Helium: Journal of Science and Applied Chemistry
https://helium-fmipa.unpak.ac.id/

Volume 5, Number 2, December 2025, Page 98 - 104
e-ISSN: 2776-4508; p-ISSN: 2776-1711
Publisher : Universitas Pakuan

Synthesis of Activated Carbon from Coffee Bean Skin to Reduce BOD in Liquid Waste from Rambak Cracker Industry

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DOI: *https://doi.org/10.33751/helium.v5i2.17*
Article history: *received: 27-01-2025; revised: 25-05-2025; accepted: 12-12-2025; published: 13-12-2025*

ABSTRACT

Liquid waste from the rambak cracker industry in Tulungagung primarily contains organic compounds that can lead to high levels of Biological Oxygen Demand (BOD). The objectives of this study were: 1) to determine the characteristics of activated carbon from coffee bean skins synthesized from coffee bean skins with different concentrations of H_3PO_4 activator. 2) to determine the effect of activated carbon mesh size on BOD levels of liquid waste from the Rambak Cracker Industry. Activated carbon from coffee bean skins was synthesized with H_3PO_4 as an activator at 5, 10, and 20% concentrations, with a 200-mesh size. The synthesis results were compared with liquid waste from the rambak cracker industry to determine the difference in BOD levels. The quality of activated carbon was analyzed in accordance with SNI 06-3730-1995, including water content, ash content, volatile matter content, bound carbon content, iodine absorption capacity, and functional groups. The results of the characterization of 200 mesh coffee bean skin activated carbon obtained water content of 5.00; 5.04; 5.18%, ash content of 3.37; 3.28; 2.62%, volatile matter content of 37.78; 36.67; 35.08%, bound carbon content of 53.85; 55.01; 57.12% and iodine absorption capacity of 998.51; 1011.21; 1023.9 mg/g. The results of functional group identification by FTIR showed that the functional groups detected in activated carbon were aliphatic CH, C=O, C-C, C=C, and C-O. BOD levels before and after contact with activated carbon were 2841.15 and 355.23 mg/L. The study concluded that 200-mesh coffee bean skin-activated carbon with a 20% H_3PO_4 activator can reduce BOD levels in liquid waste from the Rambak cracker industry by 87.5%.

Keywords: BOD, activated carbon, coffee bean skin, liquid waste

1. Introduction

In the modern era, there are many industries in both the home and factory sectors. It is not uncommon for industries to be located close to residential areas. The location of industries close to residential areas is inseparable from the negative impacts of the waste they produce. Liquid waste from industrial processes can introduce significant amounts of organic material into waterways, thereby affecting water quality around waste-disposal sites [1].

Tulungagung produces superior Rambak Crackers made from cow and buffalo skin. The Rambak Cracker household industry generates substantial organic liquid waste during processing. Liquid waste from the Rambak Cracker Industry primarily contains high levels of organic pollutants. Regarding waste from the rambak cracker industry, a study reported that liquid waste generated by a production facility in Bantul, Yogyakarta, exhibited a biochemical oxygen demand (BOD) value of 5,680 mg/L [2]. The amount of organic waste dumped into the aquatic environment will decrease the oxygen levels needed by marine organisms. This is due to the increased oxygen demand of microorganisms decomposing large amounts of organic waste; if oxygen is insufficient, they will die, leading to an unpleasant odor. Researchers have observed this situation during visits to and passage through the Ngrowo River in Tulungagung. Therefore, it is necessary to find a way to reduce or prevent excessive BOD levels in water.

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e-ISSN: 2776-4508; p-ISSN: 2776-1711
Publisher : Universitas Pakuan

One method to reduce the organic waste content in household industrial waste from rambak crackers before discharge into water is to adsorb these substances using adsorbents. Activated carbon is one of the adsorbents with high adsorption capacity. Activated carbon is widely used in water purification processes, both in drinking water production and in waste processing [3], as well as in the method for reducing BOD levels discussed previously. The particle size of activated carbon affects the level of adsorption capacity. The smaller the particle size of the activated carbon used, the greater the surface area available for adsorbing liquid waste, enabling optimal adsorption. Previous studies reported that activated carbon with a 60-mesh particle size reduced biochemical oxygen demand (BOD) by 51.83% [4]. In addition, activated carbon with a 120-mesh particle size achieved a higher BOD reduction of 64.705% [5]. Based on previous studies, research will be conducted on activated carbon of different sizes to determine the optimal size for reducing BOD levels. Activated carbon can be produced from carbon-containing materials from agricultural waste such as shells, fruit skins, roots, stems, bark, and leaves [6].

