The effect of physical activation temperature on physical and electrochemical properties of carbon electrode made from jengkol shell (Pithecellobium jiringa) for supercapacitor application

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The effect of physical activation temperature on physical and electrochemical properties of carbon electrode made from jengkol shell (*Pithecellobium jiringa*) for supercapacitor application

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ABSTRACT

The effect of physical activation temperature in the preparation process of activated carbon electrode for supercapacitor application has been analyzed successfully. The activated carbon made from jengkol shell (Pithecellobium jiringa) with a combination of chemical and physical activation. The physical activation temperature varied of 800 °C, 850 °C, and 900 °C. High specific capacitances of activated carbon electrode as high as 220F g^{-1} was found in physical activation temperature of 850 °C. Additionally, physical properties of activated carbon also evaluated such as density, degree of crystallinity, surface morphology and chemical content. These excellent results show great potential for jengkol shell in developing energy storage devices for supercapacitor application.

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1. Introduction

Biomass is a renewable organic material derived from plants and animals that can function as an energy source. Biomass waste consists of various proportions of hemicellulose, cellulose and lignin called lignocellulose material, and small amounts of simple sugars, proteins, starches and lipids [1]. The previous analysis can be seen that biomass is chemically composed of carbon, hydrogen, oxygen, nitrogen, sulfur and chlorine [2]. This composition varies from one plant species to another. High lignocellulose and carbon have potential as renewable materials for energy sources. One of the uses of lignocellulose is as a source of carbon for supercapacitor electrodes which are reported such as durian shell [3], waste tea [4] and willow catkins [5] and its produce specific capacitance of supercapacitor as high as 103.6F g^{-1}, 203F g^{-1}, and 340F g^{-1}. Jengkol (*Pithecellobium jiringa*) is a food ingredient that is favored by Indonesian people. Jengkol production in Indonesia reaches of 57,404 tons/year and is predicted to continue to increase [6]. The high production of jengkol certainly produces biomass waste such as jengkol shell. Jengkol shell is one of the many wastes in Indonesia that has a high lignocellulose of 56.84% [7]. Jengkol shell contains several active compounds such as alkaloids, flavonoids, antrakinone glycosides, tannins, triterpenoids/steroids, and saponins, besides its also has a high carbon element of 44.02% [7,8] which is believed to be the most important element can be used as a activated carbon, adsorbents, bioherbicides and biolarvasides.

In this study, we convert Jengkol (*Pithecellobium jiringa*) shell waste into activated carbon using effective-cost, simple methods for supercapacitor electrodes as high-performance energy storage devices. One stage integrated pyrolysis both carbonization and physical activation and $ZnCl_2$ impregnation are selected to convert lignocellulose jengkol shell into activated carbon. Electrodes were then fabricated in the coins type with 1 M H2SO4 as aqueous electrolyte. The results show that the activated carbon exhibits nanofiber structures. Furthermore, the different activation temperature successfully enhanced the specific capacitance from 200F/g to 220F/g at a scan rate of 1 mV/s in a two-electrode system. These unique properties enable the jengkol shell to become a high potential for activated carbon electrode materials as supercapacitor energy storage.

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2. Materials and methods

2.1. Preparation of activated carbon electrodes

Jengkol shell (*Pithecellobium jiringa*) is collected from the traditional Pekanbaru market. The initial preparation of activated carbon starts with the process of drying and pre-carbonizing of jengkol shell. Jengkol shell is sun dried for two days. The carbonization process is carried out using an oven at temperature of 250 ℃ with periodic rises temperature. Pre-carbonized samples were converted to powder using mortar and milling to be chemically activated using 0.5 M $ZnCl_2$. Carbon powder of jengkol shell was changed into monolith form using a hydraulic press instrument. The pyrolysis process starts with carbonization in N_2 gas atmosphere for room temperature to 600 ℃ followed by physical activation in CO_2 gas atmosphere [9,10]. The physical activation temperature as main focus of discussion in this study. Activation temperatures used include 800 ℃, 850 ℃, and 900 ℃. Based on differences in physical activation temperatures, all sample electrodes are coded CJS-800, CJS-850, and CJS-900. Supercapacitor cells are arranged based on jengkol shell electrodes in the form of sandwiches consisting of jengkol shell carbon electrodes, separator and current collector [11,12]. In detail, the preparation of activated carbon electrodes shown in Fig. 1.

