

Optimizing the Eco-Friendly Electrode Analysis from *Theobroma cacao* Peels Activated Carbon

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Abstract: An investigation has been successfully performed on cocoa pods activated carbon as a fundamental component for electrodes of supercapacitor. The process of producing activated carbon begins by drying cocoa pods in the sunshine and continues with drying in the oven, pre-carbonization, grating, filtering, activation of chemical with Kalium Hydroxide (KOH) at concentrations of 0.3 and 0.4 Molar, pellet sizing, continue carbonization at 600°C is followed by pellet polishing and physical activation at 600, 700, 800, and 900°C for a duration of 4 hours. Resulting supercapacitor electrodes are characterized by SEM-EDX, XRD, CV, XRF, and BET. Physical activation data showed that the best conditions were acquired at 700°C using a 0.4 Molar Kalium Hydroxide activator, the electrode density was obtained at 0.801 g/cm³. According to SEM analysis, the holes ranged in size from 0.44 μm to 0.98 μm, and the EDX data indicated that the electrode sample's carbon content was 91.49%. The data of XRD showed that the carbon electrode's structure was amorphous by angles of diffraction (2θ) of 23.569° and 44.781°. The surface area obtained from the BET analysis is 360.290 m²/g. The best specific capacitance of 140.2 F/g with an activation time of 2.5 hours was found by electrochemical property analysis using the cyclic voltametric technique. The characterization's findings indicate that cocoa pod activated carbon could potentially find usage as a foundational substance for supercapacitor electrodes.

Keywords: Cocoa shell, Carbon electrode, Supercapacitor, Temperature, Activation.

1 Introduction

Electro-chemical double-layer capacitors (EDLC), also called supercapacitors, are a particular kind of storage for energy technology. that is presently under development [1,2]. A supercapacitor is an electrochemical device with high power density in storing and discharging charges which have thousands of times storage capacity and higher energy compared to conventional capacitors. A supercapacitor with hundreds of Farad capacity may be recharged in 30 seconds. The supercapacitor's surface is made up of activated carbon and a light covering of electrolytes which operate as an electric and charge the barrier. An electrode made of an anode and a cathode is one of the components of a supercapacitor.

A base material for a supercapacitor electrode is usually made of carbon aerogel [3], nanocomposites [4], metal oxides [5], and conductive polymers [6]. Currently, supercapacitor electrode materials are being developed from converted organic materials into activated carbon. Organic materials are widely used because they are accessible, guaranteed for its continuity, environmentally friendly,

affordable, easily synthesized, can be manufactured in the form of powdery, fiber, and composite, vast surface area, and adjustable porosity. The assurance and environmentally friendly are the main factors of organic to be a future energy source in terms of green technology (green chemistry) [7,8,9].

Organic containing a high carbon is a key aspect in determining the aptitude to store the electrical charge of a supercapacitor. In the previous research, the supercapacitor electrodes were made from varied organic materials like rice husk [10], rubber wood sawdust [11], shochu distillery waste [12], durian peel [13], banana stem waste [14] and shochu waste [15]. These materials have different specific capacitance values (Csp), such as: sago pulp, rubber wood, banana stem waste, rubber wood sawdust, durian peel, and elephant grass flower with their respective specific capacitances: 132.09 F/g, 115 F/g, 104 F/g, 50.65 F/g, 66 F/g, and 43 F/g.

Cocoa pods are agricultural waste that can be used as supercapacitor electrodes in accordance with the cocoa fruit peel' content of hemicellulose cellulose and lignin and the other component are each 21.06%, 20.15%, 51.98%, and

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6,81% [16]. From the initial study, the capacitance value of cocoa pods with a 0.3 Molar concentration of KOH activator at a 700°C temperature was 90.2 F/g [17]. These findings suggest that the activated carbon electrode of cocoa shell has an opportunity to be employed as the fundamental material of the electrode of supercapacitor. However, the results of this capacitance are not optimal. Further research is still needed to obtain the best results in order to make it feasible to be used as a promising supercapacitor electrode in the future, such that it decreases the amount of carbon electrode supplied by the Indonesian industry. Besides that, the process of cocoa pods waste into useful and economically valuable goods is considered significant as well which is included into one of the [Sustainable Development Goals](#) (SDGs) programs.

The use of biomass waste into activated carbon for supercapacitor electrode base materials is not only to reduce the impact of environmental damage, but also has an economic impact. Because with the availability of environmentally friendly electrode base materials, it will reduce electrode imports that have been carried out by supercapacitor entrepreneurs and industries.

