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The physical and electrochemical properties of activated carbon electrode made from pandanus tectorius

E Taer[1], A Apriwandi[1], Krisman[1], Minarni[1],R Taslim[2], A Agustino[1]and A Afrianda[1]

[1]Department of Physics, University of Riau, 28293 Simpang Baru, Riau, Indonesia
[2]Departement of Industrial Engineering, Islamic State University of Sultan Syarif Kasim, 28293 Simpang Baru, Riau, Indonesia

Email: erman_taer@yahoo.com; apriwandi95@gmail.com

Abstract.This research focused on analyzed the effects of carbonization and activation on the physical and electrochemical properties of carbon electrode made from pandanus tectorius. Carbon electrodes were variated in four different activation types such as non activated, chemical activation, physical activation, and chemical-physical activation. The samples were carbonized at temperature of 600 ˚C using N_2 gas. Chemical activation was using 0.8 M KOH, and physical activation was done using CO_2 gas at a temperature of 850 ˚C for 2.5 h. The density of the carbon electrode was analyzed by measuring mass and volumes The morphology of the carbon electrode was reviewed by the Scanning Electron Microscope (SEM) method. Chemical element composition and Purity of carbon electrode was determined by Energy Dispersive X-ray (EDX). The degree of crystallinity was characterized by the X-ray Diffraction (XRD). The surface area of the carbon electrode can be evaluated based on XRD data. Electrochemical properties was evaluated using Cyclic Voltametry by testing two electrode mechanism in 1 M H_2SO_4 aqueous electrolyte. The activated carbon electrode based on pandanus tectorius with chemical-physical activation provides maximum surface area and maximum capacitance of 1144.82 m^2g^{-1} and 56 Fg^{-1} respectively.

1. Introduction

Pandan(pandanus amaryllifolius)is one type of shrub, with pandanaceae family [1]. This type of family have 600 species of various sizes and shapes [2,3]. In the Indonesian forest, pandanus is often found as pandanus with thorns or pandanustectorius. Pandanleavesis used for handicrafts such as made as mats and others [4]. In addition, the chemical composition of pandan leaves consists of 37.3% cellulose, 37.4% haemicellulose, 14.4% pentosans, 24% lignin and ash, and 2.5% extractive [5] so that pandan leaves become one of the potentially biomass material for activated carbon production at many application [5,6,7]. Biomass-based activatedcarbonshow some physical properties such as amorphous structure, a high degree of porosity, an extended surface area, chamical stability and good conductivity [8].The good physical properties of being a strong reason for the researchers to focus the research in electrochemicalfield as energy storage device component such as electrodes material in batery and supercapacitor [9]. Some of the activated carbon electrode from biomass used several preparation methods for supercapacitor applications, among others, Zhang *et al*.(2018) produces activated carbon from bamboo by KOH activation and high temperature of physical activation, resulting in high specific surface area of 2221.1 m^2 g^{-1} and highest capacitance of 293 F g^{-1} [10]. Hierarchically porous and heteroatom doped carbon derived from tobacco rods reported by Zhao *et al.*(2016)and it shows the highest specific surface area of 2898 m^2g^{-1} and specific capacitance of 266 F g^{-1} [11]. Ong*et al.*(2012) produces activated carbon made from durian shell that was modified by the combination of ultrasonication and microwave irradiation techniques, the highestspecific surface area as high as648.64 $m^2g^{-1}$and highest electrode capacitance of 103.6 Fg^{-1} [12]. Pandan leaves has been used as activated carbon[5,6,7,13] but no one has reported it as a supercapacitor electrode. Preparation of activated carbon from the pandan leave raw material has been successful by using integrated carbonization and activation methods. The monolithic carbon electrodes prepared by variation of chemical and physical activation. Chemical activation is using 0.8 M KOH, whereas physical activation is performed by using CO_2 gas. Our results show that biomass-based activated carbon has a large surface area that provides ion transport between electrolyte and the carbon, resulting in good electrochemical properties. The results indicate that the activated carbons from pandanustectorius as electrode materials would be promising for supercapacitor applications.

