

THE EFFECT OF CHEMICAL ACTIVATOR ON THE EFFECTIVENESS OF ACTIVATED CARBON FROM CORN COBS

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ABSTRAK

Penambahan aktivator kimia dimaksudkan untuk meningkatkan luas permukaan karbon dan membuka pori-pori yang tertutup, sehingga meningkatkan kapasitas adsorpsi karbon aktif. Penelitian yang menggunakan tiga aktivator kimia yang berbeda - HCl, NaOH, dan Na₂CO₃ - bersama dengan aktivasi fisik pada suhu 750°C menunjukkan peningkatan kualitas dibandingkan dengan proses tanpa aktivasi. Ketika memproduksi karbon aktif dari tebon jagung, hasil yang tinggi sebesar 80-82% dapat dicapai. Uji evaluasi terhadap karbon aktif menunjukkan parameter berikut: kadar air berkisar antara 6% hingga 8,79%, kadar abu antara 2,89% hingga 4,49%, dan bilangan iodin bervariasi dari 751,11 hingga 812,16. Hasil ini memenuhi standar yang ditetapkan oleh SNI 06-3730-1995. Temuan ini mengindikasikan bahwa HCl merupakan aktivator kimia yang paling efektif untuk mensintesis karbon aktif dari tongkol jagung. Kesimpulan ini didukung oleh karakterisasi FTIR dan XRD. Analisis FTIR menunjukkan adanya gugus fungsi yang khas dari arang aktif, seperti O-H, C=O, dan C-C. Selain itu, analisis XRD menunjukkan adanya SiO₂ amorf, yang merupakan sifat yang menguntungkan untuk adsorben yang terdefinisi dengan baik, yang diamati pada sudut 2θ 20-30 derajat.

ABSTRACT

The addition of chemical activators is intended to increase the surface area of carbon and open closed pores, thereby enhancing the adsorption capacity of activated carbon. Research utilizing three different chemical activators—HCl, NaOH, and Na₂CO₃—along with physical activation at 750 °C demonstrated improved quality compared to processes without activation. When producing activated carbon from corn stover, a high yield of 80-82% was achieved. Evaluation tests on the activated carbon revealed the following parameters: the moisture content ranged from 6% to 8.79%, the ash content was between 2.89% and 4.49%, and the iodine number varied from 751.11 to 812.16. These results meet the standards set by SNI 06-3730-1995. The findings indicated that HCl is the most effective chemical activator for synthesizing activated carbon from corn cob. This conclusion is further supported by FTIR and XRD characterization. FTIR analysis revealed the presence of functional groups typical of activated charcoal, such as O-H, C=O, and C-C. Additionally, XRD analysis revealed the presence of amorphous SiO₂, a favorable property for a well-defined adsorbent, observed at 2θ angles of 20-30 degrees.

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INTRODUCTION

Activated carbon is produced from carbon-containing materials. Activated carbon is interesting to develop due to its wide range of applications. This material, which has an amorphous structure and pores with a large surface area, is derived from various precursors through a thermal process (Daouda et al., 2021). Precursors derived from lignocellulosic biomass are potential raw materials to be developed. Exploration of natural materials remains a concern for researchers, primarily due to the high demand for raw materials, the need for renewable sources, and the desire to obtain inexpensive materials.

East Java is the largest corn-producing province. Since the waste produced is also relatively high in this area, one of the main contributors is corncobs. This biomass is composed of cellulose (41%), hemicellulose (36%), lignin (6%), and other compounds commonly found in plants (Kusuma et al., 2020). Additionally, the carbon content exceeds the ash content, thereby increasing the potential of corncob-activated charcoal. The process of making activated carbon from biomass waste has three important stages, namely the preliminary process (washing and drying), the carbonization process, and the activation process (Sriatun et al., 2020).

The carbonization process is a method of breaking down organic matter into carbon without the presence of air. Carbonization is generally carried out in the temperature range of 400-600 °C (Rahmadani & Kurniawati, 2017). The results of this process are expected to open the pores of the activated carbon surface (Erawati & Fernando, 2018). The characteristics of activated carbon are strongly influenced by the composition of the precursor, the activation method (physical or chemical), the oxidizing agent, and the treatment process.