2 Research Methods

The materials needed in this study to make the supercapacitor electrode were cocoa shells, KOH 0.4 M, CO₂ gas, sandpaper, aquades, nitrogen gas, rice paper, copper plates, and separators. While the equipment used were: oven furnace, mortar grinding, ball milling, sieve size 53 µm, SEM (JEOL JSM 6510 LA), XRD (X-Pert Powder), BET SURFACE AREA ANALYZER - BELSORP MR1, Physics CV UR Rad-Er 5841, XRF (PANalytical, Type: Minipal 4), CV (UR Rad-Er 5841) hydraulic press, analytical scale, hotplate stirrer, porcelain cup, universal indicator, Teflon, measuring cup, stainless steel, caliper, and sandpaper.

Activated Carbon Manufacturing

Initially, the preparation process of the sample was performed by collecting cocoa pods (sliced, sun-dried, pre-carbonized, crushed, sifted, chemically activated, washed, dried, mashed, and produced activated carbon). After that, the carbon electrode pellets were printed, carbonized, physically activated. Then, these printed pellets were polished, washed, and finally dried. The process of the series of activities has been explained in previous publications [17].

The Chemical and Physical Characteristics of Carbon Electrodes

The characterisation of electrode physical behavior was aimed to determine the morphology, porosity and crystal structure of carbon electrodes from cocoa pods obtained through the measuring of mass, diameter, thickness, and density. In addition, Thermogravimetry Analysis (TGA) and Differential Thermogravimetry (DTG) measurements was

aimed to identify the appropriate thermal temperature in the sample to be tested while measuring the mass decrease as a temperature operation, where the results of the analysis have been described in the previous article [17].

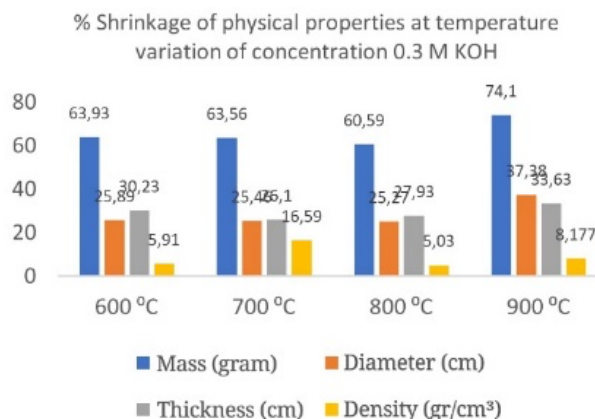
The characterization with SEM-EDX was aimed to determine the morphological structure and analyze the pore size formed in the carbon electrodes, as well as to determine the composition of compounds contained in the material. To support the EDX results, the analysis by XRF was conducted. The process was continued by BET measurements in order to calculate the particular area of surface of the produced active mass of the electrode, and XRD measurements to identify the phase produced on the electrode of carbon.

Furthermore, the measurement of electrochemical properties by Cyclical voltammetry (CV) technique was aimed to figure out the particular capacitance values of the supercapacitor cell via the CV UR Rad-Er 5841 Physics device which was regulated by the cyclic voltammetry CV v6 software with prospective width from 0 mV to 500 mV and a scanning rate of 1 mV/s. The Cyclic Voltammogram data collected was represented in specific capacitance curves (Csp) to the voltage in the velocity of scanning fluctuation.

3 Results And Discussion

Physical characteristics of carbon electrodes

The density, mass, diameter, and thickness for activated carbon are all measured during the physical property characterisation process. A carbonization and activation process results in bulk and dimensional contraction. Mass shrinkage occurs due to the breaking of bonds in compounds or moisture content is one example of materials other than carbon, oxygen and nitrogen. Meanwhile, dimensional shrinkage is caused by the rearrangement of carbon atomic groups throughout the carbonisation and the process of activation [15]. These mass reduction and dimensional shrinkage certainly cause a change in density shrinkage, which is indicated by the appearance of porosity in the sample [13].



