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The Effect of Alkaline Base Activator Type on Biochar Character of Coconut Shell as a Heterogeneous Catalyst Support

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ABSTRACT

Biochar derived from coconut shells shows promise as a support for heterogeneous catalysts. This study evaluated the effectiveness of alkaline activators (KOH 2 M, NaOH 2 M, and $Ca(OH)_2$ 2 M) in enhancing biochar properties. Biochar was produced by pyrolysis at 500 °C for 2 h, followed by chemical activation and calcination at the same temperature. The materials were characterized using FTIR, XRD, TGA, and Surface Area Analysis. Results indicate that alkaline activation modifies the biochar surface by increasing O–H, C=O, and C–O functional groups, improving polarity and adsorption capacity. The samples were predominantly amorphous and thermally stable, with minimal mass loss (0.149 mg). Among the activators tested, NaOH 2 M was the most effective, yielding a surface area of 306.233 m^2/g, pore volume of 1.841 cc/g, and pore diameter of 1.202 nm. These properties suggest that NaOH-activated coconut shell biochar is a suitable carrier material for catalytic applications.

Keywords: biochar, coconut shell, alkaline activator, heterogeneous catalyst

1. INTRODUCTION

As an agrarian country, Indonesia has great potential in producing alternative resources in the form of biomass, especially in coconut plants. Indonesia is the largest coconut producing country in the world. Based on data from the Central Statistics Agency (BPS) of the Ministry of Agriculture in 2023, Indonesia has an area of 3,290.30 thousand ha[1] with coconut production in Indonesia of 2,854.30 thousand tons.[2] Along with the high amount of coconut production, the amount of coconut shell waste produced is directly proportional. The coconut shell waste produced is 360 thousand tons per year. Where usually coconut shell waste is only thrown away or burned to be used as fertilizer or briquettes that produce CO_2 and CH_4 emissions that have a negative impact on the environment. Coconut shells if processed using the pyrolysis method have a low ash content, high volatile substances, lignin and cellulose in large quantities, so it has a high calorific value. The energy produced from coconut shell waste biomass has the potential to be a potential renewable and environmentally friendly energy source and has a lower price.[3]

Nowadays, coconut waste biomass is processed into biochar, which is a black, carbon-rich, and highly porous material with a high degree of aromatization and strong anti-decomposition properties. The processing of coconut biomass waste into biochar can be done in several ways such as pyrolysis, gasification, and carbonization. So far, pyrolysis has been chosen to produce biochar because it produces a higher amount of biochar than other methods. Coconut shell biochar has good proximate and ultimate characters and surface functional groups that have the potential to be used as catalysts or catalyst supports. However, untreated biochar generally has a limited surface area and surface functionality, so an activation process is required to improve its quality.[4]

Khuenkaeo and Tippayawong, 2020 reported that coconut shell biochar only has a BET surface area of 55.69 m^2/g with a pore diameter of 9.13 nm and a pore volume of 0.25 cm^3/g.[5] Where to use as a catalyst carrier relies heavily on accessibility to the active site and the quantity of the active site to support its catalytic performance. Biochar activation can be done physically or chemically. Chemical activation was chosen because of the higher biochar yield, higher surface area, greater porosity and lower activation temperature. Activation with alkali can increase or develop porosity, pore volume, surface area and fixed carbon by removing any impurities found on the surface of the biochar, such as mineral ash, metals, organic matter and volatile carbon. In addition, alkaline activation can improve the surface properties of biochar with the addition of surface function groups. Alkaline activation results in a positive surface charge which in turn aids in the adsorption of negatively charged species and adds a hydroxyl function group (–OH) to the surface of the biochar which increases the capacity of the biochar as an adsorption material. The most common alkalis used to activate biochar are KOH and NaOH.[4]

In the activation of coconut shell biochar using KOH, the pore structure of the coconut shell biochar improved as the surface area of the BET increased from 97.40 to 486.00 m^2/g and the pore volume increased from 0.115 to 0.236 cm^3/g.[6] Meanwhile, Sujiono, et al., 2020 reported that NaOH-activated coconut shell biochar had a surface area of 516 m^2/g with a pore volume of 0.160 cm^3/g and a pore diameter of 1.79 nm[.7] Research conducted by Zhou et al., 2021 using $Ca(OH)_2$ as an alkaline activator produces good compressive strength, porosity, and pores.[8] Likewise, research conducted by Bian et al., 2021 also used $Ca(OH)_2$ as an alkaline activator to activate slag also showed good results in terms of compressive strength, crystallinity, and pores and was more cost effective.[9] Therefore, this study aims to determine the most effective alkaline activator to activate biochar from coconut shells as a heterogeneous catalyst.

