

Experimental procedure

Activated carbon adsorption testing experiments were carried out in 2 different procedures: 1) batch system; 2) continuous system. The practical steps in the batch system included: 1) preparing 11 beaker glasses (samples O, B1, B2, B3, B4, B5) and each filled with as much as 1 L of waste; 2) adding activated carbon powder on samples B1 (40 g), B2 (55 g), B3(70 g), B4(85 g), B5(100 g); 3) stirring the sample at 400 rpm for 24 hours; 4) filtering the sample with Whatman paper; 5) pH, TSS, NH_3-N, and COD testing on each sample. The data obtained create the Langmuir isotherm and the Freundlich isotherm equations.

Practical steps on a continuous system involved: 1) preparing 10 L of waste in a container; 2) preparing filtration devices (anion cartridges, 100 g coconut shell activated carbon, 0.5-micron filters); 3) pumping sewage and passing it through filter cartridge with a discharge of 1.33 L/min; 4) taking waste samples according to the variable number of revolutions: K1 (2 rounds), K2 (3 rounds), K3 (4 rounds), K4 (5 rounds), K5 (6 rounds); 5) pH, TSS, NH_3-N, and COD testing on each sample.

Testing of the ability of activated carbon remove NH_3-N, and COD

The ability of activated carbon to remove NH_3-N and COD in wastewater is expressed in % removal of total nitrogen (NH_3-N) (Equation 1) and % removal of total COD (Equation 2).

$$\% \, removal \; total \; nitrogen = \\ = \frac{tot \; nitrogen \; awal - \\ -tot \; nitrogen \; akhir}{tot \; nitrogen \; awal} \times 100\% \tag{1}$$

$$\% \, removal \; total \; COD = \\ = \frac{COD \; awal - COD \; akhir}{COD \; awal} \times 100\% \tag{2}$$

Freundlich and Langmuir isotherm adsorption equation test

Freunlich assumed heterogeneous surfaces with different adsorption energies. According to Equation 3, K_F and n are also Freundlich constants, which determine the adsorption capacity and intensity, respectively. This constant can be obtained from the intercept and slope of the log diagram q_e versus C_e (Nowruzi et al., 2020).

$$q_e = K_F C_e^{\frac{1}{n}} \tag{3}$$

where: q_e – the equilibrium capacity of NH_3-N, i.e., the amount of NH_3-N adsorbed per unit mass of activated carbon (mg/g), is the equilibrium concentration of NH_3-N;
C_e – in solution after activated carbon (mg/L) adsorption;
K_F and n – empirical constants (Basu et al., 2018; Kusdarini et al., 2021; Kusdarini et al., 2018). The constants K_F and n can be found by Equation 4.

$$\log q_e = \log K_F + \log C_e^{\frac{1}{n}} \tag{4}$$

Plotting data using the Freundlich Equation obtained data on constants K_F and n, may be used to form an adsorption equation of activated carbon isotherm to NH_3-N contained in artificial waste.

The Langmuir isothermal adsorption equation is presented in Equation 5.

$$\frac{C_e}{q_e} = \ + \frac{C_e}{q_m}\frac{1}{bq_m} \tag{5}$$

where: C_e (mg/L) – the equilibrium concentration of NH_3-N;
q_e – the equilibrium capacity of NH_3-N. The capacity of adsorption of activated carbon is the weight of NH_3-N adsorbed per unit weight of activated carbon (mg/g); b (l/mg) and q_m (mg/g) are the Langmuir constant a constant relating to the adsorption energy, and maximum adsorption capacity of the adsorption is determined from the intercept and slope of the diagram, respectively.

RESULTS

Initial laboratory waste parameters

The results of the analysis of laboratory waste samples of the fertilizer industry before processing are presented in Table 1. The parameters are focused on TSS, NH_3-N, COD, and pH. Table 1 shows the TSS, NH_3-N, COD, and pH levels from fertilizer industry laboratory waste. From the table, it appears that the level of NH_3-N 752 mg/l is more than 750% of the standard, COD 1592 mg/l is much higher than the standard of 200 mg/l, as well as the pH is still very low, i.e. 1.05, even though the quality standard is only between 6–9. The initial condition of the fertilizer industry's laboratory waste is processed to achieve standard quality conditions.