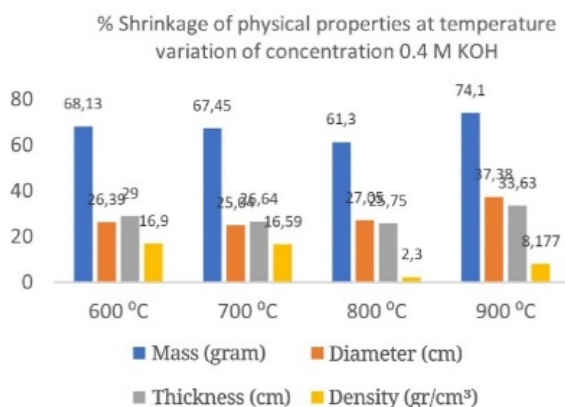


Fig. 1: Percentages loss of mass, diameter, thickness, and density with variation of temperature and activators concentration

Indeed, the predominant mass shrinkage will influence the diameter and thicknesses shrinkage. There was a considerable difference in thickness, where the thickness shrinkage in the 0.3 M sample at 600, 700, 800, and 900°C was greater than the 0.4 Molar sample at the identical temperatures. While a diameter shrinkage showed the adjacent value for a 0.3 M concentration of sample at similar temperatures. The presence of those physical values for parameters obviously resulted in a reduced density in the carbon electrode upon activation compared to prior carbonization [14]. Figure 1 depicts how the overall process of mass loss, diameter, and thickness in the sample generates a decrease in the value of density following carbonization and activation. It can be said the decrease in density in the carbon electrode is related to The growth of pore geometry arises from each stage of activation carried out. [12-14].

Another study on the manufacturer of supercapacitor electrodes about the mass and density of carbon electrodes reduced as the temperature of activation increased for banana stem waste [14]. The increase in activation temperature leads to an increase in energy to release compounds other than carbon, and reduction in mass is related to reduction in density [15].

Thermal Stability of Cocoa Shell Biomass

Figure 2 illustrates the DTG/TG curve for cocoa pods powders. The blue line shows a reduction in sample mass as time passes (TG), whereas the colored line shows a temperature shift over time (DTG). The TG curve shows that there is the occurrence of three points of mass shrinkage, signifying the establishment of three significant steps of sample mass shift. The initial point is a reduction in mass with a percentage of 7.06% from 30°C to 110.1°C indicates that water from the sample has evaporated. The following step is at temperatures of 110.1°C to 300°C, with a mass reduction of 17.14% C. This indicates that molecules of complex including hemicellulose, lignin, and cellulose, have started to breakdown into carbon at the stated temperature [16]. Obviously, hemicellulose decomposed at 200 to 390°C,

but lignin at 160 to 900°C [14]. On the other hand, there was another decrease in mass from on the third stage at a temperature of 300°C to a temperature of 552.8°C by 30.35%. This implies that there's presently an ingredient of cellulose or lignin being degraded at a temperature of greater than 552.8°C. Eventually, the overall mass lost during the three stages is roughly 54.55%. and leaves carbon material derived from cocoa pod peels as the remaining sample.

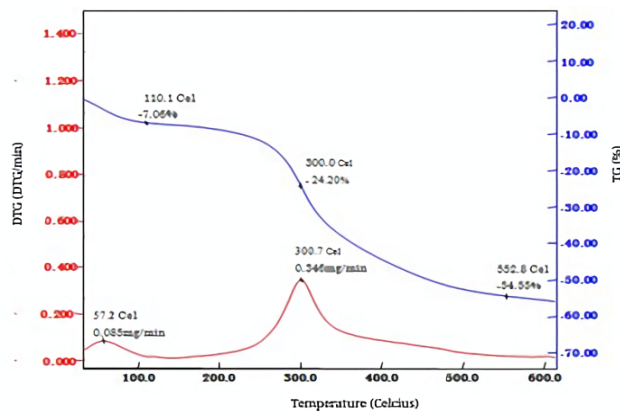


Fig. 2: DTG and TGA curves of cocoa pods

The DTG graphic exhibits data in the shape of a line, indicating the appearance of 2 peaks, specifically at a temperature of 57.2°C and 300.7°C. Apparently, the creation of peaks on a curve signifies mass reduction in terms of time. The graph shows that the content of water in the sample began to evaporate on the first peak, and there was an occurrence of significant shrinkage of cocoa pod shell mass at a rate of 0.346 mg/min at 300°C on the second peak. The carbonization process at this level of temperature should be kept for an hour to produce enhanced carbon. HenceIt is reasonable to deduce that the produced heat temperature is 300.7°C.

