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Utilization of Siwalan Waste (Borassus flabellifer L.) as a Prototype of Active Carbon-Based Batteries Using Brunauer Emmett Teller (BET) and Analysis of Variance (ANOVA) Method

Achmad Gufron[1], Husna Zaka Anshori[2], Tivani Nava Arier[3], Izra Noor Zahara Aliya[4], Dwi Mulyati Ningrum[5], Isna Nugraha[6*]

[1,3,6] Department of Industrial Engineering, Faculty of Engineering and Sains
[2]Department of Chemical Engineering, Faculty of Engineering and Sains
[4]Department of Information Systems, Faculty of Computer Science
[5]Department of Environmental Engineering, Faculty of Engineering and Sains
Universitas Pembangunan Nasional Veteran Jawa Timur, Jl. Rungkut Madya, Gunung Anyar Surabaya, Jawa Timur 60294 Indonesia

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A B S T R A C T

Currently, electricity use in Indonesia has soared and continues to increase every year. New renewable energy will be the main alternative for the country as a source of electricity. Alternative energy sources can come from biomass such as Siwalan shell waste. This waste has a composition of 18.52% hemicellulose, 11.90% cellulose, 0.23% lignin, 0.1% nitrogen, 13.80% water, 4.46% ash, 44.58% carbon and 6.41% volatile matter. % which is the chemical component of the basic ingredient for making activated carbon. Thus, the aim of this research is to determine the capacitance of a battery prototype from activated carbon from Siwalan shell waste. The method used is a quantitative method by taking data from the results of the Brunauer Emmett Teller test and the Cyclic Voltammetry test. Carbon is activated using KOH with varying concentrations of 1 M; 1.5 M; 2M; 2.5 M. The results of the surface area of activated carbon in the BET test were 13,636 m2/g; 20,941 m2/g; 28,191 m2/g; and 29,871 m2/g and in manual calculations using the BET method it was obtained 13,647 m2/g; 20,950 m2/g; 28,197 m2/g; and 29,875 m2/g. From testing and manual calculations, Siwalan shell carbon at a KOH concentration of 2.5 M has the largest surface area and has the potential to be the best active carbon material for making battery prototypes.

*Corresponding Author
Isna Nugraha
E-mail: isna.nugraha.ti@upnjatim.ac.id

1. INTRODUCTION

The demand for electricity in Indonesia is continuously increasing in line with population growth. It is estimated that Indonesia's electricity demand will grow by approximately 6.2% per year. Although the primary source of electricity in Indonesia is still dominated by coal-fired power plants, there are concerns about the decline in coal reserves (Yana, 2022). Therefore, renewable energy has become a

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focus in efforts to meet future energy needs. Renewable energy, such as biomass energy, holds promise as an alternative. Biomass, especially plant waste, is an abundant energy source found throughout Indonesia (Radhiana, 2023). Tuban Regency in East Java Province is one of the Indonesian regencies blessed with abundant natural resources. One of these natural resources is the lontar or Siwalan palm tree (Borassus flabellifer Linn.). Siwalan fruit is commonly sold along the roads leading to the city of Tuban. The Siwalan fruit is sold, but the shells are often wasted. Siwalan fruit shells contain various types of polysaccharides, such as cellulose (11.90%), water (13.80%), ash (4.46%), carbon (44.58%), and volatile substances (23.85%) (Ihsan, 2022). With this composition, Siwalan shells can be transformed into activated charcoal.Because of its large pore structure, activated charcoal can absorb a wide range of liquid and gas molecules. From lithium batteries to fuel cell catalysts, activated charcoal finds a wide range of applications (Saputra et al., 2022). The fact that activated charcoal is renewable is one of its main benefits, and coconut shells are starting to emerge as a possible supply.

The process of making activated charcoal involves two main stages, namely carbonization and activation. Carbonization aims to release carbon from biomass components such as cellulose, hemicellulose, and lignin, resulting in a high-calorific value fuel (Handayani and Suryaningsih, 2019). The analysis of the surface area of activated charcoal is important as it influences electrical conductivity (Khairiah, 2019). In this research, the BET method is used to determine the surface area and pore size distribution of activated charcoal produced from Siwalan shells. Additionally, capacitance testing using cyclic voltammetry is performed to evaluate the capacity for storing electrical charge (Elgishi et al., 2018).

The use of Siwalan waste to produce activated charcoal is an innovative step in the effort to generate environmentally friendly electricity. Through BET analysis and capacitance testing, this research can provide valuable insights into the development of alternative energy sources that can reduce dependence on conventional energy sources.

