Synthesis and Characterization of used Oil Bio-Adsorbent Material based on Corn Husk Activated Carbon

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Abstract *– Used oil waste is classified as hazardous B3 waste that poses a significant environmental threat. To address the hazards posed by used oil waste, synthesizing activated carbon as an adsorbent for used oil is necessary. This research aims to synthesize and characterize activated carbon from corn husk waste as an adsorbent material for used oil. The methods employed in this research include dehydration, carbonization, and activation using HCl and NH4OH. The structure was analyzed using XRD, UV-visible spectrophotometer, and Oswald viscometer, followed by variations in mesh sizes of 60 and 100 and the addition of PEG. Based on the research results, variations in 60 and 100 mesh have nearly identical X-ray diffraction patterns, with values of 2θ at 19.2074° and 23.0729° in the 60mesh variation, and 2θ at 19.1333° and 23.2161°, which are indicative of graphite diffraction patterns as they fall within the ~25° range and match the CIF 9014004 data for phase C Graphite with space group p6/mmm. In UV-Visible spectrophotometer testing, variations of 60 mesh without PEG, 100 mesh without PEG, 60 mesh with PEG, and 100 mesh with PEG showed absorbance values of 2.3, 1.58, 1.394, and 0.966, respectively, and viscosity values of 22.089, 20.089, 21.09, and 19.21 cP. The 100mesh sample with the addition of PEG is the variation that can effectively adsorb used oil.*

Keywords*: Absorbance; Activated Carbon; Corn Husk; PEG; Viscosity.*

INTRODUCTION

The rapid development in the industrial and transportation sectors has led to a significant increase in waste production. This is particularly evident in industry and transportation, where machines and components require oil as a lubricant. Oils have a finite lifespan and need to be replaced after a certain period of use, resulting in them becoming waste products (Aisyah *et al.*, 2021). Used oil waste is highly hazardous to living organisms and is classified as B3 waste. According to Government Regulation No. 16 of 1999 concerning the Management of Hazardous and Toxic Materials (B3 Waste), oil is a chemical substance used in motor vehicles to minimize wear on engine components. The majority of oil usage is for engine oil. Typically, oil consists of 90% base oil and 10% additives (Wu *et al.*, 2021). To address the issue of industrial waste, researchers have explored various purification methods, including adsorption. In general, adsorption is the attraction of atoms on the surface of a solid substance that can absorb (adsorb) specific substances in a fluid phase during the separation process (Hafidoh, 2021). Research on adsorbents in the form of agricultural biomaterial waste has been extensively studied. Waste as a bio adsorbent is exciting because it is readily available, renewable, and abundant (Adewuyi, 2020). Bio-adsorbents containing components such as cellulose have the potential for adsorption due to their active sites, such as hydroxyl groups (OH-) (Xiang *et al.*, 2022). Research on cellulose, which is contained in fibers and can reduce the concentration of methylene blue in batik wastewater, has been conducted (Zakaria *et al.*, 2023). Additionally, cellulose in peanut husks containing 59.58% cellulose can be an adsorbent for iron (Fe) in mineral water and adsorb up to 0.7467 mg/L (Ischak, Fazriani

and Botutihe, 2021). One of the materials used for adsorption is activated carbon.

Activated carbon has a porous solid structure containing 85-95% carbon content. It is produced from materials with carbon content through high-temperature treatment using gases, steam, and chemicals to open up pores in the activated carbon. Activated carbon is an excellent adsorbent material widely used due to its large surface area and micro-pore volume, and it is relatively easy to regenerate (Zhang *et al.*, 2021). The production of activated carbon involves stages of dehydration, carbonization, and subsequent activation (Ge *et al.*, 2023). Activated carbon can adsorb gases and certain chemicals, and its adsorption properties can be selective depending on the size or volume of its pores and surface area. The pore structure of activated carbon is related to its adsorption capacity; the more pores on the surface, the higher its adsorption capacity, resulting in faster adsorption rates (Ariyanto, Lestari and Kharismadewi, 2022). Some raw materials used for activated carbon production include wood, coconut shells, coal waste, wood processing waste, and agricultural waste such as coffee bean husks, cocoa bean husks, rice husks, straws, corn cobs, and corn husks (Hidayat, 2021).