Analysis of Cyclical Voltammetry

The CV data collected are shown as a voltamogram graph with an effects velocity of 1 mV/s and the potential range of 0-500 mV. Figure 3 shows the CV curve for CO₂ treatment of activation. The image in Figure 3 shows the area formed between the currents Charge (I_c) and current discharge (I_d) indicating the magnitude of the carbon-based electrode produces a specified the value of capacitance. I_c is a current that occurs during the charging process (charge) shown by the upper curve, while I_d is the current formed during the discharge process (discharge) illustrated by the bottom curve. The larger the area of the I_c-I_d curve that is formed, the higher the specific capacitance value of the carbon electrode generated [15, 17]. The I_c-I_d curve demonstrates that the maximum specific capacitance value is found in a 0.4 Molar 700°C carbon electrode study. In addition, the 0.3 Molar 900°C specimen was found to have the lowest specific capacitance value, because of the smallest curve area of it has an area of I_c-I_d.

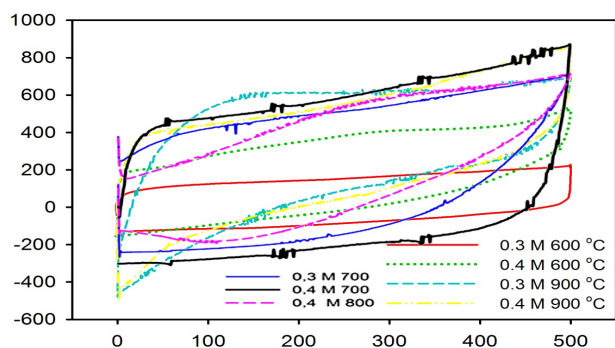


Fig. 3: Cyclic curve voltammetry

When compared to other samples, the 0.4 Molar 700°C carbon electrode exhibits an improved capacitor sensibility, as evidenced by a larger central potential region, an improved capacitor sensibility at 0 V voltage, and a horizontal discharge curve shape. The second form of the 0.4 Molar at 700°C CV slope sample is in the form of a square shape and is considered as the optimal shape for activated carbon electrodes [13, 18]. Figure 3 illustrates that raising the concentration of Kalium Hydroxide from 0.3 to 0.4 Molar may improve the quantity of porosity in the electrode of carbon, so that the quantity of ions moving into the electrode rises, as indicated by the breadth of the charge-discharge slope. The change in the value of the increased scan rate presents a narrow central promising area. The largest central potential region is obtained at a scanning rate of 1 mV/s, where the process of charging on activated carbon is sluggish, therefore the amount of charge transported from the solution of electrolytes into the electrode porosity is higher. Therefore, a specific capacitance value (Csp) of Kalium Hydroxide concentration is increasing from 0.3 to 0.4 M at 700°C activation temperature.

Figure 3 also shows that the rise in temperature of activation from 700°C to 800°C and 900°C at a concentration of 0.4 M resulting the decrease of the specific capacitance value obtained through the electrode of carbon and characterized by a decreasing charge-discharge curve. The addition of physical activation temperatures of 600°C, 700°C, 800°C and 900°C results in a rise in the quantity of porosity in the electrode of carbon but the carbon electrode becomes brittle and reduces the specific capacitance value [13]. The data from the calculation of the specific capacitance value of the electrode of cacao pods activated carbon could be shown in Table 1.

Table 1: Capacitance of specific as a function of KOH and temperature

Temperature (°C)	0.3 Molar KOH				0.4 Molar KOH			
	Mass (average)	Ic (µA)	Id (µA)	Csp (F/gr)	Mass (average)	Ic (µA)	Id (µA)	Csp (F/gr)
600	0.0173	153	-86	13.78	0.0167	378	-9	22.93
700	0.0132	707	-483	90.20	0.0112	993	-577	140.20
800	0.0172	539	-415	45.90	0.0164	299	-303	50.80
900	0.0178	608	90	29.20	0.0163	551	52	30.55

Regarding prior research on the manufacturing of electrodes

for supercapacitor using biomass basic materials, such as rubber wood [12], coconut shells [18], sago pulp [15], durian peels [13], and banana stems [14] with specific capacitance values were 54 F/gr, 10 F/gr, 132.09 F/gr, 88.38 F/gr, and 104.2 F/gr, respectively. Thus, the findings of the research on supercapacitor electrodes with cocoa peels base materials obtained the maximum of capacitance specific value is 140.2 F/g. Cocoa pods have considerable an opportunity as a fundamental substance for electrodes supercapacitor devices. An analogy of specific capacitance levels from the results of measuring the variation in the activation concentration can also be seen in Figure 4.