Table 1. Parameters of fertilizer industry laboratory waste

Parameter	Unit	Waste	Quality standards*
TSS	mg/L	189	200
NH_3-N	mg/L	752	100
COD	mg/L	1592	200
pH	-	1,05	6–9

Note: * (East, 2013).

Waste parameters after processing

Fertilizer industry laboratory waste processed by batch and continuous processes is analyzed. The sample analysis results of industrial waste after processing are shown in Figure 1–4.

Figure 1 shows the concentration of activated carbon as an adsorbent in fertilizer laboratory waste. The figure shows that increased activated carbon can remove TSS but increases TSS levels at higher concentrations. This condition happens because the type and size of activated carbon used are not appropriate and increase the TSS of the waste. Activated carbon with a large surface and microscopic pores can capture organic molecules and solid particles residing in water. When water flows through the activated carbon medium, solid particles suspended in the water stick to the surface of the activated carbon and become trapped inside the pores. The content of solid particles trapped in activated carbon increases, in addition to greater TSS in water and desorption. Therefore, it is crucial to carry out periodic maintenance and replacement of activated carbon to ensure the effectiveness of the adsorption process and prevent an excessive increase of TSS in the water produced (Zhang et al., 2019).

Figure 2 shows the effect of activated carbon concentration added as an adsorbent on fertilizer industry laboratory waste. It was seen that increasing the concentration of activated carbon from 40 g/l tar to 100 g/l can reduce NH_3-N levels from 13.1 and 8.9 mg/l. The percent of NH_3-N removal reached 98.3% to 98.7% with activated

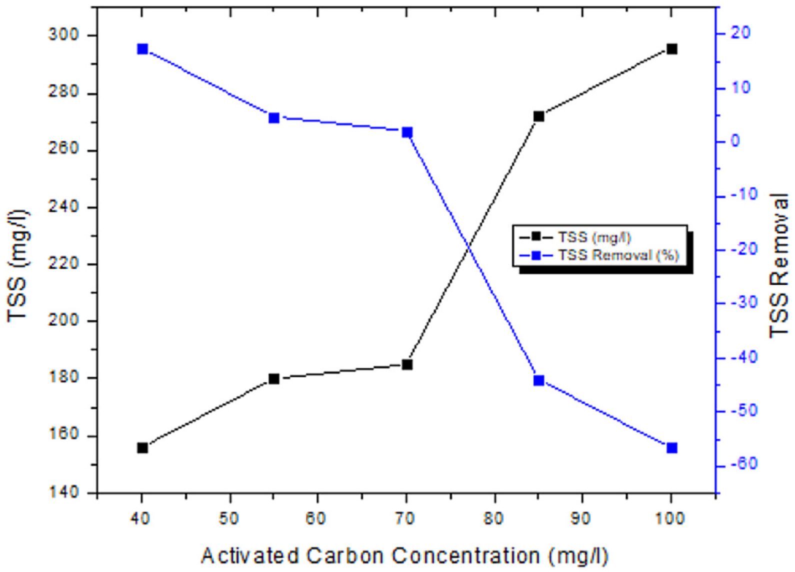

Figure 1. The effect of activated carbon concentration on TSS concentration and % TSS removal in waste with initial NH_3-N concentration was 752 mg/l

