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Effect of Contact Time and Adsorbent Dosage and Contact Time of Activated Carbon of Eucalyptus Oil Refining on Ammonia Levels (NH_3) Leather Industry Liquid Waste

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Abstract

Indonesia's industry expands to suit domestic demand as the population increases. One of these is the leather processing business, which heavily relies on both chemicals and water in its operations. Adsorption was the approach employed in this investigation. The purpose of this study is to establish the ideal contact dosage and contact time for ammonia waste adsorption. The Adsorbent characterisation test results have been discovered. The findings of the ammonia adsorption test with dose variation showed that, at a dosage of 2.5 grams and a removal rate of 92%, HCl 0.36 M activated carbon was more efficient than other activated carbon. The HCl 0.036 M activated carbon was shown to be more efficient than activated carbon in the time variation ammonia level adsorption test, with an elimination rate of 82.48% after 25 minutes. Activated carbons and HCl 0.36 M activated carbons follow the pattern of the Langmuir isotherm equation and are reversible, according to the results of isotherm tests of dosage and time variations (reversible).

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Introduction

The lining business in Magetan is one of the leather sectors that need specific care. Domestic work, which on a big scale necessitates water and chemicals, employs the bulk of Magetan's population. Waste from the Magetan leather encapsulation industry is produced as liquid waste, which has a disagreeable odor. Due to the high levels of ammonia in the liquid waste produced by the leather manufacturing industry, this may occur. Ammonia is a toxic substance or contaminant. In accordance with the Republic of Indonesia's Ministry of Environment and Forestry's rules No. 21 of 2018 regarding wastewater for businesses and activities related to the leather industry and ammonia levels of 2 mg/L, (Aditya dan Lubis, 2021). Ammonia itself has the characteristic that it has a very sticky smell and is not colour the result of microbial activity or industrial activity. Adsorption is the process of subtension absorption on the surface of a solid matter, which is one of the most efficient methods of reducing the color, odor, oil, and organic content of waste.

In the research to reduce ammonia levels in the waste using the adsorption method with activated charcoal adsorbents, waste leaf refining wood oil was used. Activated charcoal has a good rate of adsorption but has the weakness of being relatively expensive. Therefore, there is a need for alternatives to adsorbents at a more economical price, including using the waste leaves of the refining process of the White Wood Oil

Factory under the shade of BUMN, especially in Eastern Java, among them Ponorogo, Mojokerto, and Nganjuk. With a factory capacity of between 3,000 and 12,000 tons per years. The refining process of white wood oil produces leaf-shaped waste leaves that are grown over time. The waste has been partially utilized, one by putting 50% of the waste as fuel into the refinery, and the rest is still not properly treated, so it's stuck in the mountains. The study aims to conduct an adsorbent study of the ability of activated charcoal wastes from the refining process to adsorb ammonia levels (NH_3) of affected leather industries with dose and contact time variations.

Methods

Determining the influence of dosage variation on concentration

The density of the waste leaf of the refining process of activated charcoal oil is weighed at 0.5; 1.0; 1.5; 2.0; 2.5 grams. Subsequently inserted into an erlenmeyer containing 50 mL of ammonia (0.1 mg/L). Then coated using a 250 rpm shaker for 15 minutes, filtered and added 1mL nitroprusside sodium solution, 2.5 mL oxidation solution, and 1 mL phenol solution, waited about 5 minutes, and then measured its adsorption using UV-Vis spectrometry, triple ammonium levels tested. The above will apply the same treatment to activated charcoal, activated carbon HCl 0.36 M.

Determining the influence of contact time on concentration

Weigh each 2.5 gram of activated charcoal and 2.5 gram of HCl for a time variation of 5 minutes, 10 minutes, 15 minutes, 20 minutes, or 25 minutes. Then insert into an erlenmeyer containing 50 mL of 0.1 mg/L ammonia. Then in a shaker with a 5-minute time variance. Set in a sample shaker filtered using filter paper, and then add 1 mL of phenol solution, 1 mL of sodium nitroprusside solution, and 2.5 mL of oxidation solution. Wait about 5 minutes until the color changes to blue. This trial was done in three ways. The treatment applies for time variations of 10 minutes, 15 minutes, 20 minutes, and 25 minutes.

