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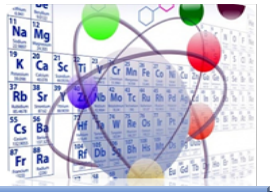

The Effect of Acid Activator Type on Character Biochar from Coconut Shell as a Heterogeneous Catalyst Carrier

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ABSTRACT

This study examines the effect of acid activators (H_3PO_4, H_2SO_4, and HCl) on the characteristics of coconut shell biochar as a heterogeneous catalyst carrier. Biochar was produced through pyrolysis at 500°C, 2M acid activation at 60°C, and calcination at 550°C, then characterized using TGA, FTIR, XRD, and BET. Acid activation increased the surface area from 4.025 m^2/g to 369.335 m^2/g (HCl), 359.557 m^2/g (H_3PO_4), and 324.938 m^2/g (H_2SO_4). FTIR showed modification of P-O, S=O, and hydroxyl-carbonyl functional groups. The crystal structure remained amorphous with HCl producing the highest intensity. HCl gave the best results with a surface area of 369.335 m^2/g, a micropore area of 631.7 m^2/g, and superior thermal stability (mass loss of 0.587 mg), indicating potential as a heterogeneous catalyst carrier.

Keywords: biochar, coconut shell, acid activation, heterogeneous catalyst, surface area

1. INTRODUCTION

Indonesia, as a country with abundant biomass resources, has great potential to develop innovative solutions in converting biomass into high value-added materials.[1] Based on data from the Central Statistics Agency (BPS), coconut plantations in Indonesia cover 3,417,951 hectares, with an annual production of 3 million tons, resulting in approximately 360,000 tons of coconut shell waste annually. With a chemical composition similar to wood, consisting of lignin, hemicellulose, and cellulose, coconut shells can be used to make various products such as crafts, fuel, and briquettes.[2] One alternative to address and increase the economic value of coconut shell waste is to convert it into alternative fuel. Techniques frequently used to convert coconut shells include pyrolysis, gasification, torreficiation, and carbonization.[3]

Pyrolysis is a thermochemical conversion method and can handle a variety of biomass-based materials. Pyrolysis is a thermochemical process that thermally degrades biomass at high temperatures into biochar, bio-oil, and syngas at different temperatures, heating rates, retention times, and inert gas flow rates. In the first stage, the complex molecular bonds of lignin, cellulose, and hemicellulose in biomass undergo cleavage to form carboxyl, carbonyl, and hydroxyl groups on the surface biochar. In the second stage, by continuously applying heat energy, larger molecules or heavy

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compounds of biomass undergo several chemical reactions to produce biochar, bio-oil, and syngas such as methane (CH_4), hydrogen (H_2), carbon monoxide (CO) and carbon dioxide (CO_2).[4] Over the past few decades biochar has attracted global attention due to its high potential for use in various fields such as wastewater treatment, water purification, CO capture$_2$, electricity generation, energy storage, and soil improvement to increase soil fertility and carbon sequestration, the building sector, to chemical catalysis. Biochar is a carbon material produced through the pyrolysis process of biomass under controlled conditions.

Biochar which is carbonized above the degradation temperature of cellulose, lignin and hemicellulose, namely 400 ^oC and 500 ^oC shows pores with an average diameter of 1.7 nm with pore type I which is a monolayer adsorption type. Biochar carbonization results at 500°C shows a fairly high pore surface area, namely 32.565 m^2/g compared to the carbonization results at 400°C which has a pore surface area of 3.573 m^2/g. Optimizing pore structure and surface area, increasing surface reactivity, and developing efficient modification methods are the main focuses of ongoing research in this field. Acid activation has emerged as a promising approach to address these challenges. The activation process aims to remove impurities from carbon after biomass carbonization; therefore, the surface area is increased and the chemical functional groups are incorporated.[5]The development of high-performance heterogeneous catalysts is a major focus of catalysis research. Surface modification of carrier materials by adding new functional groups using acid activators such as sulfuric acid, phosphoric acid, and hydrochloric acid has been shown to significantly improve catalytic performance.[6]