2.2. Physical and electrochemical chraracterization

Characterization carbon electrodes reviewed in physical and electrochemical properties. Physical characterization such as density analysis based on a standard formula of mass and volume electrode data, degree of crystallinity analysis using the X-ray diffraction method with the Shimadzu 7000 instrument (Integrated Laboratory, UNHAS, source of Cu Kα with λ = 0.154 nm) at the scattering angles (2θ) of 10-60. The layer parameters such as interlayer spacing (d_{002} and d_{100}) was calculated by Bragg's equation ($2d \sin \theta = n \lambda$), layer height ($L_c$) and layer width ($L_a$) were evaluated by using the Debye-Scherer equation ($L_c = 0.90\lambda/\beta \cos \theta_{002}$ and $L_a = 1.94 \lambda/\beta \cos \theta_{100}$). Surface morphology analysis using Scanning Electron Microscopy method at a magnification of 4000 times with the JEOL JSM 6510 LA instrument (Laboratory of SEM-EDS ITB). Chemical composition analysis using the energy dispersive X-ray method with the JEOL-JSM 6510 LA instrument (Laboratory of SEM-EDS ITB). Electrochemical characterization was evaluated using the cyclic voltammetry method with the CV UR Rad-Er 5841 instrument at a relatively low scan rate (Laboratory of materials physics, UNRI). However, the specific capacitance, energy density and power density were calculated using standard equations [13,14].

3. Result discussion

3.1. Physical properties analysis

The density analysis is focused on before and after pyrolysis of carbon electrode made from jengkol shell as shown in Fig. 2(a). The physical activation temperature significantly affects the electrode density. The density of the carbon electrodes shows a decreasing trend from 0.981 g cm^{-3} to 0.61 g cm^{-3}. Higher activation temperatures indicate a relatively lower density. The lowest density was found in the CJS-900 electrode of 0.61 g cm^{-3}. This phenomenon occurs due to the process of physical activation at high temperatures allowing the decomposition process of complex compounds to evaporate completely. Similar analysis was also found in other studies with different biomass materials such as pineapple crown [15] and durian shell [10].

Fig. 2(b) shows the XRD spectrum of all carbon electrodes. The XRD spectrum presents strong wide peaks and several weak sharp peaks. Two wide peaks are found at the 2 theta angle diffraction of 23° and 44° which are identical to low crystallinity between graphite and amorphous carbon [16]. On careful observation, the graphitic peak expansion of carbon electrodes is seem in CJS-800, CJS-900 and CJS-850 which indicate a decrease in electrodes crystallinity [17]. Furthermore, the sharp peaks indicate the presence of other elements with a crystal structure. This discussion is further explained in the chemical composition analysis of electrodes. The layer parameters such as interlayer spacing (d_{002} and d_{100}) was calculated by Bragg's equation, layer height (L_c) and layer width (L_a) were evaluated by using the Debye-Scherer equation based on XRD data, as shown in Table 1. All layer parameters show irregular data on the physical activation temperature. Interlayers spacing are considered normal for amorphous carbon structures from biomass as raw materials. Layer width and layer height indicate inversely proportional data. L_a data decreased consecutively from CJS-800, CJS-900 and CJS-850, indicating good pore properties. This analysis is consistent with the D. Qu, 2002 [18] study which states that small L_a data indicate high surface area and specific capacitance.