Results

Characteristic Carbon

The characteristic test on this study aims to know the true conditions of active carbon. From such testing, it is expected that the active carbon from the waste of the eucalyptus leaves is functioning properly. The study conducted tests on active carbon characteristics involving water concentration, ash determination, density, volatile matter determination, and fixed carbon.

Table 1. Characteristic Activated Carbon and 0.36M HCl Activated Carbon

Adsorbent	Activated Carbon	0.36M HCl Activated Carbon	SNI.06-3730-1995
Density	0.1438 mg/L	0.01052 mg/L	Max 0.3 mg/L- 0.35 mg/L
Water concentration	4.20%	2.27%	Max 15%
Ash determination	5.78%	3.74%	Max 10%
Volatile matter	25%	13.67%	Max 25%
Fixed carbon	65%	79.35%	Minimum 65%

Determination of Optimum Ammonia Adsorption Dosage Variation

The study used mass variation of activated coal adsorbents and activated charcoal HCl 0.36 M 60 mesh. Weighed absorbances of 0.5 gr, 1 gr, 1.5 gr, 2 gr, and 2.5 gr It aims to determine the correct or optimal adsorbent mass to absorb the ammonia parameter (NH_3). The concentration used to test this is 0,2 mg/L of 50 mL of the adsorbent that has been weighed and mixed into an erlenmeyer containing 50 ml of ammonia (0,2 mg/l). Then activated coal and activated carbon HCl 0,36 M were shaken in the shaker at a speed of 250 rpm and a contact time of 15 minutes. This triple study was conducted to measure the adsorption of the ammonia solution. Then the adsorption was measured using the Genesys 10S UV-Vis spectrophotometer three times.

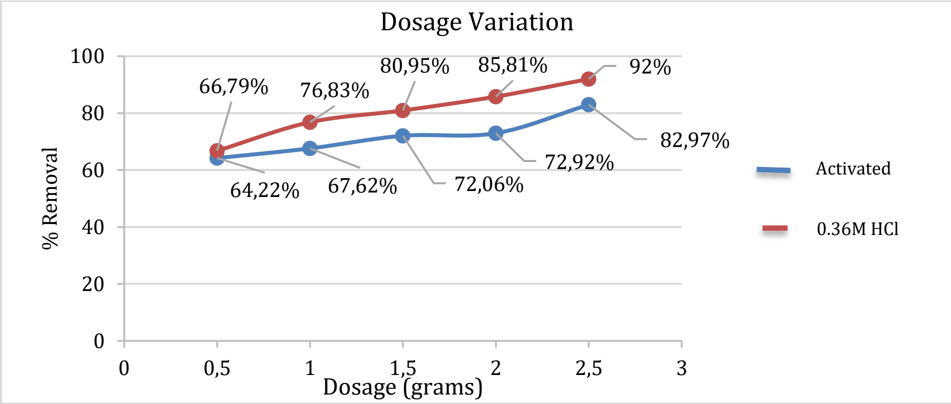


Figure 1. Graphic Optimum Dosage of Activated Carbon and 0.36M HCl Activated Carbon Refining on Ammonia (NH_3)

Based on Figure 1, it is stated that the activated carbon % removal of ammonia solution (NH_3) is 82.97% at a dosage of 2.5 grams and 0.36M HCl activated carbon % removal of ammonium solution (NH_3) is 92% at a dosage of 2,5 grams. This is because the dosage of the adsorbent is proportional to the increase in the number of particles and the surface area of the absorbent, so the capacity of adsorption is also increased. The results are consistent with the research carried out by Rio (2022), which found that the larger the mass of the adsorbents, the more the substance is adsorbed. The removal of 0.36M HCl activated carbon is larger than the removal of activated carbon. This is because 0.36M HCl activated carbon is water-binding, so the water that is tightly bound in the carbon pores will not release during the carbonation process. Closide acid (HCl) as an activator enters the pores and opens the closed coal surface, so that the absorbent compounds in the pore will disappear. If the surface area grows, then the interaction between the adsorbents and the absorbents occurs effectively.