Based on this background, the purpose of this study was to determine the effect of acid activators as carriers for heterogeneous catalysts. The innovation in this study lies in the type of acid activator used by comparing the best activators in catalyst carriers, where the activator used in the study is H_2SO_4, H_3SO_4, and 2M HCl with a ratio of 1:4 (w/v) between biochar activator at a temperature of 60 ^oC at 500 rpm for 3 hours. Characterization was then performed to determine functional group analysis, crystal structure, specific surface area, pore volume, and pore size distribution.

2. EXPERIMENTAL

2.1. Chemicals, Equipment and Instrumentation

Materials: The materials used in this study were coconut shells, distilled water, Phosphoric Acid (H_3PO_4) 85%, Hydrochloric acid (HCl) 37%, Sulfuric acid (H_2SO_4) 98%, filter paper, pH indicator, and nitrogen gas. Tools: The tools used are pyrolysis reactor, chemical glassware, 2-neck flask, condenser, mortar and pestle, 200 mesh sieve, spatula, analytical balance, magnetic stirrer, whatman filter paper, stirring rod, watch glass, glass funnel, stand and clamp, aluminum foil, oven, electric grinder, hotplate,*Fourier Transform Infra-Red* (FTIR), *X-Ray diffraction* (XRD), *Brunauer-Emmet-Teller* (BET), *Thermogravimetric Analysis* (TGA)

2.2. Research Procedure

2.2.1. Material Preparation

The coconut shells are first dried in the sun. Then, the dried coconut shells are cleaned of fibers and remaining coconut flesh. The cleaned coconut shells are then shredded and dried in an oven at 110°C for 12 hours.

2.2.2. Coconut Shell Pyrolysis

Next, the pyrolysis process is carried out by putting 100 grams of coconut shell into the reactor and then pyrolyzing it at a temperature of 500 ^oC for 2 hours with nitrogen gas flowing through it.*Biochar*The pyrolysis products were then ground using a grinder and sieved using a 200-mesh sieve. They were then characterized using FTIR, XRD, and BET.

2.2.3. Biochar Activation with Phosphoric Acid (H_3PO_4)

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20 gram biochar pyrolysis results are added with H_3PO_4 2M with a ratio of 1:4 (w/v) in a bottom flask. Reflux for 3 hours at a temperature of 60 °C at a speed of 500 rpm. After the reflux process is complete, let the mixture cool and then filter the mixture using filter paper to separate the biochar which has been activated, washed biochar which has been activated using distilled water until neutral (pH 7) and dried at 110°C using an oven for 2 hours and then characterized by TGA to determine its thermal stability.

2.2.4. Biochar Activation with Sulfuric Acid (H_2SO_4)

20 gram biochar pyrolysis results are added with H_2SO_4 2M with a ratio of 1:4 (w/v) in a bottom flask. Reflux for 3 hours at a temperature of 60 °C at a speed of 500 rpm. After the reflux process is complete, let the mixture cool and then filter the mixture using filter paper to separate the biochar which has been activated, washed biochar which has been activated using distilled water until neutral (pH 7) and dried at 110°C using an oven for 2 hours and then characterized by TGA to determine the stability

2.2.5. Biochar Activation with Hydrochloric Acid (HCl)

20 gram biochar The pyrolysis results were added with 2M HCl with a ratio of 1:4 (w/v) in a bottom flask. Refluxed for 3 hours at a temperature of 60 °C at a speed of 500 rpm. After the reflux process is complete, let the mixture cool and then filter the mixture using filter paper to separate the biochar which has been activated, washed biochar which has been activated using distilled water until neutral (pH 7) and dried at 110°C using an oven for 2 hours and then characterized by TGA to determine the stability

2.2.6. Biochar Calcination

As much as 20 grams biochar activated calcined at a temperature of 550 °C with nitrogen gas for 2 hours. Then characterized by FTIR, XRD, and BET.

