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The Influence of Variation in Dosage and Contact Time of Activated Carbon Adsorbent from Clove Leaf Waste on the Concentration of NH_3 in Liquid Leather Industry Waste

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Abstract

Leather industry waste is one of the industries that contains hazardous substances due to the presence of organic and chemical contaminants, including NH_3 waste. Adsorption method is the most effective and economical way in leather industry waste management. This study aims to determine the effectiveness of varying doses and contact times in reducing NH_3 levels in leather industry waste. The activated carbon used in this study is obtained from waste clove oil distillation leaves, carbonized at 500 °C for 2 hours, and activated using 0.1 M KOH. For the adsorption process, different doses of activated carbon (0.5; 1; 1.5; 2; 2.5) grams and various contact times (5, 10, 15, 20, 25) minutes are used, with the addition of 0.3 mg/L NH_3 in 50 mL solution. The adsorption results for NH_3 are analyzed using UV-Vis spectrophotometer and the phenate method. The most effective NH_3 reduction is achieved with a 2-gram dose, resulting in a 43.45% decrease, while the optimal contact time is 10 minutes, leading to a reduction of 68.9%. The adsorption isotherms used for activated carbon from non-activated and KOH-activated clove leaves follow the Freundlich isotherm. For non-activated carbon, the values are N = -0.5203, kf = 0.0019715 for KOH-activated carbon, the values are N = -1.1885, kf = 0.0144 for varying contact times.

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Introduction

Indonesia's leather sector is expanding quickly as a result of strong export demand. This industry grew at a pace of 41,26% in 2022, and the value of leather products and footwear exported was USD 4.62 billion (Kemenprin, 2022). But there are also detrimental effects of the leather business on the environment. The leather business produces liquid waste that smells bad and includes harmful and dangerous compounds. The Leather Industry Zone (LIK) in Magetan hosts 35 small-scale leather processing businesses. The liquid waste from these businesses is categorized as hazardous due to its content of toxic and carcinogenic chemicals (Mahendra, 2019). The Magetan Environmental Agency recognized in 2021 that the leather business was contaminating waterways. Ammonia in liquid waste is one dangerous parameter that can harm water bodies and have an adverse effect on human health (Hamonangan & Yuniarto, 2022). Numerous techniques for treating waste have been used, such as electrocoagulation, precipitation, and adsorption. Adsorption is said to be the most advantageous option among them for small-scale leather tanning businesses (Kuncoro & Soedjono, 2022).

The process of chemicals being absorbed onto another surface is known as adsorption. Adsorbents with the capacity to effectively absorb a variety of waste kinds include activated charcoal (Hendrasarie, 2021). Results of studies using banana peels as activated carbon have indicated that the Barito River's ammonia reduction ranges from 38.28% to 96.68% (Alawiyah et al., 2022). According to a research by Saputra et al., (2021) using activated carbon can lower the amount of ammonia in liquid waste from tanning leather at PT. XYZ by 30.44% to 85.98%. Activated carbon may be produced from a variety of plant wastes, including the leaves of soapbark and mahogany trees.(Haris et al., 2019), tea leaf waste (Nurafriyanti et al., 2017), oil palm leaf waste (Farma & Tondang, 2019), pineapple leaf waste (M. I. Sari et al., 2021; Setiawan et al., 2017; Sibarani et al., 2022), teak leaf waste (Hydhayat et al., 2022), and clove leaf waste (Kusuma et al., 2023; Sudarni et al., 2021). Clove leaf waste can also be utilized to produce activated carbon (Kusuma et al., 2023; Sudarni et al., 2021). The research methodology employed UV-Vis spectrophotometry to measure NH_3 content in water, adhering to the Indonesian National Standard (N. P. Sari, 2020). Research and development of waste treatment technologies, such as adsorption utilizing activated carbon from waste clove leaf, indicate substantial potential to mitigate the negative environmental effects of the leather industry.

Based on these problems, the researcher plans to look into how the NH3 concentration of liquid waste from the leather sector is affected by different dosages and contact periods of activated carbon adsorbents made from clove leaf waste. The range of dose adjustments for activated carbon will be (0.5, 1, 1.5, 2, 2.5) grams, and the contact durations will be (5, 10, 15, 20, 25) minutes.

Methods

Equipment and Materials

The equipment used for this research includes a UV-Visible spectrophotometer, furnace, magnetic stirrer, hot plate, analytical balance, shaker, stirring rod, filter paper, 60 mesh sieve, 250 ml and 1000 ml beakers, 50 ml and 100 ml Erlenmeyer flasks, 50 ml, 100 ml, and 1000 ml measuring flasks, 25 ml measuring cylinder, glass funnel, dropper pipette, 5 ml and 10 ml volumetric pipettes, micropipette, porcelain crucible, crucible tongs, blender.