Determination of Optimum Contact Time Variation

This test uses an adsorbent that is absorbed by carbon from eucalyptus leaf waste with a particle size of 60 mesh. 0.36 M HCl activated carbon adsorbents weighed 2.5 grams, then were mixed with a concentration of 0.2 mg/L of 50 mL into an erlenmeyer. After mixing the dishaker solution at a speed of 250 rpm with time variations of 5 minutes, 10 minutes, 15 minutes, 20 minutes, and 25 minutes, measure its absorption using the Genesys 10S UV-Vis spectrophotometer (NH_3).

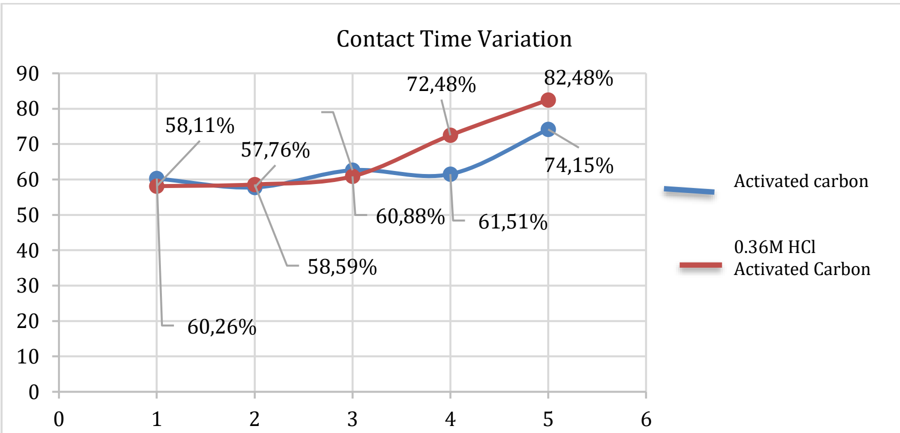


Figure 2. Graphic Optimum Contact Time Variation of Activated Carbon and 0.36M HCl Activated Carbon Refining on Ammonia (NH_3)

Based on Figure 2, time variations of 5 minutes, 10 minutes, 15 minutes, 20 minutes, and 25 minutes show that both activated carbon and 0.36M HCl activated carbon experience %removal increases as contact time increases. Based on Figure 2, the percentage removal of the ammonia solution reaches its optimal capacity in 25 minutes, with a percentage removal of activated carbon is 74.15% and 0.36M HCl activated carbon is 82.48% are respectively. The statement is consistent with the theory, stating that the longer the contact time, the better the diffusion process and the absorption of the adsorbed molecules. But it can also be influenced by the particle size the smaller the size of the particles that have activated carbon, the greater the adsorption speed at 25 minutes, it has reached the optimal time. From the above data, it can be concluded that the optimal time will be achieved with the addition of contact time.

Isotherm Model Activated Carbon Adsorbent of Dosage Variation

Langmuir's isotherm explains that the maximum adsorbent capacity occurs because there is a single layer of adsorbent on the surface of the absorbance. (Rio,2022). On the Langmuir isotherm chart with Ce as the y-axis and Ce/Qe as the x-axis, a line equation (y = bx + a) will be obtained, which will determine the values of Qm and Kl, where Qm is 1/a and Kl is the value of b. From the data from the adsorbent dose variation test results, the maximum absorption capacity value can be calculated. Below is a diagram of Langmuir's isotherm on Figures 3 and 5, and Freundlich's dosage variations can be seen in Figures 4 and 6.