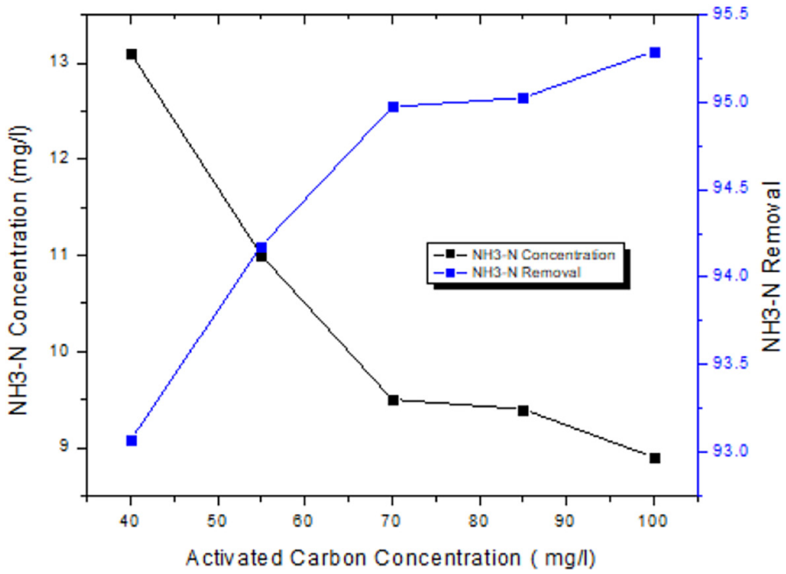

Figure 2. The effect of activated carbon concentration on NH_3-N concentration and % removal of NH_3-N in waste with initial NH_3-N concentration was 752 mg/l

carbon. This data shows that a 250% increase in activated carbon from 40 g/l to 100 g/l has little effect on percent removal. This condition is likely because, at a small concentration of NH_3-N, 13.1–8.9 mg/l is an area that is not sensitive for activated carbon to absorb NH_3-N. The results of this study are better than that in the studies of Hong et al. 2015 and Giri et al. 2013, which only achieved 80% and 72% percent removal. The NH_3-N removal rate of this study is almost the same as the removal rate conducted by Hao et al. in 2013, which is 89–99% (Li et al., 2013). The % removal of NH_3-N this study is also much better, when juxtaposed with research using activated carbon from young coconuts, which only reached less than 50% (Mangkurat et al., 2019)

Figure 3 shows the effect of activated carbon concentration used to treat fertilizer industry waste with an initial COD of 1592 mg/l on COD and % COD removal. The picture shows that COD decreased with 92.5–97.0% removal due to absorption by activated carbon. The increase in activated carbon levels from 40 g/l to 100 g/l only increased the percentage of COD removal to 4.5% from 92.5%. This condition shows that the working area of activated carbon against this

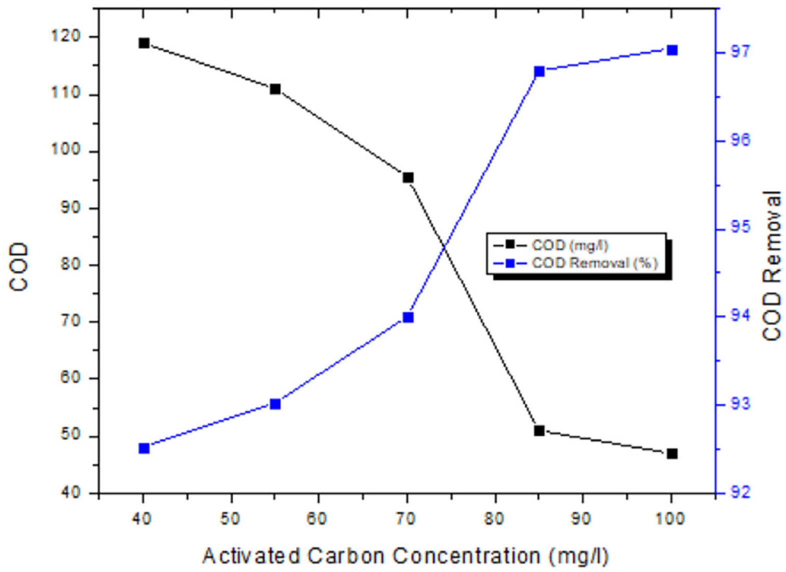

Figure 3. The effect of activated carbon concentration on COD and % COD removal in waste with initial COD was 1592 mg/l

