

Fabrication of activated carbon electrode synthesized from sacred lotus leaf natural materials for supercapacitors

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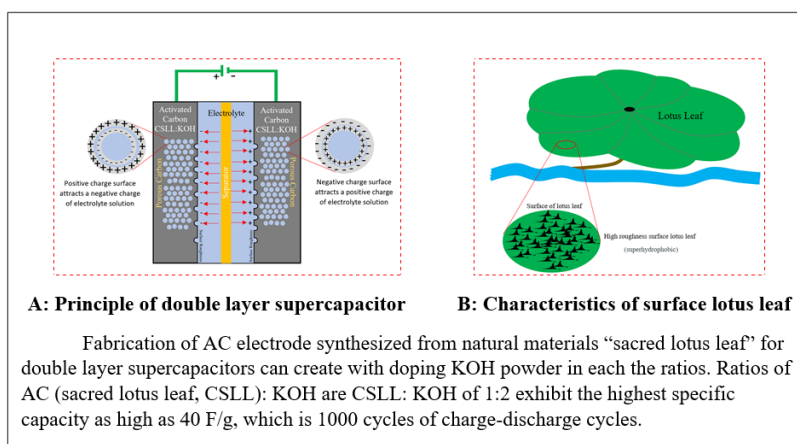
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Abstract

Supercapacitor has been the interesting issue in electric energy storage system. Supercapacitors carbon electrode was synthesized from a sacred lotus leaf. The none activated carbon sacred lotus leaf powder (CSLL) and the carbon sacred lotus leaf were mixed with potassium hydroxide (KOH) in the ratio of 1 : 1, 1 : 2, and 1 : 3 which were called CSLL, CSLL-1 : 1, CSLL-1 : 2 and CSLL-1 : 3, respectively. The structural, morphological properties and element component were analyzed with x-ray diffraction (XRD) technique, the field emission scanning electron microscope (FESEM) and energy dispersive x-ray spectroscopy (EDX), respectively. Electrical properties were measured by cyclic voltammetry (CV) and charge–discharge techniques. JCPDS 01-072-2091 data file confirmed the carbon-like (110) plan at 2 theta of 29.43° CSLL-1 : 1 and CSLL-1 : 2 showed high crystalline sizes. Morphology of CSLL-1 : 1 and CSLL-1 : 2 samples exhibited corrosion of surface clearly nevertheless carbon cluster adhered continuously on surface affect to higher the surface area. Carbon element of CSLL, CSLL-1 : 1 and CSLL-1 : 2 samples were obtained as high as of 74.50, 79.30 and 76 % by atomic, respectively which it was suitable characteristic of activated carbon electrode. The highest specific capacitance of CSLL-1 : 2 electrodes displayed approximately 40.85 F g⁻¹ at the scan rate of 20 mVs⁻¹. Moreover, the charge–discharge time of CSLL-1 : 1 and CSLL-1 : 2 electrodes showed the long discharge time more than the discharge time of CSLL-1 : 3 and CSLL electrodes. The performances of electrode demonstrated with charge-discharge of 1,500 and 1,000 cycles found that the CSLL-1 : 1 and CSLL-1 : 2 electrodes exhibited high stability. The suitable conditions ranges depicted between from the CSLL-1 : 1 to CSLL-1 : 2 ratios; furthermore, a sacred lotus leaf can fabricate the carbon electrode for supercapacitor.



Keywords: Carbon electrode; Supercapacitors; Sacred lotus leaf

Introduction

The current demand for electric energy has been likely to increase. Especially, electricity energy has been the necessary in human life. In present, alternative energy brought replace petroleum, coal and natural gas. Although, electricity production gets a high quantity, unless can't store energy into usable enough. Therefore, it is necessary to have

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energy storage devices. The devices used in an energy storage such as batteries, capacitors and supercapacitors [1]. Supercapacitors are very interesting using an energy storage device because advantage properties are high power density, high energy density, high speed charging and ability. Supercapacitors can be dividing the three groups which are

the carbon double layer capacitors, the pseudo-capacitors and the hybrid capacitors [2]. Carbon double layer capacitors were studied due to a simple structure, easy fabrication and low-cost materials [3]. Structure of carbon double layer capacitor consists of the carbon electrode sandwich, electrolyte and separated paper [4]. The significant factor which would affect to develop a high performance of the carbon double layer capacitors is their own carbon electrodes. The suitable carbon electrodes could be studied and synthesized by activated carbon (AC) which must have a high purity of carbon, surface area, corrosion resistance and conductivity [5]. The low cost activated carbon (AC) can be synthesized from natural materials such as a wool felt, coconut waste, lotus leaf, mangosteen peel, biochar, orange peel, bamboo, bamboo shoots and etc. [6 – 11]. Although, the activated carbon electrodes were made from a low-cost material but the efficiency of energy storage obtained with a high-power density and energy density [12].

