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Characterization of Activated Charcoal Produced Using Green Chemistry Principle Approach Number 7

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Abstract
Charcoal is an important component in the effort to provide clean water. One of the water qualities that need to be considered is the level of hardness. Reducing the level of hardness can be done with activated charcoal. The production of activated charcoal uses many synthetic materials. Therefore, aim of research is to produce, test the performance, and characterization of activated charcoal made with natural acids. The stages of this research are charcoal preparation and natural acid activator, carbonization, activation with natural acids (*Averrhoa bilimbi L. and Citrus aurantifolia S.*), and characterization. The production results were tested for adsorbing ability and characterization using FTIR, SEM, and XRD to determine the functional groups, structure, and components. The results showed that activated charcoal produced using natural acids had better performance in adsorbing. Characterization with FTIR showed the absorption of C-H functional groups shown at 785 cm^{-1}, 1100 cm^{-1} for C-O functional groups, 3368 cm^{-1} for O-H stretching vibration groups. Typical active charcoal groups were also detected at wave numbers 1704 cm^{-1} for C=O functional groups and 1575 cm^{-1} for C=C groups. Characterization with XRD showed the same pattern between charcoal produced with natural acids and the control. SEM images showed clear pores on the surface of the activated charcoal. Thus, natural acids can really be a candidate for activator replacement in the production of activated charcoal that applies the principles of green chemistry.

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INTRODUCTION

Activated charcoal is an important component in the water supply process(Niu et al., 2021). Charcoal plays a role in absorbing chemical components that reduce water quality (Reza et al., 2020). Nowadays, the water supply process has become very vital because the amount of clean water is decreasing and water is the most important component for living things (Ajala et al., 2022). Water is one of the primary needs that humans need in living their daily lives, such as cooking, bathing, washing clothes, etc. According to data from the Basic Health Research (Kemenkes) in 2013), most of the water used for household purposes and drinking water comes from 29.9% protected wells, 24.1% boreholes or pumps, 19.7% PDAM water, and 27% springs. On average, 32.9% of urban households used water sources from boreholes or pumps and 28.6% from PDAM tap water, while 32.7% of rural households used water sources from protected wells. From this, the source and quality of water is important for consideration in choosing the source of water used. There are still many people who use well water for daily purposes without considering the quality of the water (Fung et al., 2021).

One of the water quality parameters can be seen from the ion content in it, including ions that can affect the level of water hardness. The ions contained in water and causing hardness are Ca^{2+} and Mg^{2+} ions (Mariana et al., 2021). Water hardness is classified into two types, namely temporary hardness and permanent hardness. Temporary hardness is caused by the presence of bicarbonate compounds (H_2CO_3), which when heated will decompose into CO_2 and H_2O. The calcium and magnesium minerals will leave a white precipitate and can be separated (Li et al., 2023). Temporary hardness can be removed by boiling and can cause scaling of the pan. Permanent hardness is caused by calcium ions Ca^{2+} or magnesium ions Mg^{2+} that bind with Cl^- , SO_4^{2-}, and NO_3^- then form refractory compounds, or heat-resistant compounds. Fixed hardness can only be removed by the addition of certain substances or by special treatment (Bykowska-Derda et al., 2023).

As a handling effort to improve water quality for the community that can be done effectively, easily and at low cost is to use the filtration method using activated charcoal which acts as an adsorbent (Fung et al., 2021). Adsorbent is a medium used in adsorption methods (Sujiono et al., 2022). The best type of adsorbent used in the adsorption process is activated charcoal (Zellner et al., 2019).

Activated charcoal is a porous solid produced from carbon-containing materials by high temperature heating (S. Yadav et al., 2020). The manufacture of activated charcoal currently utilizes many natural materials. It was found that the results reached around 70% active charcoal content and ash content of around 8% (Ashtaputrey & Ashtaputrey, 2020). The research from Khandaker et al., (2021) which produces activated charcoal from natural materials has similar values. Both percentage results when measured with SNI 06-3730-1995 the results are in accordance. To analyze the adsorption ability of activated charcoal can use UV-Vis instruments, where the adsorbent used to reduce the level of water hardness is tested for absorbance with a wavelength of 200 nm - 800 nm (Wazir et al., 2020).