Corn is one of the staple foods consumed by the Indonesian population. Each year, corn production in Indonesia experiences growth. According to data from the Central Statistics Agency, in 2022, corn production in Indonesia reached 23 million tons, leading to a significant amount of agricultural waste generated from crop harvesting. However, the potential of corn husks has yet to be fully utilized; they are mainly used as animal feed, food packaging, and traditional crafts. Therefore, further research on corn husk waste is needed (BD *et al.*, 2023). Corn husks are known to have a relatively high cellulose content. The composition of corn husks consists of cellulose (36.81%), ash (6.04%), lignin (15.7%), and hemicellulose (27.01%) (Maghfirah *et al.*, 2023). Previous research has explored the use of corn husks as adsorbents for filtering chemical and inorganic parameters. For example, studies have examined the filtration of oil and fat from hotel waste, achieving filtration efficiencies and capacities of 63.74% and 19.95 mg COD/g of artificial solutions, respectively (Abuzar and Dewilda, 2014).

Additionally, research by Indah et al. (2016) investigated corn husks as adsorbents for iron removal, resulting in an adsorption efficiency of 0.499 mg Fe/g (Sholahuddin, Yuwita and Faila, 2023). Corn husk waste has also been used as an adsorbent to reduce COD and BOD levels in healthy water (Walanda *et al.*, 2022). Based on previous research, it is evident that corn husks have the potential to serve as a bioadsorbent for used oil waste.

To utilize corn husk waste as an adsorbent for used oil, further research is needed to characterize the crystal structure of corn husks using XRD (X-ray diffraction), assess adsorption quality using UV-visible spectrophotometer methods to obtain activated carbon absorbance values within the 400-800 nm wavelength range and perform adsorption testing on used oil using an Oswald viscometer. This research can contribute to developing a bio-adsorbent utilizing corn husk agricultural waste to produce activated carbon nano-membranes for oil filtration.

RESEARCH METHODS

Sample Preparation

Sample Preparation was done by washing the dried corn husks to remove any dirt adhering to them. The cleaned corn husks were then dried under direct sunlight

to reduce the moisture content. The corn husks were burned in a furnace at 100°C for 2 hours. The resulting corn husk charcoal was ground into powder using a blender and mortar. After grinding, the charcoal passed through a sieve with mesh sizes of 60 and 100. Each sample of sieved and carbonized corn husk charcoal powder was weighed at 10 grams, mixed with 80 ml of HCl, and stirred for 20 minutes. After 20 minutes, titration with NH_4OH was performed using 40 ml, and stirring continued for another 30 minutes. The precipitate was washed with distilled water until it reached a pH of 7. The charcoal powder samples from corn husks were then dried at 110°C in an oven. The process of sample preparation showed in figure 1.