This research was aimed to synthesize the super-capacitors by using the activated carbon (AC) mixed with the sacred lotus leaf. A sacred lotus leaf surface could be created the high surface roughness which corresponding to a high surface area on AC electrodes. Dominant features of the scared lotus leaf are a low-cost materials, eco-friendly resource, a lot of fiber, surface roughness abundantly and nanotube structural leaf. The AC powder would be synthesized from sacred lotus leaf via chemical activation at 800 °C for 2 h under argon (Ar) gas. The powder was doped with potassium hydroxide (KOH) which the ratio of 1 : 1, 1 : 2 and 1 : 3. The structural, morphological and element compound were examined by XRD technique, FESEM and EDX, respectively. Furthermore, the AC electrodes were fabricated with doctor-bled method on nickel (Ni) foam substrate. Moreover, electrical properties were measured by CV and charge–discharge techniques.

Materials and Methods

The research processes were divided into three processes as following 1) synthesis of activated carbon from a sacred lotus leaf, 2) fabrication of activated carbon electrodes from a sacred lotus leaf and 3) analysis process.

Synthesis of a sacred lotus leaf

Drying of sacred lotus leaf materials were dried in the air for 1 – 2 weeks. Then, sacred lotus leaf materials were grinded with mortar and then taken to sintered at 800 °C for 2 h in furnace (NOVA SPS70, Samheung, Korea). The AC

powder was synthesized from sacred lotus leaf by chemical activation at 800 °C for 2 h under argon (Ar) gas. The AC powder was prepared from a sacred lotus leaf powder mixed with potassium hydroxide (KOH) in deionized water (DI) dissolve about of 125 mL and stirred by magnetic stirrer for 24 h at 100°C. The conditions were varied with the KOH doping. The none-activated carbon is a sacred lotus leaf powder (CSLL) and a carbon sacred lotus leaf were mixed potassium hydroxide (KOH) in the ratio of 1 : 1, 1 : 2, and 1 : 3 which white instead CSLL (mass of AC powder, 1 g), CSLL-1 : 1 (mass of AC powder, 1 g : KOH, 1 g), CSLL-1 : 2 (mass of AC powder, 1 g : KOH, 2 g), and CSLL-1 : 3 (mass of AC powder, 1 g : KOH, 3 g), respectively. Complete mixed AC powder was sintered at 200 °C for 6 h, and was grinded by mortar again. Then, AC powder was sintered at 800 °C for 2 h, and was gained by mortar again. Finally, AC powder was adjusted the PH value of 10 with DI water and it was annealed at 70 °C for 24 h, and was gained by mortar again.

Fabrication of activated carbon electrode from a sacred lotus leaf

Substrates of electrodes were prepared from nickel foam sheet (1 × 2 cm², size) and nickel foam substrates were weighed by analytical balance. Cell area was fixed with Kapton tape (1 × 1 cm², size). CSLL, CSLL-1 : 1, CSLL-1 : 2 and CSLL-1 : 3 samples were mixed with carbon black in the ratio of 9 : 1 (the mass loading of the active material for the electrode, 0.90 g : carbon black 0.10 g). After that, these AC powder were dissolved in polyvinylidene fluoride (PVDF) and N-Methyl-2-Pyrrolidone solutions of 4 ml with conditioning mixer. Start processes, the nickel foam substrates were annealed at 70 °C for 2 h. The second, AC gel were coated on nickel foam substrate which replete coated approximately of 3 time. After that, the AC electrodes were annealed at 70 °C for 13 h. Then, Kapton tape was removed from AC electrode and these were weighed by analytical balance.

Characteristics of activated carbon electrode from a scared lotus leaf

This research was divided two parts as following 1) AC powder samples were analyzed the structure, morphology and element compound, respectively and 2) the AC electrodes were measured the current density – potential characteristic and charge – discharge curve. The structural, morphological and element compound properties were analyzed by XRD (PANalytical, EMPYREAN) technique,

FESEM (FEI, Helios NanoLab G3) and EDX, respectively. Then, the AC electrode was fabricated with doctor-bleed method on nickel (Ni) foam substrate. Electrical properties were measured by CV (CS350 EIS Potentiostat / Galvanostat, Corrtest Instruments) and charge – discharge techniques (CS350 EIS Potentiostat /Galvanostat, Corrtest Instruments).

