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Agricultural Bio-waste-derived Hierarchical Porous Carbon Nanofiber for High-performance Supercapacitors

Journal:	*Advances in Natural Sciences: Nanoscience and Nanotechnology*
Manuscript ID	Draft
Manuscript Type:	Original Article
Date Submitted by the Author:	n/a
Complete List of Authors:	Taslim, Rika; State Islamic University Sultan Syarif Kasim, Department of Industrial Engineering Taer, Erman; University of Riau - Bina Widya Campus, Department of physics Halbi, Suryandri; State Islamic University Sultan Syarif Kasim, Department of Industrial Engineering Apriwandi, Apriwandi; University of Riau - Binawidja Campus, Department of Physics
Keywords:	Nanofiber, Porous carbon, electrode material, Supercapacitor
Classification numbers:	5.16, 5.18

Agricultural Bio-waste-derived Hierarchical Porous Carbon Nanofiber for High-performance Supercapacitors

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Abstract. Activated carbon-derived biomass materials widely used as energy storage supercapacitors have a hierarchical pore 3D structure, large specific surface area, controllable surface morphology, as well as an in-expensive and non-complex approach. Therefore, this study aims to develop a novel, simple, efficient, and low-cost method to prepare hierarchical porous carbon nanofiber derived from corn silks (CSAC) through a one-step carbonization-physical activation process. The carbon precursors were activated by KOH solution at a high pyrolysis temperature to prepare activated porous nanofibers as an electrode material for supercapacitors without using binders. This study focused on the effect of different activation temperatures of 600, 700, 800, and 900°C on the production of highly porous carbon nanofiber. An enhancement mechanism is proposed, which not only performed high nanofiber structures to possess the large specific active surface area to enhance energy density but also achieve micro-mesopores combination to realize fast ion-transport channels for boosting high power density. A maximum specific surface area of approximately 1096.951 m^2 g^{-1} was achieved by CSAC7. Furthermore, the electrochemical performance was evaluated using 1 M H_2SO_4 solution as an electrolyte through a novel two-electrode binder-free system. The electrode materials produced a maximum specific capacitance of 237 F g^{-1} at a current density of 1 A g^{-1}. These excellent characteristics show that the synthetic approach has a great potential for fabricating high-performance supercapacitors.

Keywords: Nanofiber, porous carbon, electrode material, supercapacitor.

Classification numbers: 5.16, 5.18

1. Introduction

Supercapacitors are globally considered as superior storage devices due to the higher energy and power density compared to batteries, capacitors, and fuel cells [1]. In addition, they function as suitable materials for electrical components, pulse laser systems, and other electronic devices [2]. Supercapacitors are also directly utilized or combined with batteries for certain electrical facilities, such as electric braking, starters, vehicle, and generators [3]. The electric double-layer capacitors (EDLC) are considered to be the best type due to the variable and adaptable active materials, low cost, abundant availability, superior material properties, maximum electrolyte flow rate, high operating temperature and conductivity, compatible with a variety of low and high current components, environmentally friendly, and relatively safer [4]. However, supercapacitors still have various challenges such as the relatively low energy compared to the high power density, expensive active materials, and complex synthetic methods. In different studies, the energy density of supercapacitors has been increased with various approaches, but with a reduced power density. The main key to improving the performance of supercapacitors is the active electrode material, followed by the electrolyte and separator [5]. Three groups of active electrode materials have been reported including conduction polymers, metal oxides, and carbon [6]. Conducting polymer materials and metal oxides were used to successfully increase the energy density up to 200 Wh kg^{-1} [7]. This is several times greater than the result from other studies in the last decade. However, both materials require complex, complicated, corrosive, and toxic instruments, hence, they are not recommended for environmentally friendly mass production. Therefore, activated carbon from biomass and bio-organic waste is more promising as it has superior and attractive characteristics including abundant availability, renewability, easy fabrication, and low cost [8,9]. Activated carbon materials also have a good performance in increasing and maintaining energy density, although the energy density produced is still relatively low