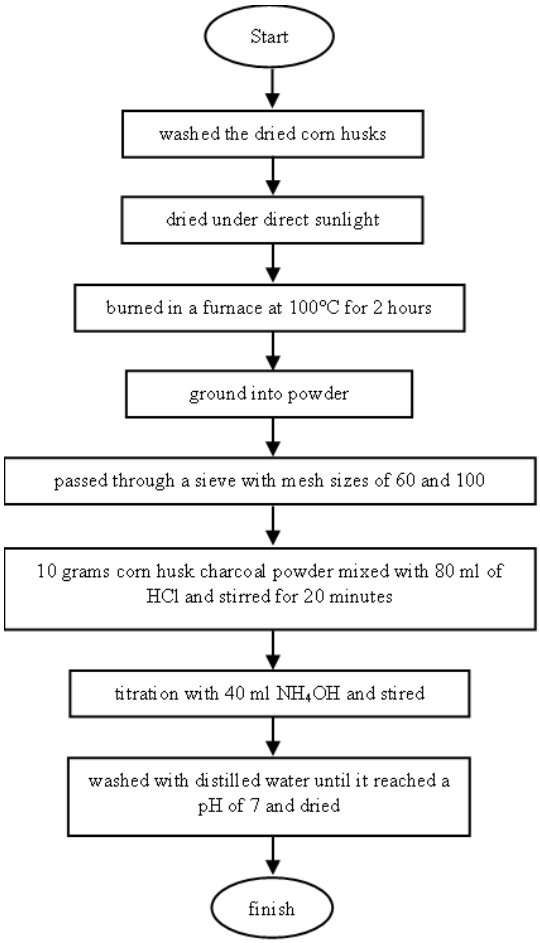

Figure 1. flowchart of sample preparation

XRD Sample

The finely ground activated carbon from corn husks was then used as a filler to produce activated carbon solid membranes. The matrix consisted of a mixture of polyethylene glycol (PEG), polyvinyl alcohol (PVA), and distilled water in respective compositions of 0.3 gr, 5 ml, and 5 ml. The filler, mixed with the matrix, was prepared in variations of 1 gram with mesh sizes of 60 and 100 for the activated carbon. The matrix and filler were mixed using a magnetic stirrer at 500 rpm and a temperature of 60°C for 20 minutes. The mixture was then cast in 10 mm diameter paralons and air-dried for 48 hours, followed by an additional 2-hour oven-drying at 105°C to remove any trapped moisture within the membrane.

Absorbance Sample

A total of 0.5 grams of 60 and 100 mesh activated carbon and 40 ml of distilled water were placed in a beaker and stirred for 1 hour. After stirring, the activated carbon was allowed to settle, and the absorbance value of the resulting solution was measured using a UV-visible spectrophotometer. In this test variation, 0.3 PEG was added and stirred at 60°C with a speed of 500 rpm.

Viscosity Sample

Chemically activated corn husk waste charcoal, 1.5 grams, was placed in an Erlenmeyer flask with variations in mesh sizes of 60 and 100, with and without adding PEG. Subsequently, 50 mL of used oil was added to each Erlenmeyer flask. The mixture was then shaken for 15 minutes, and the used oil, which had been adsorbed, was transferred to an Ostwald viscometer. The oil used in the Ostwald viscometer was drawn up using a ball pipette until it reached or passed the marked limit. The time taken for the used oil to flow from one marked limit to the next was recorded. The following equation was used to determine the viscosity value:

$$\eta_2 = \frac{\rho_2 t_2 \eta_1}{\rho_1 t_1} \quad (1)$$

Note:
η1 = Viscosity of water (cP)
η2 = Viscosity of the oil sample (cP)
ρ1 = Density of water (g/cm3)
ρ2 = Density of oil (g/cm3)
t1 = Flow time of water (seconds)
t2 = Flow time of oil (seconds)

Activated Carbon Analysis

The phase analysis of the activated carbon sample was conducted using X-ray diffraction at angles ranging from 2θ 10°-90° with a step size of 0.02°. Phase identification was based on matching the diffraction pattern data with references and determining lattice parameters using match software. Absorbance testing was performed using a UV-Vis spectrophotometer, and the effect of activated carbon on the viscosity of used oil was assessed using an Oswald viscometer.

RESULTS AND DISCUSSION

The research results are described first, followed by the discussion section to facilitate understanding and reading. The results and discussion subtitles are presented separately. This section must be at most part, at least 60% of the entire body of the article.