Coffee produced in 2022 in Tulungagung Regency was 239 tons. In the coffee processing process, coffee skin waste accounts for 40-45% of total production [7]. Coffee bean skin waste that is not used correctly and is discarded can potentially become organic waste that causes environmental pollution. In this research proposal, coffee bean skin will be used as activated carbon to increase its economic value, as it contains the main components of activated carbon, such as cellulose, hemicellulose, and lignin [8].

Table 1. Activated carbon standard (SNI) 06-3730-1995 [10]

Requirement Type	Parameter
Water content	Maximum 15%
Ash content	Maximum 10%
Bound carbon content	Minimum 65%
Absorption capacity for I_2	Minimum 750 mg/g

Researchers use coffee skin waste as the basic material for making activated carbon because coffee skin contains a total of 47.8-58.9% carbon, total nitrogen 1.9-2.3%, ash 0.43-1.6%, and cellulose 21%, so it has excellent potential to be used as a precursor for activated carbon, which can be used as an adsorbent [9]. One of the standards used to determine the quality of activated carbon is SNI 06-3730-1995 (Table 1).

The adsorption capacity of activated carbon not only depends on the surface area, pores, and surface characteristics but also on the presence of functional groups on the pore surface [11]. Activated carbon is produced in two stages: carbonization and activation [12]. In addition to the basic materials used, the synthesis of activated carbon is also influenced by the preparation process. Activated carbon activation usually uses several types of activators. Based on this, it is necessary to conduct research on the potential of activated carbon derived from coffee bean skins to reduce BOD levels, especially in liquid waste from the rambak cracker industry.

2. Methods

This type of research is truly experimental, aiming to synthesize activated carbon from coffee bean skin to reduce the BOD levels of liquid waste from the Rambak cracker industry. In this study, 200-mesh activated carbon was activated with H3PO4 solutions at 5, 10, and 20%.

2.1. Materials and Tools

The tools used in this study were a furnace flask, 200 mesh sieve, oven, desiccator, magnetic stirrer, porcelain cup, vacuum filter, 250 mL Erlenmeyer flask, 250 mL beaker glass, measuring flask, volume pipette, 50 mL burette, stand, clamp, analytical balance, pH meter, Winkler bottle, incubator cabinet, diluent container, measuring cup and FTIR Shimadzu 8201 PC. The materials used included coffee bean skin, distilled water, 5%, 10%, 20% H_3PO_4 activator solution, iodine solution, 0,025 N $Na_2S_2O_3$ solution, filter paper, starch indicator, diluent solution ($MgSO_4$, phosphate buffer, $CaCl_2$, $FeCl_3$), alkali iodide azide, $MnSO_4$, NaOH, microbial seeds, and the concentrated H_2SO_4 solution.

2.2. Making Activated Carbon

The manufacture of activated carbon consists of two stages, namely:

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e-ISSN: 2776-4508; p-ISSN: 2776-1711
Publisher : Universitas Pakuan

2.2.1. Dehydration and Carbonation

At this stage, the coffee bean skin is washed until clean and dried in the sun. The dried coffee bean skin is weighed. Then, carbonation is carried out in a furnace at 400 °C for 2 hours [13]. The resulting carbon powder is then cooled and weighed until it reaches a constant mass. After that, it is sieved through a 200-mesh sieve.

2.2.2. Activation

The sifted carbon powder was activated with 5, 10, and 20% H_3PO_4 solutions. The activation process was carried out by soaking the carbon powder in the activator solution for 24 hours at a 1:5 ratio. The powder was then filtered and washed with distilled water until the pH was neutral.

2.3. Activated Carbon Characterization

The standards for the quality of activated carbon are in accordance with SNI 06-3730-1995. Characterization of activated carbon includes testing of water content, ash content, volatile matter content, bound carbon content, iodine absorption capacity, and identification of functional groups.

2.3.1 Water content

At this testing stage, 1 g of activated carbon was weighed and placed into a previously weighed porcelain cup, and its mass was known. Then, the cup containing activated carbon was heated in an oven at 105 °C for ± 1 hour. After the heating step, a 15-minute cooling step in a desiccator was performed, and the sample was weighed until it reached a constant mass.