3. RESULTS AND DISCUSSION

3.1. Preparation of Coconut Shells into Biochar and Activation Process

The coconut shells were dried in the sun, cleaned, chopped, and then oven-dried at 110°C for 12 hours to reduce the water content. The samples were then pyrolyzed at 500°C for 2 hours to produce *biochar*. *Biochar* ground with a grinder and sieved through 200 mesh to increase the adsorption surface area. Biochar activated using three types of acids with a concentration of 2M (H_3PO_4, H_2SO_4, HCl) with a ratio of 1:4 (w/v) through a reflux method at 60°C for 3 hours with stirring at 500 rpm. Acid activation aims to increase porosity biochar. After reflux biochar filtered, washed with distilled water until the pH is neutral, then dried at 110°C for 2 hours. Biochar The carbon was analyzed by TGA to determine thermal stability, then calcined at 550°C. This temperature is optimal for removing volatile compounds and tar, developing a microporous structure, and increasing thermal stability without damaging the carbon structure. Characterization was carried out using FTIR to identify functional groups, XRD for crystal structure and amorphous phase analysis, and BET to measure surface area, pore volume, and pore distribution biochar.

3.2. Thermogravimetric Analysis (TGA) Characterization Results

TGA (Thermogravimetric Analysis) This test is carried out to determine the thermal stability and degradation characteristics of the material. Thermal stability is a very important parameter for processing and use biochar Coconut shell. Calcination process biochar requires heating at high temperatures, so the degradation effects on the carbon structure can produce undesirable results such as pore damage and reduced surface area. The results of the TGA-DTA analysis of activated biochar can be seen in Figure 1.

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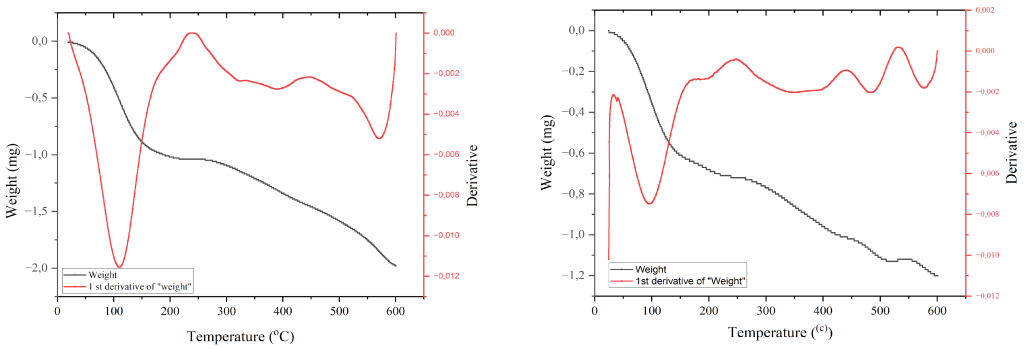
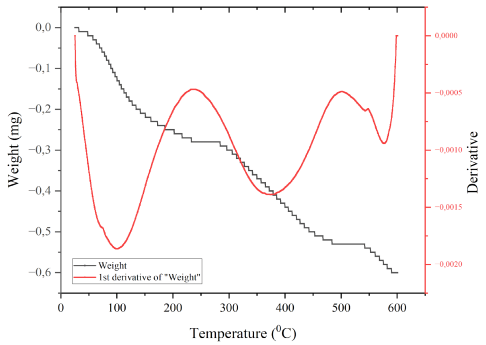


(a) (b)



(c)

Figure 1. TGA-DTA analysis, (a) Activated Biochar H_3PO_4, (b) Activated Biochar H_2SO_4, (c) HCl Activated Biochar