2. Experimental Method

Pandanus monolithic activated carbon is produced by the preparation method previously reported [14]. Monolithic activated carbon was prepared in four different activations, such as KOH activation, CO_2 activation, combination of KOH-CO_2 activation and a sample without treatment as a basis. Based on activation variations, the samples were labeled as follows: AC/KOH, AC/CO_2, AC/KOH-CO_2 and AC/Untreatment. The KOH concentration is used 8 M. The pyrolisis process includes carbonization and physical activation carried out simultaneously in one step as previously reported [15]. Carbonization was carried out at a temperature of 600 °C using N_2 gas atmosphere with a flow rate of 1.5 $Lmin^{-1}$ followed by physical activation using CO_2 gas at a temperature of 850 °C for 2.5 hours with a flow rate of 0.5 $Lmin^{-1}$. The pyrolysis process for the AC/Untreatment and AC/KOH samples are only carbonized at a temperature of 600 °C. The capacitivepropertiesof supercapacitor cells was elucidate by using two electrodes system. The arrangement of supercapacitor cell electrodes such as activated carbon electrodes, current collector, separator and electrolite are arranged in sandwich form [8]. The current collector used is steinless steel 316-L type which produced by Goodfellow Cambridge Ltd., England. The separator is used as a duck eggshell membrane [16] while the electrolyte used is H_2SO_4 1 M [17]. Characterization of the sample consists of physical and electrochemical properties. The physical properties of electrodes analyzed included density, degree of crystallinity, surface area, surface morology and element content. Density is calculated by measuring the dimensions and mass of the carbon electrodes. The degree of crystallinity was characterized by using the XRD method with the X-Pert Powder Panalytical instrument with the Cu k-α light source and a wavelength of 1.5418 Å. The microcrystallite dimensions and interlayer spacing are calculated by using standard formulas [18,19] and bragg equation [20]. The surface area of the carbon electrode was calculated using the standard formula from microcrystalline height data obtained from XRD analysis [21]. The surface morphology was reviewed by using the SEM method with 5000 and 40000 magnifications. Theelement content was characterized by using the EDX method. The SEM-EDX characterization method uses the JEOL JSM 6510 LA. The electrochemical properties was measured by using the Cyclic Voltametry (CV) method with the CV UR Rad-Er 5841 instrument and it calibrated with a 1280 solartron device. Specific capacitance is calculated using cyclicvoltammogram data with the formula [22, 23];

$$C_{sp} = \frac{\Delta I}{sxm} \tag{1}$$

Where I= electric current, s = scan rate and m = mass of electrode.

3. Result and Discussion

3.1. Density analysis

The density of the activated carbon from pandanustectorius is shown in the Figure 1. Density is calculated by measuring the diameter, thickness and mass of carbon monolith (not shown here). The sample density is presented with graphs before and after pyrolysis. The carbon sample without treatment has the highest density of 0.846 g cm^{-3}. As different activation treatments show the different density in the activated carbon sample. Samples with combination of KOH-CO_2 activation treatment had the lowest density of0.685 g cm^{-3}, while a single activation sample such us AC/KOH or AC/CO2 produces density in the range of untreatment and combination KOH-CO_2 activation samples. The decrease in density is caused by the shrinkage mass and volume when chemical and pysical activation process was performed. The activation process removal of elements other than carbon which causes the development of porous structures and increased carbon content. The mass decrease is due to the reaction of the carbon atoms with the activator agent while the volume decrease is related to the rearrangement of carbon atoms during the activation process [24] so the density wasdecreases.