compared to metal oxides and conduction polymers. [10,11] Additionally, biomass precursors rich in structurally diverse porosity and significant heteroatoms provide a large number of active ion contacts and facilitate the transport of electrolytic ion charges in various pores including micro-, meso-, and macro- leading to a high performance of energy and balanced power density [12]. Moreover, the 3D hierarchical pore structure obtained from the biomass-derived activated carbon has been shown to increase the energy density up to 5 times coupled with a high power density [13]. Hierarchical porous carbon consisting of micro-, meso-, and macropores are obtained from various biomass precursors such as orange peel [14], pineapple leaves [15], rice husk [16], ginger waste [17], peanut shells [18], bamboo stem [19], and jujube fruit [20]. The peanut shell agricultural biomass produced activated carbon having a 3D hierarchical pore structure as well as abundant micropores and mesopores with surface area=2014.6 m^2 g^{-1}. Activated carbon is prepared by a new and low-cost method using $ZnCl_2/CO_2$ activation which produced a specific capacitance of 310 F g^{-1} in a 3-electrode system [21]. Shang *et al.,* (2021) obtained activated carbon from chitin (*Portunus trituberculatus* Crab) waste [22], which was converted into hierarchical porous carbon through ZIF-8 nanoparticles bio-template followed by high-temperature carbonization, leading to a specific capacitance of 182 F g^{-1}. Similar results were also obtained from different biomass precursors such as cauliflower which produced a unique hierarchical pore structure using KOH activation at 700°C high-temperature simple which possessed a high surface area of 2061 m^2 g^{-1} [23]. However, the activated carbons obtained from the above precursors are prepared in powder form, hence, they require synthetic binders and insulators to test the electrochemical properties. Although the counter electrodes used are good conduction materials, it is still considered to reduce the efficiency. Also, the template/bio-template method often used to ascertain the 3D pore structure is a relatively risky technique because the final step requires more treatment to remove the by-products. On the other hand,

corn silk agricultural biomass has high potential as a hierarchical porous carbon precursor. The basic fibrous structure allows the discovery of dense nanofibers on activated carbon, thereby adding to its renewability. Although the use of corn silk as a starting material for activated carbon in supercapacitors has been previously reported, the method of preparation using binders through a relatively complicated technique is considered to limit its novelty. Therefore, this study aims to prepare binder-free activated carbon through a novel one-step activation of KOH/CO_2. The chosen strategy has been shown to produce higher capacitance compared to Mitravinda *et al.* (2018) [24] which used different methods and possesses higher energy density compared to a study by Zhou *et al.* (2020) [25]. In addition, we selected H_2SO_4 aqueous electrolytes because of their advantages including the highest ionic concentration, highest conductivity, and low viscosity compared to other aqueous electrolytes. In result, the optimum specific capacitance of activated carbon obtained is 237 F g^{-1} with a maximum energy density of 18.19 Wh kg^{-1} in an aqueous electrolyte of 1 M H_2SO_4.

2. Materials and methods

2.1. Materials

The corn silks (CS) were initially obtained from the traditional plantations of the Kampar community, Riau and then cut into small pieces within a range of 2 cm, cleaned, and sundried for 48 hours to obtain dry samples. Furthermore, the samples were pre-carbonized and crushed using a crusher machine to obtain dried corn silk powder. The KOH activating agent, HCl, and aqueous electrolyte H_2SO_4 were obtained from Merck KGaA, 4271 Darmstadt, Germany, and Panreac Quimica Sau, Espana, while deionized water (DI) as a material for neutralizing samples was made on a lab scale. The organic separator from the duck eggshell membrane was extracted with 1 M HCl solution.