The specific capacitance can be calculated formular as follows eq. (1);

$$C_s = \frac{\int I dV}{2 \times v \times \Delta V} \quad (1)$$

where C_s is the specific capacitance ($F\ g^{-1}$), I is the current density ($A\ g^{-1}$), ΔV is the potential vs. Ag/AgCl (V), and v is the scan rate ($mV\ s^{-1}$).

Results and Discussion

Photography and structural properties of activated carbon power from a sacred lotus leaf by XRD technique

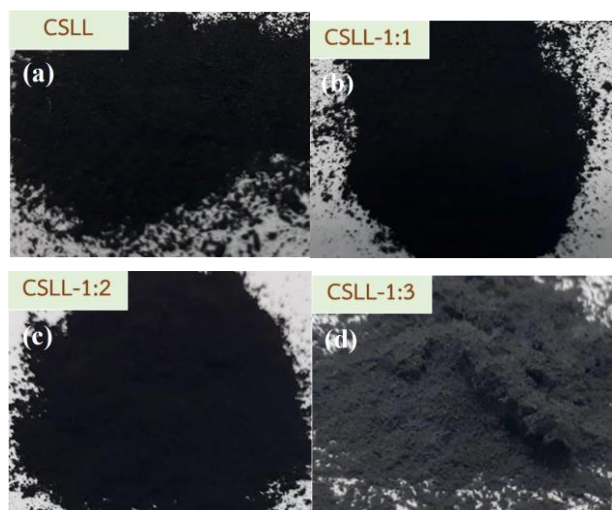


Fig. 1 Photography of the CSLL, CSLL-1 : 1, CSLL-1 : 2 and CSLL-1 : 3, respectively.

Photography of CSLL, CSLL-1 : 1 and CSLL-1 : 2 samples were showed powder thoroughly and uniform black colour more than the CSLL-1 : 3 sample clearly as shown in

the Fig. 1(a) – 1(c). CSLL-1 : 3 sample was exhibited gray colour clearly as shown in the Fig. 1(d) due to the surface area was corroded with KOH amount abundantly cause large occur and multitude a porous gap. Moreover, incomplete carbonization process was occurred for the CSLL-1 : 3 sample. The purpose of good AC electrode must be the high purities carbon (C) whereas high purities carbon were showed the black colour for the CSLL, CSLL-1 : 1 and CSLL-1 : 2 samples. The uniform black colour of the CSLL, CSLL-1 : 1 and CSLL-1 : 2 samples were indicated which one of properties of activated carbon which similar to [10] reference. Thus, the CSLL, CSLL-1 : 1 and CSLL-1 : 2 samples were obtained suitable will be made the AC electrode for double layer supercapacitors.

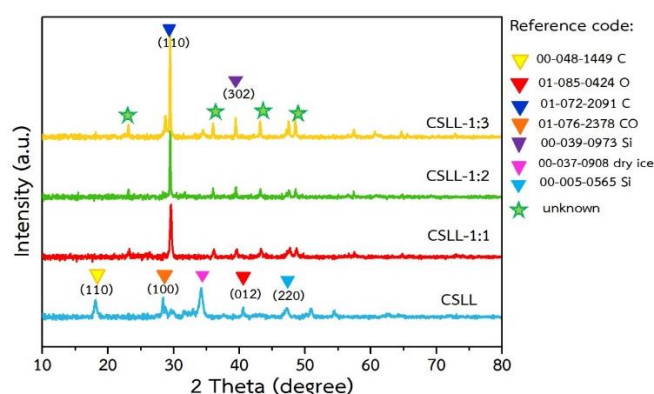


Fig. 2 XRD results of the CSLL), CSLL-1 : 1, CSLL-1 : 2 and CSLL-1 : 3, respectively.

The XRD results of CSLL, CSLL-1 : 1, CSLL-1 : 2 and CSLL-1 : 3 samples were showed crystalline of (110) carbon plane at 29° as show in the Fig. 2, respectively. Especially, crystal of CSLL-1 : 1, CSLL-1 : 2 and CSLL-1 : 3 samples were higher than CSLL sample due to influence of KOH doping. KOH interaction cause carbon particles adhere itself, and were removed from AC powder after. Moreover, the crystallite size of carbon was calculated by Scherrer's formula found that crystallite sizes are 25.99, 68.28, 110.27 and 143.33 nm for CSLL, CSLL-1 : 1, CSLL-1 : 2 and CSLL-1 : 3, respectively as show in the table 1. Crystallite sizes of carbon were tended larger when KOH amount increased, show that KOH corrosion on carbon powder surface effect to crystalline recombination.