Activated charcoal is generally produced using synthetic acid or base activators. This is not in accordance with green chemistry principle number 7, namely the use of new renewable materials (Zimmerman et al., 2020). In addition, the use of synthetic acids and bases will also violate the principle of reducing the amount of waste, so modifications are needed in the activation process and here lies the urgency of conducting research on the production of activated charcoal in accordance with the principles of green chemistry (Thohir, 2023). Research has been conducted that duplicates the use of synthetic acids and bases with natural materials, such as acids from *Averrhoa bilimbi L.* and *Citrus aurantifolia S.* (Mosharrof et al., 2021). Previous research showed that the adsorption performance of activated charcoal produced with natural acids is not different from synthetic acids (Thohir, 2023). So it is important to continue the search related to the characterization of activated charcoal produced with natural acids.

Characterization is done to see the optimization of active charcoal used as an adsorbent. Some characterizations that can be used such as functional group analysis using FTIR (Ali et al., 2020), surface morphology analysis using SEM (Wazir et al., 2020), and testing with XRD (Wazir et al., 2022). Characterization using FTIR on activated charcoal from corncob and areca nut husk states that the test results both have absorption on aromatic C-H, C-H aliphatic, C=O carboxylic acid, C=O carbonyl, and also N-H and C-C aliphatic absorption on corncob charcoal, while in aromatic C=C on areca nut husk (Njewa et al., 2022).

Other tests characterize activated charcoal using FTIR and find absorption in coconut shells in the form of C=O, C=C, C-C and C-H (Ali et al., 2020) and O-H, C=O, C-O absorption in animal bones (Jawad et al., 2019). The SEM test on the morphological identification of HCl-activated corn cob activated charcoal states that each concentration variation and activator shows a different shape and pore size (Liu et al., 2020). The characterization results in this study will be able to explain the performance of activated charcoal produced using natural acid activators that work equally effectively when compared to charcoal produced with natural acids.

METHOD

Tools and Materials

The tools used in this research include a set of glassware, furnace, analytical balance, mortal pestle, 200 mesh sieve, magnetic stirrer, hot plate, filter paper, spatula, glass funnel, dropper pipette, measuring pipette, FTIR, XRD, SEM. The materials used include corn stover, *Citrus aurantifolia S.* juice solution, *Averrhoa bilimbi L.* juice solution, distilled water, Ca^{2+} solution and Mg^{2+} solution 10 ppm, EBT-NaCl indicator, EDTA, aluminum foil, ammonia buffer pH 10.

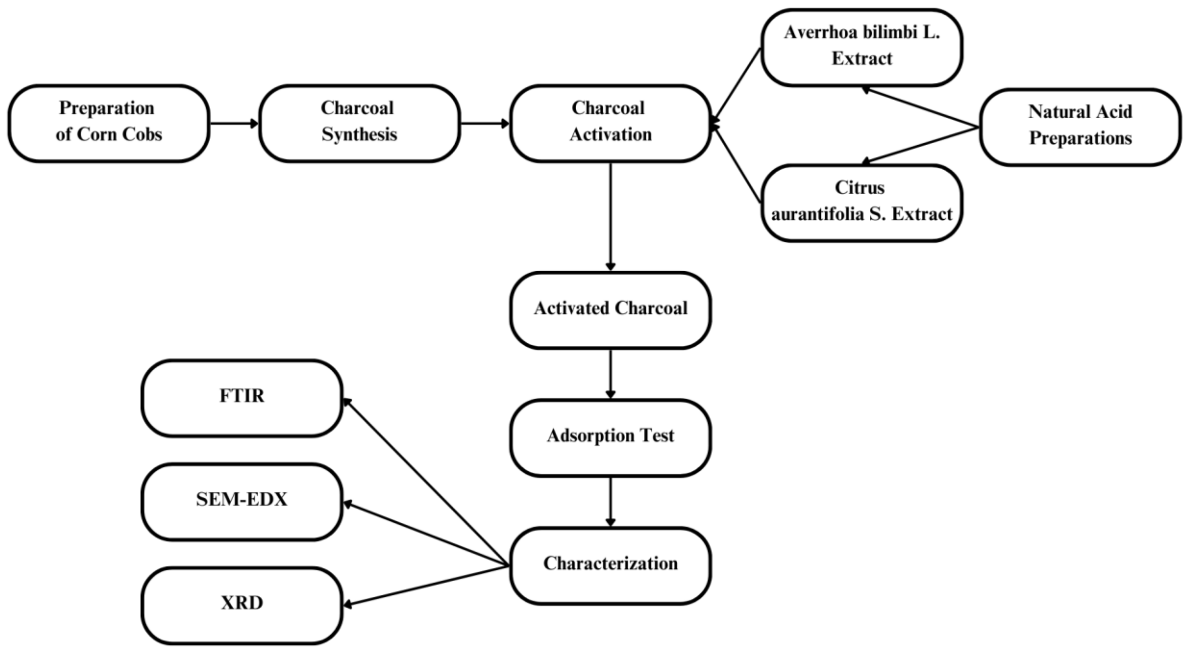

Figure 1. Research procedures

Charcoal Preparation

Corn cobs were ground to reduce their size and placed in a porcelain cup to be fired. The furnace was set to 400 °C for 10 minutes. The charcoal that has been formed is pulverized with a mortar and pestle. Once smooth, it is sieved with a 200 mesh sieve (Thohir, 2023).