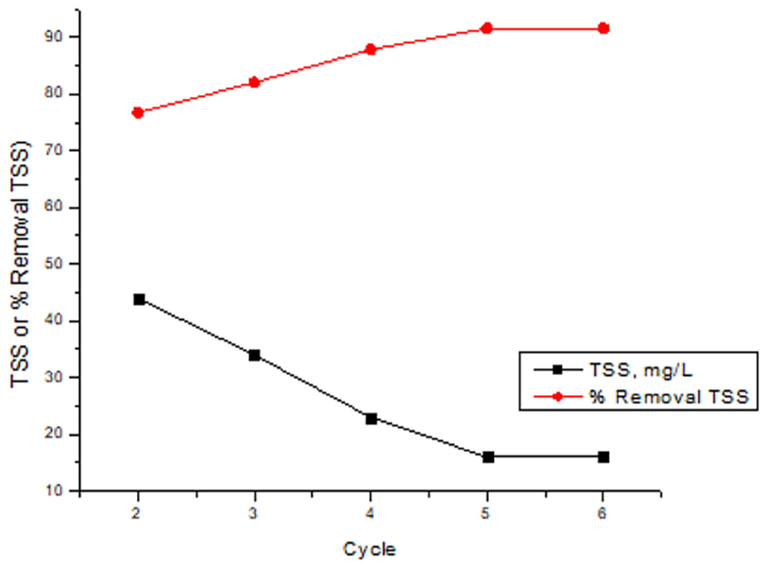

Figure 4. The effect of the number of feed cycles in the series of filtration devices with activated carbon and cation exchangers on TSS and % TSS removal in waste with initial TSS was 198 mg/l

concentration has reached optimal conditions, so it requires other methods. The COD removal results of this study are better than activated carbon from palm shells and sugarcane bagasse(19) COD levels This waste has met quality standards and must be released into the environment (East Java Governor Regulation No. 52, 2014) (Mustafa et al., 2022; Mustefa et al., 2021).

Figure 4 is the TSS value and % TSS removal of lime water after treatment using adsorption filtration and cation exchanger with various recycle variants. The figure shows that TSS has experienced a sharp decrease in cycles. The highest TSS removal % reached 91.5%. These removal results are almost identical to the TSS removal from biodiesel wastewater (24). This result is different only if using activated carbon, as an adsorbent causes an increase in the TSS value.

Figure 5 shows the effect of the number of process cycles on the concentration of NH_3-N and NH_3-N removal in waste. The figure shows that the NH_3-N levels drop with the number of cycles, indicating that repeatability of the process can decrease NH_3-N levels, but this decrease is small relative to the number of cycles cycle. The decrease in the NH_3-N levels occurs due to NH_3-N

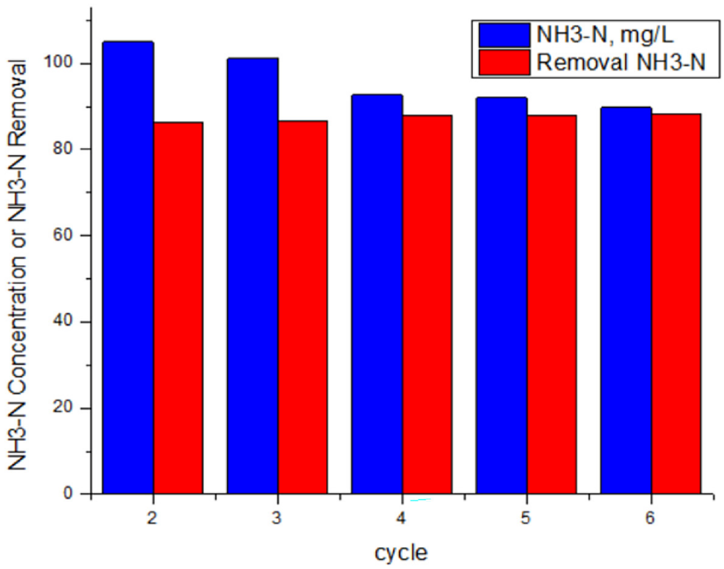

Figure 5. The effect of the number of cycles of the adsorption process using continuous adsorption equipment on NH_3-N and NH_3-N removal levels