The materials used in this research are waste from clove oil distillation leaves obtained from Kare village, Kec. Kare, Kab. Madiun, KOH, distilled water, phenol (C_6H_5OH), sodium nitroprusside ($C_5FeN_6Na_2O_2$), oxidizing solution (sodium citrate ($C_6H_5Na_3O_7$) and sodium hypochlorite (NaClO)).

Procedure

1. Preparation of Clove Leaf Waste Sample

After two days of sun drying, the leftover leaf material from the distillation of clove oil is mixed. It is then sieved using a 60 mesh sieve.(Sudarni et al., 2021).

2. Production of Activated Carbon

The waste sample of sieved clove leaves is carbonized in a furnace for two hours at 500°C. Using a basic solution, KOH, the carbon is activated in accordance with the cited study. Weighing 100 grams of waste carbon from clove leaves, the carbon is submerged in a 0.1 M KOH solution. A magnetic stirrer is used to agitate the mixture for one hour at room temperature at 400 rpm. It is allowed to stand for a whole day. Following activation, distilled water is used to rinse the solid activated carbon until it achieves a neutral pH. It is then dried for an hour at 110°C in an oven. (Sudarni et al., 2021).

3. Characterization of Activated Carbon

a. Moisture Content

The water content in the carbon is related to its hygroscopic properties. The procedure involves weighing 1 gram of the sample and placing it in a crucible. The crucible is then put in an oven at +110°C, cooled in a desiccator, and weighed until a constant weight is obtained (Almira et al., 2021).

b. Ash Content This analysis is performed by weighing 1 gram of the sample in a porcelain crucible. The crucible is placed in a furnace at a temperature of 750°C for 3 hours, cooled in a desiccator, and then weighed until a constant weight is achieved (Almira et al., 2021).

c. Volatile Matter Content

1 gram of the sample is placed in a crucible and covered with a known-weight crucible lid. It is then heated to 900°C in a furnace for 15 minutes. The crucible is taken out of the furnace and weighed until a constant weight is reached (Almira et al., 2021).

d. Fixed Carbon

Indicates the amount of carbon remaining in the material after volatile matter is removed (Almira et al., 2021).

e. Bulk Density

To determine the bulk density of the activated carbon, a 25 mL pycnometer is used. The pycnometer is dried and weighed before use. Then, the pycnometer is filled with distilled water and weighed to determine its weight with water. Approximately 5 mL of water is removed before adding around 1 gram of activated carbon to the pycnometer. Distilled water is added until it's full, and the pycnometer is weighed again (Sagala, 2018).

4. Creation of Calibration Curve

A calibration curve was created for this investigation utilizing a number of standards that varied in concentration from 0.1, 0.2, 0.3, 0.4, and 0.5 mg/L. A 100 mL measuring flask containing a 1 mg/L NH3 stock solution was used to construct the standard series. The solution was then mixed with phenol solution, sodium nitroprusside solution, and oxidizing solution, and allowed to sit at room temperature for approximately one hour. Next, a UV-Visible spectrophotometer was used to measure each standard series' absorbance at a wavelength of 640 nm (Kurniawan et al., 2022). This was done to make sure the detector could pick up the corresponding absorbance values based on the absorption data.

2.5 Determination of Optimal Dose Variation of NH_3 on Non-Activated and KOH-Activated Clove Leaf Waste Activated Carbon

The amounts of non-activated and KOH-activated waste activated carbon from clove leaves were measured and ranged from 0.5 to 2.5 grams. They were then mixed with 50 mL of ammonia at a concentration of 0.3 mg/L in a sample solution. For fifteen minutes, the mixture was agitated using a shaker set to 250 rpm. The mixture was then filtered after being allowed to stand for 15 minutes. The UV-Visible spectrophotometer was used to determine the amount of NH3 present in the filtrate. There were three iterations of the treatments.

2.6 Determination of Optimal Contact Time of NH_3 on Non-Activated and KOH-Activated Clove Leaf Waste Activated Carbon

The residue of non-activated and KOH-activated clove leaves from the distillation of clove oil was weighed at two grams and mixed with 50 milliliters of ammonia at a concentration of 0.3 milligrams per liter. A 250 rpm shaker was used to agitate the mixture. After shaking for different amounts of time—5, 10, 15, 20, and 25 minutes—the material was filtered. The UV-Visible spectrophotometer was used to determine the amount of NH3 present in the filtrate. There were three iterations of the treatments.