2. LITERATURE REVIEW

Siwalan

"Siwalan" (Borassus flabellifer Linn.) is a type of palm tree that offers various benefits from almost every part of the plant. It thrives in arid regions and is not overly concerned with seasons. Siwalan is distributed in areas including East Java, eastern parts of Central Java, Madura, Bali, West Nusa Tenggara, East Nusa Tenggara, and Sulawesi. The physical characteristics of the Siwalan tree include broad leaves centered at the top of the trunk, resembling a coconut or royal palm tree, with a single trunk that can reach heights of several meters (Aperti, 2018).

People commonly use various parts of the Siwalan tree for various purposes. The fruit flesh is used as food, its flowers are utilized for making sugar, its leaves are used for weaving, the stem can replace rattan, and the trunk of the tree is useful for bridge construction. However, Siwalan shells are seldom utilized and are often considered as waste. Nonetheless, research has revealed that Siwalan shells contain an intriguing chemical composition, including hemicellulose 18.52%, cellulose 11.90%, lignin 0.23%, nitrogen 0.1%, water 13.80%, ash 4.46%, carbon 44.58%, and volatile matter 6.41%. This composition contains potential raw materials for the production of activated charcoal (Wasana, 2022).

Activated carbon

Activated carbon is a form of carbon that has undergone activation using CO2 gas, steam, or certain chemicals, which opens up its pores and enhances its ability to adsorb substances on its surface (Huda, 2022). The production of activated carbon involves two main stages, namely the carbonization process and the activation process. The carbonization process is the stage in which carbon is released from biomass components such as cellulose, hemicellulose, and lignin, with the aim of producing a high-calorific value fuel (Handayani and Suryaningsih, 2019). In other words, activated carbon is essentially regular carbon that has undergone activation treatment using specific chemicals such as KOH, lemon juice, or vinegar. This activation process increases the surface pores of the carbon, which, in turn, enhances its ability to adsorb liquids and gases. Additionally, activated carbon has high electrical conductivity properties. The following presents a comparison of surface area measurements of activated carbon as well as its ability to generate electricity in batteries from previous research.

Table 1. Comparison of results from previous activated carbon measurement studies

Measurement Method	Surface area (μm)	mWatt of battery (mW)	Reference
Multimeter	99,327	11,82	Tumimomor et al., 2018
Multimeter	839,01	13,010	Hutapea et al., 2017
Multimeter	11,42	1,030,336	Pahlevi, Junaidi and Hc, 2020

Battery

A battery, often also referred to as an accumulator, is an electrical cell where electrochemical processes take place that can occur back and forth with high efficiency (Pasaribu and Reza, 2021). A battery consists of a number of separate elements that are then combined in a box made of hard rubber or plastic. Each battery cell consists of basic components that form positive and negative plates. Batteries are typically divided into two types: secondary batteries and primary batteries.

Secondary batteries are rechargeable batteries, such as those used in mobile phones. On the other hand, primary batteries are single-use batteries because the electrode materials in these batteries cannot be reused after they are depleted (Nasution, 2021). A primary battery consists of three main components: a carbon rod as the anode (positive pole of the battery), zinc (Zn) as the cathode (negative pole of the battery), and paste as the electrolyte, which functions as a conductor. Batteries contain various chemical substances, including mercury, manganese, lead, nickel, lithium, and cadmium. These substances are found in secondary batteries, while manganese content is often found in primary batteries (Nasution, 2021).

Brunauer Emmett Teller (BET) Test

The Brunauer Emmett Teller (BET) theory was first discovered in 1938 and provided important insights into the adsorption of gas molecules on solid surfaces. This theory explains that energy is absorbed through dipole induction into non-polar gases, resulting in the formation of bonds between the layers of molecules adsorbed on the solid surface. The BET method has become a commonly used analytical tool to measure the surface area and porous structure of a material. The success of the BET theory lies in its ability to predict the number of adsorbate molecules present on the first layer covering the solid surface, known as monolayer adsorption capacity (Sinha et al.,2019). The BET theory is used after conducting tests with a device called the Surface Area Analyzer (SAA). SAA is used to determine the diameter and volume of pores, as well as the specific surface area of a material. The working principle of SAA involves the mechanism of gas adsorption, typically using gases such as nitrogen, argon, or helium, on the surface of the solid material to be characterized.