Activation is the process of forming activated carbon by enlarging the diameter of carbon pores that have been formed from the carbonization process. It also opens new pores to increase the volume that will be absorbed in

the carbon pores (Erawati & Fernando, 2018). Generally, carbon activation can be done by physical and chemical activation. Compared to physical activation with high temperatures, chemical activation at room temperature produces more activated carbon yield, does not pollute the environment, and also has wider utilization (Hisbullah et al., 2022). In chemical activation, the precursor is reacted with an acid, base, and or salt solution activator (Ho, 2022). Reagents commonly used as activating agents include NaOH, ZnCl₂, KOH, HCl, H₃PO₄, Na₂CO₃, etc. (Hisbullah et al., 2022).

Activated carbon with HCl activation has been produced as an adsorbent for Cd(II) ions by (Rizkyi et al., 2016). This research shows that the adsorption power of activated carbon is 30.658 ppm. The addition of this activator aims to dissolve impurities on the surface of activated charcoal, particularly lignin, thereby allowing the pores to open and expand the surface of the activated charcoal. Acid treatment of activated carbon converts crystalline cellulose into an amorphous form, resulting in mesoporous carbon with a significantly large surface area. Another study (Rachmawati & Mujiburohman, 2021) provided methylene blue adsorption power of 21.876 ppm with NaOH activator. The addition of this activator can degrade the structure of lignin, hemicellulose, and other substances, making the pores more open and enhancing the adsorption power. The use of Na₂CO₃ activator for the manufacture of activated carbon from corn stalks for samples of dug well water into clean water, has been carried out by (Anggraini et al., 2023). Some of the above studies show that these activators have the potential to provide better activated carbon properties than without activation. Additionally, the use of these activators is related to the porosity and volume distribution of the carbon pores (Medhat et al., 2021).

Therefore, this study aims to identify the optimal conditions for synthesizing activated carbon from corn cob raw materials by varying the type of chemical activator, using HCl (acid,

ACA), NaOH (base, ACB), and Na₂CO₃ (salt, ACG). This research is expected to produce high-quality activated carbon that meets the needs of activated carbon in Indonesia.

METHODS

Materials

The materials used in this research are corncob waste from Kawengan Village, Bojonegoro, using gloves and polyester plastic containers. For activator materials, pro-analysis solutions were used, including HCl 1 M, NaOH 1 M, Na₂CO₃ 1 M, KI, I₂, and Na₂S₂O₃, and distilled water.

Tools

The equipment used in this research includes an analytical balance (JA3003 electronic balance), furnace (Manual Operation B-one mini muffle furnace 1210), mortar pestle, beaker (Herma), mohr pipette (Iwaki CTE33 ashashi glass, made in Indonesia), magnetic stirrer (Cimarec), suction ball (D & N, made in Germany), spatula, 100 mesh sieve, Whatmann filter paper, and Uv-Vis Spectrophotometer (722 Spectrophotometer-Vis). Furthermore, it was characterized using a Thermo Nicolet iS10 FT-IR spectrophotometer, which was used to determine the functional groups of corncob-activated carbon. The analysis was carried out at wavelengths between 4000 and 400 cm⁻¹. In addition, it was characterized using PANalytical's X-Pert PRO X-ray diffractometer (XRD) with a diffraction angle range of 5° to 60° to examine the crystal structure of corncob-activated carbon.

Sample Preparation

The initial step in sample preparation is manual washing and drying. This procedure follows the method from (Anwar, 2020). Before use, corncob samples were cleaned with clean water. Then, they were cut and dried in the sun for 3 days. After drying, the corncobs are furnace at 120°C for 2 hours, so that they are entirely free from water content (Anwar, 2020). This process is also known as the dehydration process. After dehydration, the corncobs are then carbonized using a furnace at 750°C for 2 hours (Rachmawati & Mujiburohman, 2021). The corncob ash (ABJ) is then pulverized and sieved using a 100 mesh sieve.

Activation of Carbon from Corn Cob Waste

A total of 10 grams of ABJ was mixed using a variety of activator solutions, including 1 M HCl, 1 M NaOH, and 1 M Na₂CO₃. The mixture ratio of activated carbon to activator was 1:10. It was then homogenized using a magnetic stirrer for 24 hours at a rotating speed of 300 rpm. After that, it was filtered and washed with distilled water until the pH became 7 (neutral). Then dried in a furnace at 120°C for 2 hours.

Quality Test of Corn Cobs Carbon

To determine the quality of the corncob-activated carbon, the parameters of moisture content, ash content, and iodine number were measured. There were three repetitions for each treatment carried out.