Preparation of *Citrus aurantifolia S.* and *Averrhoa bilimbi L.* as Natural Acids

Several *Citrus aurantifolia S.* were prepared and the juice was extracted by squeezing without adding water. After that, filtering is done and the filtrate is taken. This continues to be repeated until getting a volume of 100 mL of *Citrus aurantifolia S.* acid. The same thing is done with *Averrhoa bilimbi L.* (Thohir, 2023).

Activation of Charcoal with *Citrus aurantifolia S.* and *Averrhoa bilimbi L.*

Charcoal that has been taken 5 grams is added to 100 mL of acid from *Citrus aurantifolia S.*. Soaking charcoal with acid from *Citrus aurantifolia S.* is done for 24 hours in a 200 mL beaker glass covered with aluminum foil. This mixture was then filtered and the residue on the filter paper was taken. The residue was refurnished at 110°C for 3 hours. The same treatment is carried out on charcoal that will be activated with *Averrhoa bilimbi L.*, by replacing the *Citrus aurantifolia S.* juice solution with *Averrhoa bilimbi L.* juice (Thohir, 2023).

Characterization of Activated Charcoal

Characterization of active charcoal was carried out using several instruments, namely FTIR Spectrophotometry with wave numbers 4000 - 400 cm^{-1} for charcoal samples, active charcoal with synthetic activator, active charcoal with *Citrus aurantifolia S.* activator, and active charcoal with *Averrhoa bilimbi L.* activator. Characterization with XRD for charcoal samples, activated charcoal with synthetic activator, activated charcoal with *Citrus aurantifolia S.* activator, and activated charcoal with *Averrhoa bilimbi L.* activator. SEM-EDX characterization with 5000 times magnification for activated charcoal with *Citrus aurantifolia S.* and *Averrhoa bilimbi L.* activators.

RESULTS AND DISCUSSION

Charcoal Synthesis and Adsorption Ability

Activated charcoal is a porous solid produced from carbon-containing materials by high temperature heating. Activated charcoal needs to be activated to remove impurities from its voids. The activation process is significant as it affects many things (Vinayagam et al., 2022). What can be affected by the activation process morphologically is the pores of the charcoal. The activation process will make the charcoal pores clean and with a uniform shape (Islam et al., 2022). In addition, when viewed from the resulting properties, activation will affect the adsorbing ability and adsorbing capacity (M. S. Yadav, 2020).

In addition to activation, the source of carbon as the main ingredient of charcoal is also important to consider, so far activated charcoal has been made using useless post-production materials, such as rice husks, tree trunks, and corn cobs. The selection of waste materials as a source of activated charcoal is also in accordance with the principles of green chemistry because it will minimize waste production (Zimmerman et al., 2020).

In the Bojonegoro, a lot of land is managed into corn plantations, so the number of cobs is also very large and can be used to overcome water problems. Corn stalks are the part of the corn where the seeds are attached. This part is usually thrown away because its utilization is still lacking and becomes waste. Corn cob waste that will be disposed of will be a serious problem for the community environment because it causes environmental pollution (Rahma et al., 2019). From here the researchers made innovations in dealing with problems from problems that are in the environment, namely utilizing corn cobs as activated charcoal for adsorbents that can reduce water hardness. Activated charcoal contains several components including water, ash, bound carbon, nitrogen, and sulfur (Patel, 2020). Corn cobs were charred using a temperature of 400 °C for 10 minutes. Activated charcoal is used as an adsorbent because it has a good absorption capacity, which is 1 gram per 500 m^2 measured from the adsorption of nitrogen gas (N_2). Activated charcoal can perform adsorption because it has pores that can expand its surface in adsorbing substances and odors (Riaz et al., 2020).