Measurements are taken at a constant temperature, usually at the boiling point of the gas used. This instrument essentially measures the amount of gas that can be absorbed by the solid surface at specific pressure and temperature conditions (Putri, 2019). The gas adsorption process in SAA involves the uptake of gas (such as nitrogen, argon, or helium) on the surface of a solid material at a constant temperature. By knowing the volume of gas that can be adsorbed by the solid surface at specific pressure and temperature, as well as the theoretical surface area of one adsorbed gas molecule, the total surface area of the solid can be calculated (Rizki, 2021). This makes the BET method and the SAA instrument essential tools in the characterization of the surface area and pore structure of a material.

3. RESEARCH METHOD

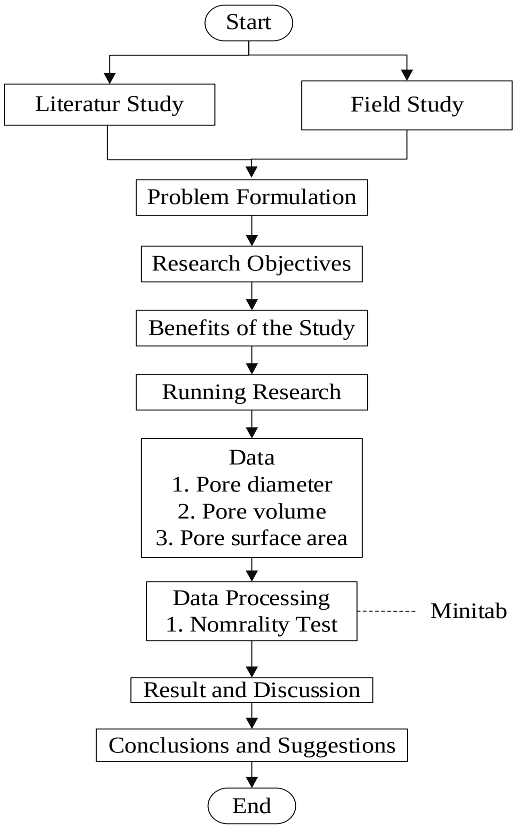

Figure 1. Research flow chart

Data analysis is conducted on the data collected using quantitative methods. The obtained data is presented in the form of tables and graphs to facilitate the analysis process. The analysis carried out in this research is quantitative analysis using the BET (Brunauer Emmett Teller) method. BET testing is performed to determine the specific surface area and pore size distribution of a material (Fitri, 2022). The BET theory can be applied after conducting tests using the Surface Area Analyzer (SAA). This instrument is used to determine pore diameter, volume, and the specific surface area of the material, which is the surface area per unit gram (Kusumaningtyas, 2017). BET testing is conducted using a Quantachrome apparatus, with an initial heating not exceeding the material's specified temperature. The initial heating at a temperature of 450°C results in a significant specific surface area (Fitri, 2022).

Following the BET analysis, cyclic voltammetry testing is carried out to determine the capacitance value with respect to the amount of electric charge stored at an electrical potential, represented as current (I) and voltage (V) (Elgishi, et al., 2018). Once the data is collected, data analysis will be performed using Analysis of Variance (ANOVA). ANOVA is a specialized form of statistical analysis commonly used in experimental research. This analysis method was developed by R.A. Fisher. ANOVA is a statistical hypothesis test where conclusions are drawn based on data or inferential statistical groupings (Marpaung, 2017). In this study, ANOVA is used to determine the impact of variations in the concentration of the KOH activator and the volume of the NaOH electrolyte on the specific surface area and capacitance of Siwalancarbon.

4. RESULT AND DISCUSSION

Analysis of Pores using BET (Brunauer Emmett Teller)

The analysis of the pores in Siwalan carbon includes the measurement of diameter, specific surface area, and pore volume, which are related to testing using the Surface Area Analyzer (SAA). The four samples were tested in powder form with a weight of approximately 0.07 grams at a temperature of 77.35 K for 1 hour using nitrogen gas as the adsorbate. Nitrogen gas, obtained from liquid nitrogen, transitions into the gas phase at room temperature. The principle of the Surface Area Analyzer (SAA) involves degassing and analysis. The characteristics of the pores can then be automatically determined using software (QuantachromeNovaWin-Data Acquisition and Removal) on a computer connected to the instrument. The test results, including the specific surface area, were determined using the BET method on each sample and then compared with the calculated results. The calculation of the specific surface area used the theoretical BET equation. The BET theory is employed because it offers advantages over previous theories, particularly the Langmuir theory, as conceptually, the BET method is an extension of the Langmuir theory. The BET theory can be applied to multiple layers, and physical gas molecules will adsorb onto solids up to an unlimited number of layers (Hwang and Barron, 2011).