Moisture content

The moisture content was determined by weighing the sample, which was as much as 1 gram, and then placing it in a previously weighed porcelain cup. Then the cup was heated in an oven at 105°C for 1 hour. The cup was then cooled in a desiccator and weighed until a constant weight was achieved. Moisture content was calculated with equation 1.

$$\text{Moisture content}(\%) = \frac{B_a - B_b}{B_a} \times 100\% \quad (\text{Eq. 1})$$

B_a = initial mass of sample (g)

B_b = final mass of sample (g)

Ash content

Ash content was determined by weighing 1 gram of the sample and placing it in a porcelain cup of known weight. The sample was then refurnished at 750°C for 1 hour. The sample was then cooled in a desiccator and weighed until the weight was constant. The determination of ash content was calculated based on Equation 2.

$$\text{Ash content}(\%) = \frac{B_b}{B_a} \times 100\% \quad (\text{Eq. 2})$$

B_a = initial mass of sample (g)

B_b = final mass of sample (g)

Iodine number

Iodine number was determined with a

0.25-gram sample in an Erlenmeyer flask and 25 mL of 0.1 N iodine standard solution was added. The mixture was stirred for 15 minutes and filtered. A total of 10 mL of filtrate was transferred to another Erlenmeyer flask and titrated with 0.1 N $\text{Na}_2\text{S}_2\text{O}_3$ until the color

$$\text{Iodine number} = \frac{10 - \left(\frac{B \times C}{D} \right) \times 12,693 \times 2,5}{W} \quad (\text{Eq. 3})$$

B = The volume of sodium tiosulfate for titration (mL)

C = sodium tiosulfate (N)

D = iodine (N)

W = mass of activated carbon (g)

12,693 = amount of iodine corresponding to 1 mL of 0.1 N sodium thiosulfate solution

changed to pale yellow. Add 1% amylose indicator, and titration continues until the solution turns clear. Record the volume of the used peniter. The data obtained is used in the calculation according to Equation 3.

Data Analysis

Activated carbon that has been tested for quality is then analyzed according to the quality standards of activated charcoal that apply in Indonesia. Then, proceed with characterization using FTIR to identify its functional groups and characterization with XRD to assess its crystallinity.

RESULT AND DISCUSSION

Activated Carbon Sample Preparation

A series of sample preparation procedures is required to achieve an effective and fast production process. The biomass as starting material should be free of impurities by washing with deionised water and then dried, pulverised, and sieved to obtain a uniform particle size (Ukanwa et al., 2019). This enables a faster carbonization process by lowering the thermal gradient.

Table 1. Yield results of activated carbon with various activators

Activator	Initial Weight (g)	Final Weight (g)	Yield (%)
HCl	25	20,45	82
NaOH	25	20,28	81
Na_2CO_3	25	20,00	80

Chemical activation of biomass can improve the overall efficiency of activated carbon. Furthermore, it can provide opportunities for sample applications due to the presence of activating agents for the process (Ukanwa et al., 2019). Yield results of activated carbon with various activators are presented in Table 1.

NaCl , H_2SO_4 , Na_2CO_3 , HCl, and NaOH solutions are some of the activators used for activated carbon activation. In this study, HCl (ACA), NaOH (ACB), and Na_2CO_3 (ACG) were used as activators. The use of an ACA activation agent in the synthesis of activated carbon aims to remove lignin and allow the formation of more pores (Ho, 2022). In addition, the addition of strong acids to activated carbon also results in changes to cellulose crystals, making them amorphous. Meanwhile, the use of ACB as an activating

agent in the manufacture of activated carbon is to increase porosity. The use of ACB also produces low ash content, as well as good absorption capacity. Meanwhile, the use of ACG as an activator can remove impurities that contribute to pore formation.

To determine the effectiveness of activated carbon that has been activated, it is necessary to conduct a quality test. The relationship between the type of activator and the quality of corncob carbon was investigated by analyzing water content, ash content, and iodine number testing. The quality test results are described as follows,

Water Content

Water content analysis was conducted to determine the ability of the activator as a hydrating agent in the carbonization process. Figure 3 shows the moisture content of activated

carbon with various activators.

