

# THE ADSORPTION PERFORMANCE AND CHARACTERIZATION OF ACTIVATED CHARCOAL OF BONE CHAR AGAINST ACID ORANGE 7

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# ABSTRACT

The use of Acid Orange 7, a synthetic dye, in the textile industry harms the environment because it is carcinogenic. This research aims to remove the Acid Orange 7 dye dissolved in the water. This study used cow bone charcoal as an alternative adsorbent made by the carbonization method. In addition, the batch adsorption method was applied in the bleaching process of the synthetic dye waste, Acid Orange 7. Several tests, such as SEM, EDX, BET, XRD, and FTIR, were carried out to determine the characteristics and ability of activated charcoal from cow bones as an adsorbent for acid Orange 7 dye waste. Other organic adsorbents, such as graphene oxide and activated carbon, were used to compare the results. Bone char adsorption Acid Orange 7 62.2% efficiently. The adsorption efficiency of activated carbon is 79.8%, while graphene oxide has an adsorption rate of 89.4%. The findings revealed that bone char could be used to cure synthetic dye waste, Acid Orange 7, as an alternative. Additional treatment and purification operations are required to improve the adsorption efficiency of bone char.

Keywords: Bone char; Azo Dyes; Acid Orange

## INTRODUCTION

The textile industry is developed. However, the liquid waste produced by the industry contains several types of pollutants, such as dispersants, levelling agents, salts, acids, alkalis, and various dyes [1]. The hazardous liquid waste is generated using synthetic dyes in the colouring process. Synthetic dyes are often used in industry because they are more stable, easy to obtain, and inexpensive. However, using synthetic dyes in colouring has drawbacks because the waste produced is difficult to degrade, causing contamination [2]. Acid Orange 7 is a synthetic dye used in the dyeing process in the textile industry. Acid Orange 7 has water-soluble properties, so it can cause environmental problems in the form of a decrease in water quality and have an unfavourable impact on living things because this dye is carcinogenic [3]. This dye has a tautomer one structure, a mono-azo acid dye with the molecular formula C<sub>16</sub>H<sub>11</sub>N<sub>2</sub>NaO<sub>4</sub>S, which contains an azo chromophore group. Therefore, acid orange 7 can cause cancer if disposed of as environmental waste [1].

Chemical and physical waste treatment can be carried out to handle the waste generated from acid orange 7. However, the high costs and the sludge that can be generated have become material for reconsideration because they are considered less efficient. Therefore, many types of alternative methods are used to treat waste. Among various methods, adsorption is one of the main methods in the chemical industry. Adsorption is the adhesion of atoms, ions or molecules from a gas, liquid or dissolved solid to a surface. This process creates a film of the adsorbate on the surface of the adsorbent.

Activated charcoal is one type of adsorbent that can be used as an alternative to textile industry waste treatment using the adsorption method. Activated charcoal is considered potent enough to remove contaminants contained in water. In addition, activated charcoal can remove metal content, such as lead, cadmium, nitrate, and chloride ions, in the solution [4]. Activated charcoal is a porous solid containing 85-90% carbon produced from carbon-containing materials through a high-temperature heating process [5]. This non-graphite and non-graphitizable carbon have a very irregular microstructure. The porosity and large surface area make activated charcoal often applied in water treatment, wastewater, odour removal, and some heavy metals. The surface area of activated charcoal particles is up to >1000 m<sup>2</sup>/g. The surface area is one of the properties that can indicate good potential for removing pollutants from aqueous solutions. The large surface area makes the adsorption efficiency better. It is because there are more adsorbing sites when we increase the surface area. The adsorbing site can also increase van der Waals forces. [6,7]. Activated charcoal contained in bone charcoal could adsorb Pb ions with excellent absorption efficiency. The use of activated carbon and graphene oxide as a comparison in handling synthetic dye waste acid orange 7 has been carried out by Sumarghandi (2015) [8]. Activated charcoal is very abundant because it can be produced from agricultural and livestock wastes such as cow bones [9], chicken bones [10], corn cobs [11], coconut shells [12], and rice husks [13].