Activation of charcoal to open the pores of charcoal can be done using heating and the addition of chemicals such as acids and bases (Heidarinejad et al., 2020). Such as the use of sulfuric acid (H_2SO_4) and base (NaOH) as activators for charcoal activation and the results of water content, ash. The charcoal obtained has an optimum absorption capacity with basic activation better than acid activation. Other activators such as using hydrochloric acid (HCl) with a concentration of 0.1 N proved effective for the absorption of lead (Pb), from his research conducted the addition of 40 mg of activated carbon can absorb lead with a percentage of 94.70% (Gao et al., 2020). However, synthetic activators are not in accordance with the principles of green chemistry number 7, so in this study, synthetic activators were substituted with natural acids, namely *Citrus aurantifolia S.* and *Averrhoa bilimbi L.* (Thohir, 2023).

Activation of activated charcoal using natural acids yielded satisfactory results. There is no significant difference between charcoal made with synthetic acids and natural materials from the adsorption ability factor, as shown in Figure 2.

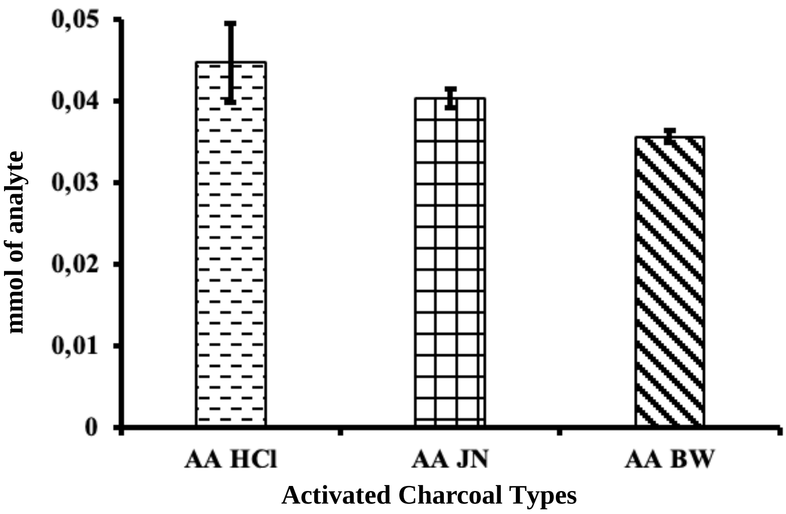

Figure 2. The number of moles that active charcoal can adsorb (AA HCl: active charcoal HCl activator, AA JN: active charcoal *Citrus aurantifolia S.* activator, AA BW: active charcoal *Averrhoa bilimbi L.* activator) (Thohir, 2023).

The activation ability of natural acids is inseparable from the chemical properties contained in them. *Citrus aurantifolia S.* and *Averrhoa bilimbi L.* are natural acids that replace inorganic acid or base activators that are more environmentally friendly. *Citrus aurantifolia S.* contains citric acid, malic acid, and ascorbic acid which are the most content in *Citrus aurantifolia S.*. *Citrus aurantifolia S.* even contains as much as 7-7.5% citric acid per 100 grams of fruit. The pH content in *Citrus aurantifolia S.* is an acidic pH of 2.48-2.5 (Mosharrof et al., 2021). From Figure 2, it can be seen that the remaining analytes absorbed with the activated charcoal from natural acid have a smaller value, meaning that the activated charcoal from natural acid activator has a better adsorbing ability (Thohir, 2023).

Citrus aurantifolia S. is proven to be able to accelerate the evaporation of water content, resulting in the lowest water content in effective acid pretreatment of eggshell powder (Mosharrof et al., 2021). Meanwhile, *Averrhoa bilimbi L.* is a fruit that contains a lot of aliphatic acid compounds, (Z)-9-octadecanoic acid, hexadecanoic acid, sulfate and feroxide (Setyawan et al., 2021). These natural acids in *Citrus aurantifolia S.* and *Averrhoa bilimbi L.* can be used as substitutes for inorganic acids or bases in the activation of activated charcoal. Ultimately, the production of activated charcoal using natural acids has great potential for the future, as it considers environmental sustainability by minimizing the use of synthetic acids.

Charcoal Characterization

The characterization of activated charcoal was done using three instruments, namely FTIR, SEM-EDX, and XRD. FTIR is used to see the functional groups formed in activated charcoal, SEM-EDX is used to see the morphology and atomic composition in the material, and XRD is used to see the lattice of activated charcoal.

FTIR

We display the IR absorption of functional groups in 2 spectra, namely specifically for the absorption of fingerprint regions (fingerprint) and main groups. We separate this so that the readings do not overlap and know the role of each spectral region according to the regions that have different properties.