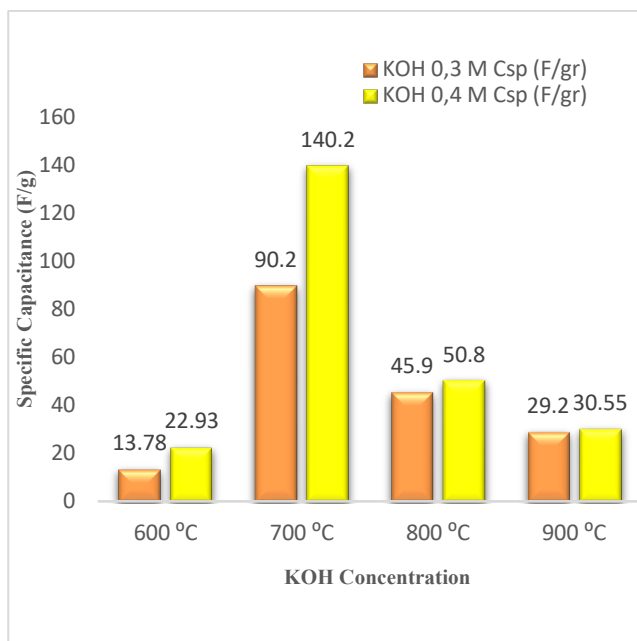


Fig. 4: The value of specific capacitance based on variation temperature and KOH concentration

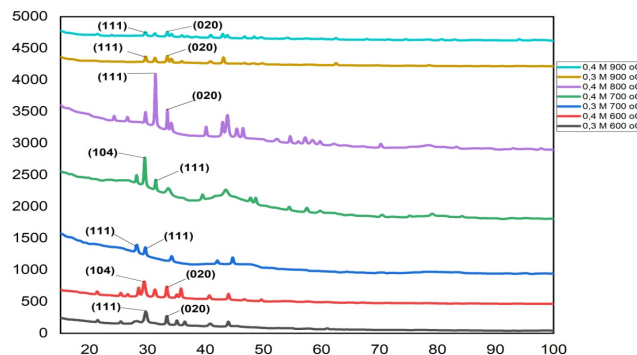


Fig. 5: Patterns of X-ray Diffraction

Diffraction of X-rays study.

A carbon XRD pattern for sample cocoa shell biomass by concentration activator 0.3 and 0.4 M KOH at 600°C, 700°C, 800°C, and 900°C, respectively, could be illustrated in Figure 5. The chart depicts the connection among X-ray intensity and angle of scattering (2θ) in eight sample of carbon samples from cacao pod. The fitting yielded data on scatter

angle, peak height, and peak width.