2.3.2. Ash content

At the ash content testing stage, approximately 1 g of activated carbon was accurately weighed and transferred into a previously cleaned, dried, and weighed porcelain crucible. The initial mass of the empty crucible was recorded before sample addition. Subsequently, the crucible containing the activated carbon sample was placed in a muffle furnace and heated to 650 °C for 4 hours to ensure complete combustion of the organic components. After ashing, the crucible was carefully removed from the furnace and allowed to cool in a desiccator for 15 minutes to prevent moisture absorption from the surrounding air. The crucible was then weighed, and the heating–cooling–weighing cycle was repeated until a constant mass was obtained, indicating that the ashing process was complete.

2.3.3. Volatile matter content

1 g of activated carbon was heated in a furnace at 950 °C for 15 minutes, then cooled in a desiccator and weighed.

2.3.4. Bound carbon content

The bound carbon content of activated carbon from coffee bean skin is the difference between the total percentage and the sum of the percentages of water, ash, and volatile matter.

2.3.5. Iodine absorption capacity

Activated carbon was weighed up to 0.5 g, added to 25 mL of 0.1 N iodine solution, and shaken for 15 minutes. Then let it stand for a while before filtering. Furthermore, 10 mL of the filtrate was taken and titrated with 0.1 N $Na_2S_2O_3$ solution until a straw-yellow solution was obtained, with 1% starch solution as an indicator. Then, it is titrated until the solution is colorless.

2.3.6. Identification of functional groups

At this testing stage, the carbon before and after activation was each subjected to functional group analysis using FTIR over the wave number range 500-4000 cm^{-1}.

2.4. Adsorption stage

At this stage, the industrial wastewater from rambak crackers was divided into three 250 mL beakers, each containing up to 100 mL. Then, 5 g of 200 mesh activated carbon was added to each sample and left for 1 hour. Furthermore, the mixture was filtered using filter paper. The resulting filtrate was analyzed using BOD parameters.

2.5. Analysis by Winkler Titration Method

A total of 1-2 mL of the filtered sample was placed in a 1000 mL Erlenmeyer flask, and a diluent solution was added to a final volume of 700 mL.

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e-ISSN: 2776-4508; p-ISSN: 2776-1711
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Then, it was homogenized by shaking. After being homogeneous, it was put into 2 Winkler bottles until it touched the mouth of the bottle. One of the bottles was closed and incubated for five days, then its dissolved oxygen was measured.

The remaining bottle was measured for dissolved oxygen on day zero by adding 1 mL of $MnSO_4$ and 1 mL of alkali iodide azide reagent. Then the bottle was closed and shaken until homogeneous. After being homogeneous, the mixture was left for 10 minutes. After that, 1 mL of concentrated H_2SO_4 solution was added. Then the bottle was closed and shaken until homogeneous. After being homogeneous, the mixture was left for 8 minutes. After that, 100 mL of the mixture was transferred to an Erlenmeyer flask and then titrated with $Na_2S_2O_3$ 0.025 N solution until straw yellow. After that, 2-3 drops of starch were added, and the solution was titrated again with a 0.025 N $Na_2S_2O_3$ solution until colorless. Furthermore, the BOD calculation and the decrease in BOD of the liquid waste before and after treatment were performed [14].

3. Results and Discussion

3.1. Characteristics of Coffee Bean Husk Activated Carbon

The quality of activated carbon is influenced by the raw materials used. Activated carbon derived from different raw materials will produce varying attributes.

3.1.1 Water content

The water content is determined by heating activated carbon in an oven at 105 °C for 3 hours to ensure maximum dehydration. The lower the water content, the more pores are available for adsorbate molecules [15].

In Table 2, the water content of 200-mesh coffee bean skin-activated carbon meets SNI 06-3730-1995, with a maximum value of 10%. The water content of CA-AP_5, CA-AP_{10}, and CA-AP_{20} 200 mesh is 5.00%, 5.04%, and 5.18%, respectively. The acidity level of the activator can affect the amount of water required in the washing process. The more acidic the activator is, the more water is needed for washing, which causes the activated carbon to absorb more water [9].

Table 2. Results of the water content of the activated carbon of coffee bean skin

Sample	Water content (%)
CA-AP_5	5,00
CA-AP_{10}	5,04
CA-AP_{20}	5,18

3.1.2. Ash content

Ash content is the amount of metal oxide residue left after activated carbon is burned at high temperatures (usually around 650–1000 °C) in the absence of oxygen. In this test, activated carbon is heated in a furnace at 650 °C for 4 hours.