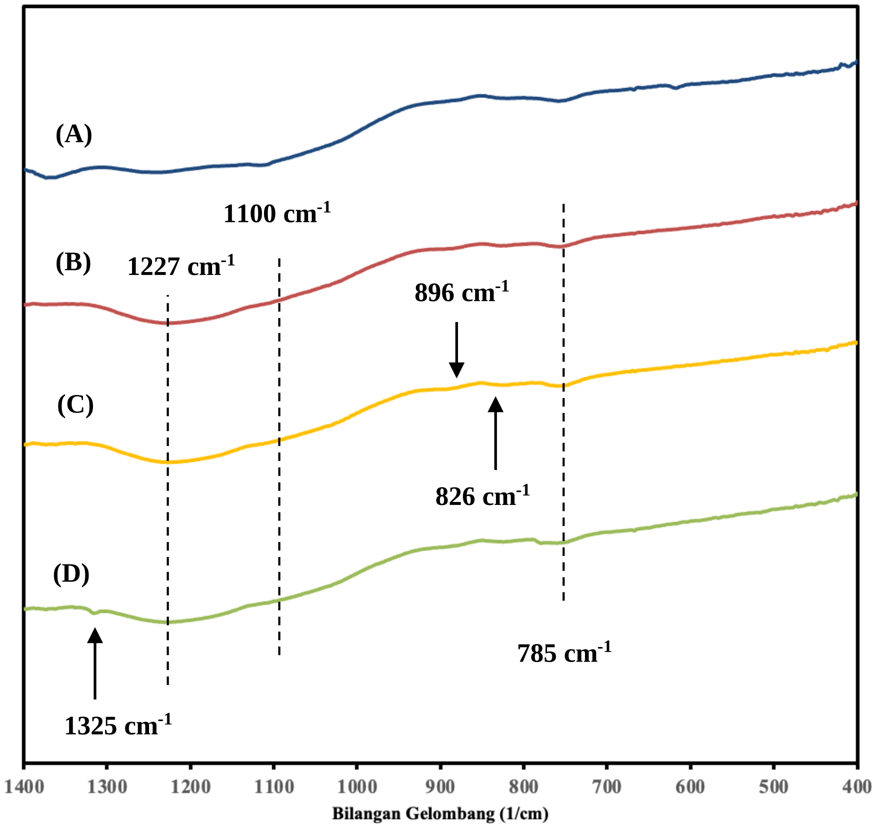

Figure 3. FTIR spectra of fingerprint region (A) Charcoal (B) Activated charcoal with synthetic activator (C) Activated charcoal with *Citrus aurantifolia S.* activator (D) Activated charcoal with *Averrhoa bilimbi L.* activator

In the fingerprint area, for charcoal activated with *Averrhoa bilimbi L.*, there are several absorptions that appear such as in the 785 cm^{-1} area which is the absorption of the C-H group. 1100 cm^{-1} for the C-O functional group. And at a wavelength of 1325 cm^{-1} is the C-N group. There is a specific absorption owned by charcoal activated with blimbing wuluh is in the 1325 cm^{-1} region (Ali et al., 2020), this can be an indication of the emergence of new bonds and which becomes potential as an active side of adsorption. Because the N atom has a free electron pair (PEB) which has the potential to be used as an ion exchange medium (S. Yadav et al., 2020).

While for activated charcoal activated with *Citrus aurantifolia S.*, there are several absorptions in the fingerprint area, including the C-H group at wave number 760 cm^{-1}, wave numbers 896 cm^{-1} and 826 cm^{-1} indicate the presence of a typical C-H group bending vibration (Wazir et al., 2020). And wave number 1227 cm^{-1} indicates the presence of C-O stretching vibration group (Mariana et al., 2020).

If you look closely, there is actually an increasing trend downward, especially in the 760 cm^{-1} region, this shows that the activation process using natural acids has the same ability as synthetic acids, and even better. The greater the absorption that appears shows the more bonds that are open.

As for the main region, there are many functional groups identified. Some that can be identified include the O-H group in the 3272 cm^{-1} absorption band. Absorption in the 1575 cm^{-1} region which indicates the presence of C=C groups. C=C groups are typical groups on activated carbon. Meanwhile, 1575 cm^{-1}; 1936 cm^{-1}; and 2250 cm^{-1} identify C-N groups (S. Yadav et al., 2020).

While for charcoal activated by using *Citrus aurantifolia S.*, there are several absorptions that appear. The absorption for the O-H stretching vibration group bond is at wave number 3368 cm^{-1} (Syamsu et al., 2020), wave number 2324 cm^{-1} shows the presence of CH and CH_3 functional groups which indicate the presence of fat and ammonia, wave number 2167 cm^{-1} shows the presence of CO_2 bonds, wave number 1704 cm^{-1} shows the C=O (aldehyde) functional group (Khandaker et al., 2021), wave number 1594 cm^{-1} indicates the presence of C=C stretching vibration group, wave number 1423 cm^{-1} indicates O-H group (Jawad et al., 2019).