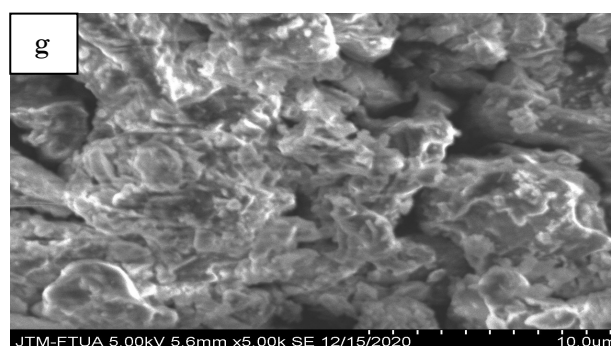
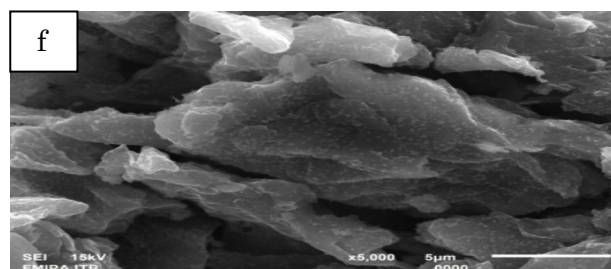
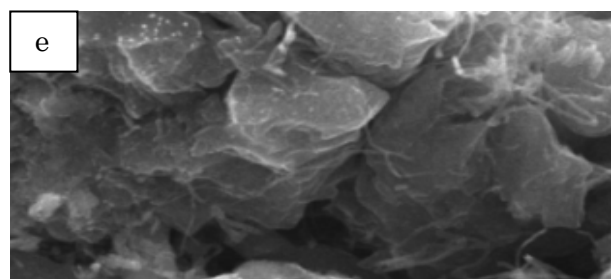
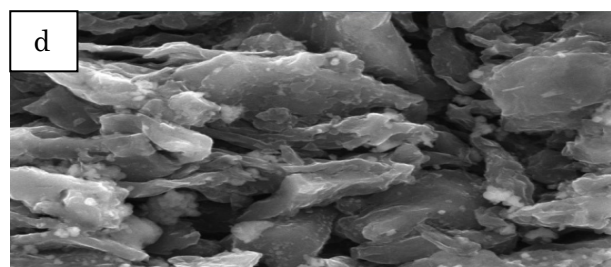
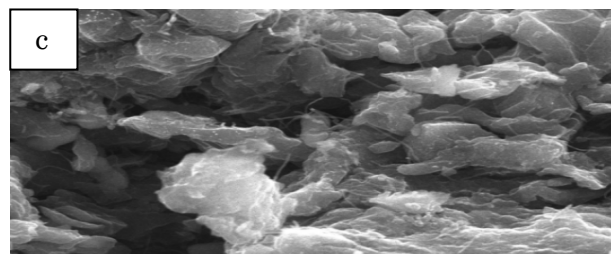
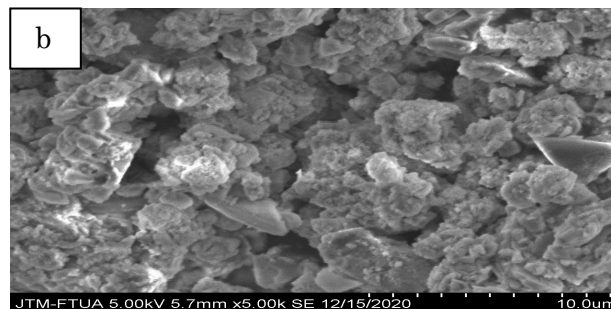
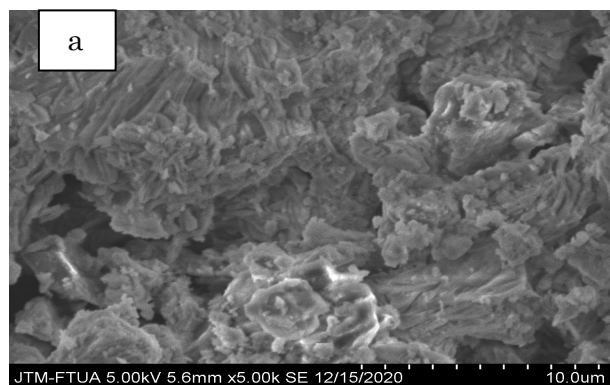
The 0.4 M 800°C graph shows the crystal peaks clearly immoderate compared to the other samples, at the 0.4 M 800°C graph diffraction angle. This occurrence shows the crystal peaks at the diffraction angles 2θ that is: 29.62°, 31.34°, 33.40°, 40.08°, 42.92°, 43.74°, 45.36°, and 46.43°. with the peak heights being 133.93, 604.30, 253.85, 114.86, 169.32, 243.13, 102.42, and 124.35, followed by a 0.4 M 700°C sample. Based on the XRD graph of the five samples, it can be seen the presence of carbon material and a graphite crystal structure as the phase formed [19].

Table 2: Applying diffraction of X-ray findings to the cocoa pod electrode.

Sample	Initial	2θ	θ	h	k	l	$d (\text{Å})$	$a (\text{nm})$
600°C	0.3	29.66	14.83	1	1	1	3.01	5.22
		33.29	16.65	0	2	0	2.69	5.38
600°C	0.4	29.39	14.70	1	0	4	3.04	12.53
		33.28	16.64	0	2	0	2.69	5.38
700°C	0.3	28.12	14.06	1	1	1	3.17	5.50
		29.59	14.80	1	1	1	3.02	5.23
700°C	0.4	29.49	14.75	1	0	4	3.03	12.48
		31.39	15.70	1	1	1	2.85	4.94
800°C	0.3	29.54	14.98	1	2	1	2.20	4.57
		32.24	15.79	0	2	1	2.80	5.20
800°C	0.4	31.34	15.69	1	1	1	2.85	4.94
		33.58	16.79	0	2	0	2.67	5.34
900°C	0.3	29.64	14.82	1	1	1	3.01	5.22
		33.38	16.69	0	2	0	2.68	5.37
900°C	0.4	29.61	14.80	1	1	1	3.02	5.23
		33.37	16.69	0	2	0	2.69	5.37

The characterization results indicate two big corner points with angles of 2θ ranging from 25 to 35. This range corresponds to the planes of diffraction (002) and (100) utilizing Mocrical Origin Ware to get the 2θ angle at each peak. The data was fitted utilizing the Lorentzian distribution function resulting in the data on scattering angle, peak height and peak width. The distance values between the fields of dhkl, and the sample miller index are shown in Table 2.