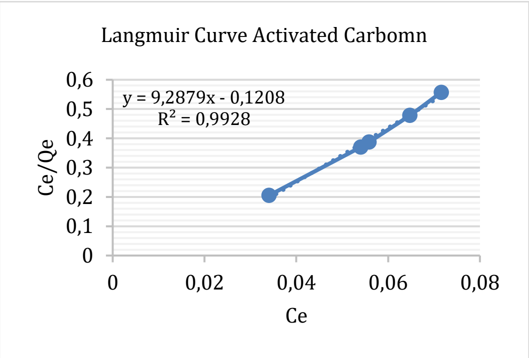
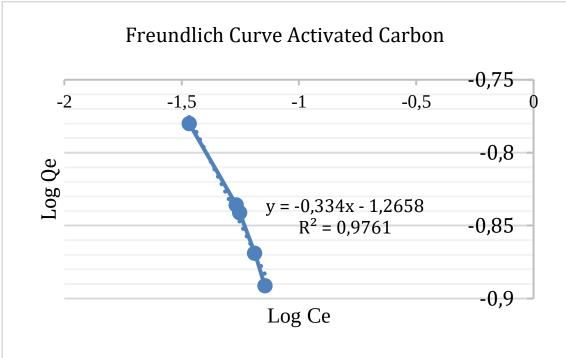


Figure 3. Graphic Isotherm Langmunir Activated Carbon Dosage Variation



.**Figure 4.** Graphic Isotherm Freundlich Activated Carbon Variation Dosage

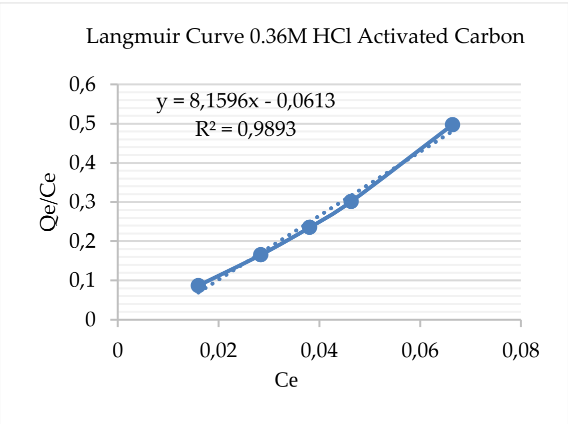


Figure 5. Graphic Isotherm Langmuir 0.36M HCl Activated Carbon Dosage Variation

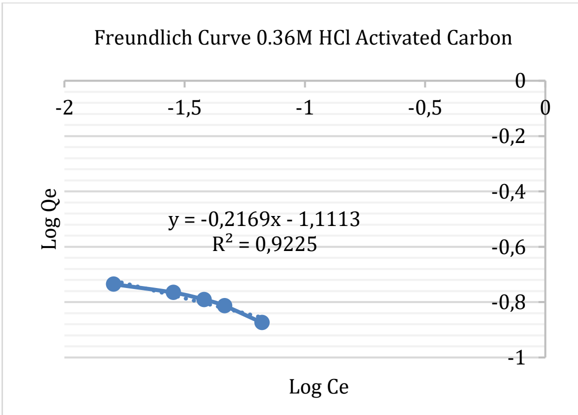


Figure 6. Graphic Isotherm Freundlich 0.36M HCl Activated Carbon Dosage Variation

Based on Figure 3 isotherm of Langmuir activated carbon above, the equation y = 9,2879x -0,1208 with slope R^2 = 0,9928 was used to calculate Qm of activated carbon adsorbents. Calculate the Qm value as follows:

Qm = 1/a
=1/0,1208
= 8,2781 mg/gr

As for 0.36M HCl activated carbon isotherm Langmuir curve can be seen in Figure 5. Based on the graphic, the equation y = 8,1596x - 0,0613 with slope R^2 = 0,9893 is obtained, so that the Qm of HCl is 0.36 M activated carbon, as follows:

Qm = 1/a
= 1/ 0,0613
= 16,3132 mg/gr

Capacity derived from the activated carbon adsorbent waste leaf purification eucalyptus oil with physical activation occurs when absorption of activated carbon Qm reaches 8.2781 mg/gr and Q to 0.36M HCl activated carbon of 16.3132 mg/g. Freundlich's isotherm explains the type of adsorption formed on the part of the layer and its weak bond. Freundlich isotherm is heterogeneous at the place of adsorption. (Rio,2022). Determining the Freundlich isotherm equation is done by calculating the log price Ce and Log Qe subsequently distributed and obtained the equation for activated carbon is y = -0,334x – 1,2658 with slope R^2= 0,9761 and the 0.36M HCl activated carbon equation is y= -0,2169x -1,1113 with slopes R^2 = 0,9225. From this equation, we get the constant value as follows:

Table 2. Value of the Freundlich Activated Carbon and 0.36M HCl Activated Carbon

Freundlich Constant		
	Activated Carbon	0.36M HCl Activated Carbon
1/n	0,9761	0,9225
n	1,0244	1,0840
ln k	1,2658	1,1113
k	0,2357	0,10553

The negative mark of the obtained Freundlich equation indicates the gradient of the curve's inclination to the left. Figures 4 and 6 show that the obtained graph is in the reverse position this is due to the resulting very low efficiency values. The adsorption values obtained are very small at low concentrations, but the absorption will increase with the increasing concentration of the solution. This is related by Dea's (2019) statement in his research, stating that the smaller the concentration, the lower the effectiveness values of the adsorption, but the adsorption will rise as the solution concentration increases.

The test of the Freundlich and Langmuir adsorption equations is shown with the activated carbon curve in Figures 3 and 4, whereas the 0.36M HCl activated carbon curve in Figures 5 and 6 with the value of the determination coefficient $R^2 \geq 0,9$ (near the number 1) has met the Langmuir and Freundlich equations. From Figure 3 and the active carbon charts 4, it is seen that the determinant coefficient value R^2 = 0,9928 in the Langmuir equation is greater than the determining coefficient value Freundlich R^2 = 0,9761. Whereas based on Figure 5 and the 6 0.36M HCl activated carbon showed that the factor determinant R^2 = 0.98 in the 93 equation of your Langmuir is larger compared to the determinants of Freundlich R^2 = 0.9225. This proves that the adsorption of activated carbon is a pattern of Langmuir's isotherm equation, so it can be concluded that the active adsorption of the leaf of eucalyptus oil against ammonia occurs chemically, i.e., only one layer lasts and the surface is homogeneous because each active site can only absorb one molecule. So describe the state of balance between the surface and the solution that can be reversible.

Isotherm Model Activated Carbon Adsorbent of Contact Time Variation

The Langmuir isotherm explains that the maximum adsorbent capacity occurs because there is a single layer of adsorbed material on the surface of the adsorbed material. On the Langmuir isotherm chart with Ce as the y-axis and Ce/Qe as the x-axis, a line equation (y = bx + a) will be obtained, which will determine the values of Qm and Kl, where Qm is 1/a and Kl is the value of b. From the data from the adsorbent dose variation test results, the maximum absorption capacity value can be calculated. Below is the graphic isotherm of Langmuir on Figures 7 and 9, and Freundlich's time variations can be seen on Figures 8 and 10.