2. EXPERIMENTAL

2.1. Chemicals, Equipment and Instrumentation

The ingredients used in this study were coconut shells from a jenang candy factory in Langkat Regency, North Sumatra, aquades (H_2O), nitrogen gas (N_2), potassium hydroxide (KOH) 99% (Merck), sodium hydroxide (NaOH) 99% (Merck), and calcium hydroxide ($Ca(OH)_2$) 99% (Merck). The tools used in this study are erlenmeyer, beaker, measuring cup, measuring gourd, double-neck flask, analytical balance, oven, pyrolysis reactor, grinding machine, 200 mesh sieve, hot plate, magnetic stirrer, spatula, condenser, statif and clamp, filter paper, Universal pH indicator, nitrogen gas (N_2), Fourier Transform Infrared Spectroscopy (FTIR), X-Ray Diffraction (XRD), Thermogravimetric Analysis (TGA), and Surface Area Analyzer (SAA).

2.2. Research Procedure

2.2.1. Preparation of Coconut Shell Biochar

The coconut shells taken from the jenang candy factory are cleaned from the fibers that stick to the surface of the shell. Then it is cleaned using running water and dried in the sun for 24 hours. The dried coconut shells are chopped and dried again using an oven at 110°C for 12 hours.

2.2.2. Pyrolysis of Coconut Shell

A total of 100 grams of coconut shells were put into the reactor and then dyrolysis at 500°C for 2 hours with nitrogen gas. The pyrolysis results in the form of biochar were then mashed using a grinder machine, then sifted using a 200 mesh sieve and characterized using FTIR, XRD, and SSA.[10]

2.2.3. Activation of Coconut Shell Biochar

A total of 20 grams of coconut shell biochar were activated with alkaline activators (KOH, NaOH, and $Ca(OH)_2$) concentrations of 2M [11] with a ratio of 1:4 (b/v) refluxed at a temperature of 60°C at a speed of 500 rpm for 3 hours. Then neutralized using aquuades to pH 7 and dried using an oven at 110°C for 2 hours. Then the results were characterized using FTIR and TGA instruments.

2.2.4. Calcination of Coconut Shell Biochar

Activated biochar (KOH, NaoH, and $Ca(OH)_2$) is calcined at 550°C for 2 hours with nitrogen gas flow. Then the results were characterized using FTIR, XRD, and SAA.

3. RESULTS AND DISCUSSION

3.1. Function Group Analysis Using FTIR

In the *basic biochar* of coconut shells, vibrations were recorded in bands 3031.13 cm-^{1} to 2924.02 cm^{-1} indicating the presence of $C–H_2$ groups, in bands 1874.71 cm^{-1} indicating the presence of aromatic C=C groups, in high-intensity bands 1583.42 cm^{-1} indicating the presence of C=O, in bands 1368.71 cm^{-1} to 1164.94 cm-^{1} which shows the presence of a C–O group, and in the 610.92 cm^{-1} band showing the C–H group correlated with data [12] which shows that in the *basic biochar* of the coconut shell there are C=C, $C–H_2$, C–H, C=O, and C–O, which can be seen in the following figure 1.

