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Synthesis of Functional Groups on Surface-Modified Activated Carbon for Nitrate Removal from Tofu Home Industry Wastewater

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ABSTRACT

This research successfully converted spent coffee grounds (SCG) into powdered activated carbon (AC) through carbonization at 400°C. The surface functional groups of this carbon were subsequently modified using hydrochloric acid as an activating agent to enhance its adsorption properties. The purpose of this research was to evaluate the performance of this modified activated carbon (MAC) as an adsorbent for reducing nitrate content in tofu industry wastewater, which is characterized by high concentrations of nitrate ions. The MAC was characterized using SEM and FTIR analysis to determine its surface morphology and functional groups, which are critical for adsorption process of capturing atoms, ions, and molecules on a materials surface. The batch adsorption experiments using AC and MAC as adsorbent were then performed for reducing nitrate content in tofu industry wastewater. Batch adsorption experiments were conducted under optimal conditions: a pH of 7±0.2, room temperature, a stirring rate of 150 rpm, and an adsorbent dose of 1 g/L. The results demonstrated a significant decrease in nitrate concentration from 28.6 mg/L to 1.2 mg/L, achieving a 95.80% removal efficiency at an equilibrium contact time of 180 minutes. The performance of the MAC was comparable to, and even more favorable than, that of unmodified activated carbon (UAC) used as a control that only achieved 58.74% percentage removal of nitrate. This remarkable nitrate removal efficiency is attributed to the distinct morphology and enhanced surface properties imparted by the hydrochloric acid modification. Therefore, modified activated carbon from coffee grounds shows high potential as an effective adsorbent for wastewater treatment.

Keywords: adsorbent, adsorption, nitrate removal, spent coffee grounds, surface modification.

1. INTRODUCTION

Spent coffee grounds (SCG) are an abundant organic waste product generated from the coffee extraction process. Due to their high organic matter content and large production volume, they represent a feasible and promising precursor for manufacturing activated carbon (AC). This AC from spent coffee grounds can subsequently be used as an adsorbent in water and wastewater treatment applications [1,2]. In this study, spent coffee grounds were converted into powdered activated carbon through high-temperature carbonization. To enhance its adsorption properties, hydrochloric acid (HCl) was used as an activating agent to modify its surface functional groups for use as a commercial-grade adsorbent [3].

The need for effective water treatment is critical, particularly for pollutants like nitrate ions. The presence of nitrates in water bodies, primarily from human activities such as agricultural runoff and industrial discharge, leads to severe environmental problems like eutrophication and water quality degradation. It also poses significant health risks, including methemoglobinemia ("blue baby syndrome"), diabetes, and cancer [4-25]. Consequently, regulatory bodies like the World Health Organization (WHO) and the

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Indonesian Environmental Protection Agency have set strict limits on nitrate concentrations in water. One notable source of nitrate-rich wastewater is the tofu manufacturing industry, a prevalent home-based enterprise in many East and Southeast Asian countries [21,26].

While various physical, biological, and chemical methods exist for nitrate removal including reverse osmosis, biological denitrification, and ion exchange, adsorption process stands out as a particularly promising methof for water and wastewater treatment. It is favored for its technical and economic feasibility, design simplicity, and effectiveness at low contaminant concentrations [3,11,13,20,26-28]. The adsorption capacity of activated carbon can be significantly enhanced by modifying its surface functional groups, which can bond with heteroatoms like oxygen, nitrogen, and halogens on the carbon surface [3,20,26-28]. Hydrochloric acid was selected for this modification due to its high solubility, volatility, and minimal impact on the final composites properties [3].

While the use of AC for water treatment is well-established, its application for targeted nitrate (NO_3^-) removal from specific industrial wastewater streams like tofu wastewater remains inadequate and faces significant limitations. Therefore, this study aims to investigate the use of HCl-modified, spent coffee ground-based activated carbon for adsorbing nitrate ions from tofu wastewater. The effects of surface modification and solution pH were evaluated, and the underlying removal mechanisms were also assessed.