Surface Morphology Electrode Supercapacitor Activated Carbon Cocoa Pods



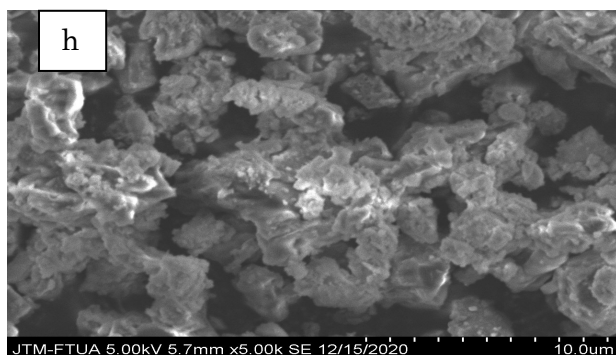


Fig. 6: SEM results of magnified electrodes of carbon 5000X (a) 0.3 Molar temperature 600°C, (b) 0.4 Molar 600°C, (c) 0.3 Molar temperature 700°C, (d) 0.4 Molar temperature 700°C, (e) 0.4 Molar temperature 800°C, (f) 0.4 Molar temperature 800°C, (g) 0.3 Molar temperature 900°C and (h) 0.4 Molar temperature 900°C

The surface morphology analyzed by SEM, magnified by 5000 times seen in Figure 6. Figures 6a, b, c, d, e and f show the morphology of the 0.3 and 0.4 Molar carbon electrodes at temperatures of 600, 700, 800°C. The pores between the particles are unevenly distributed and have irregular shape formed upon the surface of sample. There remain minor elements present on the sample surface indicating the occurrence of materials other than carbon or free particles which incompletely evaporated during the carbonisation and process of activation. It can be shown that the larger the concentration of the activator, the larger the pore size with an uneven size, denser, and more pronounced visible particles.

The SEM results of the carbon electrodes 0.3 and 0.4 Molar at 900°C are shown in Figures 6g and 6h. The obtained morphology shows a surface with more irregular carbon particles and shapes indicating a very small carbon value. The formed particles are more visible even though the pores are uneven among the particles. The increase in activation temperature causes more carbon water evaporation reactions and will increase the quantity and pores size. Therefore, the particles would be damaged, and make them brittle [13].

All of the samples from the SEM results above show a fairly large, irregular pore shape and unequal size. Contrasting with prior experiments on electrodes of carbon using durian shells [13], the morphology of the image surface of electrode samples with the same magnification was achieved through varied physical activation time.

To retrieve more complete data, measurements of distance and particle size were carried out using ImageJ software, the results of which are presented in Table 3. The table provides information that the spacing and particle size of the carbon electrodes are uneven, and this indicates that the smallest particle size is 24.7726 μm and the distance between the particles is 0.8404 μm , which is best formed at 700°C 0.4 M. This indicates that when the particle size decreases, the surface area and porosity among the elements grow. The

exposed porosity prompts the decrease on the carbon electrode density, so the specific capacitance increases [20].

Table 3: Distance and size of particles after activation with KOH

No	Temperature (°C)	KOH (M)	r (μm)	Xc (μm)
1	600	0.3	0.9718	73.0000
2		0.4	0.9814	60.0000
3	700	0.3	0.7882	53.3565
4		0.4	0.8404	24.7726
5	800	0.3	0.4311	54.3427
6		0.4	0.4444	34.1113
7	900	0.3	0.7882	53.3566
8		0.4	0.8445	50.0000

Analysis of Activated Carbon Chemical Composition

The EDX data demonstrate the high quality of carbon in the cocoa pod carbon electrode sample as well as other components included inside. The proportion of elemental content in the carbon electrode sample is shown in Table 4.