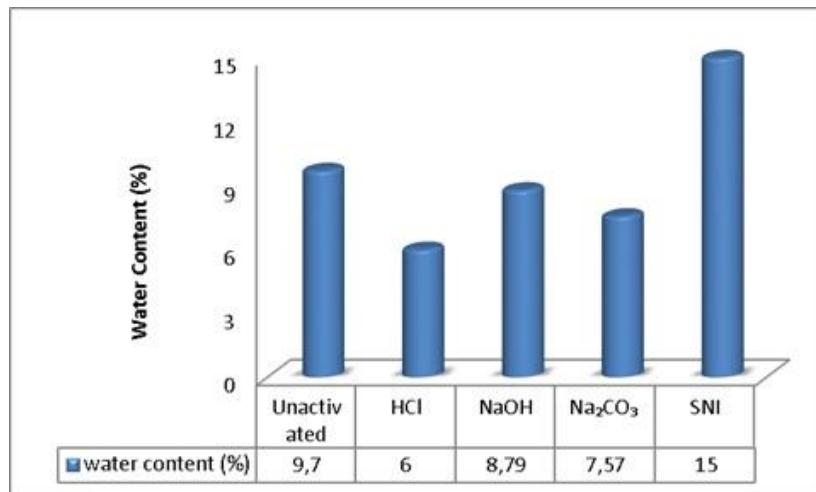


Figure 1. Water content of activated carbon with various activators

In the test data (Figure 1), the lowest water content is when using ACA. This is related to the effect of the pH of the activator and its behavior when reacting with water during the washing process. The hygroscopic nature of HCl can reduce moisture content due to its ability to bind water more completely. Lower water content indicates that less water remains in the activated charcoal. Thus, activated charcoal can have a larger surface area, and its adsorption effectiveness is also better than that unactivated one (Safitri et al., 2024). The test results on all samples (Figure 3) show that the activated carbon produced meets the quality standards of SNI 06-3730-1995 for water content, which is a maximum of 15%.

Ash content

Ash content is the number of materials and minerals that are not burned during the carbonization process. Testing the ash content is necessary because the biomass base material contains not only carbon compounds but also several other minerals. According to Hisbullah et al. (2022), a lower ash content value in activated carbon provides better quality. This is because the presence of ash content can result in pore closure on the surface of activated charcoal, so it will reduce its adsorption ability. The results of the ash content test in this study are presented in Figure 2.

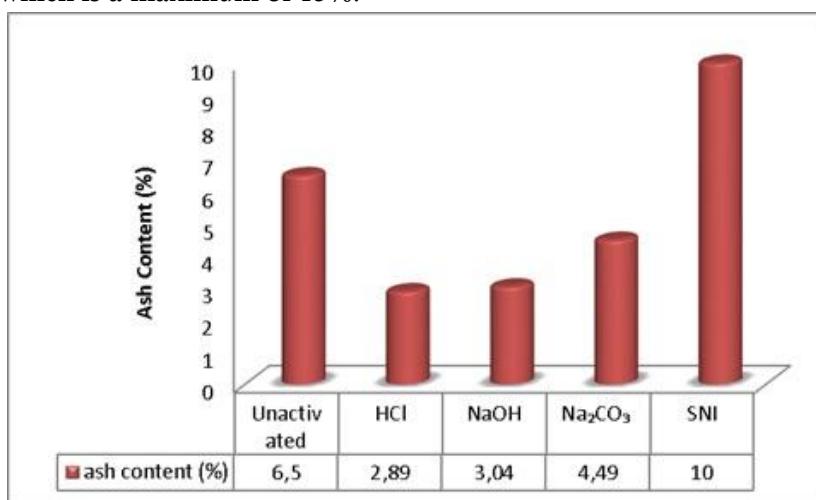


Figure 2. Ash content of activated carbon with various activators

The results of the study, as shown in Figure 2, indicate that acidic activators are more optimal for expanding the surface of activated

carbon, thereby forming more pores. This is because the ACA dissolves minerals by degrading cellulose into carbon. So that the ash

content in ACA becomes lower than the other activators. The test results on all samples indicate that the activated carbon produced meets the quality standards of SNI 06-3730-1995 for ash content, with a maximum of 10%.

Iodine Number

The purpose of the absorption of iodine number is to determine the adsorption ability of

coloured solutions. The absorbency of activated carbon to iodine is the number of milligrams of iodine adsorbed by one gram of activated carbon. The higher the iodine number absorbency value, the higher the quality of activated carbon. Therefore, the iodine number absorbency is one of the important indicators in producing activated carbon.