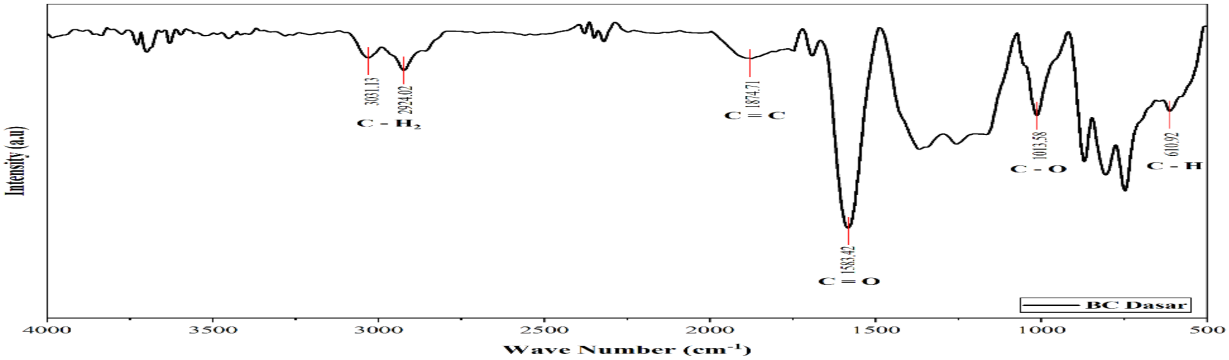


Figure 1. FTIR Spectrum of Coconut Shell Biochar

In the calcined biochar of KOH, absorption was recorded in the band 1586.41 cm^{-1} which indicates the presence of the C=O group, in the band 1117.10 cm^{-1} which indicates the presence of the C–O group, but the presence of the O–H and C–H groups was not recorded which showed calcination in the KOH-activated biochar causing the release of the O–H and C–H functional groups in the biochar which is above the temperature of 600°C groups O–H begins to decompose and produces CO gas on the surface as evidenced by the increase in the C–O group after calcination. With an increase in temperature 400–600°C, the results of the C–O, O–H, C=O, C–O, and –COOH groups decrease significantly because the increase in temperature induces the deoxygenation reaction. The decrease in the C–O group is likely due to the transformation of the C=O group into the C–O group through the hydrogenation reaction at higher temperatures, which also leads to the increase in the C–O group [13]. This is appropriate because according to the reaction that occurs after the biochar is activated will produce potassium carbonate. Potassium hydroxide (KOH) will be reduced to metal K and carbonate K_2CO_3 during the thermal process. The resulting carbonates and alkali metals are intercalcined into a carbon matrix that expands and stabilizes the space between the layers of carbon atoms. Higher temperatures also cause potassium to diffuse into different layers of carbon and form new pores in the carbon. Therefore, additional porosity may be generated by further reactions between potassium carbonates and carbon, which can increase carbon combustion.

In the calcined biochar of NaOH, the uptake was recorded at band 3421.82 cm^{-1} indicating the presence of O–H, at band 1582.33 cm^{-1} indicating the presence of a C=O group, and at band 1162.91 cm^{-1} indicating the presence of a C–O group. Activation using NaOH significantly increased the O–H functional group and the C–O group, although there was a slight decrease after calcination. At 550°C, the yield of the C–O, OH, –O C–O, C–O, and –COOH groups decreased significantly as the increase in temperature prompted the deoxygenation reaction. The survival of the C=O and O–H groups is due to the fact that the NaOH activators are not as aggressive as KOH to attack the O group.

The correlation between the content of the main organic compounds (phenol and aromatic) and the specific surface area of the active biochar catalyst is that the phenol content shows a positive linear correlation with the specific surface area of the active biochar catalyst, while the aromatic content shows a negative linear correlation with the surface area. This may be due to the fact that the porosity that develops on the active biochar catalyst facilitates the diffusion of intermediate pyrolytic compounds into the pores, causing a reaction with the O-active group within the pores. Meanwhile, the large surface area provides more room for reactions between pyrolytic intermediates and oxygen-containing groups, so the reactions that form phenols are increased as the surface area increases, leading to the formation of additional phenols.

A negative linear correlation between aromatic content and surface area may be due to significantly enhanced aromatization and polymerization reactions, as well as some aromatics moving to carbon on active *biochar* catalysts through aromatic polycondensation, leading to a decrease in aromatic content at high surface area. It can be attributed to the high reactive activity of the O-H group contained in the active *biochar* which reacts with the phenol methoxyl to break down the side chain.[14]

In $Ca(OH)_2$ calcined biochar, the uptake that appeared was recorded at the 1585.73 cm^{-1} band indicating the presence of a C=O group, and in the 1054.14 cm^{-1} band indicating the presence of a C–O group. This is because the increase in temperature makes the C–O, O–H, C=OO, C–O, and –COOH groups decrease significantly because the increase in temperature encourages the deoxygenation reaction. The decline of the C–O group is likely caused by the transformation of the C=O group into the C–O group through the hydrogenation reaction at higher temperatures, which also leads to the increase of the C–O group. Based on the three activators used, the difference in the functional group whose vibrations can be seen in figure 2 below.