XRD Analysis

The X-ray diffraction (XRD) patterns obtained from the activated carbon powder samples of corn husks with mesh sizes of 60 and 100 are shown in Figure 2. The X-ray diffraction (XRD) patterns obtained from corn husk activated carbon powder samples with sizes of 60 and 100 mesh exhibit similar patterns with signs of crystallinity forming at 2θ = 10-30 degrees, which are not sharp and narrow. However, the crystallinity formed is still low. The peaks shown in the XRD results can be observed in Figure 1. In the 60mesh variation, the diffraction peaks are located at 2θ = 19.2074º and 23.0759º, while in the 100mesh variation, they are located at 2θ = 19.1333º and 23.2161º, indicating the presence of graphite carbon.

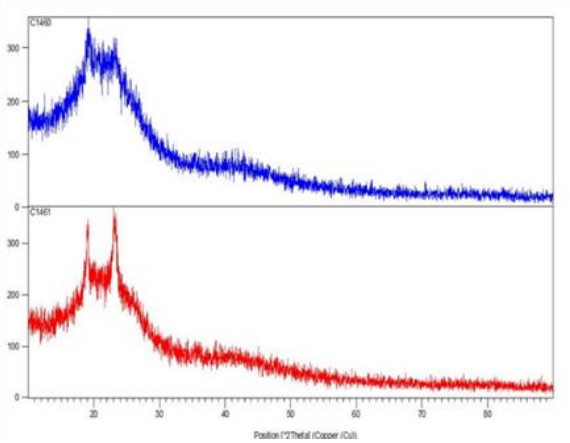

Figure 2. X-ray Diffraction Patterns of Corn Husk Activated Carbon Powder 60 (A) and 100 (B) Mesh.

When there are only two peaks in the X-ray diffraction results, determining the exact percentage of the crystalline structure formed is impossible. However, the presence of these two peaks in the X-ray diffraction results of corn husk-activated carbon powder samples can already indicate the formation of activated carbon in the corn husk sample. The indication of the formation of standard activated carbon is observed at 2θ = 23.0759º in the 60 mesh variation and 2θ = 23.2161º in the 100 mesh variation because these values fall within the 2θ range of approximately 25º. There is a match between the diffraction peak results and the CIF 9014004 data for the C Graphite phase with space group p 6/m m m.

Based on the analysis of the X-ray diffraction patterns, it is evident that the mesh sizes of 60 and 100 do not significantly affect the crystal structure of activated carbon. In the XRD pattern analysis, the patterns of the 60 mesh and 100 mesh samples are relatively similar. However, in the 100 mesh sample, there is an increase in diffraction peak height compared to the 60 mesh sample. Additionally, a shift in the 2θ

peak occurs between 60 mesh and 100 mesh. This is indicated by the diffraction peak at 2θ for 100 mesh activated carbon, which is located at 2θ = 19.2074º and 23.0759º, compared to the 60 mesh activated carbon, which is located at 2θ = 19.1333º and 23.2161º. This difference is due to the varying particle sizes of activated carbon, which affects the uniformity of the activation process in smaller-sized activated carbon (Chen *et al.*, 2023).

A comparison of the X-ray diffraction patterns produced from the corn husk sample activated carbon reveals the presence of the same phase, which is the amorphous phase. The amorphous phase is a state where the arrangement of atoms in the material has yet to be structured regularly and periodically (Lan *et al.*, 2021). The amorphous phase is usually found in materials without heat or chemical treatment that can alter their phase. In the corn husk samples, heat treatment at 110°C was applied. This temperature was not sufficient to change the atomic arrangement in the sample. The activated carbon sample from corn husks is considered semi-crystalline because there are already visible peaks in the X-ray diffraction pattern. The presence of two peaks indicates that atoms are beginning to arrange more regularly but at a low percentage of repetition. Therefore, the X-rays emitted onto the sample only affect the atoms that have started to arrange regularly. Another study result shows that at the diffraction peak of 2θ = 23º, there is still a presence of silica in the sample. The 2θ = 23º angle range shows the diffraction pattern of amorphous hydrated silica. In amorphous silica, constituent atoms are randomly arranged with low regularity (Wang *et al.*, 2023).