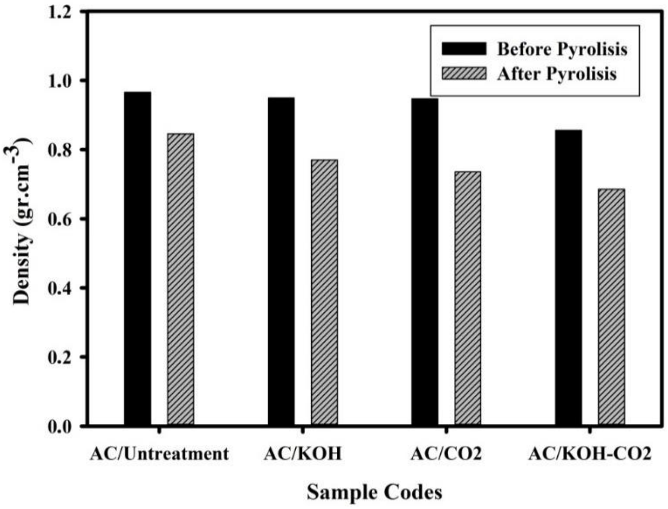

Figure 1. Density of carbon electrode from pandanus tectorius

3.2. Degree of crystallinity analysis

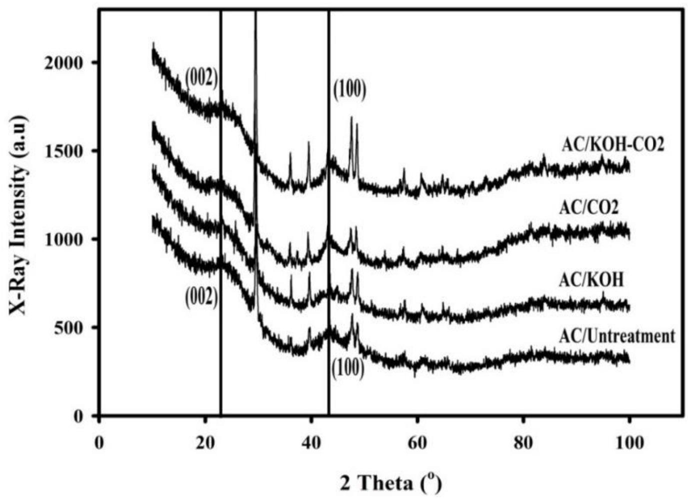

Figure 2.X-Ray Diffraction curve

The XRD curves of carbon electrodes prepared by different activations are shown in Figure 2. All samples generally present the same curve. This curve is identical to amorphous carbon with two broadening peaks [25]. The result of X-ray diffractions are used to evaluate interlayer spacing and microcrystallinity dimention by using Microcal Origin software. The 2θ angles of the 002 and 100 reflection planes for the samples in range of 24.443˚-25.603˚ and 44.929˚-45.852˚, respectively. These data suggest that the samples have a good peak coverage for carbon materials. In addition, the curve also shows sharp peaks at 29-30 ˚ and 47-48 ˚ angles. This sharp peak indicates the presence of other elements such as $CaCO_3$ caused by the residue from the pyrolisis process.

The sample structure parameters such as the interlayer spacings and microcrystallite dimensions are calculated and listed in Table 1. The microcrystallite height, in the range of 8.024-11.73 Å are almost identical to those of the activated carbon from other biomass materials, such as the durian shell, in the range of 10.58-36.21 Å [23]. These data are still in range of activated carbon. The different activation process give different effect on the interlayer spacing and microcrytallite dimention. The AC/KOH shows the smallest diffraction angleof 2θ and the AC/CO_2 and AC/KOH-CO_2 obtained the largest diffraction angle of 2θ. The diffraction angle 2θ 001 does not indicate a significant change in each sample treatment. Microcrystallite height produces varying data. The sample without treatment has the greatest L_c value. Along with the activation of KOH and CO_2 given in the sample, the Microcrystallite height are decreased. The smallest Microcrystallite height is in the AC/KOH-CO2 sample. Activation of KOH-CO_2 allows carbon electrodes to produce increased crystallinity due to the addition of KOH-CO_2 at higher temperatures. The increasing of crystallinity means better conductivity. The increasing in baseline in the low angle area for activated carbon is probably to originate from the presence of micropores that are rich in carbon framework.

Table 1. Diffraction angle (2θ), interlayer spacing (d), microcrystallinity height (L_c) and microcrystallinity width (L_a)

Sample codes	$2\theta_{(002)}$ (^{0})	$2\theta_{(100)}$ (^{0})	$d_{(002)}$ (Å)	$d_{(100)}$ (Å)	L_c (Å)	L_a (Å)
AC/Untreatment	24.936	45.214	3.56794	2.00386	11.7256	20.3456
AC/KOH	24.443	45.852	3.63878	1.97745	10.2181	22.4143
AC/CO_2	25.161	44.968	3.53655	2.01425	9.6355	26.5307
AC/KOH-CO_2	25.603	44.929	3.47649	2.01591	8.02419	10.2419

The microcrystallite height can be used to determine the specific surface area (SSA) of the electrode samples using empirical formula and it is shown in the Table 2. Based on the Table 2, The microcrystallite height is strongly associated with the surface area. A small microcrystallite height is required to produce a high specific surface area. Samples without treatment has the smallest specific surface area of 804.26 m^2g^{-1}. The addition of KOH allows the development of good carbon pores so that the specific surface area increases to 942 m^2g^{-1}. The CO_2 Activation at a temperature of 850 °C indicates the presence of rich micropores in a carbon framework and produces a specific surface area of 970 m^2g^{-1}. The combination of KOH-CO_2 activation produces the highest specific surface area of 1145 m^2 g^{-1}. The addition KOH and CO_2 activation developed micropores and more carbon pore so the carbon sample shown the higest specific surface area.