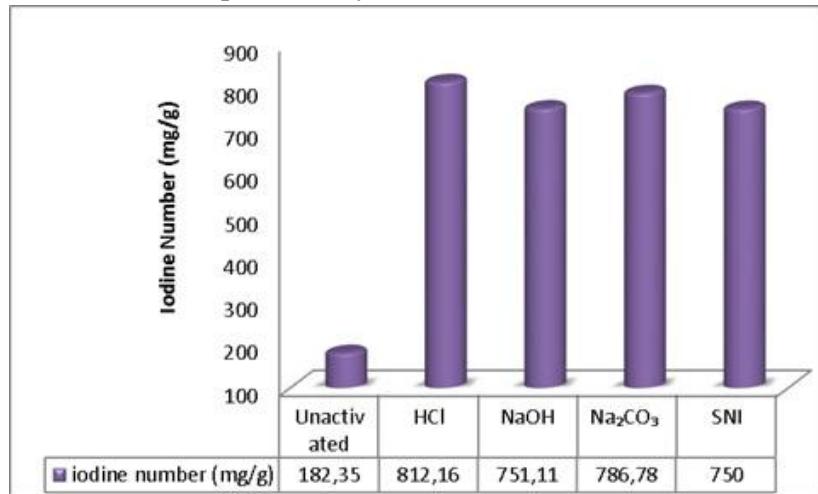


Figure 3. Iodine numbers absorbency of activated carbon with various activators

The results of the iodine number absorbance test are presented in Figure 3. The data in Figure 3 shows that all activated carbons gave higher results than the unactivated carbons. However, HCl activator is the best activation agent for corncob sample preparation compared to other activators. This is because

acidic substances can bind water more perfectly to dissolve organic and inorganic substances bound in hexagonal-structured carbon material to obtain carbon with cleaner and more open pores (Sriatun et al., 2020). This aligns with the value of water content.

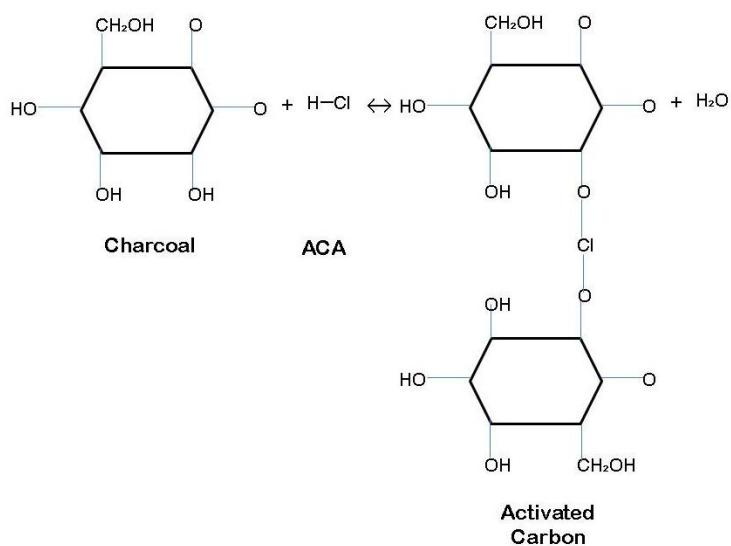


Figure 4. Reaction mechanism of activated charcoal with HCl activator

When activating with HCl, Cl⁻ will enter through the hexagonal carbon bonds and capture impurities that cover the pores. HCl also

acts as an oxidising agent on the surface of the activated carbon molecules. The reaction mechanism of activated charcoal with HCl

activator is presented in Figure 4.

Activated Carbon Characterization

To determine any changes in chemically activated carbon, the samples were then characterized. In this study, FTIR and XRD were used to characterise the changes in the samples. Figure 5 shows the results of the FTIR spectra of corncob-activated carbon, where the spectra of activated carbon are slightly different from the spectra of carbon without activation, there is a change in the shape of the spectra, shifting, reducing the intensity and adding new peaks after the activation process, possibly due to the carbonization and activation process causing dehydration, decomposition of complex lignocellulose groups into simpler groups (Efifyanti, 2020).

The absorption region at 3000 cm^{-1} indicates the presence of OH stretching functional groups, resulting from hydroxyl groups with hydrogen bonds. The OH group vibration peak looks quite sharp, indicating free OH bonds that may come from hydrogen bonds, so that it is possible to interact with water molecules adsorbed by activated carbon

(Efifyanti, 2020). The 2000 s cm^{-1} absorption region shows the presence of $\text{C}\equiv$ functional groups from stretching alkynes.