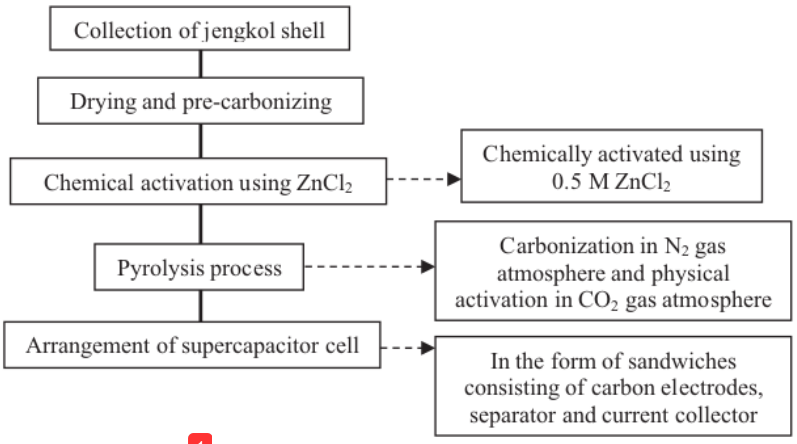

Fig. 1. Schematic diagram for the preparation of activated carbon.

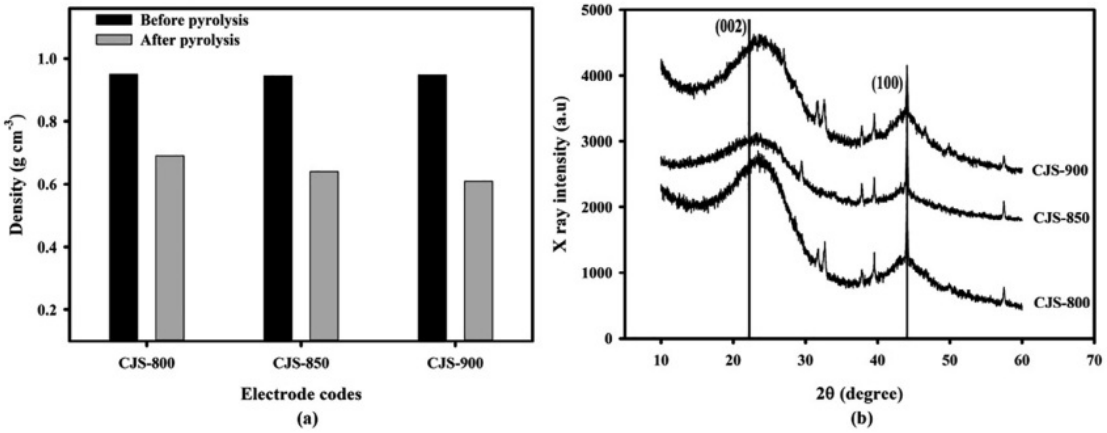

Fig. 2. (a) The density of carbon electrodes for pyrolysis process, (b) XRD pattern for CJS-X electrodes.

Table 1
The layer parameter of carbon electrodes made from jengkol shell.

Electrodes	$2\theta_{002}$	$2\theta_{100}$	d_{002}(Å)	d_{100}(Å)	L_a(Å)	L_c(Å)
CJS-800	23.172	44.198	3.835	1.972	35.668	13.272
CJS-850	23.641	45.144	3.788	2.049	14.775	14.907
CJS-900	23.184	43.447	3.833	2.081	31.584	14.395

Surface morphology analysis of carbon electrodes was reviewed using the scanning electron microscopy method with a high magnification of 40000x as shown in Fig. 3. The SEM micrograph presents the surface morphology in the form of particles and carbon fiber. Carbon fibers experience different reductions in length and diameter at each electrode due to large volume contractions during the carbonization and physical activation processes. Fig. 3(a) shows a SEM micrograph for the CJS-800 electrode. The CJS-800