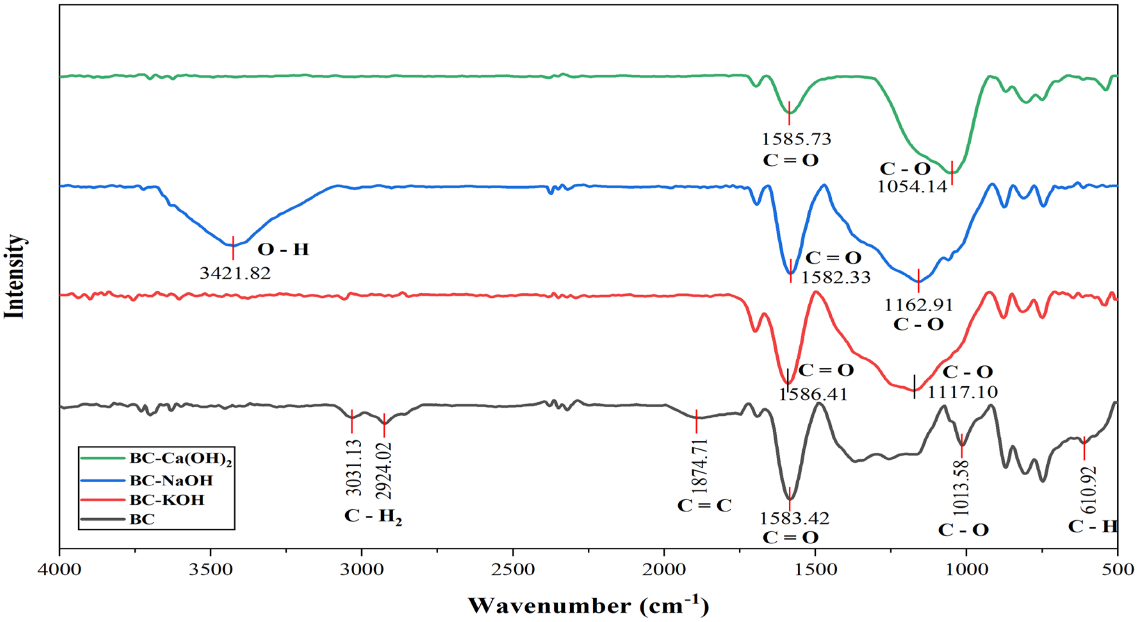


Figure 2. FTIR Spectrum of Biochar Activated with KOH, NaOH, and $Ca(OH)_2$

Based on the FTIR spectrum above, it can be seen that the absorption of the bands on the spectrum shows several functional groups which can be seen in table 1 below.

Table 1. Alkaline Activated *Biochar* FTIR Spectrum Data

Material	**Wave Number (cm^{-1})**	**Function Group**
Biochar	3031.13 – 2924.02	$C–H_2$
	1874.71	C=C
	1583.42	C=O
	1013.58	C–O
	610.92	C–H
BC-KOH	1586.41	C=O
	1117.10	C–O
BC-NaOH	3421.82	O–H
	1582.33	C=O
	1162.91	C–O
BC-$Ca(OH)_2$	1585.73	C=O
	1054.14	C–O