Furthermore, changes in the presence of functional groups on activated carbon are presented in Figure 6, both for activated carbon without activation and activated carbon with several activators. In addition, the crystallinity is also shown in Figure 6.

FTIR (Fourier Transform Infrared) Characterization

The adsorption capacity of activated carbon is influenced by several factors, including water content, ash content, and iodine number, as well as its chemical composition. One key aspect is the presence of functional groups, which serve as the active components of activated carbon. To identify the functional groups present in the compounds found in corncob, FTIR analysis was performed. The spectra of FTIR Characterization results on corncob adsorbent before and after activation can be seen in Figure 5.

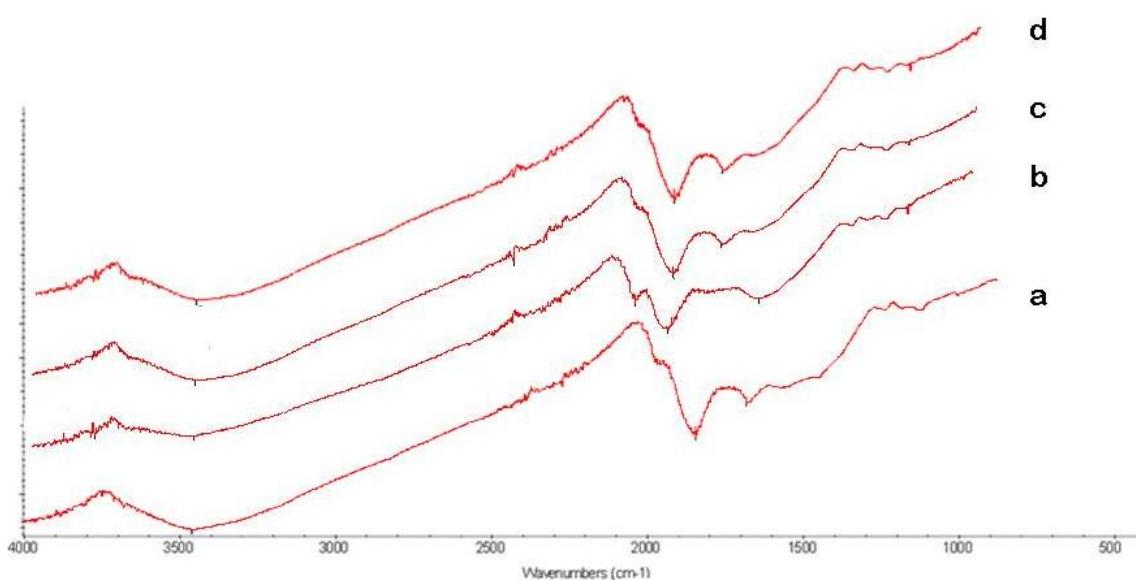


Figure 5. FTIR spectra (a) Without Activation, (b) Activation of HCl 1 M, (c) Activation of NaOH 1 M, (d) Activation of Na_2CO_3 1 M.

Figure 5 presents patterns of peak absorption at wavenumber 3450 cm^{-1} , 1897.59 cm^{-1} , and 1643 cm^{-1} . Changes in functional groups and absorption bands mark the chemical structure differences between carbon and

activated carbon. The OH group absorption band at 3450 cm^{-1} in non-activated carbon is wider than in activated carbon, indicating that non-activated carbon retains more OH groups. The $\text{C}=\text{O}$ functional group appears at a

wavenumber of about 1897.59 cm^{-1} and enhances the adsorption performance. The $\text{C}=\text{O}$ group is generally a description of stretch vibrations from carboxylic acids, ketones, or lactones (Belhamdi et al., 2019). Using an acidic activator decreases peak intensity and narrows the adsorption band, indicating a loss of volatile compounds (Sriatun et al., 2020).

Hydroxyl and carbonyl groups are polar, making the surface of activated carbon hydrophilic and increasing the hydrophilic properties of activated carbon (Efifyanti, 2020). The FTIR spectra patterns of activated carbon in this study have similarities with Hisbulah et al., 2022; Sriatun et al., 2020. The aromatic ring

$\text{C}=\text{C}$ functional group is shown at a wave number of 1643 cm^{-1} .

a) XRD (X-Ray Diffraction) Characterization

Characterization of samples using X-Ray Diffraction XRD to determine the crystal structure or information on the presence of crystalline and amorphous phases of the basic materials that have been synthesized, both in the form of elemental content and in the form of compounds. The presence of sharp peaks characterizes the crystalline phase while the formation of wide peaks or humps with low intensity characterizes the amorphous phase (Saban, 2023).