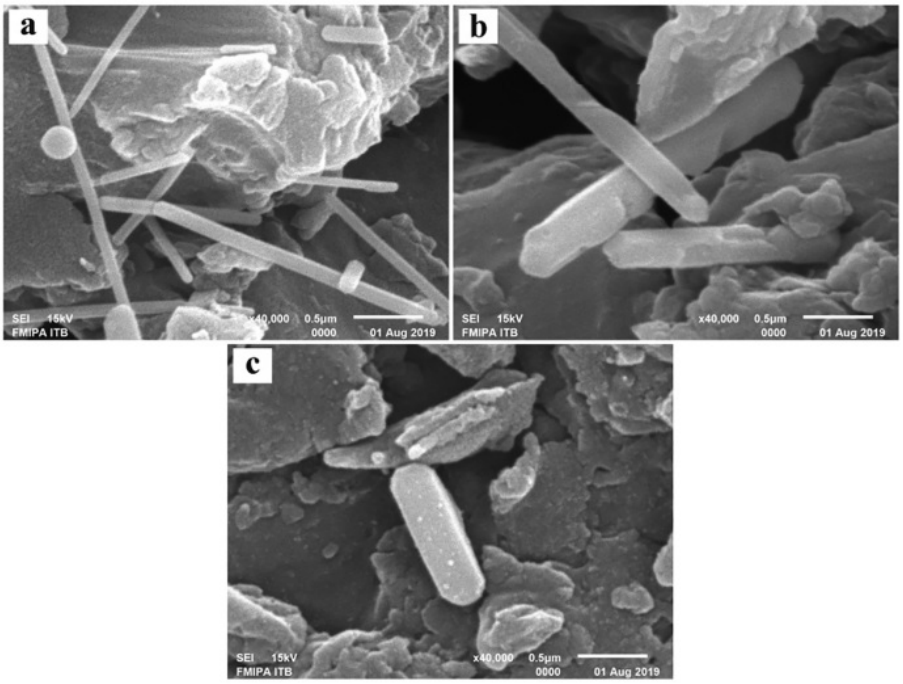

Fig. 3. SEM micrograph with magnification of 40000x for (a) CJS-800, (b) CJS-850, and (c) CJS-900.

electrode can maintain particle structures with carbon scattered evenly without evident damage. The length and diameter fibers were in the range of 710–2090 nm and 64.51–99.33 nm, respectively. Reductions in length, diameter and fiber distribution are evident when the physical activation temperature is greater at the CJS-850 electrode as shown in Fig. 3(b). The fiber length and diameter in the CJS850 in ranges between 490 and 1480 nm and 174.59–318.17 nm. Further addition of physical activation temperature to the CJS-900 electrodes shows a surface morphology with no fiber at all. High physical activation temperatures cause carbon fibers to degrade and turn into chunks of particles. The particle size of the electrodes ranges from 340 to 1040 nm. Several similar studies with different biomass sources also show the surface morphology of carbon fiber such as banana stem [11] and willow catkins [19].

The chemical composition of the electrodes after pyrolysis process is evaluated using the energy dispersive spectroscopy method as shown in Table 2. The chemical elements of the electrode consist of carbon and oxygen which are dominated by carbon. The carbonization and activation treatments in this study can produce extraordinary carbon with a percentage of 98.34–98.56%. The addition of physical activation temperature can increase carbon on the electrodes as shown on the CJS-800 and CJS-850 electrodes. The results of this study are similar to several other studies in different biomass resources such Pitch [20] and Wheat straw [21]. The addition of physical activation temperature further can decrease carbon content of the electrodes such as the CJS-900 electrodes. This phenomenon is caused by higher temperatures damage the arrangement of carbon in the electrodes so that the percentage of carbon has decreased.