2. RESEARCH METHODS

2.1. Tofu Wastewater

Tofu wastewater collected from one of the home-based tofu industries in Martapura, South Kalimantan. This study reports that tofu wastewater contains high organic compounds, with a temperature of 32±2°C, a pH of 4.6±0.2, and nitrate content of approximately 28.6±0.4 mg/L. The characteristics of the tofu wastewater used in this study exceeds quality standards. The quality standard of temperature of 25°C, pH of 6–9, and nitrate content of 20 mg/L.

2.2. Preparation of Activated Carbon and Modified Activated Carbon from the Spent Coffee Grounds Using Hydrochloric Acid as Activator

The activated carbon adsorbent employed in this study is generated from wasted coffee grounds. After brewing the coffee, the grounds were oven-dried for 24 hours at 80ºC. Following that, it was carbonized for 1 hour at 400°C and chemically activated by immersing 50 g of the activated carbon in 150 mL solution of hydrochloric acid, HCl (Sigma Aldrich) at concentrations of 0.5, 2, and 5 N for 24 hours. The presence of the activator in relation to the amount of water in the solution acts as a dehydrating agent [3,20]. It removes bound water to enhance the porosity of the raw material, enlarging the pores of the activated carbon and increasing its adsorption surface area; also, the surface functional properties were changed [3,5]. Additionally, the weight of the activated carbon yield is computed to establish the percentage of shrinkage, and a water content test is conducted to assess the residual water and ash content in the activated carbon.

2.3. Characterization of Activated Carbon and Modified Activated Carbon from the Spent Coffee Grounds Using Hydrochloric Acid as Activator

The surface morphological structure of unmodified activated carbon (UAC) and modified activated carbon (MAC) are investigated by Field Emission Scanning Electron Microscopy (FE-SEM, JOEL JSM-6500F). Fourier Transform Infrared Spectrometry was used to determine the functional groups on the surface of the samples (FT-IR, bio-rad, digilab FTS-3500).

2.4. Batch Adsorption Process for Nitrate Removal Using Activated Carbon from the Spent Coffee Grounds

Batch adsorption tests were conducted by adding 100 mL tofu wastewater solution containing nitrate ions to a 250 mL HDPE bottle and adjusting the pH with NaOH (1 N, Sigma Aldrich) or HNO_3 (1 N, Panreac, 65 percent solution) until determined pH. Following that, weighed quantities of AC (UAC, Unmodified AC and MAC, Modified AC) were introduced to plastic bottles. After that, the mixture was placed in a shaker equipped with a water bath (Firstek Scientific) and kept at a pre-determined temperature of 25±2°C for a certain contact duration of adsorption process. Centrifugation (EBA 3S, Hettich) and filtration over a 0.22 μm pore size of PVDF membrane filter (Sigma-ALdrich) were used to separate the solution. Finally, residual nitrate concentrations in the filtrate were determined using nitrate concentrations in the supernatant solution were determined using a UV–Vis spectrophotometer (DR 500) set to 220 nm in accordance with the standard procedure for the investigation of water and wastewater [4,10,20,24,25]. The difference between the initial and equilibrium adsorbate concentrations was used to compute the amount of nitrate adsorbed. The adsorption tests were repeated three times and the average value was calculated.

The following equations (1) and (2) were used to compute the adsorption capacity and percentage:

$$\text{Adsorption capacity, } q_e = \frac{(C_i - C_e)}{m} \text{ x } V \quad (1)$$

$$\text{Percentage removal, } \% = \frac{(C_i - C_e)}{C_i} \text{ x } 100 \quad (2)$$

Where q_e is adsorption capacity (mg/g), C_i and C_e are the initial and final concentration of respective anions (mg/L), m is the mass of the adsorbent (g) and V is the volume of the respective solution (L).