Table 1 Parameter of AC powder from scared lotus leaf with XRD analysis.

Peak	2 Theta (degree)	hkl	FWHM (θ)	d-spacing (\AA)	Crystallite size	size
CSLL	18.0797	110	0.6240	4.9025	25.99	
	34.1608		0.7488	2.6226	22.31	
CSLL-1 : 1	29.5748	110	0.2496	3.0182	68.28	
CSLL-1 : 2	29.4330	110	0.1560	3.0322	110.27	
CSLL-1 : 3	29.4046	110	0.1248	3.0351	143.33	

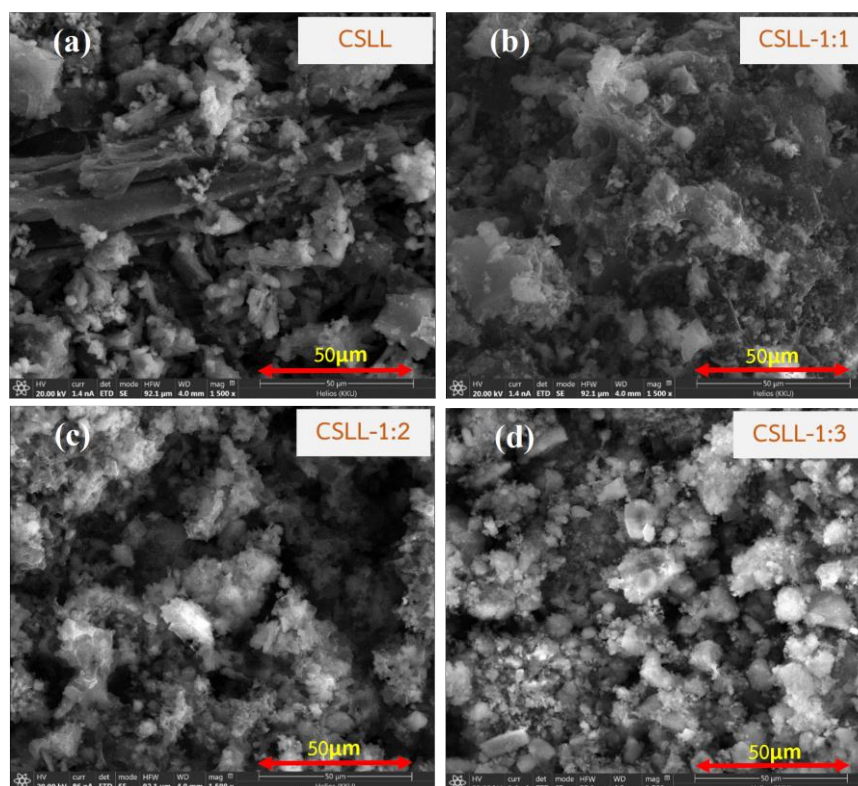


Fig. 3 SEM image of morphology surface of (a) CSLL, (b) CSLL-1 : 1, (c) CSLL-1 : 2 and (d) CSLL-1 : 3, respectively.

Morphology surface characteristics and elemental of activated carbon powder from a sacred lotus leaf by FE-SEM and EDX techniques

Powder surface morphological of CSLL, CSLL-1 : 1, CSLL-1 : 2 and CSLL-1 : 3 samples were exhibited the roughness surface due to the corrosion of KOH. Found that, CSLL-1 : 2 sample was corroded abundantly caused the large porous occurred. Moreover, CSLL-1 : 3 sample was

showed uniform roughness which corresponding to increasing of KOH. In order hand, surface of CSLL, CSLL-1 : 1 samples were corroded by KOH decreased as shown in the Fig. 3(a) – (d) which mentioned reason was supported Shanshan Qu et. al. research in [10] reference. However, EDX mapping of CSLL-1 : 3 sample found that dispersion of carbon non-uniform when these sample was compared with CSLL, CSLL-1 : 1 and CSLL-1 : 2, respectively as

shown in the Fig. 4(a) – (d). Show that, the motivations of the AC electrode are high surface area, high carbon purities and carbon dispersive. Moreover, element compound (% Atomic) were exhibited in the table 2 which indicated carbon

amount. Thus, CSLL-1 : 2 sample was obtained high surface area, uniform dispersion carbon and high carbon amount approximately of 76% by atomic which the suitable properties for the AC electrode.