Table 3. Results of the activated carbon ash content of coffee bean skin

Sample	Ash content (%)
CA-AP_5	3,37
CA-AP_{10}	3,28
CA-AP_{20}	2,62

In Table 3, the ash content of 200-mesh coffee bean skin-activated carbon meets the requirements of SNI 06-3730-1995, with a maximum value of 15%. The ash content of CA-AP_5, CA-AP_{10}, and CA-AP20 200 mesh samples is 3.37, 3.28, and 2.62%. The higher the concentration of H_3PO_4, the lower the ash content produced. Activators at high concentrations can release metal oxides from activated carbon, resulting in lower ash content than with low-concentration activators [16].

3.1.3. Volatile matter content

The volatile matter content measures the amount of compounds that have not evaporated during the carbonation and activation stages, but evaporate at 950 °C.

In Table 4, the volatile matter content of the 200-mesh CA-AP_5, CA-AP_{10}, and CA-AP_{20} samples is 37.78%, 36.67%, and 35.08%, respectively. The volatile matter content of activated carbon from coffee bean skin 200 mesh still does not meet SNI 06-3730-1995, which sets a maximum limit of 25%. This is due to the presence of hydrocarbon compounds that were

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not decomposed during activation. Activated carbon with high volatile matter content contains more organic compounds that can reduce adsorption capacity, because some of the pore space is filled with compounds that are not effective in adsorption.

Table 4. Volatile content of activated carbon in coffee bean skin

Sample	Volatile matter content (%)
CA-AP$_5$	37,78
CA-AP$_{10}$	36,67
CA-AP$_{20}$	35,08

3.1.4. Bound Carbon Content

The bound carbon content measures the fixed carbon content in activated carbon after a high-temperature heating process. During heating, volatile matter will evaporate, while ash and fixed carbon will remain as residue.

Table 5. Results of carbon levels bound to the activated carbon of coffee bean skin

Sample	Bound carbon content (%)
CA-AP$_5$	53,85
CA-AP$_{10}$	55,01
CA-AP$_{20}$	57,12

In Table 5, the bound carbon content of the 200-mesh CA-AP$_5$, CA-AP$_{10}$, and CA-AP$_{20}$ samples is 53.85%, 55.01%, and 57.12%, respectively. The bound carbon content in this study did not meet the requirements of SNI 06-3730-1995, with a minimum of 65%. Higher activator concentrations tend to produce activated carbon with higher bound carbon, because a more intensive activation process removes more volatile components, leaving more fixed carbon.

3.1.5. Iodine Absorption Capacity

Iodine adsorption is one indicator of activated carbon quality. On the surface of activated carbon, there are active sites that bind adsorbate; the adsorption capacity of these sites is tested using iodine solution. The greater the activated carbon's adsorption capacity for iodine, the better its adsorption ability.

Table 6. The results of the iodine absorption capacity of activated carbon from coffee bean skin

Sample	Iodine absorption (mg/g)
CA-AP$_5$	998,51
CA-AP$_{10}$	1011,21
CA-AP$_{20}$	1023,90

In Table 6, the iodine absorption capacities of the 200-mesh CA-AP$_5$, CA-AP$_{10}$, and CA-AP$_{20}$ samples are 998.51, 1011.21, and 1023.90 mg/g, respectively. In this study, the iodine adsorption capacity of coffee bean skin-activated carbon met the requirements of SNI 06-3730-1995, with a minimum value of 750 mg/g.

3.1.6. Identification of Functional Groups

Identification of functional groups of 200 mesh coffee bean skin activated carbon using FTIR (*Fourier Transform Infrared Spectroscopy*) in the wave number range of 4000-500 cm^{-1} is presented in Figure 1.

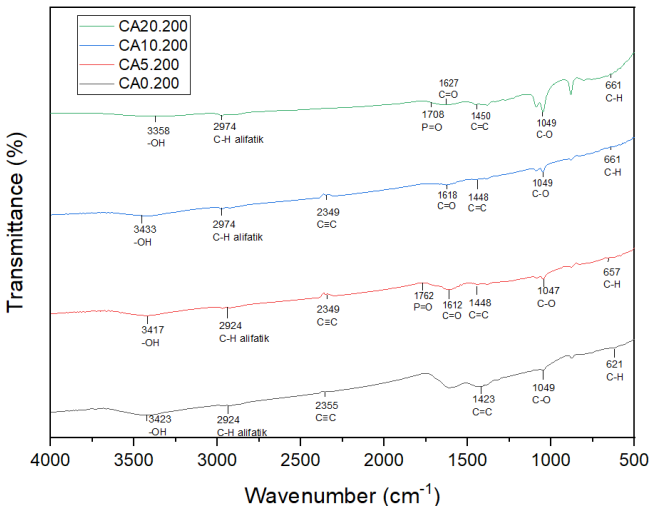

Figure 1. Spectra of activated carbon from coffee bean skin

From the spectral data, all activated carbons with various H_3PO_4 concentrations exhibit O-H (hydroxyl) functional groups with absorption bands at 3500-3200 cm^{-1}, as seen in Figure 1. There is a C=O group, which is a typical group in activated carbon, at the absorption peak of wave numbers 1820-1600 cm^{-1} [17]. The absorption at 1600-1400 cm^{-1} is attributed to