3. RESULTS AND DISCUSSION

3.1. Characterization of Activated Carbon from Spent Coffee Ground

The results of the activated carbon characteristic analysis show that test parameters used are in accordance with Indonesian National Standards Agency (SNI) No. 06-3730-1995 for activated carbon. The moisture content of the activated carbon from spent coffee ground used in this study of 3.7% (max. 15%) and ash content of 1.69% (max 2.5%). This indicates that activated carbon made from coffee grounds has the potential to be used as activated carbon.

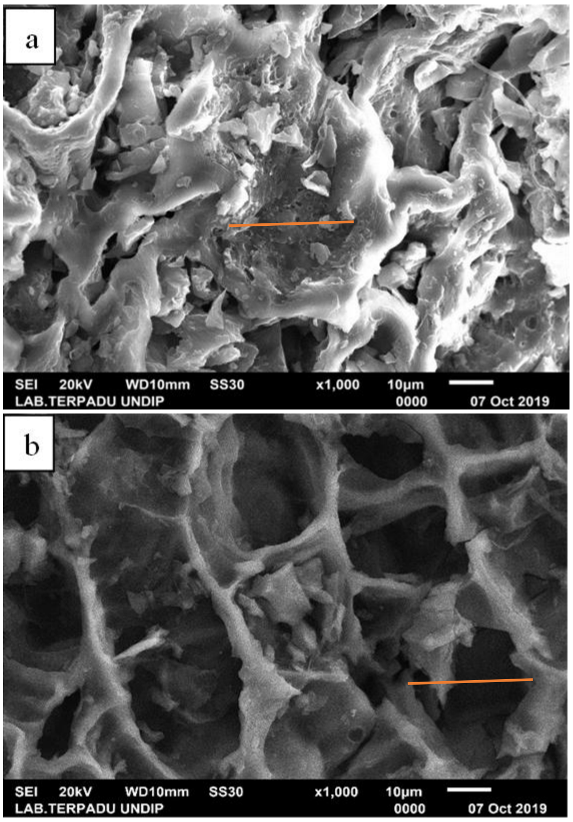

Figure 1. The FE-SEM morphology images of activated carbon from spent coffee ground (a) unmodified AC (UAC) and (b) modified AC with 2 N HCl (MAC).

The surface morphology and porosity of obtained activated carbon were observed by SEM images as shown in Figure 1. Seems in Figure 1b that activated carbon that has been activated using HCl (MAC) has larger pores than that which is not activated (UAC, Figure 1a), because HCl as activator agent is a water-binding agent that perfect for dissolving organic and inorganic substances bound in carbon material. It shown more pores are formed and the adsorption ability of activated carbon from the spent coffee grounds is will be increased. These differences may account for increased adsorption performance of MAC (BET surface area of 16.314 m^2/g) for nitrate anions compared with UAC (BET surface area of 10.125 m^2/g). Increasing the surface area of AC improves its ability to adsorb NO_3^-. The capacity for NO_3^- is increased by the surface roughness, high porosity, and large surface area associated with the porous structure [15].