Table 2 Parameter of element compound (% Atomic).

Sample	Element compound (% Atomic)								
	C	O	K	Ca	Cl	Si	Mg	P	Al
CSLL	74.50	19	3.40	2	1.0	-	-	-	-
CSLL-1 : 1	79.30	17.10	0	2.20	-	5	0.50	0.40	-
CSLL-1 : 2	76	18.90	0.50	2	-	7	0.60	0.40	-
CSLL-1 : 3	33.40	47.80	3.20	4.20	-	8.30	1	-	21

Table 3 Significant of AC electrode.

Sample	Significant capacitor, C_v (F g ⁻¹)				
	5 mV s ⁻¹	10 mV s ⁻¹	20 mV s ⁻¹	50 mV s ⁻¹	100 mV s ⁻¹
CSLL	39.76	35.36	29.80	20.93	0.30
CSLL-1 : 1	37.95	37.89	33.48	24.32	16.13
CSLL-1 : 2	31.90	32.89	40.85	35.45	28.40
CSLL-1 : 3	5.34	4.74	4.41	3.98	3.67

Electrochemical properties of electrodes by CV technique

Fig. 5 (a) – (f) were shown the cyclic-voltammogram of sample electrodes at scan rate of 5 mV s⁻¹, 10 mV s⁻¹, 20 mV s⁻¹, 50 mV s⁻¹, and 100 mV s⁻¹ and presents CSLL-1 : 2 sample only, respectively. The current density of CSLL, CSLL-1 : 1 and CSLL-1 : 2 samples were obtained higher than the CSLL-1 : 3 sample because CSLL-1 : 3 sample was a low purity of activated carbon (AC) from image and EDX results. Moreover, the maximum specific capacitance (C_s) value is 40.85 F g⁻¹ for CSLL-1 : 2 sample electrode at the scan rate of 20 mV s⁻¹ corresponding to the FESEM and EDX data base help confirm. However, the specific capacitance of CSLL, CSLL-1 : 1 electrodes were shown high value as same as the CSLL-1 : 2 electrode which were 39.76 and 37.97 F g⁻¹ at scan rate of 5 mV s⁻¹. Meanwhile, the CSLL-1 : 3 electrodes was the lowest specific capacitance (as shown in the Table 3) due to abundantly KOH doping give rise to low purity AC powder was supported from EDX data. Thus, the suitable

ratios activated carbon per KOH are 1 : 1 to 1 : 2 due to properties as following a high surface area, nano-scale crystalline size, high current density and high specific capacitance.

Electrochemical properties of electrodes by CD technique

The galvanostatics charge and discharge characteristic were shown the charge and the discharge electric of CSLL, CSLL-1 : 1, CSLL-1 : 2 and CSLL-1 : 3 electrodes, respectively. Found that, scan rate at 1 mVs⁻¹, CSLL-1 : 2 electrode were exhibited behaviour capacitor-like, although the charge time longer than CSLL, CSLL1;1 and CSLL-1 : 3 but the discharge time more than as shows in the Fig. 6 (a), so efficiency of CSLL-1 : 2 sample was confirmed suitable condition in preparation of AC electrode synthesized from sacred lotus leaf for double layer supercapacitor. Meanwhile, Fig. 6 (b) and (c) were shown the charge – discharge at scan rate of 2 and 5 mV s⁻¹ the results tendency as same which the electric discharge time range of CSLL-1 : 2 sample was shown longest times.