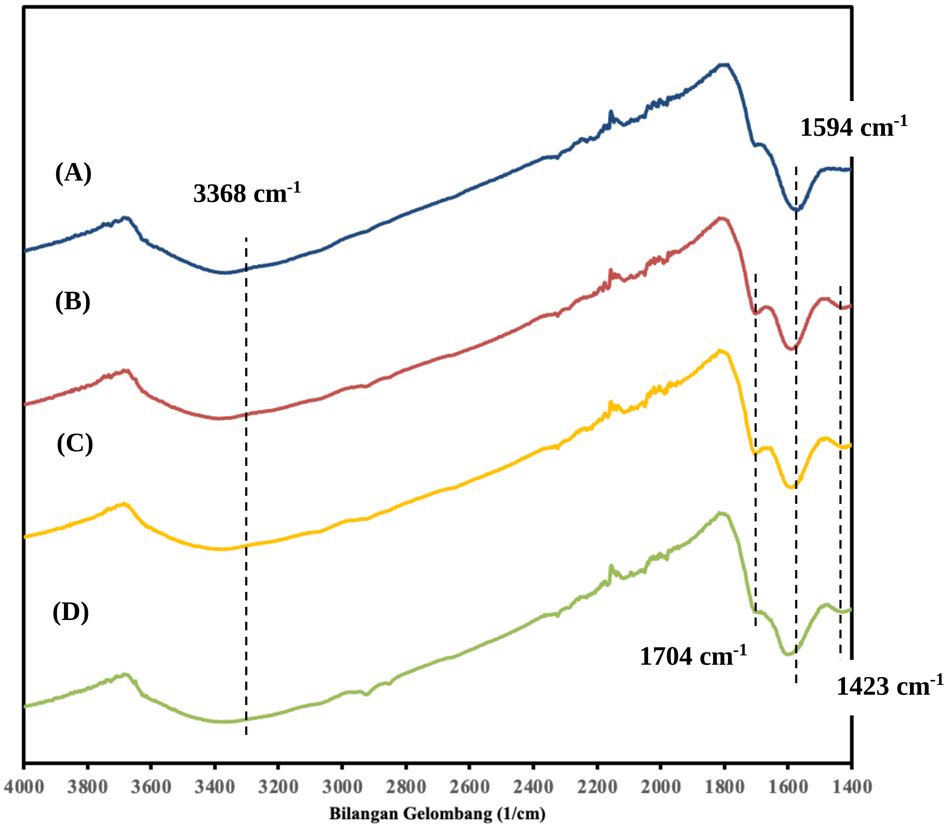

Figure 4. FTIR spectra of main groups of (A) Charcoal (B) Activated charcoal with synthetic activator (C) Activated charcoal with *Citrus aurantifolia S.* activator (D) Activated charcoal with *Averrhoa bilimbi L.* activator

There is a cluster that only appears on activated charcoal, and the downward trend is clearer in the 2800 to 3000 cm^{-1} region. The cluster that appears in the absorption is associated with C-H. The cluster is also emphasized because it has appeared in the fingerprint area.

In addition to the C=C group which is typical of active charcoal, the C=O group is also a typical group found on active charcoal, this means that active charcoal is successfully activated by natural acid compounds from *Citrus aurantifolia S.*.

SEM

The morphological data of activated charcoal surface for *Citrus aurantifolia S.* and *Averrhoa bilimbi L.* showed impressive results (Vinayagam et al., 2022). When viewed from the surface results obtained, there are a large number of pores on the surface with uniform size and evenly distributed. In the pores formed, it can be seen that the pores are made perfectly and there are no closed holes (Harmawanda et al., 2023). This indicates that the main work of the activator as a pore maximizer works well (Niu et al., 2021). From these results it can also be seen that the use of natural acid activators is also able to work optimally (Islam et al., 2022).