Table 4: Chemical composition in carbon electrodes at various temperatures

Content	Temperature (°C)							
	600		700		800		900	
	%Mass	%Atom	%Mass	%Atom	%Mass	%Atom	%Mass	%Atom
C	86.75	90.62	87.05	91.02	87.87	91.49	91.33	94.05
O	10.72	8.73	10.12	7.95	9.65	7.54	6.75	5.22
Mg	0.93	0.39	0.75	0.39	0.94	0.48	0.68	0.35
Ca	2.07	0.58	2.07	0.65	1.55	0.48	1.23	0.38

Table 4 indicates that the substances in the sample of carbon electrode are primarily composed by the element carbon (C), which has the percentage of mass of more than 86% and an atom percentage greater than 90%. The high C concentration suggests that the sample of carbon electrode is exceptionally pure [8]. Carbon electrode samples contain components other than carbon, including oxygen (O), magnesium (Mg), and calcium (Ca). Following component C, element O has the second greatest proportion, due to the incomplete decomposition or the occurrence of bonding in the activation process on the carbon sample by the time of carbonization of the oxygen content [21, 22]. Mg and Ca are classified as elements with atomic percentages less than 0.66% and mass percentages less than 2.1%, respectively. The carbon electrode's Ca concentration is caused by the chemical composition in the cocoa pod, one of that contains Ca [23]. The element Mg is described as coming from dirty steel balls while ball milling, and consists of component K because of the incomplete decomposition on the chemical activation [21, 24]. Increasing the concentration of Kalium Hydroxide activator from 0.3 to 0.4 Molar causes further breakdown of elements other than carbon, resulting in a higher production of the C element.

To determine the presence of other elements in the activated carbon of cocoa pods, a follow-up analysis with XRF was carried out. Tests were performed for samples of 600°C 0.4 M and 900°C 0.4 M. The test results show that in the carbon electrode sample of cocoa pods there are other elements whose percentage content can be seen in Table 5. This may

be due to the oxygen content was deserted by carbonization imperfections or there may also be bonding in the activation process [13, 25].

Table 5 contains information on the elements found in the carbon electrode samples have high elements (K) and (Ca) with a concentration percentage of (K) 61.83% and (Ca) 24.65% for the 600°C 0.4 Molar sample, as well as the 900°C 0.4 Molar sample with 56.44% of (K) concentration and 30.84% of (Ca).

Table 5: Compound content in carbon electrode samples at 600°C and 900°C with KOH 0.4 M

Element	Concentration (%)	
	600°C	900°C
Magnesium	4.87	3.78
Aluminium	0.81	1.46
Silicon	1.22	1.36
Phosphor	2.03	2.01
Sulfur	1.01	0.43
Chlor	0.11	0.13
Kalium	61.83	56.44
Calcium	24.65	30.85
Titanium	0.04	0.09
Chrome	0.03	0.01
Mangan	1.06	1.23
Ferrum	0.41	0.52
Nickel	0.01	0.01
Cuprum	0.06	0.09
Zinc	0.46	0.04
Rubidium	0.16	0.26
Strontium	0.05	0.07
Argentum	1.14	1.15
Barium	0.07	0.07

Analisa Surface Area (BET)

The results of BET provide information that the sample with concentration 0.4 M at the temperature of 700°C has a correlation coefficient of 0.99 with a fairly good surface area of 360.290 m²/g. This result is supported by the results of image processing with ImageJ software, where the smallest pore size is also obtained for samples with the same conditions. These findings demonstrate that the smaller the particle size, the greater the area of surface, so the higher the specific capacitance [20, 26].

4 Conclusion

It has been successfully carried out manufacture of electrodes of supercapacitor produced from activated carbon cocoa shells. The concentration of KOH activator and activation temperature determine the percentages of mass shrinkage, diameter, thickness, and density. Evidently, the higher the temperature, the greater the shrinkage percentage. SEM analysis reveals microscopic gaps among the elements on the surface of cocoa pod's carbon electrode. These gaps are predominant, reaching 94.05% C atoms in the 0.4 Molar sample at a temperature of 700°C as shown by EDX

characterization. This shows that activation using CO₂ at 700°C can increase the carbon purity of the samples, and obtains 2θ values ranging from 25°-35° and 42°-44° on the reflection planes 002 and 100 for XRD measurements. The sloping peak at 2θ lies between 30°-35° and 42°-44°, showing a structure that is amorphous in the electrode of carbon. The maximum capacitance of specific value obtained was 140.2 F/g at a temperature of 700°C with a KOH activator concentration of 0.4 M. Indeed, the optimum condition is obtained at the activation of KOH 0.4 Molar at a temperature of 700°C.

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