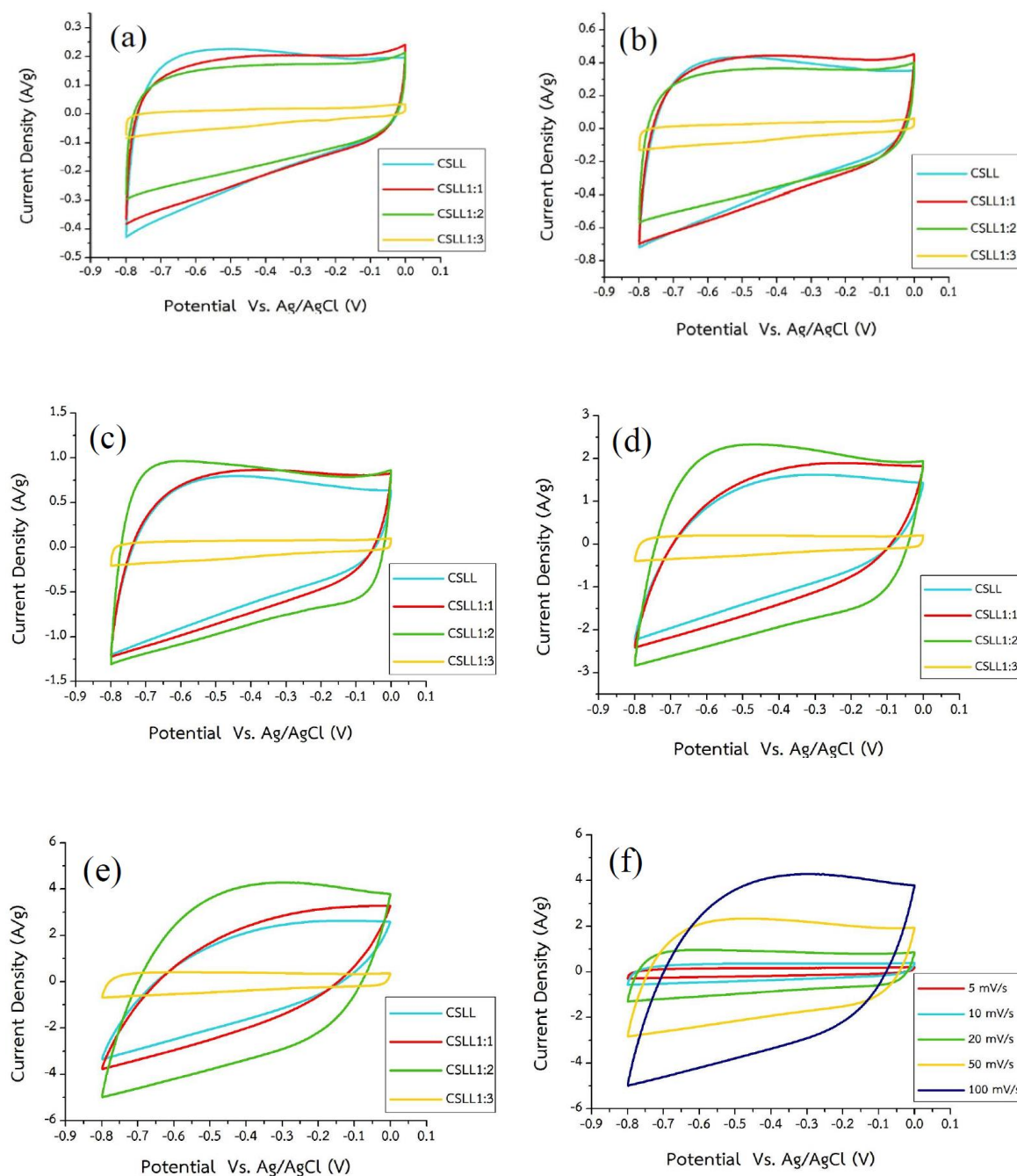


Fig. 5 Galvanostatic charge and discharge results of samples at the scan rate of (a) 1 mV s⁻¹, (b) 2 mV s⁻¹, and (c) 5 mV s⁻¹, respectively.