2.2. Synthesis of carbon pellets

30 g of dried corn silks powder was dissolved with a 0.5 m/L KOH solution on a hotplate at 300 rpm for 2 hours. The sample was precipitated and dried in an oven at 110 °C, while the corn silk powder was converted into pellets using a hydraulic press without using any adhesive material, hence, the adhesive properties of the pure powder were derived originally from the sample. A total of 20 pellets samples were prepared for pyrolysis using carbonization in the N_2 gas environment and physical activation with CO_2 in the furnace tube. In addition, the pyrolysis process began from a temperature of 30^o C to 600 °C in N_2 gas and then increased to 900 °C in a CO_2 environment. Four different carbonization temperatures were applied namely 600 °C, 700 °C, 800 °C, and 900 °C. Moreover, to facilitate data interpretation, samples were labeled CSAC-x, CSAC was corn silk activated carbon (CSAC), while x was the number 6, 7, 8, and 9 indicating carbonization at the four temperatures. The pelleted carbon samples were neutralized using DI water.

2.3. Characterization of materials

The carbon pellets' density was evaluated based on changes in mass, thickness, and diameter during the pyrolysis process through standard equations. Furthermore, the amorphous properties of the samples were characterized using the XRD (X-ray diffraction) technique at an angle range of 5-60 ° in a Cu Kα radiation source (Phillips expert powder instrument). Surface morphology and variation of sample elements were also examined through the SEM-EDS (Scanning electron microscopy-energy dispersive spectroscopy) technique using the JEOL-JSM-LA-3600 instrument at a voltage of 15Kv. Additionally, the specific surface area and pore diversity were determined through the N_2 gas absorption technique and evaluated using BET and BJH calculations.

2.4. Supercapacitor electrochemical performance

The electrochemical properties of the supercapacitor were evaluated in two-electrode configurations. The supercapacitor cells were made in the form of sandwich layers consisting

of a cell body made of acrylic, stainless steel current collector, pelleted carbon electrodes, and an organic separator from the duck eggshell membrane. The cyclic voltammetry (CV) and galvanostatic charge-discharge (GCD) methods are techniques commonly used in evaluating specific capacitance, as well as energy and power density. CV was evaluated using the CV-UR Rad-Er 1380 instrument at a constant voltage range of 0-1.0V at a scan rate of 1 mV s^{-1}, while GCD used the 2018 CD-UR Rad-Er at a fixed current of 1 A, both instruments were calibrated with a mean error of ± 6.01%.

3. Results and discussions

Crystal phase change properties of corn silk-based hierarchical porous carbon were reviewed using the powder X-ray diffraction (XRD) method. The XRD pattern with different physical activation temperatures particularly between 700 and 900 °C, are shown in Figure 1.

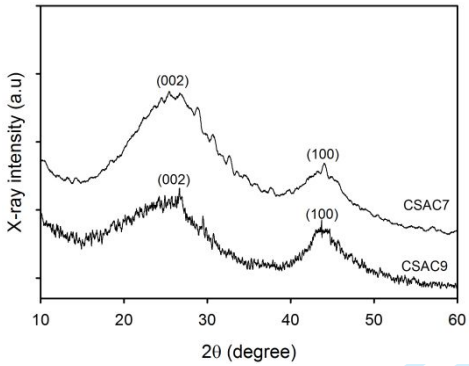

Figure 1. The XRD pattern of CSAC7 and CSAC9

The CSAC7 display two clearly confirmed broad peaks at 2θ angles 24.34° and 43.92° which correspond to the scattering planes 002 and 100. These characteristics show that the structure of the turbostratic/disturbed carbon has a high amorphous nature [26]. On the other hand, CSAC9 performed low broad peaks at angles of 2θ 24.86° and 44.13° indicating the amorphous nature degraded towards graphitization due to high-temperature treatment. Furthermore, the most obvious broad peaks in the 002 reflection plane ranging between angle 24.34°-24.86° indicate a random aromatic sheet structure, while the 100 reflection plane from 43.92°- 44.13° confirms the type of carbon that is close to the graphite structure, in this case,