Table 2. Specific surface area of activated carbon

Sample Codes	L_c (Å)	$\rho_{(xrd)}$ (gcm^{-3})	SSA (m^2g^{-1})
AC/Untreatment	11.7256	2.1202	804.26
AC/KOH	10.2181	2.0789	941.51
AC/CO_2	9.6355	2.1390	970.39
AC/KOH-CO_2	8.0242	2.1759	1144.82

3.3. Surface morphology analysis

The effect of activations on the surface morphology of activated carbon electrodes from pandanus tectorius is reviewed in Figure 3. The SEM characterization for all electrode samples using magnifications of 40000x. Figure 3.a shown the sample without treatment presents aglomeration and larger particle size compare the other sample. The size of particle for AC/Untreatment is in the range of 0.685-0.153 μm. The AC/KOH sample displays a smoother surface morofology and a visible pore between particles and shown in Figurre 3.b. The presence of pores between particles are indicated by a dark color. The particle size becomes smaller with a size range of 0.225-0.113 μm. KOH activation successfully reduces particle size because of the activation agent breaks the bonds between particles [26]. The Figure 3.c shown the CO_2 activation sample, this activation produces particle sizes in the range of 0.442-0.202 μm. Combination of KOH-CO_2 activation shows the smallest particle size morphology of 0.156-0.070 μm which shown in Figure 3.d. Combination of activation allows the reduction of the most particle size so as effect to the development of better pores for carbon electrodes from pandan leaves.

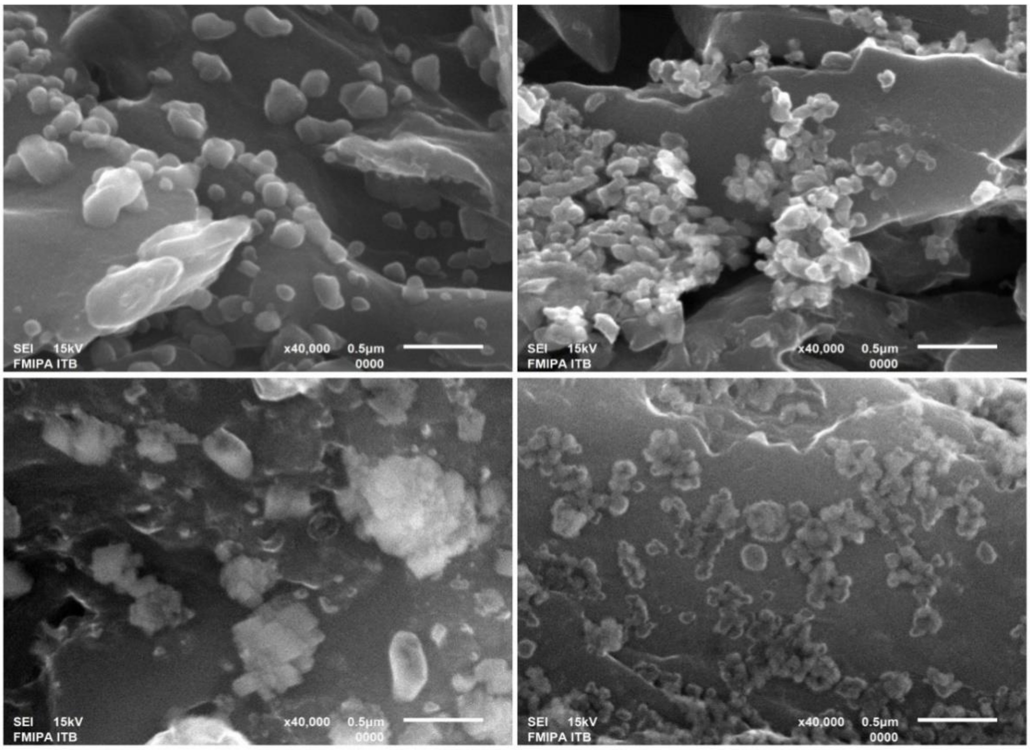

Figure 3.SEM micrographswith a magnification of 40000 times for a) AC/Untreatment; b) AC/KOH; c) AC/CO_2; d) AC/KOH-CO_2