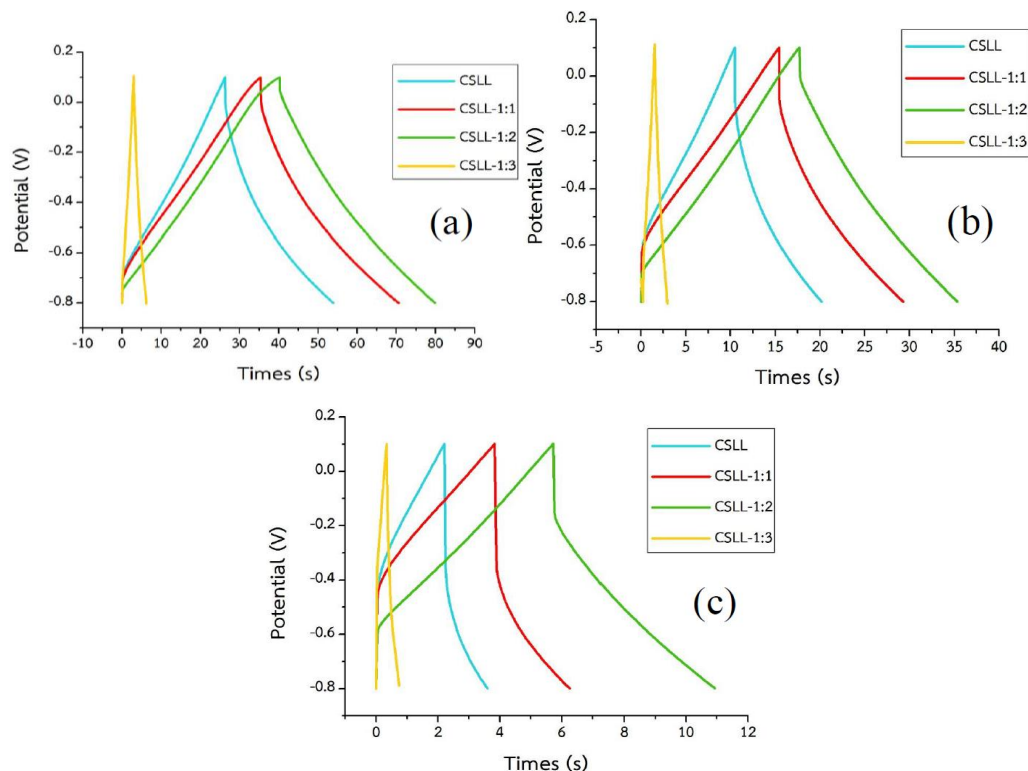


Fig. 6 Galvanostatic charge and discharge results of samples at the scan rate of (a) 1 mV s^{-1} , (b) 2 mV s^{-1} , (c) 5 mV s^{-1} , respectively.

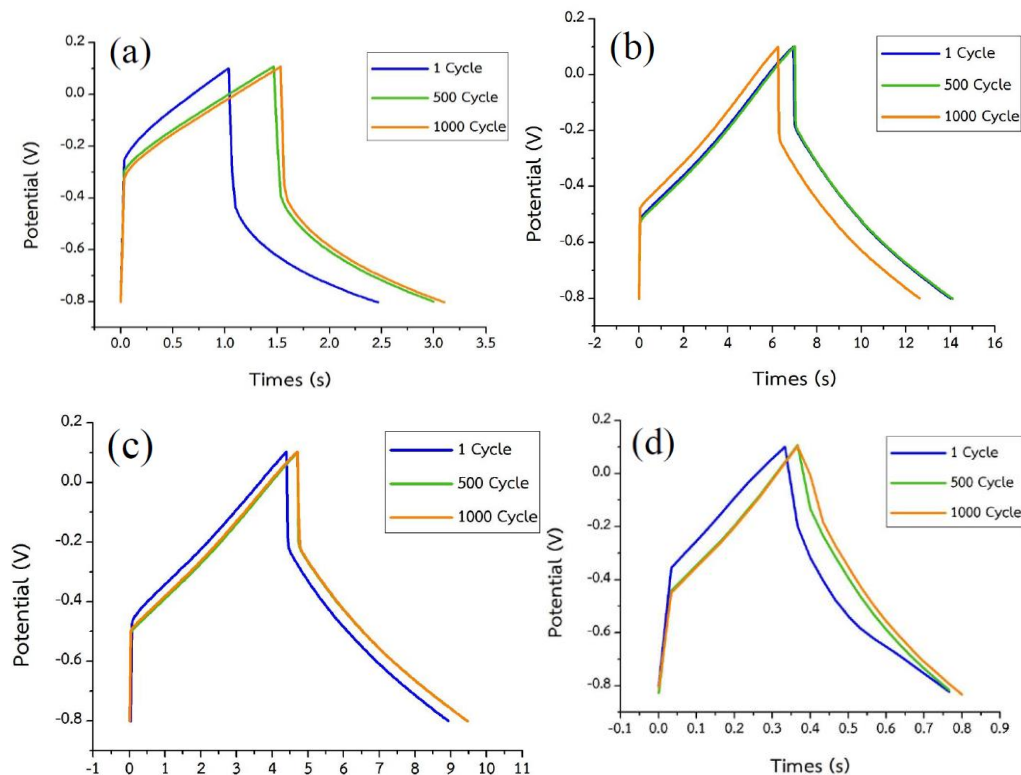


Fig. 7 Galvanostatic charge and discharge results of (a) CSLL, (b) CSLL-1 : 1, (c) CSLL-1 : 2, and (d) CSLL-1 : 3 samples, respectively at the scan rate of 5 mV s^{-1} .