Pore Diameter

The size of the pores in a carbon material is related to the ability of the pores to adsorb gases or fluids. The size of the pore diameter also affects the density and porosity of the carbon. A larger pore diameter results in a decrease in material density. The number and size of pores create space for fluids to flow, increasing adsorption capacity. This high adsorption capacity is also reflected in the volume of pores that fluids or gases can pass through. The larger the pore diameter in Siwalan carbon, the greater the pore volume and porosity.

In this study, the average pore diameter of the four Siwalan carbon samples is in the nanoscale range. Table 4.1 shows that the pore diameter value of sample D is the largest compared to samples A, B, and C. Referring to the concentration of KOH, which is 2.5 M, used as the activating agent, it becomes the factor for the formation of large pores in Siwalan carbon during the activation process.

Table 2. Pore diameter values of Siwalan shell carbon

Sample	KOH Concentration (M)	Pore Diameter (nm)
A	1	4,0867
B	1,5	4,0884
C	2	4,0926
D	2,5	4,1037

Based on these results, carbon in Siwalan can be classified as mesoporous with an approximate pore diameter of 4.1 nm. The classification of Siwalan carbon follows the specifications of the International Union of Pure and Applied Chemistry (IUPAC), which categorizes mesoporous materials as those with pore diameters ranging from 2 to 50 nm. The adsorption process yields a value that represents the ability of Siwalan carbon to adsorb nitrogen gas through its pores. Therefore, pore size can be determined through this process.

Specific Surface Area

Surface area refers to the number of pores per unit area of the sample. Specific surface area, on the other hand, is the ratio of the sample's surface area to the relative atomic mass of the adsorbate, where the relative atomic mass of nitrogen is 28.013 g. The desired outcome is the ability of Siwalan carbon to adsorb nitrogen gas. The larger the specific surface area of Siwalan carbon, the more pores are present on the unit surface area of the material. The specific surface area values of samples A, B, and C (Table 2) are lower than the specific surface area value of sample D. This indicates that with a higher concentration of KOH, the specific surface area becomes larger.

Table 3. Specific surface area values from test results and calculations of Siwalan shell carbon

Sample	KOH Concentration (M)	Surface Area Test Results	Calculation Specifications (m^2/g)	Error (%)
A	1	13,636	13,647	0,081
B	1,5	20,941	20,950	0,436
C	2	28,191	28,197	0,029
D	2,5	29,871	29,875	0,013

The specific surface area values obtained from the calculation are determined using the BET method equation. The formula used is as follows:

$$S = \frac{Xm.Lav.Am}{Mv}$$

Information:

S : Total surface area

Xm : Monolayer capacity

Lav : Avogadro's number (6.023 × 10^23 molecules/mol)

Am : Cross-sectional area of the adsorbate

Mv : Molar volume of the ideal gas, which is 22.4 liters/mol.

To find the monolayer capacity itself, the formula is as follows:

$$Xm = \frac{1}{s + i}$$

Information:

s : *Slope*

i : *Intercept*

It can be observed from Table 2 that the specific surface area values from the test results and the calculated values are nearly identical. This is supported by conducting data correction in the form of errors to determine the magnitude of calculation errors in percentage. From the four samples, the average calculation error is 0.205%, with the lowest error found in sample D, which is 0.013%.

The magnitude of the specific surface area also influences the porosity of Siwalan carbon, where a larger carbon surface area results in smaller porosity. This is because an increase in the number of carbon pores makes the spaces between the pores tighter. This pore density reduces the adsorption capacity of carbon in coconut shell. Table 2 shows the specific surface area values of Siwalan carbon. Therefore, Siwalan carbon with a 2.5 M KOH concentration has the potential to be a good adsorbent.

Volume

The amount of space inside each Siwalan carbon is represented by the volume of its pores. This has to do with carbon's capacity to absorb adsorbates that take the form of liquids or gasses, such nitrogen gas. The total amount obtained is rather small, less than 3 ml/g, according to the findings of the SAA test. The carbon from Siwalan sample A actually has the lowest space capacity, at 1.388 ml/g. Sample D has a pore volume of 2.846 ml/g, as shown in Table 3. The difference between this number and the other sample pore volumes is not very different. Due to the very small total pore volume and the micro level gram scale at which pore volume units are measured, the slight discrepancy is caused by this.