the attenuation of the graphite structure towards the formation of a disturbed carbon structure [27,28]. The reflection planes 002 and 100 were attenuated from sample CSAC7 to CSAC9 as the high-temperature pyrolysis process from 700 to 900 °C tends to increase the random disturbed structure of activated carbon, thereby initiating a better amorphous formation [29]. This property is important for improving the good hierarchical pore structures in carbon samples. The morphological structure of activated carbon-based corn silk waste was evaluated using the SEM method at a voltage of 15 kV. Figure 2 shows the morphological structure with different physical activation temperatures of 600, 700, 800, and 900 °C. In general, the outer surface structure of the sample is dominated by bulk materials, aggregation and clumps of large wrinkled particles, while some parts display a different inner surface structure for each temperature. At 600 °C (Figure 2a), the activated carbon consisted of particle aggregations with surface wrinkles. Additionally, a larger zoom in Figure 3b shows that the sample surface is relatively flat in each aggregation without any obvious pores. Furthermore, physical activation at a higher temperature above 700 °C led to a better decomposition into the basic components including hemicellulose, cellulose, lignin compared to 600°C. This indicates a relatively different surface morphology as shown in Figure 2c-d. Figure 3c shows the relatively dominant pore holes structure in the 64-7214 nm range. The diverse pore structure including mesopores and macropores were confirmed at a larger magnification area as shown in Figure 2d. Mesopores have a size ranging from 18-48 nm, while macropores are in the range of 73-136 nm. However, micropore structures were not found at this magnification. The combination of the mesoporous and macropore structures is very advantageous in the smooth ion diffusion process as well as its absorption direction in the form of 3D to increase and maintain the high energy and power density of the supercapacitor respectively [10,30]. This analysis was further confirmed using cyclic voltammetry.

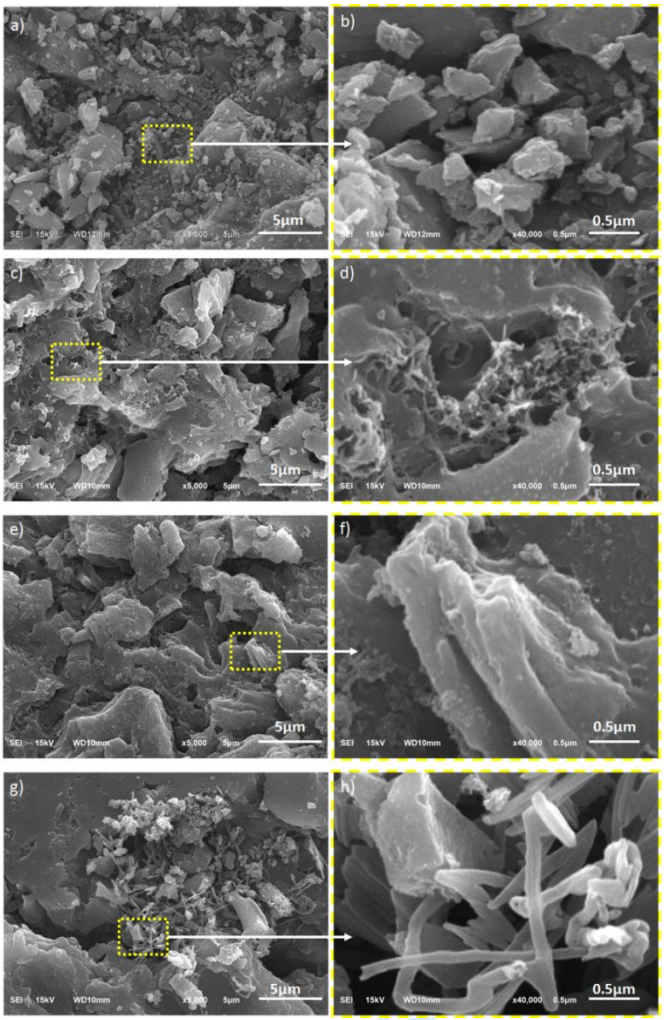

Figure 2. The SEM macrograph of CSAC6 in magnification of (a) 5000x, (b) 40000x, CSAC7 in magnification of (c) 5000x, (d) 40000x, CSAC8 in magnification of (e) 5000x, (f) 40000x, CSAC9 in magnification of (g) 5000x, (h) 40000x

An increase in the physical activation temperature of 800 °C in the CSAC8 sample shows a surface morphology filled with macropores which have a relatively large size less than 300 nm as shown in Figure 2e. In addition, Figure 2f shows a distinct morphology in which clustered tubular-rod-like structures are found. This is because the higher temperature porosity allows the breakdown of the main carbon structure derived from cellulose and lignin drastically. The rod shape is contributed from the basic structure of cellulose while the outer tubular shape is attributed to lignin [31]. This analysis has been reported in previous studies with different biomass precursors such as cassava petiole [32] and cacao shells [33].