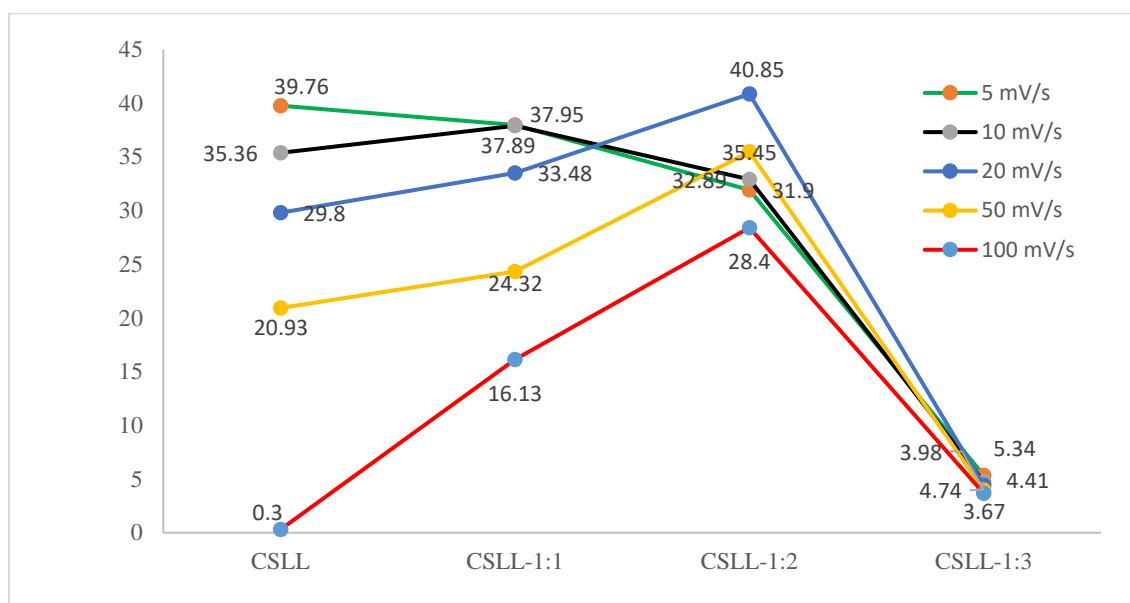


Fig. 8 The specific capacitance of samples at the scan rate of 5 mV s⁻¹, 10 mV s⁻¹, 20 mV s⁻¹, 50 mV s⁻¹, and 100 mV s⁻¹, respectively.

Finally, the AC electrodes were tested by charge – discharge cycle at 1,500 and 1,000 cycles found that the CSLL-1 : 1 and CSLL-1 : 2 electrodes represent discharge time longer than CSLL and CSLL-1 : 3 as shown in the Fig. 7 (a) – (d), respectively. the specific capacitance of samples was shown in Fig. 8 at the scan rate of 5 mV s⁻¹, 10 mV s⁻¹, 20 mV s⁻¹, 50 mV s⁻¹, and 100 mV s⁻¹ respectively. Especially, the specific capacitance has as high as 40.85 F g⁻¹ for CSLL-1 : 2 at scan rate of 20 mV s⁻¹. the performance level of the CSLL-1 : 2, was compared with other biomass carbon-based materials found that research of Qu et al. (2018) AC lotus leaf material has the specific capacitance of 379 F g⁻¹ and Zhang et al. (2017) AC bamboo material has the specific capacitance of 193 F g⁻¹. Although, the specific capacitance values were obtained from synthesis lotus leaf less more than the other research but experimental synthesis process non-complexed and low-cost materials

Conclusion

Fabrication of AC electrode synthesized from natural materials “sacred lotus leaf” for double layer supercapacitors was created with doping KOH powder in each the ratios. Ratios of AC (sacred lotus leaf, CSLL): KOH are CSLL, CSLL: KOH of 1 : 1, 1 : 2 and 1 : 3, respectively. Color characteristic of CSLL, CSLL-1 : 1 and CSLL-1 : 2 powder were exhibited the dark black and uniform dispersive. Crystal of AC powder was showed plane of carbon

obviously when KOH amount increase. Surface area of CSLL-1 : 2 powder was occurred high roughness and non-uniform effect to high surface area. Moreover, element compound of carbon in CSLL-1 : 2 sample is approximately 76 % by atomic which AC properties-like. Especially, the specific capacitance has as high as 40.85 F g⁻¹ for CSLL-1 : 2 at scan rate of 20 mV s⁻¹. Addition charge–discharge time, CSLL-1 : 2 electrodes can discharge longest times at 1,000 cycle. Thus, AC electrode was prepared from sacred lotus leaf which applied to the double layer supercapacitor.

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