Table 4. Total Pore volume of Siwalan shell Carbon

Sample	KOH Concentration (M)	Total Pore Volume (ml/g)
A	1	0,0226
B	1,5	0,0139
C	2	0,0267
D	2,5	0,0285

The Influence of Different Concentrations of KOH Activator on the Surface Area of Siwalan Carbon. In this research, BET (Brunauer Emmett Teller) testing of Siwalan carbon was performed with four variations of KOH activator concentrations: 1 M, 1.5 M, 2 M, and 2.5 M. The impact of the KOH activator concentration can be determined by comparing these concentrations to the results of the BET surface area test. The results of the BET surface area test for Siwalan carbon are presented in the Table 5.

Table 5. Comparison of the effect of KOH activator concentration

Sample	KOH Concentration (M)	Surface Area Test Results	Calculation Specifications (m^2/g)
A	1	13,636	13,647
B	1,5	20,941	20,950
C	2	28,191	28,197
D	2,5	29,871	29,875

The results of the comparison of the effect of KOH activator concentration can be determined by comparing these concentrations to the BET surface area test results. Figure 1 shows the graph of the influence of different concentrations of KOH activator on the surface area of Siwalan carbon.

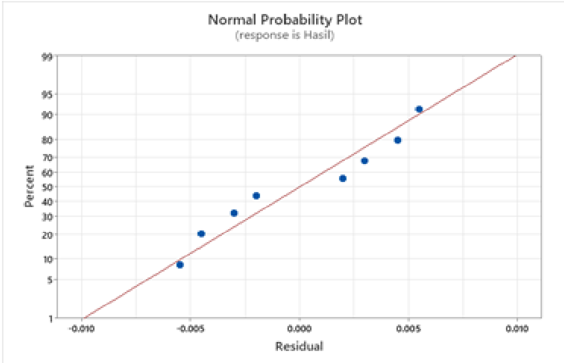

Figure 2. Normal probability plots output of the influence of different koh activator concentrations on the Carbon surface area using Minitab software

Based on the results of the normal probability plots output, it can be observed that the data points are close to the diagonal line which indicates that the residual values of the observed data are normally distributed with a P-Value of 0.1. Thus, the result obtained from Normal Probability Plots is a P-Value of 0.1 > 0.05, then H0 is accepted and the data is considered normally distributed.

Factor Information

Factor	Type	Levels	Values
KOH	Fixed	4	1.5M, 1M, 2.5M, 2M

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
KOH	3	331.823	110.608	3483702.44	0.001
Error	4	0.000	0.000		
Total	7	331.823			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
0.0056347	100.00%	100.00%	100.00%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	23.1635	0.0020	11627.26	0.001	
KOH					
1M	-9.52200	0.00345	-2759.57	0.001	1.50
1.5M	-2.21800	0.00345	-642.80	0.001	1.50
2M	5.03050	0.00345	1457.89	0.001	1.50
2.5M	6.70950	0.00345	1944.48	0.001	1.50

Figure 3. Output of the influence of different KOH activator concentrations on the carbon surface area using Minitab software

Parameters:

a. P-Value > 0.05, then H0 is accepted and H1 is rejected, indicating that the factor has no effect on the surface area of the specimen.

b. P-Value < 0.05, then H0 is rejected and H1 is accepted, indicating that the factor has an effect on the surface area of the specimen.

The graph above explains the use of KOH as an activator in the activation process of activated carbon from Siwalan with different concentrations. The result obtained from the graph is that the KOH concentration factor has a P-Value of 0.001 < 0.05, therefore, H0 is rejected and H1 is accepted, indicating that the factor has an effect on the surface area of the specimen. The higher the concentration of the KOH activator in the activation process, the wider the surface area obtained, resulting in greater adsorption capacity for the electrolyte. According to Waluyo et al., 2017, as the pore size of activated carbon increases, the capacitance becomes larger, and the internal resistance becomes smaller.

5. CONCLUSION

Based on the results of research and discussion, it can be concluded that in BET (Brunauer Emmett Teller) testing, activated carbon from Siwalan activated using KOH with concentration variations of 1 M, 1.5 M, 2 M, and 2.5 M has a surface area of 13.636 m2 /g, 20.941 m2 /g, 28.191 m2 /g, and 29.871 m2 /g, respectively. The results of manual calculation using BET technique for the same concentration fluctuation are 13.647 m2 /g, 20.950 m2 /g, 28.197 m2 /g, and 29.875 m2 /g, respectively. Siwalan carbon at a concentration of 2.5 M KOH has the highest surface area, both based on testing and manual calculations. As a result, sample D, which has a KOH content of 2.5 M, is the optimal material to be used in the manufacture of activated carbon battery prototypes. Further research on the electrical capacitance of this activated carbon is needed to further evaluate its performance and potential as an environmentally friendly solution in meeting electricity demand.

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