3.2. Crystallinity Analysis Using XRD

Figure 3 shows a comparison of basic biochar diphroctograms, KOH-activated biochar, NaOH-activated biochar, and $Ca(OH)_2$-activated biochar. It can be observed in areas 2θ = 9.1; 23.19, and 43.04° which are the typical peaks of coconut shell biochar.15 As for after being activated with KOH, it is marked to appear at the peak of regions 2θ = 9.5; 23.4, and 42.51°. After being activated with NaOH, peaks appear in regions 2θ = 7.42; 22.48, and 44.2°. After activation with $Ca(OH)_2$, peaks appear in regions 2θ = 8.21; 21.94, 26.52, and 41.5°. In addition, there is a shift in the main peak that is not too significant after activation. This is because thermal treatment includes oxidation and calcination. The presence of a widened peak can be seen in figure 3 which proves that the resulting carrier is amorphous in line with 7 and 6 which produce alkaline-activated biochar which is amorphus. And the widened shape of the crest as shown in figure 3 shows the shape of the crest amorphus according to the study.[16]

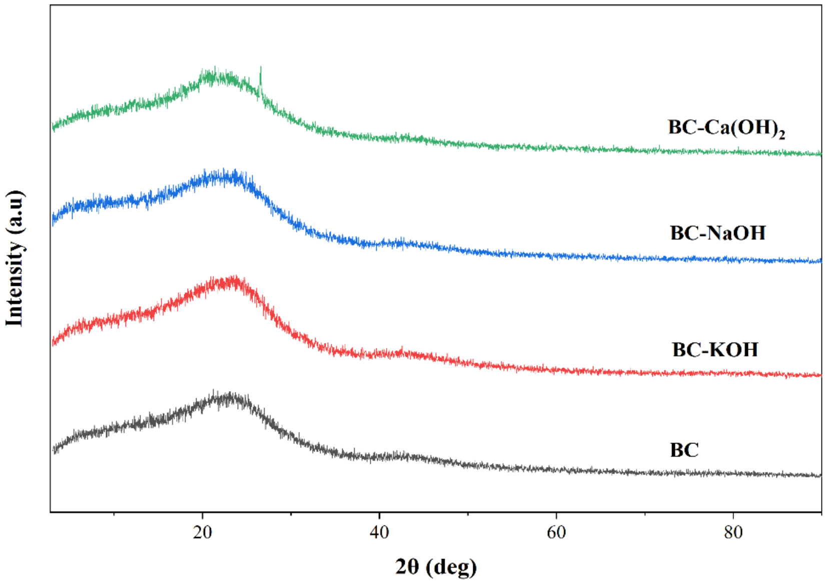


Figure 3. Comparison of Basic Biochar Diphroctograms, BC-KOH, BC-NaOH, and BC-$Ca(OH)_2$

3.3. Analisis Dekomposisi Material Menggunakan TGA

TGA analysis was performed to identify the characteristics and patterns of sample mass reduction as a function of temperature changes. The resulting curve interprets the percentage decrease in sample weight (wt%) against temperature rise (T). As the temperature increases, its weight decreases due to the loss of mass, due to thermal decomposition of the biomass. Where the decomposition of materials in alkaline activated biochar can be seen in figure 4 below.

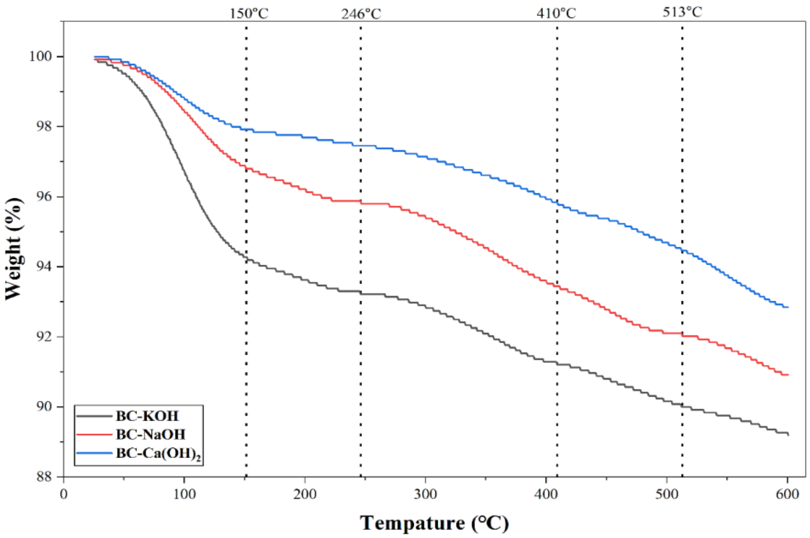


Figure 4. TGA Biochar Activated Graph KOH, NaOH, and $Ca(OH)_2$

Where the decomposition of the three activators used showed that KOH-activated biochar experienced the largest decrease with weight loss of 10.65% or equivalent to 1,018 mg, followed by NaOH-activated biochar of 8.94% or equivalent to 1,077 mg, and $Ca(OH)_2$ activated biochar of 7.13% or equivalent to 0.928 mg. The weight loss in alkaline activated biochar can be seen in the following table.