Beside surface morphology by SEM, the obtained activated carbon were also analysis by FTIR for it surface functional group characteristic. Shown in Figure 2, there are some peaks showing with high intensity in the wave number region of 3750.24 cm^{-1} which indicates the presence of O-H groups. Followed by adsorption shown at wavenumber 2935.2, 2852, 1592.8, 1380, 1250.4, 1056, 871.2 and 547.2 cm^{-1} and at a wave number of 1592.8 cm^{-1} indicate the presence of C=C vibrations in the aromatic ring. The difference can be seen after adding 2 N HCl, the intensity decreases and a new peak is produced in this figure for the modified one, the new peak produced at 750.4 cm^{-1} is the presence of a C–Cl group due to interactions that occur on the surface of the activated carbon. Then, there is absorption at wave number 1592,8 cm^{-1} which indicates the presence of aromatic C=C bonds in the activated carbon structure. At a wave number of 1380 cm^{-1}, it indicates the presence of an S=O bond strain. A wave number of 1250.4 cm^{-1} indicates the presence of a C–O stretching bond, while a wave number of 744 cm^{-1} corresponds to a C–Cl bond. The C–Cl stretching vibration typically occurs within the range of 600 cm^{-1} to 850 cm^{-1}. The intensity of the peak is typically medium to strong, driven by the significant dipole moment change during the stretch [2]. Supported by previous research [15], in the FTIR spectrum relating to the adsorption of NO_3^- anions by the adsorbent, peaks were observed at wavenumbers of 693.2, 910.4, 1007.5, 2848.9, 2924.3, 3527.4, 3651.5 and 3691.9, respectively. The modifications to the adsorbent allowed for confirmation of the functional groups in the FTIR spectrum, and the chemical structure was qualitatively considered to be satisfactory.

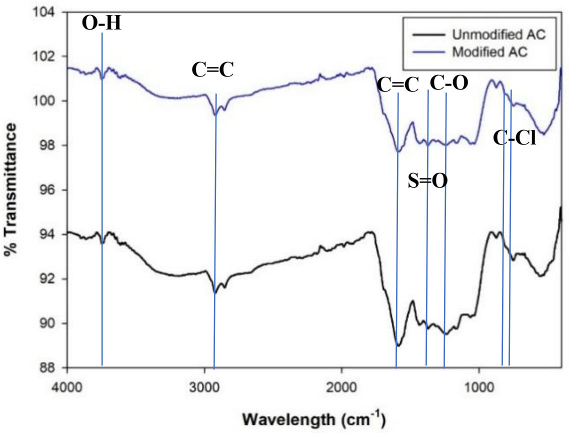

Figure 2. FT-IR spectra of UAC and MAC with 2 N HCl.

Superior textural characteristics may result in an increase in the number of available active sites and an increase in the mass transport efficiency of pollutants adsorbing [6-8]. The ability of an adsorbent to adsorb is a result of the synergy between its surface chemistry and its textural characteristics. A high surface area (MAC confirmed by BET surface area of 16.314 m^2/g and UAC of 10.125 m^2/g) is a prerequisite for high capacity, but the pore size distribution plays a crucial role in determining how well that surface area can be accessed by a specific adsorbate molecule. The optimal adsorbent is constructed analysed by FE-SEM as shown in Figure 1 with a hierarchical pore framework and customized surface chemistry to provide the highest adsorption capacity for its targeted use. FTIR results show that activated carbon that has been soaked and modified with hydrochloric acid produces more surface oxygen than the chemically activated pore structure. The hydrochloric acid treatment of adsorbent, MAC, successfully oxidized the surface of the carbon adsorbent. This is evidenced by the introduction of new and intensified infrared bands corresponding to carbonyl (C=O), carboxyl (O–C=O), and hydroxyl (O–H) functional groups, then the adsorbent has more surface oxygen. Based on the FTIR results above, it can be concluded that Figure 2 has a functional group with an activated carbon structure in general which contains O–H, C=C aromatics, C–H, S=O, C–O and C–Cl groups which are used as active groups to adsorb adsorbate.

3.2. Effect of Contact Time of Nitrate Removal

Contact time is the time required for AC from the spent coffee grounds adsorbent to be able to absorb the nitrate contained in the tofu wastewater sample. According to previous research, a longer interaction time between the adsorbent and the adsorbate allows the number of collisions to occur so that the percent adsorption increases [9].