TGA-DTA analysis showed differences in the thermal stability of activated biochars. All samples underwent three stages of degradation: (1) dehydration at 50-150°C (release of bound water and hydroxyl groups), (2) decomposition of oxygen functional groups at 200-400°C, and (3) degradation of the carbon structure at temperatures above 400°C. The H_3PO_4 activated biochar Figure 1(a) showed a total mass loss of 1.854 mg, the highest among all samples. A sharp DTA peak at 100°C indicated intensive dehydration of hydroxyl groups and bound water. Continued degradation up to 600°C with exothermic peaks at 300°C and 500°C indicated gradual decomposition of phosphate groups (P-O) and modified carbon structure. The low thermal stability was due to the labile phosphate groups and the easily decomposed C-O-P bonds at high temperatures.[7]

The H_2SO_4-activated biochar in Figure 1(b) experienced a mass loss of 1.194 mg with a more gradual degradation pattern. The strong endothermic peak at 100°C indicates the release of water and hydroxyl groups. The TGA curve shows a gradual weight loss at 200-500°C, indicating the decomposition of sulfonate (S=O) groups and oxidation of the carbon structure.[8] The exothermic peak at 400°C indicates carbon oxidation catalyzed by residual sulfuric acid groups. Moderate thermal stability indicates that sulfonic groups provide better stability than phosphate but still lower than HCl. HCl-activated biochar Figure 1(c) shows superior thermal stability with the lowest mass loss (0.587 mg).[6] A small endothermic peak at 100°C indicates minimal dehydration. The TGA curve shows a very gentle and gradual weight loss up to 600°C

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without a sharp degradation peak. Weak exothermic peaks at 200°C and 500°C indicate limited decomposition of oxygen groups and minimal carbon oxidation. The high stability is due to HCl not leaving labile groups in the biochar structure, but rather producing a stable aromatic carbon structure with more thermally resistant hydroxyl-carbonyl groups.[9]

3.3. Fourier Transform Infra-Red (FTIR) Characterization Results

Characterization of functional groups in biochar activated and calcined can be done through analysis Fourier Transform Infrared (FTIR), is an infrared spectroscopy technique that combines Fourier transforms for spectrum interpretation. FTIR spectral data from characterization results biochar acid-activated and calcined can be seen in Figure 2 and Figure 3.

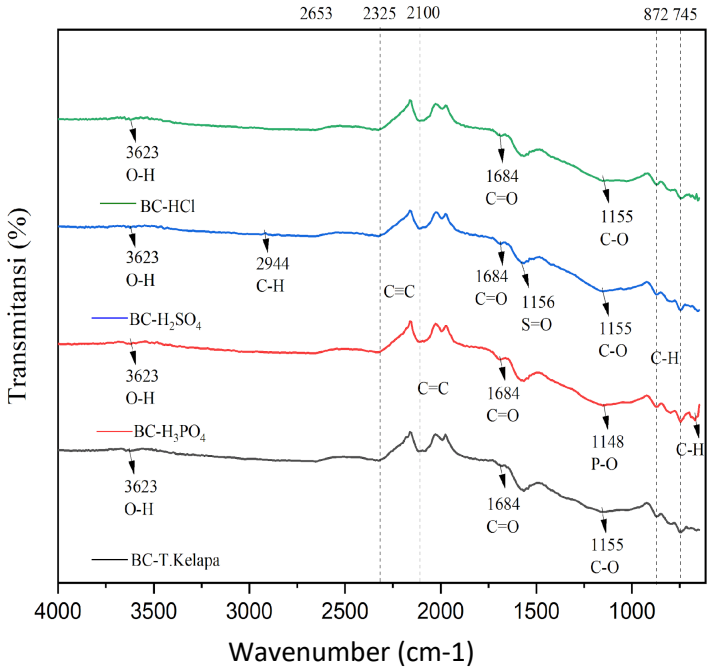

Figure 2. Infrared Spectroscopy (FTIR) Analysis Biochar Before and After Acid Activation (H_3PO_4, H_2SO_4, HCl)