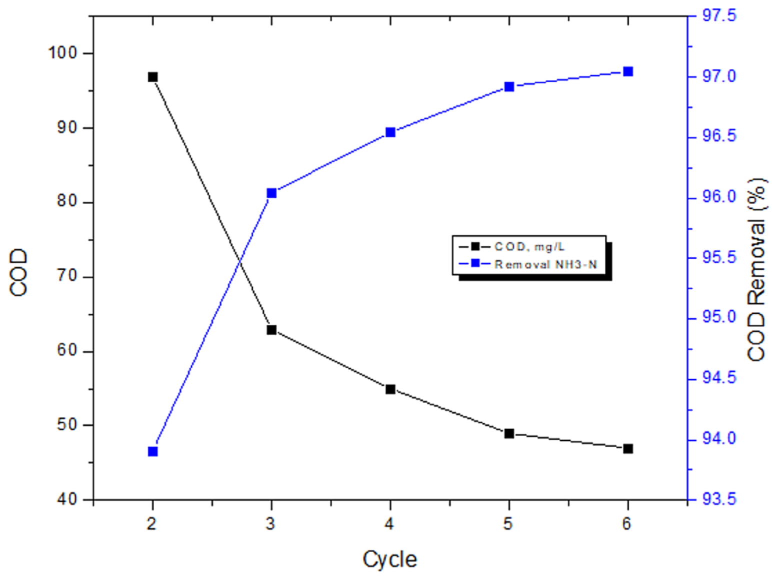

Figure 6. Effect of the number of cycles of the adsorption process on COD and COD levels removal from fertilizer laboratory waste by continuous adsorption process

adsorbed by activated carbon. The concentration of NH_3-N is 89.7–104.9 mg/l; this figure has met waste quality standards in Indonesia. NH_3-N removal is at 86.1–88.1% in this waste. The value of NH_3-N removal is better than the research of Hao and Liu (Li et al., 2013).

Figure 6 shows COD concentration and COD removal from fertilizer waste in the adsorption process at various cycles. The increasing number of cycles decreases the COD value and increases COD removal. Increasing the number of cycles increases the contact time between the waste and activated carbon. Increasing the contact time raises the amount of chemical adsorbed by activated carbon. The increased adsorbed chemicals by activated carbon decrease the concentration of dissolved and suspended chemicals. The reduction of these chemicals causes a decrease in COD values.

Freundlich and Langmuir isotherm adsorption to NH_3-N

Isotherm studies describe the behavior of adsorbents and state the relationship between the amount of adsorbate absorbed by adsorbents. Test of the adsorption behavior of activated carbon

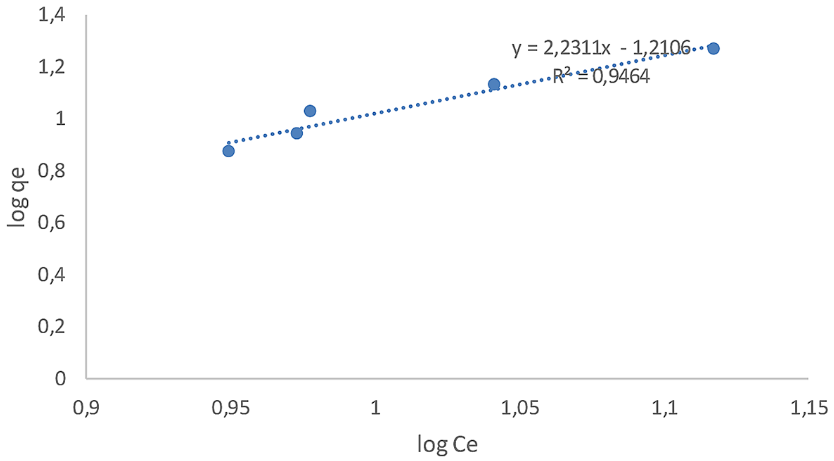

Figure 7. Plot Log(Ce) Vs. Log (qe) to test the isotherm adsorption equation of the Freundlich Model