Activated charcoal derived from cow bones was used in this study due to the high consumption of cows in Indonesia and the large amount of cow bone waste produced by the butchery industry. Cow bones can be assumed as waste or food scraps, which until now are still minimally utilized [14]. One of the uses of waste in the form of cow bones is to make it an adsorbent. Cow bones are first converted into activated charcoal through a carbonization and activation process, which is then used as an adsorbent in liquid waste. The chemical composition of bone is hydroxyapatite, collagen, glycosaminoglycans, proteoglycans, and glycoproteins [15]. Activated charcoal from cow bones is an alternative that can be used as an adsorbent because activated charcoal from cow bones has many oxide compounds, such as potassium oxide, which has a relatively open skeletal structure. Activated cow bone charcoal is very effective in the adsorption process of metals, including cadmium, chromium, copper, lead, and others [4]. Thus, using activated charcoal from cow bones as an adsorbent can be a bleaching agent in liquid waste and remove toxic metals and carcinogenic substances in water [16]. The production of activated charcoal from cow bones (bone char) used as an adsorbent for synthetic dyes acid orange 7 is still rarely done. This research was conducted to utilize cow bone and convert it into activated charcoal as an adsorbent in acid orange 7 waste and to compare the decrease in acid orange 7 dye levels with other adsorbents such as activated carbon and graphene oxide.

### METHODS

#### Material and Tools

The materials used in this study were cow bones obtained from traditional markets, NaOH, HCI 0.1M, Acid Orange 7, and distilled water. The carbonization synthesizes activated charcoal from cow bones, which consists of several stages, including dehydration, carbonization, and activation.

#### Preparation of raw materials

Cow bone as raw material is washed first to remove various impurities. After that, the cow bones are cut into cubes with a size of  $2 \times 2$  cm to make it easier for the material to enter the tube furnace. The material is then dried in the sun for two days [17].

#### Synthesis

Carbonization is carried out through a pyrolysis process of cow bones using a tube furnace. The pyrolysis process is carried out by flowing N<sub>2</sub> gas, so the operating conditions are inert at a temperature of 900 °C. The temperature was maintained for 1 hour after the process temperature was obtained. The bone charcoal received was activated using NaOH with a mass ratio between NaOH and bone charcoal of 3:1 (NaOH: Char). Mixing was carried out using a magnetic stirrer for 2 hours. Bone char is mixed with 2 litres of distilled

water, and NaOH is slowly added to the mixture. The resulting material was dried in the oven for 4 hours at a temperature of 130 °C. The further decomposition process was carried out using a tube furnace at an inert condition (flowing gas N<sub>2</sub>) and a temperature of 900 °C for 1.5 hours. The material that has been further decomposed using a tube furnace is then treated using 0.1 M HCl and washed using a hot aqua dest until a pH of 6.5 is obtained [18]. The material is then dried in an oven at 110 °C for 24 hours.

#### Characterization

Several tests were carried out to determine the characteristic of the activated charcoal material from cow bones. In addition, the research test determines the ability of activated charcoal from cow bones as an adsorbent in synthetic waste Acid Orange 7. The test is in the form of Scanning Electron Microscopy (SEM), which is used for the morphological structure of activated charcoal cow bones. Energy dispersive X-ray spectroscopy (EDX) is used to determine what elements are contained in the material. The surface area of the adsorbent was measured using the Brunauer-Emmett-Teller (BET) test. X-ray Diffraction (XRD) and Fourier-transform infrared spectroscopy (FTIR) are carried out for the initial phase and functional groups of an activated bone charcoal material cow.

#### **Data Analysis**

Various concentrations of acid orange 7 were dissolved in water. The comparison using several adsorbents, such as bone charcoal, activated carbon, and graphene oxide analyzed using Visible-spectrophotometry (VIS) to determine the decrease in the level of orange 7 dye in the solution of each type of adsorbent.