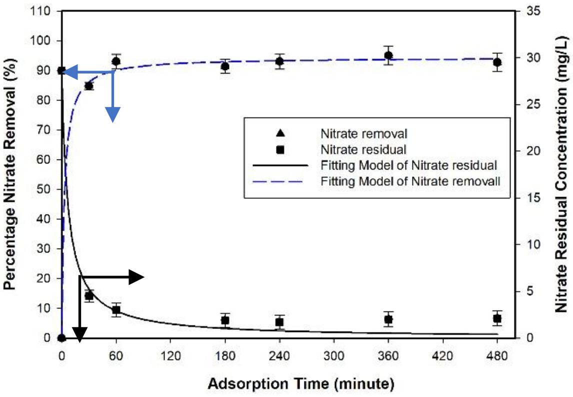

Figure 3. Effect of contact time of nitrate removal and nitrate residual at room temperature, MAC adsorbent dose of 1 g/L, pH_e of 7±0.2, and stirring rate of 150 rpm.

The effect of contact time on the adsorption of nitrate onto the adsorbent as shown in Figure 3 was investigated at room temperature, 1 g/L adsorbent, pH_e of 7±0.2, and stirring rate of 150 rpm. The residual nitrate concentration reduced dramatically from around 30 mg/L to 3.5 mg/L during the first 60 minutes, which was presumably owing to diffusion into the pores and/or adsorption onto the surface of the adsorbent. At first, the nitrate concentration gradient diffusing into and across the adsorbent's pore was quite large. Following that, the nitrate content was slightly dropped to 1.90 mg/L and remained constant for the next three hours, until equilibrium was attained. The early increase in adsorption efficiency results from the extensive surface area, abundant empty sites, high reactivity, and increased availability of binding sites close to the clay surface; however, after equilibrium is achieved, the vacant sites become occupied by NO_3^- anions, and the efficiency remains constant. Three hours (3h) of reaction time was allowed for the equilibrium adsorption studies. The optimal equilibrium period suggested that samples of tofu wastewater had saturated the activated carbon from wasted coffee grounds.

3.3. Effect of Initial pH on Nitrate Removal using UAC and MAC as Adsorbent

The amount of adsorbed nitrate depends on the distribution of N_2O and NO_3^- which are controlled by pH. It had been reported that nitrate adsorption process is pH dependent and the optimum nitrate adsorption occurs in neutral pH range, such as silica at pH 5 to 7, zirconium at pH 3 to 10, and zero valent iron at pH 5.5 to 8.5 [5,15,23,24]. The primary influencing factor on the adsorption process is pH, since it influences the charge of the surface functional groups on the adsorbent, which may have an effect on the adsorbate elimination mechanism.

The effect of equilibrium pH was examined using a three-hour reaction period, a 1 g/L adsorbent dosage, a shaking rate of 150 rpm, and a room temperature. The pH effect on nitrate adsorption density was demonstrated in Figure 4 throughout a wide pH range of 3–9. The nitrate adsorption envelop for UAC and MAC was bell-shaped, similar to that of common oxyanions. The nitrate adsorption using UAC from Figure 4a achieving a 58.74% percentage removal of nitrate (reducing the nitrate level from 28.6 mg/L to 11.8 mg/L), while at the same adsorption process using MAC shown better percentage removal of 95.8% (reducing the nitrate level from 28.6 mg/L to 1.2 mg/L).

At pH 7±0.2, adsorption was maximal. When the pH of the solution was low and the adsorbent had a positive charge, electrostatic contact was minimal. On the other hand, the force operating between the neutral charge of the nitrate molecule (N_2O) and the adsorbent surface is the weaker van der Waals interaction, independent of its composition, which explains why nitrate was adsorbed while in an acidic state. When nitrate ion (NO_3^-) is generated at a pH greater than 6, the nitrate adsorption density increases as the pH increases, the nitrate ion being adsorbed via electrostatic contact on the adsorbent surface [16].