The FTIR spectrum of Figure 2 shows the differences in the characteristics of functional groups in biochar before and after chemical activation. The broad absorption band at 3623 cm^{-1} indicates the stretching vibration of hydroxyl (-OH) groups from bound water, phenolic hydroxyls, and carboxylic groups. BC-HCl shows a sharper peak, while BC-H_2SO_4 and BC-H_3PO_4 have broader peaks with varying intensities, indicating the formation of more diverse -OH groups through oxidation reactions during activation.[10] The absorption band at 2944 cm^{-1} specific to BC-H_2SO_4 indicates aliphatic C-H groups, indicating partial cleavage of the aromatic structure. The band in the range of 2100-2325 cm^{-1} indicates aromatic C=C groups as an indicator of the degree of carbonization. Unactivated biochar shows the highest intensity (the most stable aromatic structure), while BC-H_3PO_4 shows the lowest intensity because H_3PO_4 breaks the aromatic structure to form C-O-P cross-links. The strong peak at 1684 cm^{-1} indicates the C=O group of the carbonyl which plays a role in the formation of acid sites. BC-H_3PO_4 and BC-HCl show the highest intensity, indicating a high surface oxygen content. The absorption band at 1155 cm^{-1} indicates the C-O group of ethers, alcohols, and esters.[11,12] BC-H_2SO_4 shows a shift to 1156 cm^{-1} indicating the formation of sulfonate groups (-SO_3H) as strong acid sites, while BC-H_3PO_4 displays a peak at 1148 cm^{-1} indicating the P-O group of phosphate through C-O-P bonds. BC-HCl does not show new groups from the

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activator, but has a significant increase in intensity at the oxygen group, indicating its main function in opening pores and dissolving minerals.[13]

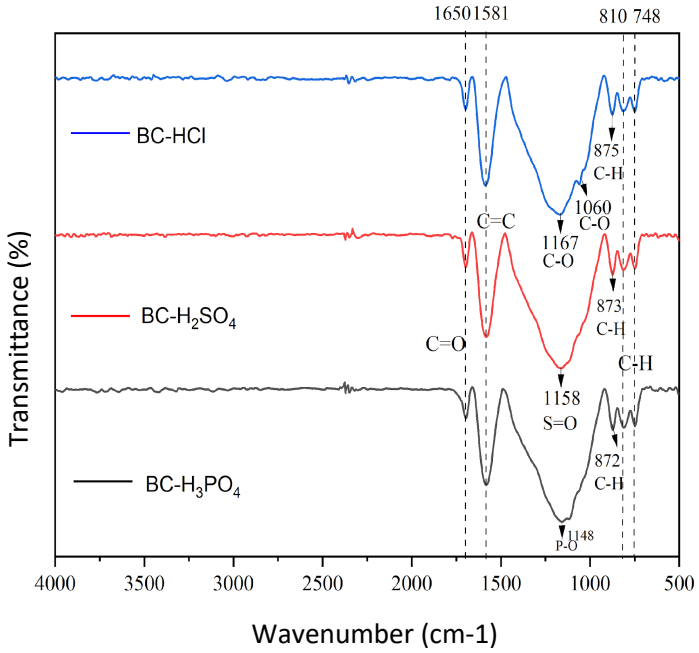

Figure 3. Infrared Spectroscopy (FTIR) Analysis of Acid-Activated Biochar (H_3PO_4, H_2SO_4, HCl) Which Has Been Calcined at a Temperature of 550°C