3.4. Chemical content analysis

The EDX spectrum presents element content of the electrode is shown in the Figure 4. The EDX analysis showed the level of element content [such as carbon and other elements] present in the electrode samples. This spectrum shows that the samples are composed of carbon, oxygen, magnesium, potassium and calcium. The highest peak was recorded for the carbon element. This high carbon peak indicates that carbon is the highest elemental content of the electrode sample which shown in percentage of 92.96%, 94.54%, 94.44% and 94.93% for AC/Untreatment, AC/KOH, AC/CO_2 and AC/KOH-CO_2, respectively. Chemical activation using KOH effects the quantity of oxygen, potassium and chlorine [26] so that the percentage of carbon changes from 92.96% to 94.54%. The CO_2 activation at high temperatures also effects carbon elements. The combination of KOH-CO_2 activation produces the highest carbon content so that a combination of activation indicated can erode and remove the other element content than carbon to the maximum for activated carbon from pandan leaves. The oxygen content is due to the presence of carbon and oxygen bonds at the

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activation process. The other elements such as potassium and calcium are the basic components of pandan leaves. Percentage composition of each element in the activated carbon electrodes is shown in Table 3.

Table 3. The chemical composition of the all samples

Element contents	AC/Untreatment	AC/KOH	AC/CO_2	$AC/KOH\text{-}CO_2$
	Atom (%)	Atom (%)	Atom (%)	Atom (%)
Carbon	92.96	94.54	94.44	94.93
Oxigen	5.54	4.79	4.96	3.50
Magnesium	0.16	0.09	0.11	-
Potassium	0.22	0.21	0.10	-
Calcium	1.12	0.38	0.56	1.57
Totals	100%			