Activated Carbon Absorbance Results

This study initially involved determining the maximum wavelength. The maximum wavelength is when a substance exhibits the highest absorption. The maximum wavelength is used because the absorbance change per unit concentration is the greatest at this wavelength, resulting in maximum intensity (figure 3).

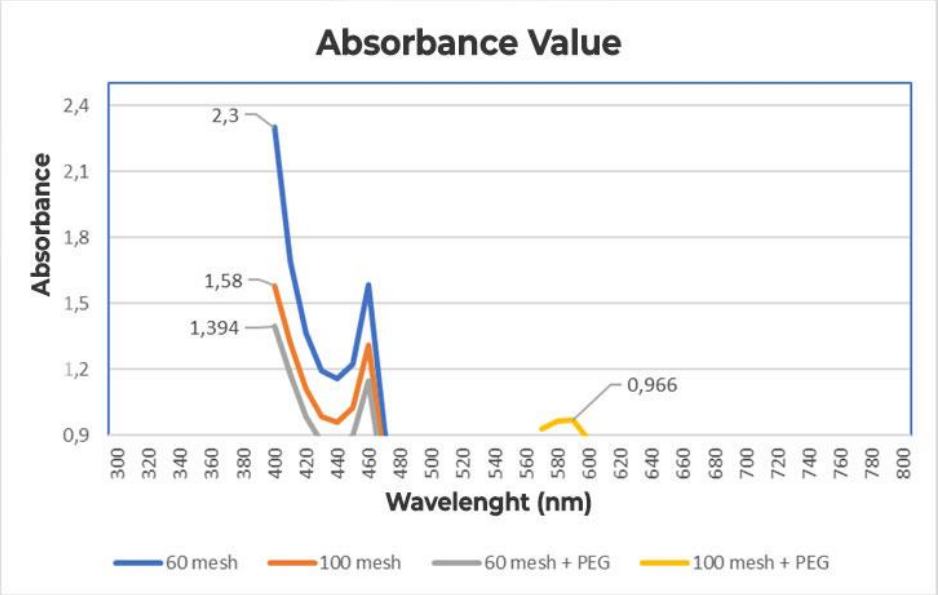

Figure 3. Graph of Activated Carbon Absorbance Values from Corn Husk Waste

The maximum wavelength of activated carbon from corn husk waste varies because the aqueous solution mixed with activated carbon has different concentrations. The activated carbon solution with 100 mesh variation and the

addition of PEG has a different concentration because the particles in the activated carbon have become nano-sized and cannot be filtered by the Whatman filter. From the measurements by UV-visible spectrophotometer, the 100 mesh sample with PEG has a maximum wavelength of 600 nm and an absorbance value of 0.966. In activated carbon variations of 60 mesh without PEG, 100 mesh without PEG, and 60 with the addition of PEG, they all have the same maximum wavelength because they have the same concentration and can be filtered by the Whatman filter. In these variations, the maximum wavelength is 400 nm, with absorbance values of 2.3, 1.58, and 1.394, respectively.

The results of the UV-visible spectrophotometer analysis show that the addition of PEG and the mesh size affect the absorbance value. UV-Vis (Ultra Violet-Visible) spectrophotometer is a commonly used instrument for chemical compound analysis. The determination using a UV/Vis spectrophotometer aims to determine the absorptivity value that has been adsorbed. Absorptivity is inversely proportional to adsorption; the more significant the absorptivity value, the smaller the adsorption, and vice versa. In the variations with the addition of PEG, there is a decrease in the absorbance value because adding PEG can reduce the particle size of activated carbon compared to the synthesis of activated carbon nanoparticles without the addition of PEG. This is because PEG is an oligomer with short, uniform chains that can be easily absorbed.