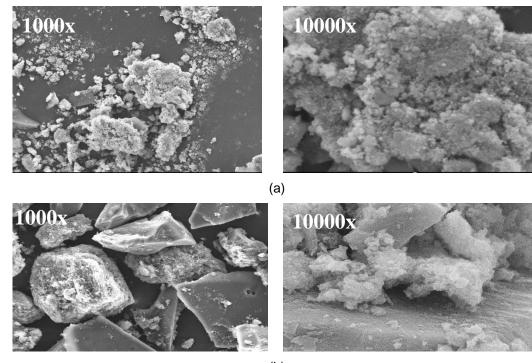


Figure 1. Activated charcoal from cow bone

Manufacturing cow bone-activated charcoal is carried out through dehydration, carbonization, and activation stages. Activated cow bone charcoal used as an adsorbent for acid orange 7 waste has been made in this study and is shown in Figure 1. Visually, activated cow bone charcoal is black and has a rough texture. Characterization and determination of absorption were carried out using tests such as XRD, SEM, FTIR, and Vis Spectrophotometry which aimed to determine the ability and absorption of active machete of cow bone as an adsorbent.

#### **Charcoal Characterization**

Activated cow bone charcoal was characterized Scanning using Electron Microscopy (SEM) to determine its morphological and typographical structure. Some of the enlargements carried out in SEM testing of activated cow bone charcoal were compared between activated cow bone charcoal before and after the adsorption process. A comparison of the results of the SEM test is shown in Figure 2.



(b)

Figure 2. Analysis Result using Scaning Electron Microscopy (SEM ) (a) Before the adsorption (b) After the Adsorption Process

# **RESULTS AND DISCUSSION**

Morphologically, there is no significant difference between the activated charcoal of cow bone before and after the adsorption process. It indicates that the adsorption process does not change the chemical properties of the activated carbon. The adsorbate does not erode or cause changes to the surface or morphology of the activated carbon. The cow bone-activated charcoal material is shown in Figure 2. Both have irregular shapes and comprise two particles, secondary and primary particles [19]. Secondary particles are particles with a large size, while primary particles are particle flakes that agglomerate into a single unit. Thedistribution of particles in the SEM results of activated charcoal cow bone also looks heterogeneous [20-22].

Table 1. Activated Charcoal Content of Beef Bones using SEM-EDX

Element	Mass(%)	Atom (%)
Na	7.5	7.9
Mg	3.4	3.4
Si	5.3	4.5
K	3.6	2.2
Ca	56.5	34

Morphological analysis of Scanning Electron Microscopy (SEM) showed that the material is porous. Seen on the SEM image with a magnification of 10000 x. The activation process using NaOH on the material oxidizes surface molecules so that the surface area of material increases and affects the the adsorption efficiency [23]. This material also has a carbon content of 23.7%, which can be seen from the results of the SEM-EDX test, as shown in Table 1. Apart from carbon materials, other materials such as sodium, magnesium, silica, potassium, and calcium were also identified in the SEM-EDX test.

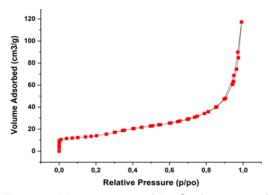


Figure 3. Measurement of the Surface area of Bone Char as an Adsorbent (N2 adsorption/desorption Isoterm)

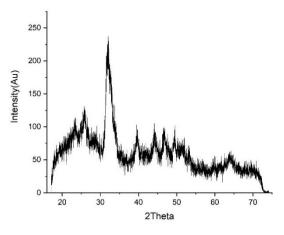


Figure 4. The results of the XRD analysis of bone char

Meanwhile, the analysis of pore measurements and surface area using the Brunauer-Emmett-Teller (BET) test produced a surface area (SBET) of 5.0876E+01 m<sup>2</sup>/g, an average pore diameter of 13.749 nm, and a total pore volume of o .1749 cm3/g. The capacitive performance of carbon materials is directly reflected in the active surface area, which determines the specific capacitance and rate capability. Therefore, nitrogen adsorption/desorption isotherms and pore size distributions were studied further to examine the porous structure of bone char [24]. Figure 3 shows the nitrogen adsorption/desorption isotherms and the pore size distribution of bone char samples. Based on the IUPAC classification, the synthesized bone char displays a type II isotherm with a hysteresis cycle, a Type H1 loop, indicating that the pores formed in the bone char sample are mesoporous in size. Mesopores are 2-50 nm, meaning that this cow bone-activated charcoal has tiny pores. Small pores lead to a large mass transfer area. The high surface area and mesoporous structure of bone char show great potential for use as an adsorption material for acid orange 7 dye waste [25].