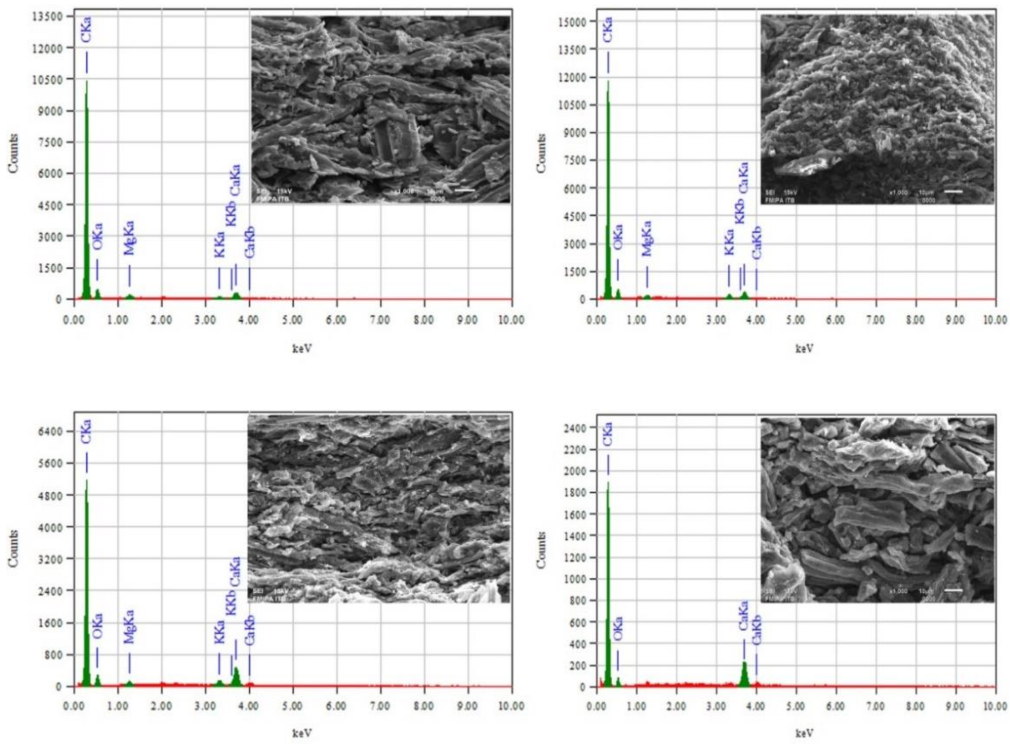

Figure 4. The EDX spectra of the all samples

3.5. The capacitive electrode analysis

The cyclic voltammetry measurements are commonly used to test the EDLC cell performances that use an activated carbon electrode. The cyclic voltammograms of all samples in 1 M H_2SO_4 aqueous electrolyte at a voltage of 0-0.5 V with a 1 mVs^{-1} scan rate. The I-V curve formed a rectangular shape for carbon electrode material [27] which are shown in Figure 5. This shape type are represents the specific capacitance produced by pure carbon electrode [28]. The rectangular shapes of the CV curves imply a quick ion diffusion and good charge propagation in all electrodes at a lower scan rate. Based on Figure 5, the specific capacitance result of 24 Fg^{-1}, 26 Fg^{-1} 43 Fg^{-1} and 56 Fg^{-1} for AC/untreatment, AC/KOH, AC/CO_2 and $AC/KOH\text{-}CO_2$ samples, respectively. The activations increase the electrode capacitive properties (from 24 Fg^{-1} to 56 Fg^{-1}). KOH activation produces new pores and increases the surface area. The large surface area provides a large medium for the ions diffusion into the carbon matrix sample [26] so specific capacitance increase from 24 Fg^{-1} to 26 Fg^{-1} for AC/KOH sample. Activation using CO_2 in longer time tend to produce samples with dominant micropores [15] and it can be result higher specific capacitance of 43 Fg^{-1}. In this study, combination of KOH-CO_2 activation shown the highest specific capacitance of 56 Fg^{-1}, it means KOH-CO_2 activation resulted the good combination pore and higher specific surface area so the specific capacitance reach maximum for carbon electrode based on pandanustectorius.

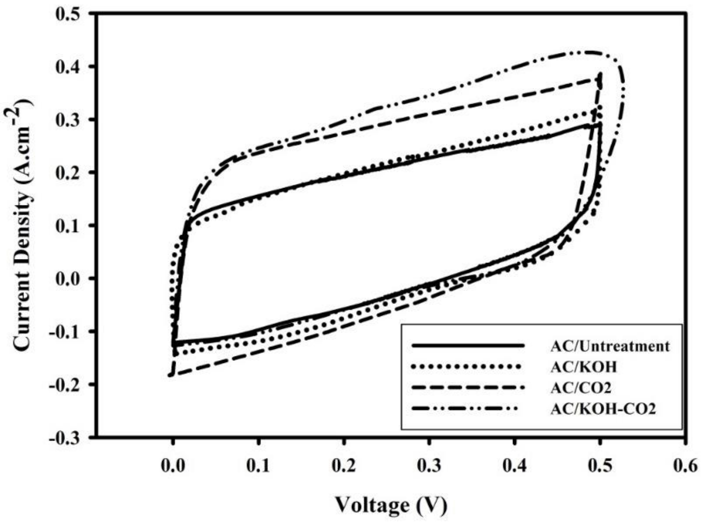

Figure 5. The CV curve for all samples

4. Conclusion

The analyzed of physical and electrochemical properties of activated carbon electrode made from pandanus tectorius has succesfully demonstrated. Activated carbon electrode prepared in four different activations such as KOH and CO_2 activation by using one step pyrolisis process simultaneously. The addition of activator agents such as KOH or CO_2 affects the physical and electrochemical properties of electrodes. Activator agents change the physical properties of the sample and improve the electrode capacitance properties. The carbon electrode has excellent physical and electrochemical properties for supercapacitor applications. The combination of KOH-CO_2 activation is shown in the best physical and electrochemical properties compared with the other activation samples. The lowest density of resulting sampel is 0.685 gcm^{-3}. Specific surface area produced reached of 1144.82 m^2g^{-1} with the highest carbon content of 94.93%. The superiority of physical properties supports good electrochemical properties with highest specific capacitance velue of 56 Fg^{-1}.

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