Table 2. Alkaline Activated *Biochar* Decomposition

Material	**Suhu (℃)**	**Penurunan Berat (%)**
BC-KOH	0-266	6.77
	267-417	2.05
	418-600	2.02
BC-NaOH	0-248	4.11
	249-500	3.79
	501-600	1.22
BC-$Ca(OH)_2$	0-240	2.17
	241-446	2.37
	447-600	2.59

3.4. Analisis Luas Permukaa, Volume Pori, dan Diameter Pori Menggunakan SAA

Further analysis was carried out using *the Surface Area Analyzer* (SSA) to determine the effect of the activator on the characteristics of the catalyst including surface area, total volume and average pore diameter. The surface area was calculated using the BET method, while the pore volume and average pore diameter were analyzed from the desorption band using the BJH method. The results of measuring surface area, pore volume and average pore diameter of each catalyst are summarized in table 3 below.

Table 3. Surface Area, Pore Volume, and Pore Diameter of the Material

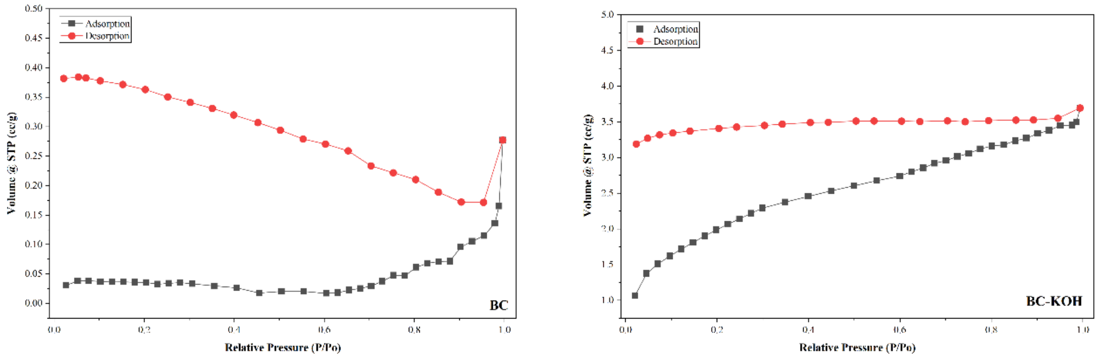
Material	Surface Area (m^2/g)	Pore Volume (cc/g)	Pore Diameter (nm)
BC	4.025	1.744	8.666
BC-KOH	148.351	1.166	1.572
BC-NaOH	306.233	1.841	1.202
BC-$Ca(OH)_2$	204.293	1.600	1.566

The data in table 3 show that the basic biochar of coconut shells has a limited surface area, which is only 4,025 m^2/g with a pore volume of 1,744 cc/g, and a pore diameter of 8,666 nm which indicates that the basic biochar of coconut shells is still dominated by macropores. This condition is caused by the presence of tar and volatile compounds that clog the surface of the coconut shell biochar.

KOH-activated biochar has experienced a significant increase where there is a 37-fold increase from basic biochar., namely from 4,025 m^2/g to 148,351 m^2/g. Although according to [6] coconut shell biochar activated using KOH has a surface area from 97.40 m^2/g to 486.00 m^2/g with a pore volume of 0.115 cc/g to 0.236 cc/g. There is only a 5fold increase in the surface area. Although it only has a surface area of 148,351 m^2/g, KOH-activated biochar has a pore volume of 1,166 and a pore diameter of 1,572 nm which is included in the micropore which is good for catalytic activity.