Analysis of the crystal structure of activated charcoal was also carried out using X-Ray Diffraction (XRD). The results of the test analysis using XRD are shown in Figure 4 (a). In this analysis, the hydroxyapatite content was confirmed by an X-ray diffractogram showing a strong peak at  $33^{\circ} 2$   $\theta$ . In addition, the  $2\theta$  -axis peak between  $25^{\circ}$  and  $60^{\circ}$  also determined the presence of an apatite phase in the cow bone-activated charcoal material analyzed [26]. In general, the bone-activated charcoal diffraction pattern was close to the other bone-activated charcoal diffraction patterns obtained from pork and chicken bones [10,27].

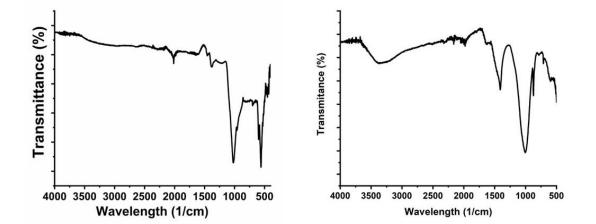


Figure 5. The results of the analysis of bone char (a) FTIR before adsorption (b) FTIR after adsorption

This cow bone-activated charcoal material was also identified using Fourier Transform Infrared Spectroscopy (FTIR) in Figure 5 (a). Bone charcoal spectrum was obtained in wave numbers 4000 cm<sup>-1</sup> to 500 cm<sup>-1</sup>. A large number of peaks in the FTIR range indicates a complex bone char structure [20]. C=C stretching is indicated by peaks obtained at 500 cm<sup>-1</sup> to 750 cm<sup>-1</sup>. The strain adsorption band indicates CO in carboxylic acids detected at 1000 cm<sup>-1</sup> to

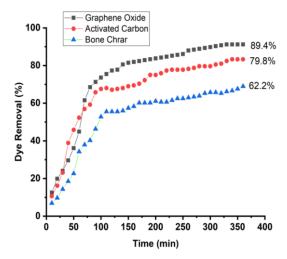
1030 cm<sup>-1</sup>. The bands indicate C-H asymmetric bending at 1400 cm<sup>-1</sup> to 1420 cm <sup>-1</sup> [28] and C-H deformation at 1455 cm<sup>-1</sup> to 1510 cm<sup>-1</sup>. The C=C is associated with peaks of 1627 cm<sup>-1</sup> to 165 5 cm<sup>-1</sup>. The vibrational strain C=C=C is related to a peak from 1987 cm<sup>-1</sup> to 2019 cm<sup>-1</sup>. The C=C=C was associated with peaks of 2068 cm<sup>-1</sup> to 2213 cm<sup>-1</sup>, the C=C strain was associated with peaks of 2309 cm<sup>-1</sup> to 2343 cm<sup>-1</sup>, O=H indicated the presence of free hydroxyl

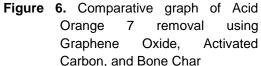
groups and intermolecular bonds were indicated by the peaks obtained from 3750 cm<sup>-1</sup> to 3692 cm<sup>-1</sup>, while the presence of C=C strain on carbon is indicated by peaks obtained from 3870 cm<sup>-1</sup> to 3940 cm<sup>-1</sup>. Figure 5 (b) shows the FTIR spectrum of bone char after it is used to adsorb AO7 dye waste. The vibrations at 3060-3030 cm<sup>-1</sup> (aromatic =C-H stretching), 1600–1450 cm<sup>-1</sup> (aromatic C=C stretching), 1621 cm<sup>-1</sup> (C=N stretching), 1508 cm<sup>-1</sup> (N-H bending), 1452 cm<sup>-1</sup> (Azo-N=N stretching), and 1250-1000 cm<sup>-1</sup> (S-O stretching and aromatic =C-H bending), 1038 cm<sup>-1</sup> (C–O–H stretching), respectively represent the characteristic absorption of AO7 in the bone char [29,30].