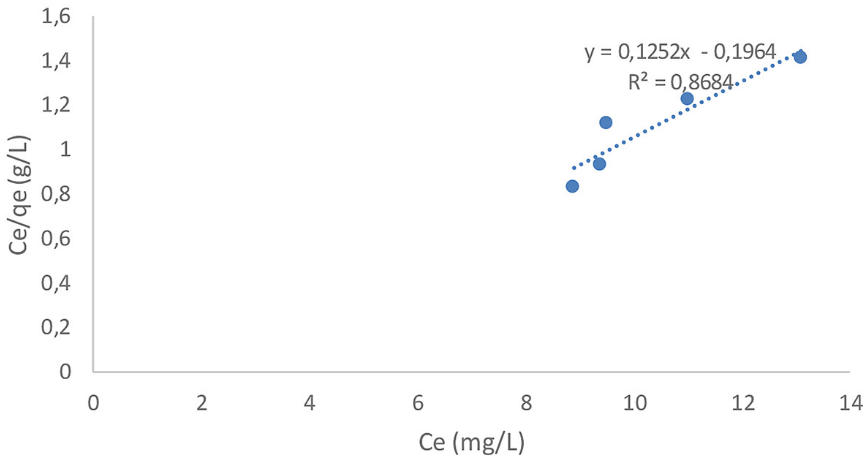

Figure 8. Isotherm adsorption plot from Langmuir Model for NH_3-N adsorption of fertilizer waste by activated carbon

against NH_3-N using Freundlich and Langmuir equations is presented in Figures 7 and 8. Equation parameters are presented in Table 2.

Figure 7 shows a test graph of the Freudlich Equation against the adsorption data of NH_3-N by activated carbon at various times. The figure shows that the resulting image is a straight line with an R^2 of 0.9464. The slope of linear regression results against the experimental data is 2.2311 and intercept -1.2106. This graph shows that the substance n = 1.0566 and K_F = 0.061574, or in other words, the Freundlich Equation of the NH_3-N adsorption process by activated carbon is:

$$q_e = 0.061574 C_e^{\frac{1}{1.0566}} \quad (6)$$

Figure 8 shows the c_e/q_e plot against c_e as a test method for the adsorption of NH_3-N from fertilizer waste by activated carbon. The graph shows the Equation of a straight line with R^2 = 0.8684 with a slope of 0.1252 and intercept -0.1964. From the data obtained, the Langmuir isotherm equation:

$$\frac{C_e}{q_e} = \frac{C_e}{7.98722} + \frac{1}{-5.09165} \quad (7)$$

Or in other words, qm = 7.98722 and value b = -0.1046. Comparison of the isothermic adsorption results using Freundlich and Langmuir isotherms can be seen in Table 2.

Table 2 shows that the adsorption of NH_3-N by activated carbon in fertilizer waste is more

Table 2. Isotherm adsorption parameters to NH_3-N by activated carbon

Adsorption System	Friendly isoterm			Langmuir isoterm		
	R^2	n	K_F, mg/g	R^2	B, l/mg	q_m, mg/g
Activated carbon	0.9464	0.4482	0.0616	0.8684	-0.1046	7.9872

representative using the Freundlich equation than the Langmuir equation.

CONCLUSIONS

Activated carbon can reduce the content of NH_3-N and COD in the liquid waste of the urea and ammonia fertilizer industry to meet waste quality standards. The waste quality standard is based on East Java Governor Regulation Number 72 of 2013. The percentage of NH_3N removal in the adsorption process by coconut shell activated carbon using the batch process is 98.26–98.82%. The use of continuous adsorption with multiple filtrations produces NH_3-N removal of 86.05–88.07%. COD in the batch system is 92.53–97.05%, while in the continuous system, it is 93.91–97.05%. NH_3-N adsorption in the batch system is better than in the continuous system. Adsorption of TSS by activated carbon in a batch system achieves COD removal of -56.6–17.5%, while using a continuous system it achieves COD removal of 93.9–97.0%. The Freundlich adsorption isotherm equation for NH_3-N yielded constants R^2, n, and K_F of 0.9464, 0.4482, and 0.0616 mg/g. The Freundlich adsorption isotherm equation is more representative than the Langmuir equation.

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