NaOH-activated biochar experienced the largest increase where there was a 76fold increase. Although based on [7] biochar coconut shells activated using NaOH have a surface area of 516 m^2/g with a pore volume of 0.160 cc/g to 1.79 nm. This is quite good because NaOH activated biochar has a surface area of 306,233 m^2/g and its pore volume increases to 1,841 cc/g with the pore diameter decreasing to 1,202 nm which is excellent at increasing catalytic activity.

$Ca(OH)_2$ activated biochar has increased 57 times with a surface area of 204,293 m^2/g. This increase is quite good where the activation of coconut shell biochar using $Ca(OH)_2$ has not been widely done. With a surface area of 204,293 m^2/g with a pore volume of 1,600 cc/g, and a pore diameter of 1,566 nm, it is quite promising in increasing catalytic activity. From the results of the SSA (Surface Area Analyzer) instrument, data was obtained in the form of an isothermal adsorption-desorption graph of each catalyst shown in the following figure 5.



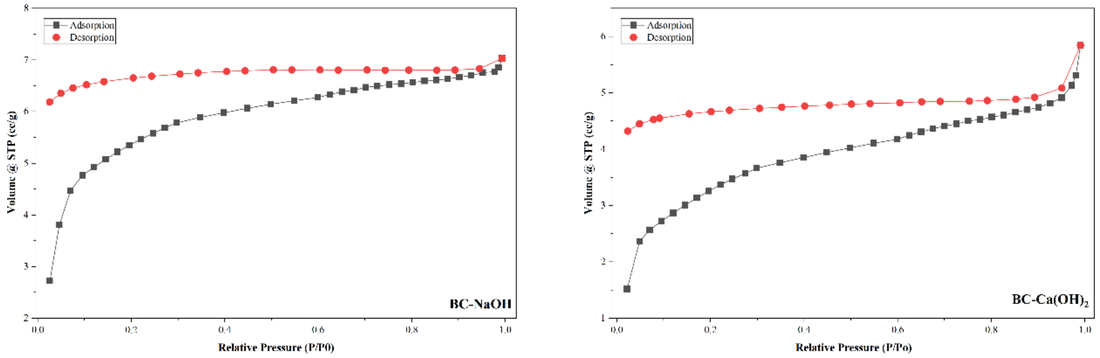


Figure 5. Comparison of N_2 Adsorption-Desorption Isotherm Graphs of BC, BC-KOH, BC-NaOH, and BC-$Ca(OH)_2$

The graph shows isothermal adsorption-desorption data measured using nitrogen at a temperature of 77.3K. Adsorption isotherm is used to evaluate gases adsorbed at different relative pressures (P/Po), where P is the vapor gas pressure, and Po is the adsorbent saturated pressure. In the basic biochar, the isothermal curve model shows a type III isotherm which indicates that the type of adsorbent quantity increases as the relative pressure increases. The absence of an arc point like in the second type is due to the stronger adsorbate-adsorbent interaction than adsorbents. Just like type II, the number of layers on the adsorbent surface is unlimited (multilayer).

Based on comparative isotherm adsorption data from the three activators shows different volume values with quite similar curve models. The adsorption isotherm curve in KOH, NaOH, and Ca(OH)2 activated biochar exhibits type II isotherms showing a linear relationship between adsorption and relative pressure at medium pressure ratios. Type II is a normal form of isothermal in non-porous adsorbents or large porous solids (macropore) with a size greater than 50 nm that exhibits monolayer–multilayer adsorption. Where the maximum number of adsorption is consecutively 3.6949; 7.033; 5.8435 cc/g with a relative pressure of 0.9930; 0.99369; and 0.99048 correlated with Sujiono et al., 2020.[15]

4. CONCLUSION

Based on the research that has been conducted, it can be concluded that different types of alkaline activators affect the physicochemical characteristics of biochar from coconut shells by modifying the surface of biochar by multiplying the O–H, C=O, and C–O groups which can increase polar properties and adsorption, materials that are amorphous, and thermally stable with a weight loss range of 0.149 mg, and the most effective captivator used to activate biochar from coconut shells judging from its surface character is Sodium Hydroxide (NaOH) 2M which produces a surface area of 306,233 m2/g with a pore volume of 1,841 cc/g and a pore diameter of 1,202 nm.

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