The FTIR spectrum of Figure 3 shows significant structural transformations in activated biochar after calcination at 550°C. The disappearance of the broad absorption band at 3200-3600 cm^{-1} indicates the thermal decomposition of hydroxyl (-OH) groups through dehydration and condensation reactions, resulting in a more consolidated and thermally stable carbon structure. The disappearance of the peak in the 2000-2500 cm^{-1} region indicates the transformation of the aromatic structure into a more ordered graphitic structure. Strong peaks at 1650 and 1581 cm^{-1} are important characteristics of all calcined samples. The 1650 cm^{-1} peak indicates the C=O vibration of the aromatic carbonyl group or quinone, while the 1581 cm^{-1} peak indicates the C=C vibration of the aromatic or graphitic structure. The shift of the C=O peak from 1684 cm^{-1} to 1650 cm^{-1} indicates the transformation of the aliphatic carbonyl group into a conjugated aromatic carbonyl.[14]

The absorption bands in the 1000-1200 cm^{-1} region show specific differences between the samples. BC-HCl displays a peak at 1060 cm^{-1} (C-O vibration of ether/epoxy groups) formed by the condensation of hydroxyl groups at high temperatures. BC-H_2SO_4 displays a peak at 1158 cm^{-1} (S=O vibration), indicating the sulfonic group (-SO_3H) is retained after calcination. BC-H_3PO_4 displays a broad and intense peak at 1148 cm^{-1} (P-O vibration), confirming the excellent thermal stability of the phosphate group at 550°C. The peaks at 810 and 748 cm^{-1} indicate the out-of-plane aromatic C-H vibration or skeletal vibration of the condensed aromatic rings, confirming the occurrence of extensive aromatic condensation. Overall, calcination at 550°C transformed biochar from a material with diverse aliphatic oxygen functional groups into a more stable aromatic carbon structure with aromatic carbonyl groups (quinones), ethers, and specific groups from activators (sulfonates and phosphates).

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3.4. Characterization Results*X-Ray diffraction* (XRD)

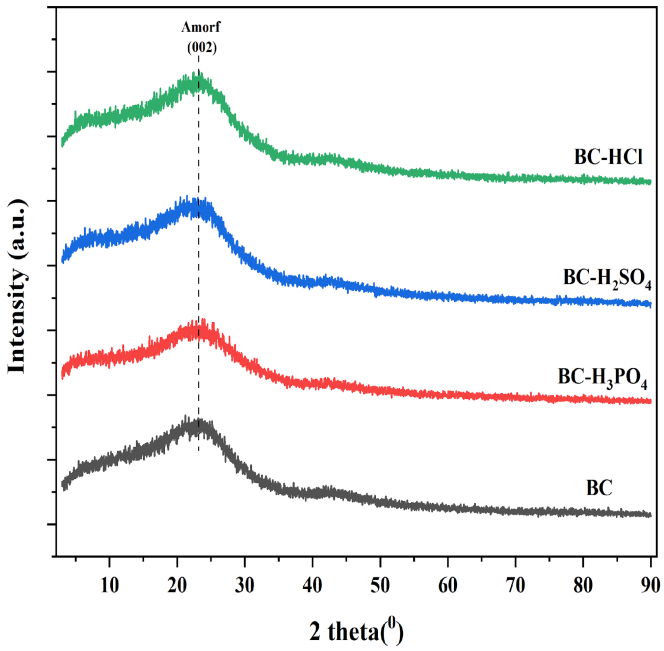

Figure 4. XRD diffraction Biochar Coconut shell BiocharActivated H_3PO_4, Biochar Activated H_2SO_4 And Biochar HCl Activated