Averrhoa bilimbi L. ***Citrus aurantifolia S.***

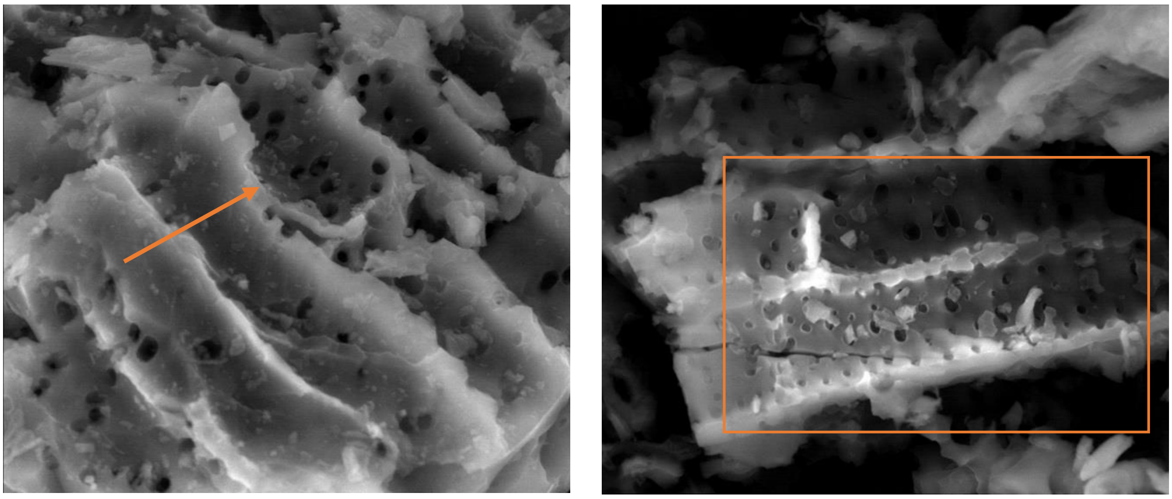

Figure 5. SEM image of activated charcoal activated with natural acid

There are many types of acids in natural materials. In addition to acids, we also know that there are many secondary metabolites in natural materials. What we need to know more is whether the secondary metabolites of natural materials also help the activation process, because it is very possible that the activation work is done not only from the acid molecules contained in natural materials. As happened in the process of making gold nanomaterials with natural materials. It turns out that components in natural materials such as tannins, terpenes, saponi and others can be a reducing agent and stabilizer at once. This is necessary and important to be examined further.

Averrhoa bilimbi L. ***Citrus aurantifolia S.***

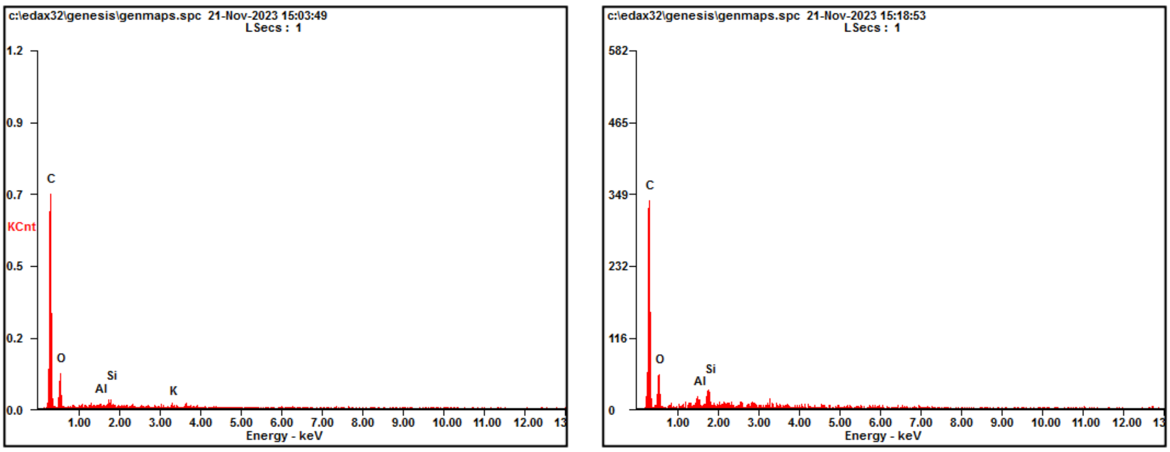

Figure 6. EDX spectra for atomic percentage in activated charcoal with natural acid activator

Table 1. Percentage of atoms in activated charcoal

Elements	Percent Weight	
	Averrhoa bilimbi L.	*Citrus aurantifolia S.*
Carbon	65,48	65,12
Oxygen	30,73	29,10
Alumina	00,91	01,82
Silica	01,64	03,96

In addition to surface data, SEM combined with EDX provides data on the percentage of atoms contained (Sujiono et al., 2022). From the test results, it shows that carbon has a percentage value of about 70%, followed by oxygen because the material is always in oxide form as much as 30%. This is in accordance with the standard of activated charcoal whose constituent is carbon with a percentage reaching 70%.