Results

Characteristics of Clove Leaf Waste Activated Carbon

Table 1. Characteristics of Clove Leaf Waste Activated Carbon

Component	Non-activated clove leaf charcoal	Activated Clove Leaf Charcoal Activated with 0.1 M KOH	SNI 06-3730-1995 (BSN, 1995)
Moisture Content (%)	0,64	0,35	15%
Ash Content (%)	10,01	5,96	10%
Volatile Matter (%)	21,34	18,49	25%
Fixed Carbon (%)	68,01	75,20	65%
Bulk Density (%)	0,31	0,17	0,3-0,35 g/mL

It is clear from Table 1 that the water content test satisfies the Indonesian National Standard (SNI) criteria. The efficacy of the chemical activation process in binding water molecules and removing water content during activation is shown by the low water content in activated carbon activated with KOH.(Previanti et al., 2015). The reduction in water content leads to the enlargement of activated carbon pores, an increase in surface area, and enhanced adsorption capacity. The research results also demonstrate that the volatile matter and carbon content in KOH-activated activated carbon conform to the Indonesian National Standard (SNI). The content of bound carbon indicates the material's purity with respect to the carbon fraction (Nitsae et al., 2020). The specific weight of activated carbon is influenced by its particle size, where smaller particle size corresponds to a larger surface area and higher adsorption capacity. The density of activated carbon remains unchanged due to particle dimensions (Sagala, 2018).

Calibration Curve

The calibration curve was made using the absorbance values that came from using a UV-visible spectrophotometer to measure the working solution's concentration. As the working solution concentration rises, so does the NH3 calibration curve. The working solution containing the lowest concentration (0.36667) at 0.1 ppm has the lowest absorbance value. The working solution with the greatest concentration of 0.5 ppm, 0.70067, has the highest absorbance value.

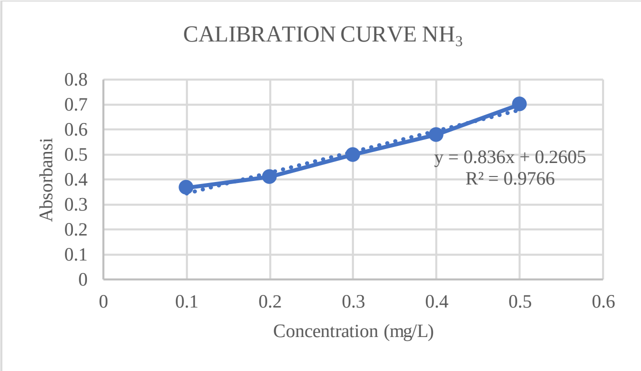

Figure 1. NH_3 Calibration Curve

Figure 1 presents the calibration graph showing the relationship between the concentration of the working solution and the instrument response, represented by a straight line indicating linearity, of the UV-Visible spectrophotometer. The calibration curve for NH3 testing is considered valid if the correlation coefficient (r) has a value of ≥ 0.97 (N. P. Sari, 2020).

Determination of Optimum Dose Variation of NH_3 on Non-Activated and KOH-Activated Clove Leaf Waste Activated Carbon

One factor that is crucial in figuring out the adsorption capacity is dose variation. Reducing the concentration of NH3 in the liquid waste from the leather industry requires careful consideration of the adsorbent dose. Thus, precise dose calculation is essential to attain the best possible adsorption efficiency. Figure 2 below displays the findings of the investigation into NH3 parameter reduction utilizing the activated carbon adsorption technique from clove leaf waste.

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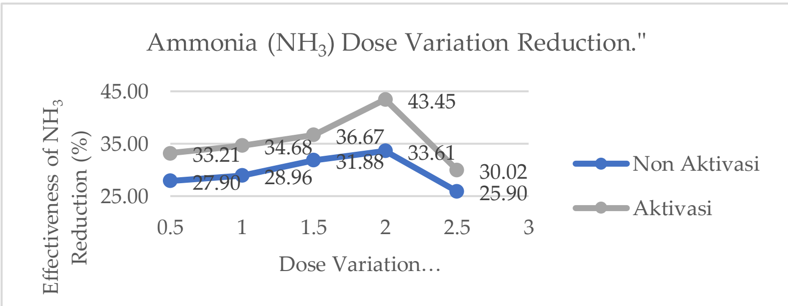

Figure 1. Ammonia (NH_3) Dose Variation Decrease Graph

The study's findings show that non-activated activated carbon reduces NH3 most effectively at a dosage of 2 grams (33.61%), and least effectively at a dosage of 2.5 grams (25.90%). Conversely, the greatest decrease in KOH-activated activated carbon was seen at a dose of 2 grams (43.45%). However, a dose of 2.5 grams (30.02%) resulted in the lowest decrease. Figure 2 shows that as the adsorbent dose is increased, the NH3 concentration of both non-activated and KOH-activated activated carbons decreases. Its adsorption capability is increased with a larger dosage of adsorbent. This may be explained by the higher dose of adsorbent, which increases the amount of surface area and pores accessible to bind adsorbate molecules during the adsorption process.