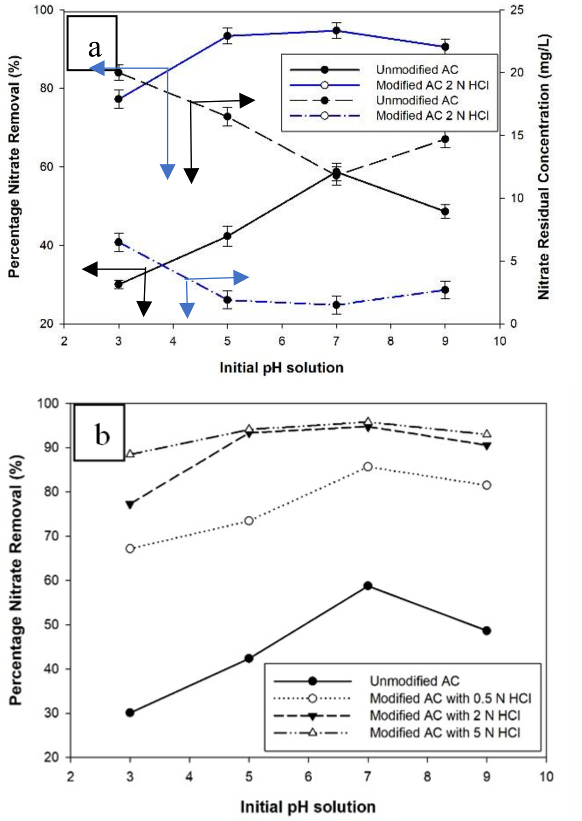

Figure 4. Effect of initial pH solution of nitrate removal and nitrate residual at room temperature, adsorbent dose of 1 g/L, adsorption time of 3 h, and stirring rate of 150 rpm: a) using UAC and MAC with 2 N HCl, b) using UAC and MAC with various HCl concentration.

MAC from coffee grounds, treated with 2 N HCl at pH 7±0.2, exhibits an optimal pH for reducing nitrate levels in tofu industrial wastewater, with a decrease of 95.8% as shown in Figure 4b (the concentration of nitrate parameters from 28.6 mg/L to 1.2 mg/L as shown in Figure 4a). When the pH of the solution was increased above 7, however, the nitrate adsorption density dropped. At this pH, the surface sites of the adsorbent are deprotonated and acquire a negative charge, rendering adsorption of negative charge nitrate (NO_3^-) species undesirable owing to repulsive force. Additionally, it may enhance the concentration of hydroxyl ion (OH^-), which has intermolecular competition with nitrate for both UAC and MAC adsorption.

The presence of an overabundance of hydroxyl ions next to the adsorbent's binding sites harmed the adsorption process [10,13]. It was found that nitrate weakly adsorbed by the adsorbent, and adsorbed via electrostatic attraction and van der Waals interaction as the adsorption mechanism of nitrate removal [8,10,17,26]. This is in accordance with the theory that states that the greater the concentration of activated carbon, the greater the surface area will be and the number of pores on the activated carbon will be more. So that, the adsorption of nitrate that occurs in tofu wastewater will be better.

4. CONCLUSION

Activated carbon was successfully synthesized from spent coffee grounds through a carbonization process at 400°C. The surface structure and functional groups of the carbon were then modified using hydrochloric acid at various concentrations. The resulting unmodified activated carbon (UAC) and modified activated carbon (MAC) were applied as adsorbents to remove nitrate ions from tofu wastewater. The equilibrium time for nitrate reduction using MAC modified with 0.5 N HCl was 180 minutes of stirring, achieving a 93.36% reduction in nitrate levels (from an initial concentration of approximately 30 mg/L to 1.9 mg/L). The optimum conditions for nitrate removal were achieved with MAC modified with 2 N HCl at a pH of 7±0.2, resulting in a 95.8% reduction and a final nitrate concentration of approximately 1.2 mg/L. Higher concentrations of the hydrochloric acid activator increased pore formation on the activated carbon, thereby enhancing its adsorptive capacity. This was evidenced by the significant decrease in nitrate levels in the wastewater. Both UAC and MAC derived from spent coffee grounds proved to be effective, non-toxic, biodegradable adsorbents suitable for removing nitrate from polluted water.

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