The XRD diffractogram of Figure 4 shows broad peaks with high background intensity, indicating a predominantly amorphous carbon structure with low crystal regularity. The main peak at a 2θ angle of about 23° corresponds to the (002) plane of the modified graphite structure. BC-H_3PO_4 shows a peak at 2θ = 23.35° with a significant peak width (FWHM = 14°), indicating a very small crystal size and irregular structure.[15] This is consistent with the activation mechanism of H_3PO_4 as a dehydrating agent that produces a large pore structure by reducing crystal regularity. BC-H_2SO_4 displays a peak at 2θ = 22.71° with higher intensity and sharper peaks than pure biochar. The strong dehydrating ability of H_2SO_4 removes volatile and tar components, and opens a more regular pore structure, although the carbon structure remains predominantly amorphous. BC-HCl shows the highest peak intensity among all samples (1485 cps at 2θ = 23.38°), indicating a carbon structure with a relatively better level of regularity. HCl functions as a cleaning agent that removes mineral impurities and opens the pore structure without causing extensive damage to the carbon structure.[16,17]

3.5 Brunauer-Emmet-Teller (BET) Characterization Results

Characterization using the method Brunauer-Emmett-Teller (BET) is carried out to determine the specific surface area, total pore volume, pore radius, pore size distribution and to determine one of the five isotherm types, namely type I, type II, type III, type IV, type V or type VI isotherms biochar before and after chemical activation. The BET characterization results are presented in Table 4.1 and the N_2 adsorption-desorption isotherms are shown in Figure 5.

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Table 1. Biochar Porosity Characteristics Before and After Activation

Sample	BET Surface Area (m^2/g)	Micropore Size $(m)^2/g$)	Pore Size (nm)
***Biochar*-Coconut Shell**	4,025 m^2/g	6,96 m^2/g	290 nm
***Biochar*-Activated H_3AFTER_4**	359,557 m^2/g	535,5 m^2/g	3,07 nm
***Biochar*-Activated H_2SO_4**	324,938 m^2/g	484,6 m^2/g	3,04 nm
***Biochar*-HCl Activated**	369,335 m^2/g	631,7 m^2/g	3,07 nm

The BET analysis results showed a significant increase in surface area and pore volume after chemical activation. Unactivated coconut shell biochar had a BET surface area of 4.025 m^2/g with a micropore area of 6.96 m^2/g and a pore size of 290 nm, indicating a dominant macropore structure with low porosity. Chemical activation increased the BET surface area where: BC-HCl (369.335 m^2/g), BC-H_2SO_4 (324.938 m^2/g), and BC-H_3PO_4 (359.557 m^2/g), showing an 80-90-fold increase compared to pure biochar. BC-HCl showed the highest BET surface area and micropore area (631.7 m^2/g), indicating the effectiveness of HCl in opening the pore structure by dissolving mineral impurities and tar without damaging the carbon structure. BC-H_2SO_4 has a micropore area of 484.6 m^2/g, indicating the strong dehydration ability of H_2SO_4 in forming pore structures. BC-H_3PO_4 displays a micropore area of 535.5 m^2/g, in accordance with the mechanism of H_3PO_4 as a dehydrating agent and C-O-P cross-linking agent that produces a complex pore network.[18]

The pore size of activated biochar decreased to 3.04-3.07 nm, indicating a transformation from a macroporous structure to a mesoporous-microporous one. The uniform pore size (3 nm) in all three activated samples indicated the formation of a regular and homogeneous pore structure. The dominance of micropores in activated biochar (micropore area reaching 90-95% of the total BET area) is very advantageous for the adsorption applications of small molecules and metal ions.[19].