Determination of Optimum Contact Time for NH_3 on Non-Activated and KOH-Activated Clove Leaf Waste Activated Carbon

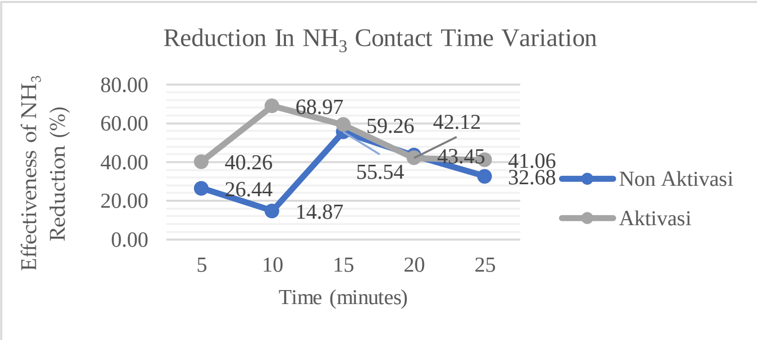

Figure 2. Graph of NH_3 Contact Time Variation Decrease

The term "contact time" describes the amount of time that passes between adding the adsorbent and filtering the NH3 filtrate from the adsorbent after rotating the NH3 sample in a shaker. In this study, the impact of contact time was examined by adjusting the duration of contact from 5 to 25 minutes. For each adjustment in contact time, the adsorbent dose was fixed at 2 grams. According to Figure 4.3, the most effective time to reduce non-activated activated carbon was found to be 15 minutes (55.54%), but the most effective time to reduce KOH-activated activated carbon was 10 minutes (68.9%). The percentage of adsorption for non-activated activated carbon was found to decrease at a contact time of 20 minutes, whereas the percentage of adsorption for KOH-activated activated carbon was shown to decrease at a contact time of 15 minutes. This suggests that the ideal duration for adsorbent removal has been attained at a contact period of 15 minutes for non-activated activated carbon and 10 minutes for KOH-activated activated carbon.