# The Absorbability of Activated Charcoal from Cow Bones Against Acid Orange 7

The absorption of activated charcoal from cow bones was determined using a Visible Spectrophotometer. At first, to determine the relationship between adsorption and concentration, a calibration curve of Acid Orange 7 was made. A calibration curve was constructed by conducting an absorbance test of Acid Orange 7 at a concentration of 1 to 10mg/L. The solution was read and plotted spectrophotometrically at a wavelength of 497 nm. According to the applicable Lambert-Beer law equation, the resulting calibration curve represents the relationship between adsorption and concentration. This law is generally used for chemical analytical measurements. Some compounds absorb ultraviolet (UV) and visible (Vis) rays.

The equation in Lambert Beer's law determines the efficiency of activated charcoal from cow bones in removing Acid Orange 7 dye. In addition, organic adsorbents such as activated carbon and graphene oxide were investigated as comparisons. The results of the comparison of the efficiency of Acid Orange 7 dye removal between cow bone activated charcoal (Bone Char), activated carbon and graphene oxide are shown in Figure 6. This absorption test was to determine the ability of activated charcoal to absorb Acid Orange 7 dye compared to the other organic adsorbents.





The adsorption efficiency of Acid Orange 7 was tested using Visible Spectrophotometry. The adsorption value obtained in the visible spectrophotometric test was recorded and used in the calculations to determine the percentage of removal efficiency of Acid Orange 7. Figure 6 shows the effect of contact time on the removal and rate of Acid Orange 7 dye using graphene, activated carbon and activated cow bone charcoal. The adsorption capacity and percentage removal of Acid Orange 7 increased rapidly during the initial stage and then decreased to reach equilibrium at a slower rate. Activated cow bone charcoal (Bone Char) has an absorption efficiency of Acid Orange 7 dye of 62.2%, as shown in Figure 6. It was obtained after 360 minutes of contact time. However, absorption efficiency by graphene oxide and activated carbon showed better performance.

Activated carbon has an efficiency of 79.8%, and graphene oxide has the highest absorption efficiency of 89.4%. The effectiveness of the adsorbent in removing Acid Orange 7 dye from the aquatic environment within a particular time, especially for graphene oxide, is related to the physicochemical and mechanical properties of the adsorbent. The large surface area and high porosity increase the absorption of the active sites available for the dye [31]. The results of this study are not much different from the research conducted by Sumarghandi [8]. About the removal of Acid Orange 7 using graphene and activated carbon. To continue this research, activated cow bone charcoal (Bone Char) is used as an alternative adsorbent. In this study, the effectiveness of activated cow bone char (Bone Char) was produced at 62.2%. It is expected because the adsorbent is obtained from waste cow bones that are no longer used. There is a need for further processing, such as purification of the carbon content in activated cow bone charcoal, the process of removing impurities, and efforts to enlarge the pores in the material so that the efficiency of absorption by activated cow bone char can be better and comparable to activated carbon and graphene oxide

#### CONCLUSION

The result of this study revealed that activated carbon of cow bone, graphene oxide and activated carbon acted effectively in the removal of Acid Orange 7 from the aqueous solution. Activated cow bone charcoal has been characterized using several test equipment such as SEM, SEM XRD EDX, BET, and FTIR. The characterization test showed that the activated charcoal of cow bones is a porous material with 27% hydroxyapatite and carbon content. Therefore, activated cow bone charcoal can be used as an alternative to organic adsorbents with an absorption efficiency of Acid Orange 7 dye in the aqueous solution of 62.2%. Furthermore, it shows good potential because the activated cow bone charcoal can decompose acid orange 7 waste with an absorption capacity that is not too far from the absorption capacity of graphene oxide and activated carbon.

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