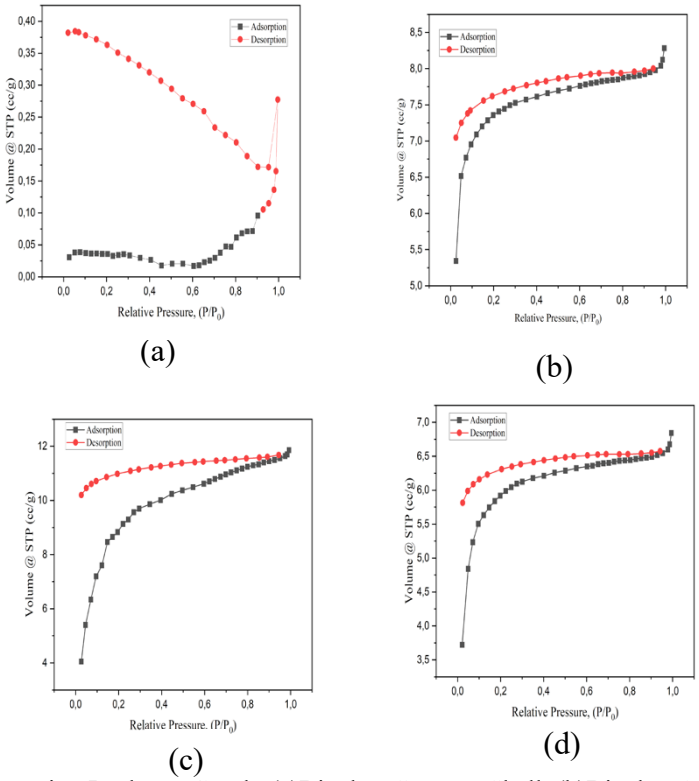

Figure 5. Adsorption-Desorption Isotherm Graph, (a)Biochar Coconut Shell, (b)Biochar Activated H_3PO_4, (c) Biochar Activated H_2SO_4, (d) Biochar HCl Activated

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Nitrogen adsorption-desorption isotherm Figure 5. provides information on the pore characteristics and adsorption mechanism of biochar. Unactivated coconut shell biochar Figure 5a shows a Type III isotherm pattern (IUPAC classification) with a very low adsorption volume (<0.05 cc/g) at $P/P_0 < 0.8$, indicating a weak interaction between nitrogen and the adsorbent surface. A significant increase in adsorption volume occurs at $P/P_0 > 0.9$, indicating multilayer condensation on the external surface or filling of macropores.[17]

Biochar activated with H_3PO_4 Figure 5b biochar activated H_3PO_4 shows the characteristics of Type I or Type I(b) adsorption isotherm, This is identified from the sharp increase in adsorption volume at very low relative pressure ($P/P_0 < 0.1$), which indicates rapid filling of the micropores biochar H_2SO_4-activated isotherm Figure 5c, shows the characteristics of Type IV adsorption isotherm. This is identified from the presence of a clear hysteresis loop between the adsorption and desorption curves, especially in the relative pressure range $P/P_0 = 0.1$-1.0, with a gradual increase at low to medium pressures, followed by an increase approaching $P/P_0 = 1.0$. According to the IUPAC classification, Type IV isotherm is characteristic of mesoporous materials with pore sizes of 2-50 nm, which is in accordance with the pore size data Biochar activated H_2SO_4 of 3.04 nm[20]. BC-HCl Figure 5d shows a Type I isotherm with a sharp increase in adsorption volume at $P/P_0 < 0.1$, indicating rapid filling of micropores, followed by a plateau at $P/P_0 = 0.2$-0.8, and a slight increase at P/P_0 approaching 1.0. A small hysteresis loop between the adsorption-desorption curves indicates a mixed pore structure with micropore dominance, in agreement with the data of pore size of 3.07 nm and very high micropore area (631.7 m^2/g)[4].

4. CONCLUSION

Effect of Acid Activator, Chemical activation increases the surface area of biochar up to 90-fold (4.025 m^2/g to 369.335 m^2/g) with a structural transformation from macropores to micropores-mesopores. Specific functional group modifications are formed: P-O bonds (H_3PO_4), sulfonic groups (H_2SO_4), and hydroxyl-carbonyl groups (HCl), with the structure remaining amorphous. HCl is the most effective activator with the highest surface area (369.335 m^2/g), the largest micropore area (631.7 m^2/g), superior thermal stability (mass loss of 0.587 mg), and a relatively regular structure (intensity of 1485 cps), making it an optimal candidate as a heterogeneous catalyst carrier.

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