Adsorption Equilibrium of NH_3 by Clove Leaf Waste Activated Carbon Adsorbent

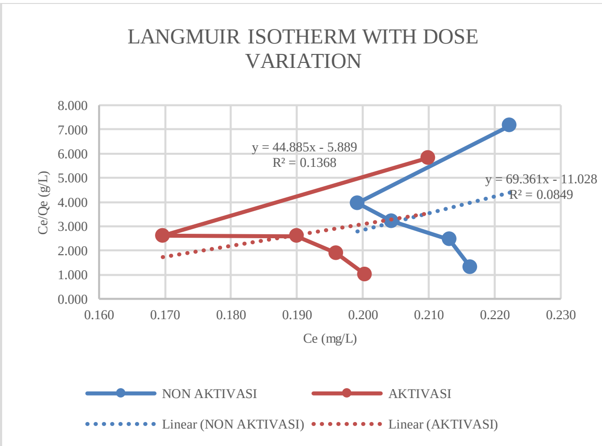

Figure 3. Langmuir Isotherm Graph with Dose Variation

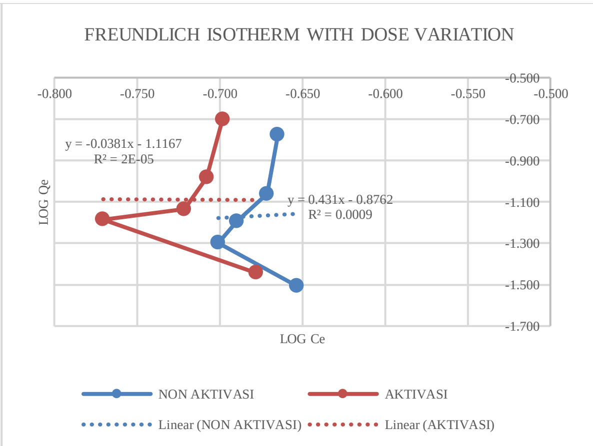

Figure 4. Freundlich Isotherm Graph with Dose Variation

Successful completion of the Freundlich and Langmuir adsorption tests is demonstrated by the linearization graphs, which show determination coefficients (R2) that are approaching or above 1. According to the observation results (Figures 6 and 7), the Freundlich adsorption equation pattern is followed by the NH3 adsorption by clove leaf waste activated carbon, with corresponding R2 values of around 0.90998 and 0.9858. The NH3 adsorption capacity is represented by the Langmuir Qmax values, which are -0.1016 mg/g for non-activated carbon and -0.6786 mg/g for carbon activated with KOH. The strength of adsorbate binding is indicated by the Kl affinity constant, which is -0.1295 L/mg for non-activated carbon and -0.0602 L/mg for carbon activated with KOH. Negative Kl values suggest that the Langmuir equation is not being followed. The relative capacity, Kf, and surface heterogeneity, n, are the expressions for the Freundlich isotherm equation.Non-activated carbon: Kf 0.0019715 mg/g, n -0.5203. KOH-activated carbon: Kf 0.01444 mg/g, n -1.1885. n <1 signifies high heterogeneity, while n >1 indicates a cooperative process (Ajemba, 2014 in Triwijaya, 2022). The results indicate that variations in NH_3 adsorption contact time follow the Freundlich isotherm, with the adsorbate existing in a multilayer, reversible fashion involving van der Waals forces and without specific activation energy.

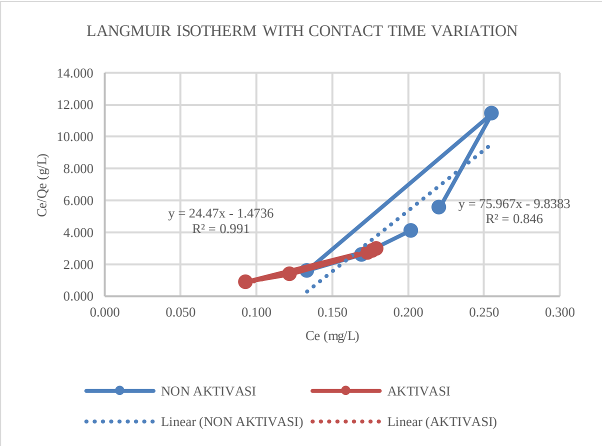

Figure 5. Langmuir Isotherm Graph with Contact Time Variation

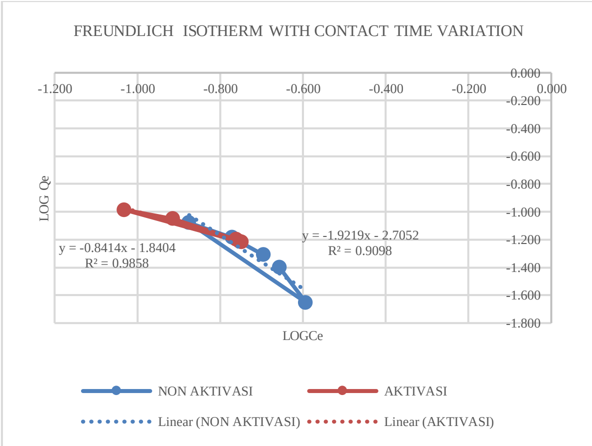

Figure 6. Freundlich Isotherm Graph with Contact Time Variation

Conclussion

This study's findings are as follows: With a decrease of around 43.45%, the most efficient dose for adsorbing NH3 is 2 grams of KOH-activated carbon. Ten minutes is the ideal adsorption duration for NH3 using KOH-activated carbon, yielding a decrease of around 68.9%. The Freundlich isotherm is an appropriate adsorption isotherm for activated carbon derived from clove leaf waste, regardless of whether it is KOH-activated or not. According to the values of the isotherm parameters, KOH-activated carbon has N = -1.1885, kf = 0.0144, and R2 = 0.9858, whereas non-activated carbon has N = -0.5203, kf = 0.0019715, and R2 = 0.9098.

The following are the conclusions drawn from this study: The dose of 2 grams of KOH-activated carbon is the most efficient in adsorbing NH3, with a decrease of around 43.45%. Ten minutes is the ideal adsorption time for NH3 with KOH-activated carbon, yielding a 68.9% reduction. Whether the activated carbon is KOH-activated or non-activated from clove leaf waste, the Freundlich isotherm is the most efficient one for adsorption.Non-activated carbon has N = -0.5203, kf = 0.0019715, and R2 = 0.9098 isotherm parameter values, but KOH-activated carbon has N = -1.1885, kf = 0.0144, and R2 = 0.9858.

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