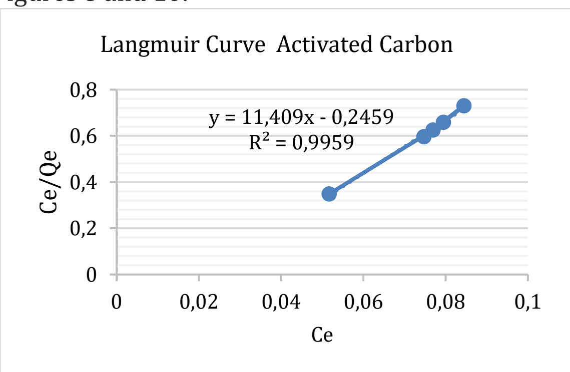


Figure 7. Graphic Isotherm Langmuir Activated Carbon Adsorbent of Contact Time Variation

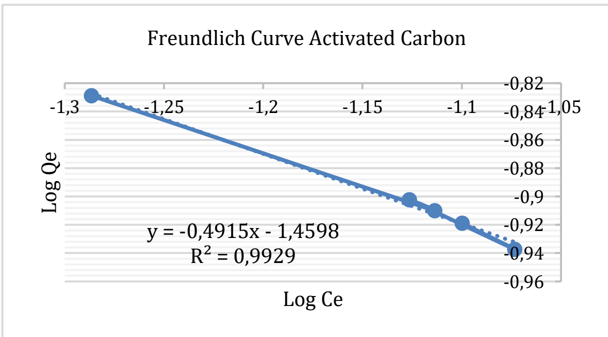


Figure 8. Graphic Isoterm Freundlich Activated Carbon Adsorbent of Contact Time Variation

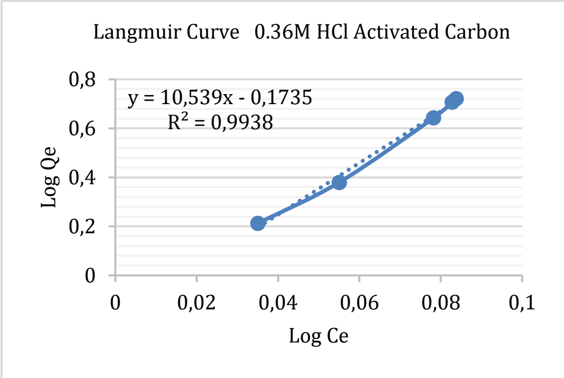


Figure 9. Graphic Isoterm Langmuir HCl 0.36M Activated Carbon of Contact Time Variation

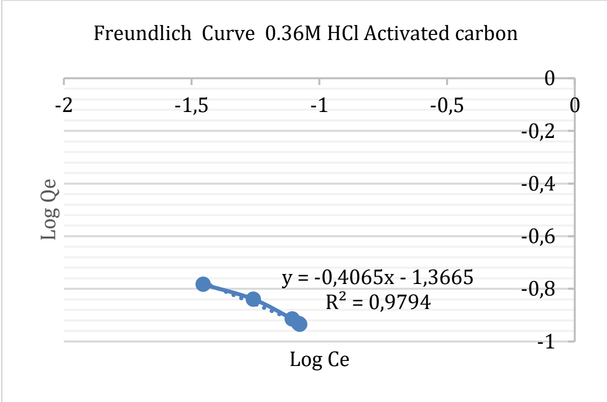


Figure 10. Graphic isotherm Freundlich HCl 0.36M Activated Carbon of Contact Time Variation

Based on the isotherm of Langmuir activated carbon in Figure 7, the equation y = 11,409x - 0,2459 with slope R^2 = 0,9959 was used to calculate the Qm of activated carbon adsorbents. Calculate the Qm value as follows:

Qm = 1/a
= 1/0,2459
= 4,0666 mg/gram

As for 0.36M HCl activated carbon isotherm graph by Langmuir can be seen in Figure 9. Based on the graphic, the equation y = 8,1596x - 0,0613 with slope R^2 = 0,9893 is obtained, so that the Qm of 0.36M HCl activated carbon adsorbent, as follows:

Qm = 1/a
= 1/ 0,0613
= 16,3132 mg/gram

The capabilities derived from the activated carbon adsorbents of waste leaves of eucalyptus oil purification with physical activation occur when the absorption of Qm of activated carbon reaches is 8,2781 mg/gr and Qm of 0.36M HCl activated carbon reaches is 16,3132 mg/g. Freundlich's isotherm explains the type of adsorption formed on the part of the layer and its weak bond. Freundlich's isotherm is heterogeneous at the place of adsorption. (Rio,2022). In determining the Freundlich isotherm equation, it is done by calculating the log price Ce and log Qe subsequently distributed and obtained. The equation for activated carbon is y = -0,334x - 1,2658 with slope R^2 = 0,9761, and the 0.36M HCl activated carbon equation is y = -0,2169x -1,1113 with slope R^2 = 0,9225. From this equation, we get the constant value as follows:

Table 3. Values of Freundlich Coal Constants

	Freundlich Constant	
	Activated Carbon	0.36M HCl Activated Carbon
1/n	0,9761	0,9225
n	1,0244	1,0840
ln k	1,2658	1,1113
k	0,2357	0,10553

The negative mark of the obtained Freundlich equation indicates the gradient of the curve's inclination to the left. Figures 8 and 10 show that the obtained graph is in the reverse position; this is due to the fact that the resulting efficiency values are very low. Factors influencing low-efficiency values include the initial concentration of the solution. The adsorption values obtained are very small at low concentrations, but the adsorption will increase as the solution concentration increases. This is supported by Dea's (2019) statement in his research, stating that the smaller the concentration, the lower the adsorption effectiveness values, but the adsorption will increase as the solution concentration increases.

The testing of the Freundlich and Langmuir adsorption equations is shown with the activated carbon curve in Figures 7 and 8, whereas the 0.36M HCl activated carbon curve in Figures 9 and 10, with the value of the determination coefficient $R^2 \geq 0,9$ (near the number 1), has met the Langmuir and Freundlich equations. From Figure 7 and Figure 8, the active coal shows that the determinant coefficient value R^2 = 0,9959 in the Langmuir equation is greater than the determining coefficient value Freundlich R^2 = 0,9929, whereas based on Figures 9 and 10, the 0.36M HCl activated carbon curve shows that R^2, 0.9938 in

Langmuir isotherm equation, is larger compared to the determinacy coefficient value Freundlich R^2. =0,97994. This proves that the adsorption of activated carbon follows the patterns of your Langmuir isotherm equation. So it can be concluded that the adsorption of activated carbon from eucalyptus oil leaves against ammonia is chemically occurring, i.e., only one layer lasts and the surface is homogeneous because each active site can only adsorb one molecule. So describe the state of balance between the surface and the solution that can be reversible.

Discussion

The results indicate that HCl 0.36 M activated carbon is highly effective in removing ammonia from wastewater in the leather processing industry. The high removal rates at both optimal dosage and contact time underscore the potential for practical application in industrial settings. The adherence to the Langmuir isotherm model further supports the reliability and predictability of the adsorption process.

This research opens opportunities for further studies focusing on the advanced characterization analysis of adsorbents from eucalyptus oil refinery waste, such as SEM and X-ray, to understand the adsorption mechanisms. It also paves the way for studies to assess the effectiveness of these adsorbents on other types of waste.

These findings have significant implications for the leather processing industry in Indonesia. By optimizing the dosage and contact time, the industry can enhance its wastewater treatment processes, reducing environmental impact and complying with environmental regulations. The use of HCl-activated carbon, in particular, offers a promising solution due to its high efficiency and relatively rapid action.

Conclusion

The results of a dose variation ammonia adsorption test showed that 0.36M HCl activated carbon at dosage 2.5 grams with % removal is 92% more better absorption capacity compared to 2.5 grams than activated carbon with %removal of 82.97%.The results of contact time variation ammonia adsorption test showed that 0.36M HCl activated carbon more better adsorbent capacity at 25 minutes with a removal rate of 82.48% compared to activated carbon at 25 minutes with %removal is 74.15%. The resultant isothermic model of carbon adsorption variation dosage and time variation on 0.36M HCl activated carbon and activated carbon follows the pattern of Langmuir's isotherm equation and is reversible.

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