3.2. Electrochemical properties analysis

Electrochemical properties of supercapacitor cells such as specific capacitance, energy density and power density were evaluated using the cyclic voltammetry method in the sandwiches form two electrode systems with 1 M H_2SO_4 aqueous electrolyte. As shown in Fig. 4, all CV curves exhibited quasi-rectangular shapes recorded at 1 mV s^{-1}, indicating ideal capacitive behavior for bio-waste materials. The area of the CV curve identifies the specific capacitance of the electrode. The CJS-850 electrode demonstrated the highest CV curve with a specific capacitance of 220F g^{-1}, followed by the CJS-900 and CJS-800 electrodes with specific capacitance of 217F g^{-1} and 202F g^{-1}, respectively. The difference in physical activation temperature affects the capacitive properties

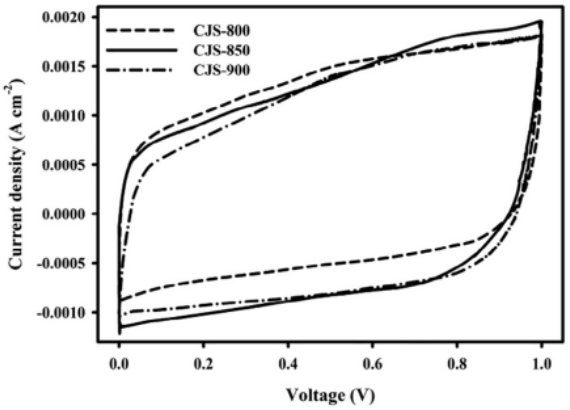

Fig. 4. The CV curve for CJS-800, CJS-850, and CJS-900 electrodes.

Table 2
The element content for all carbon electrodes made from jengkol shell based on EDS characterization.

Elements	Electrode codes		
	CJS-800 (%)	CJS-850 (%)	CJS-900 (%)
Carbon	98.3395	98.5603	98.1604
Oxygent	1.6605	1.4397	1.8396
Totals	100%		

Table 3
The different biomass resources for capasitive properties of carbon electrode supercapacitor.

Biomass	Energy density (Wh kg^{-1})	Power density (W kg^{-1})	References
Coffee bean	15	75	[22]
Dean neem leaves	55	569	[23]
Garlic skin	14.56	310.67	[24]
Gelatin	7.45	263.5	[25]
Lemon peel	6.61	425.26	[26]
Sago bark	5	400	[27]
Jengkol shell	50.56	110.11	This study

of the electrodes. The increasing in activation temperatures from 800 ℃ to 850 ℃ exhibit increasing in specific capacitance from 202F g^{-1} to 220F g^{-1}. However, the addition of a physical activation temperature of 900 ℃ shows a decrease of specific capacitance as high as 217F g^{-1}. This phenomenon is caused by higher temperatures resulting in slightly effected physical properties of the electrodes, as stated in the previous discussion so that affects the capacitive properties. Based on specific capacitance data, we can determine energy density and power density using standard equations [12,13]. The maximum energy density and power density in this study were 50.56 Wh kg^{-1} and 110.11 W kg^{-1}, respectively. These results are similar to several other studies with different biomass sources, as shown in Table 3.

4. Conclusion

In summary, we developed jengkol shell waste as a activated carbon source for supercapacitor electrodes through different physical activation temperatures. Activation temperatures of 800 ℃ 850 ℃ and 900 ℃ produce amorphous structures, in addition, jengkol shell carbon electrodes present fibers evenly on the surface morphology with a dominant carbon element of 98.56%. This good physical properties support the high electrochemical performance of supercapacitor cells. In symmetric supercapacitors, jengkol shell carbon electrodes provides good performance of specific capacitance, energy density and power density as high as 220F g^{-1}, 50.56 Wh kg^{-1} and 110.11 W kg^{-1}, respectively was found at CJS-850 electrode at the relative low scan rate in 1 M H_2SO_4 aqueous electrolytes. Therefore, jengkol shell waste is very promising as a carbon electrodes source for supercapacitor-based energy storage devices through cost-effective and easy methods with high energy and power densities.

CRediT authorship contribution statement

E. Taer: Conceptualization, Methodology, Software. **Apriwandi:** Data curation, Writing - original draft, Writing - review & editing. **R. Taslim:** Visualization, Investigation. **Agustino:** Project administration.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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