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an aromatic C=C group, indicating an increase in carbon content. The C≡C group (alkyne) at wave numbers 2309-2495 cm^{-1} is a group with high carbon purity, where the release of O and H elements that were previously bound to C occurs [18]. The absorption of wave numbers 1300-800 cm^{-1} identifies the presence of a C-O group, while the absorption at wave numbers 700-400 cm^{-1} indicates the presence of an aromatic C-H group from hydrocarbons. Figure 1 shows that new absorption bands appear at 1057 cm^{-1} and 1026 cm^{-1} when carbon is activated with H_3PO_4, corresponding to the P-O vibration and P-OH functional groups. There is also a functional group P=O at a wavelength of 1708-1762 cm^{-1}. The presence of functional groups P=O, P-O, and P-OH is thought to be due to activation using phosphoric acid (H_3PO_4), which has not been optimally washed.

3.2 Adsorption Results of Industrial Liquid Waste

The purpose of this study was to determine the reduction in BOD levels in liquid waste from the rambak cracker industry using 200-mesh activated carbon activated with H_3PO_4 at concentrations of 5, 10, and 20%. Biological Oxygen Demand (BOD) is the amount of oxygen needed to break down organic matter in liquid waste. BOD does not directly measure the amount of organic matter; instead, it measures the amount of oxygen required, making it an indicator of wastewater pollution.

Table 7. Results of reducing the BOD of liquid waste from the rambak cracker industry

Sample	BOD Before Treatment (mg/L)	BOD After Treatment (mg/L)	Decrease in BOD (%)
CA-AP_5	2841,15	426,250	85,00
CA-AP_{10}	2841,15	497,269	82,50
CA-AP_{20}	2841,15	355,232	87,50

In Table 7, the BOD of the industrial liquid waste from the rambak cracker industry is 2841.15 mg/L. After contacting activated carbon from coffee bean skin, BOD levels decreased. The BOD levels of each industrial liquid waste of the rambak after being approached with CA-AP_5, CA-AP_{10}, and CA-AP_{20} 200 mesh samples were 426.250, 497.269, and 355.232 mg/L. The lowest BOD level was observed in the liquid waste contacted with CA-AP_{20} (200 mesh), at 355.232 mg/L, representing a 87.50% decrease. In this study, the BOD reduction was higher than that reported in previous research on activated carbon derived from corn cobs, which achieved a BOD reduction of 51.83% [4]. This decrease in BOD occurs because organic materials in the liquid waste from the rambak cracker industry are adsorbed onto activated carbon, thereby reducing the oxygen required for microbial degradation. The more organic material activated carbon absorbs, the lower the BOD value in the liquid waste [4]. The smaller the activated carbon particle size, the greater the decrease in BOD in the liquid waste from rambak crackers [19]. The smaller size of activated carbon provides a greater surface area for adsorbing materials that contact the carbon, so the amount of material that can be absorbed is greater [20].

4. Conclusion

Based on the results of this study, activated carbon derived from coffee bean skin was successfully synthesized and chemically activated with H_3PO_4 solutions at 5%, 10%, and 20% concentrations, with a particle size of 200 mesh. The characterization results showed that the activated carbon met the requirements of SNI 06-3730-1995 for moisture content, ash content, and iodine adsorption capacity. However, the volatile matter and fixed carbon contents did not meet the standard. FTIR analysis indicated that the activation process did not significantly alter the functional groups present, which were dominated by aliphatic C–H, C=O, C=C, and C–O groups. The activated carbon prepared with 20% H_3PO_4 exhibited the best overall characteristics. Furthermore, the liquid waste from the rambak cracker industry before treatment had a BOD value of 2841.15 mg/L, and the highest BOD reduction (87.50%) was achieved using activated carbon activated with 20% H_3PO_4 (CA-AP20) at a particle size of 200 mesh, indicating that smaller particle sizes provide a larger surface area and enhance adsorption efficiency for reducing BOD levels in industrial liquid waste.

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Publisher : Universitas Pakuan

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