The addition of PEG affects the size of the activated carbon nanoparticle formed and its properties as an adsorbent. With the addition of PEG, the analyte concentration decreases. The decrease in analyte concentration indicates that more analytes are adsorbed by the adsorbent, in this case, activated carbon nanoparticles with or without PEG. This is due to the influence of the size of the activated carbon nanoparticle, where the relatively more significant size results in a smaller contact surface area and relatively fewer metals adsorbed on the adsorbent surface (Elvida, 2021). In variations in mesh size, there is also a decrease in the absorbance value because the particle size of activated carbon affects its adsorption capacity. Smaller particle sizes increase the surface area, enhancing adsorption capabilities (Mariana *et al.*, 2021).

Results of Activated Carbon Viscosity on Used Oil

In examining the influence of PEG on corn husk waste activated carbon as a bioadsorbent material on used oil with variations of 60 and 100 mesh, viscosity testing of used oil mixed with activated carbon was conducted to determine the effect of PEG and sieve size on adsorbing used oil waste. The oil obtained was directly adsorbed using activated carbon. The adsorption process with activated carbon aims to absorb impurities and residual metals from engine combustion that cause changes in oil viscosity. The used oil that has been adsorbed is filtered to separate the used oil itself from the activated carbon. Then, viscosity measurements were taken using an Oswald viscometer by recording the time it took for the used oil to flow through the tube in the Oswald viscometer. Before determining the viscosity of used oil, the density of used oil, the density of aquades, and the aquades flow time in the Oswald viscometer were determined as blanks (comparisons to determine the viscosity of used oil).

Table 1. Results of Viscosity Testing of Activated Carbon on Used Oil

Sample	Flow Time (s)	Viscosity Value (cP)
Used oil without activated carbon	4,07	22,42
60 mesh without PEG	4	22,089
100 mesh without PEG	3,82	21,09
60 mesh with PEG	3,67	20,26
100 mesh with PEG	3,48	19,21

Table 1 shows that activated carbon with a mesh size of 60 has a higher viscosity value than 100 mesh. The 60 mesh size cannot effectively absorb impurities and metals in used oil. In comparison, the 100 mesh size reduces the viscosity of used oil because it can absorb impurities and metals more effectively. This demonstrates that particle size affects the surface area of activated carbon, with smaller particles having a larger surface area. Surface area is one of the factors affecting activated carbon adsorption capacity. Activated carbon typically has large pores, resulting in a large surface area. Activated carbon can be used as an adsorbent for liquids or gases (Reza *et al.*, 2020).

Samples with the addition of PEG show a decrease in viscosity compared to samples without PEG. The 100 mesh sample with PEG has a lower viscosity value than the 100 mesh sample without PEG. Although the increase is not significant, it can be observed that PEG also affects the characteristics of corn husk waste activated carbon, resulting in better absorption of impurities and metals when PEG is added. Adding PEG can reduce the particle size of activated carbon, and size also affects the adsorption capacity of corn husk waste activated carbon (Yuwita and Rohmah, 2022).

CONCLUSION

The X-ray diffraction patterns indicate that all samples exhibiting broad and weak diffraction patterns are characteristics of amorphous carbon. The indication of the formation of standard activated carbon is observed at $2\theta = 23.0729^{o}$ and $2\theta = 23.2161^{o}$ because they fall within the range of $2\theta \sim 25^{o}$, and it aligns with the CIF 9014004 data in space group p 6/m m m. The absorbance values of carbon after PEG activation and different activated carbon particle sizes resulted in decreased absorbance values, with the lowest value of 0.966 observed in the 100 mesh variation with PEG. The absorbance value affects the adsorption value; lower absorbance values correspond to higher adsorption values. The viscosity of used oil decreases significantly after the addition of activated carbon, particularly in samples with smaller activated carbon particle sizes and the addition of PEG. This is because particle size and PEG affect the surface area of activated carbon, with a viscosity value of 19.21 cP observed in the 100 mesh variation with PEG.

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