XRD

The XRD data obtained showed that all components tested were amorphous (Islam et al., 2022). This is reasonable because the charcoal is produced at a relatively moderate temperature of 400 degrees and in a short time, 10 minutes. This certainly has not been able to make the components change in the crystalline phase. Carbon will begin to form crystalline at 700 degrees and within 1 hour. However, the charcoal making process does not require such high temperatures.

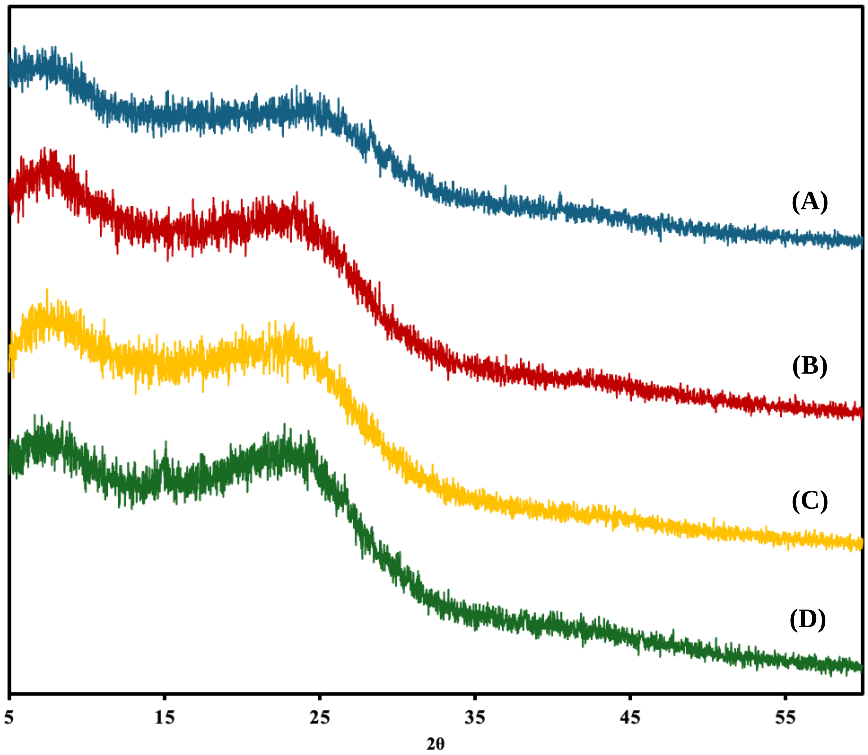

Figure 7. XRD spectra of (A) Charcoal (B) Activated charcoal with synthetic activator (C) Activated charcoal with *Citrus aurantifolia S.* activator (D) Activated charcoal with *Averrhoa bilimbi L.* activator

The results obtained show that the activation process shows an increase in peaks. On charcoal without activation, the peak formed is still sloping, but after the activation process, the peak becomes sharper. This is an indication that the activation process does make the charcoal pore holes more open and free of impurities (S. Yadav et al., 2020).

In addition, it can also be seen that activation using natural acids has the same performance, and this further consolidates what was found earlier, that natural acids can really be a candidate for replacing synthetic acids in the activation process.

CONCLUSION

The production of activated charcoal using natural acids as activators has proven successful in substituting the work of synthetic acids. This can be seen from several factors, such as adsorption ability, functional group absorption through FTIR test, morphology from SEM image, and crystal lattice from X-ray diffraction. In terms of adsorption ability, it was found that the natural acid had a better performance by being able to leave only 0.04 mmol of adsorption test analyte. FTIR data showed that the activated charcoal produced with various acids had functional groups for C-H at 785 cm^{-1}. 1100 cm^{-1} for the C-O functional group. The absorption of O-H stretching vibration group is at wave number 3368 cm^{-1}. Groups typical of activated charcoal were also detected at wave numbers 1704 cm^{-1} for the C=O functional group and 1575 cm^{-1} for the C=C group. On the surface morphology of the charcoal, it was found that the characterization results showed that the charcoal had homogeneous and repeated pores with clean and evenly distributed holes. The amount of carbon reached 70% in the EDX test. And the XRD test showed a sharper absorption after activation. These results mean that natural acids that do not violate the principles of green chemistry can replace synthetic acids.

RECOMMENDATIONS

There needs to be a deeper analysis using metabolite analysis approaches and more advanced instruments to find out what metabolites are most significant in influencing charcoal production with natural acids.

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