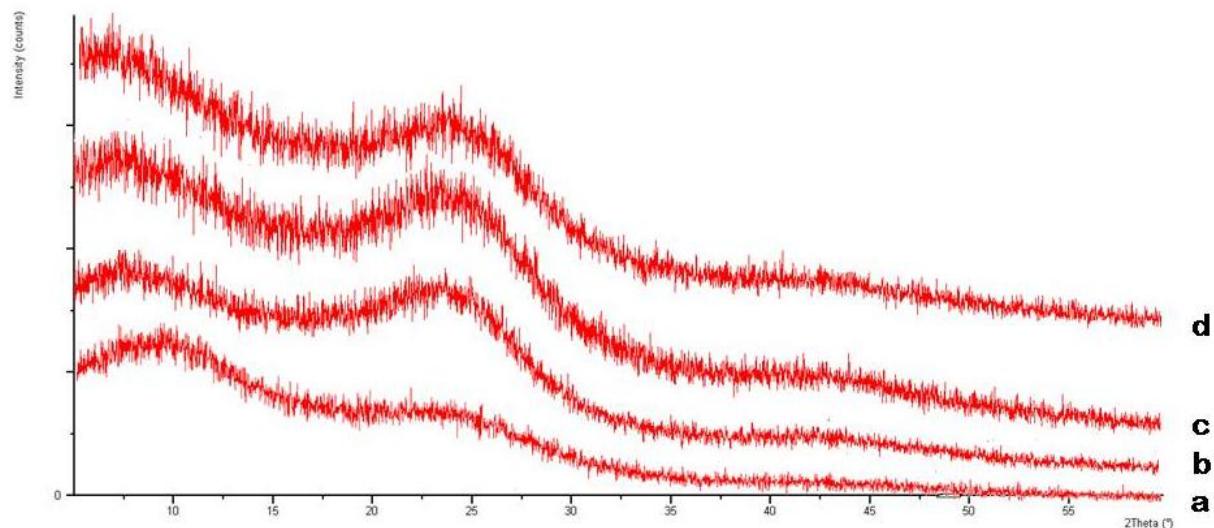


Figure 6. XRD diffractograms (a) Without Activation, (b) Activation of HCl 1 M, (c) Activation of Na_2CO_3 1 M, (d) Activation of NaOH 1 M

Figure 6 shows the X-ray diffraction of corncob-activated carbon without activation and also activated with HCl , Na_2CO_3 , and NaOH . The X-ray diffraction results show carbon with an amorphous structure, making it impossible to determine the crystalline phase of the carbon formed (Bijang et al., 2022). According to (Saban, 2023) activated carbon samples with broad peaks and the absence of sharp peaks, indicating a predominantly amorphous structure, are favorable properties for well-defined adsorbents.

XRD results showed peaks at 2θ angles of 20-30 degrees for the samples with no activation, HCl activator, Na_2CO_3 activator, and NaOH activator. There are differences in the diffractograms before and after activation.

The difference is mainly evident at the peak 2θ angle of 20-30 degrees; after activation with HCl , the peak on activated carbon widens. The purpose of activation is to remove impurity compounds and expand the pore space of activated carbon. Under normal circumstances, before activation, the pore space of activated carbon is filled with various substances, including cations and unbound water molecules. The removal of molecules from the pore space of activated carbon will expand the pores.

CONCLUSION

Research results from the use of three different chemical activators, namely HCl ,

NaOH, and Na₂CO₃, as well as physical activation at 750°C, showed a quality improvement when compared to no activation. Evaluation tests were conducted to assess parameters such as moisture content (6%), ash content (2.89%), and iodine number (812.16%), in accordance with SNI 06-3730-1995. The results showed that HCl was the best chemical activator for synthesizing corncob-activated carbon. FTIR and XRD Characterization further support this conclusion. FTIR analysis revealed the presence of typical functional groups associated with activated charcoal, including O-H, C=O, and C-C. XRD analysis showed the presence of amorphous SiO₂, an advantageous property for a well-defined adsorbent, which was observed at 2θ angles of 20-30 degrees.

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