Moreover, the temperature of 900°C maximized the basic decomposition process of cellulose and lignin to form a clear surface morphology of carbon fibers, as shown in Figure 2g-h. The diameter of the obtained nanofibers varied relatively in the range of 76 nm to 127 nm. The predominant nanofiber fiber allows well-connected pores in a relatively small size range [34]. These characteristics improve the high performance of supercapacitors. The elemental composition of porous activated carbon samples at activation temperatures of 700 °C and 900 °C for CSAC7 and CSAC9 was examined using energy dispersive spectroscopy (EDS) techniques in the energy range of 0-20 keV. Table 1 shows the elemental composition of the CSAC7 and CSAC9 samples in detail. Elemental carbon (C) has the highest percentage of approximately 83.7485-85.2755% followed by oxygen 8.0333-8.6075%, silicon (Si) 3.2471-3.1535%, potassium (K) 1.6359- 3.9428%, magnesium (Mg) 0.8317-0.7930%, and aluminum (Al) 0.4022-0.3290%.

Table 2. The elemental composition of CSAC7 and CSAC9

Element	CSCA7 (%)	CSAC9 (%)
Carbon (C)	85.2755	83.7485
Oxygen (O)	8.6075	8.0333
Silicon (Si)	3.2471	3.1535
Potassium (K)	1.6359	3.9428
Magnesium (Mg)	0.8317	0.7930
Aluminium (Al)	0.4022	0.3290

High-temperature pyrolysis of 700 C for CSAC7 showed the highest carbon composition of 85.2755%. This property is considered to increase the capacitive properties of the electrode material. Furthermore, the highest elemental oxygen was also found in the CSAC7 sample which contributed to the wettability of the material thereby initiating the pseudocapacitance effect [35]. An increase in the activation temperature up to 900C for CSAC9 reduced the percentage of carbon and oxygen in the sample. This is because the activation of high temperatures evaporates the constituent elements of the material in the

form of more H_2O and CO_2. Moreover, other elements such as Silicon, Potassium, Magnesium, and Aluminum are obtained in relatively low amounts from basic sources of biomass that have not been completely evaporated [36].

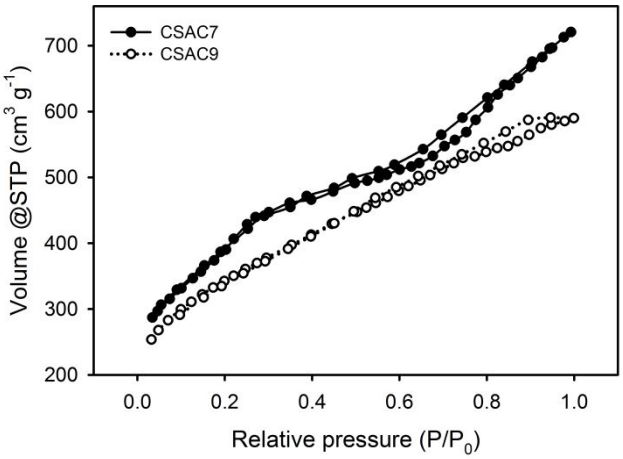

Figure 3. Nitrogen adsorption-desorption isotherms of CSAC7 and CSAC9

The analysis of nitrogen adsorption/desorption isotherms of corn silk-based hierarchical porous activated carbon at different pyrolysis temperatures are needed to evaluate the impact of pore structure and porosity properties. Figure 3 shows the N_2 adsorption-desorption isotherm curve of both samples, CSAC7 had significant N_2 adsorption at a relative pressure of $0.0<P/P_0<0.2$, thereby confirming the relatively large number of micropores. Furthermore, the H4 type hysteresis loop was clearly shown over a relatively long relative pressure range of $0.2<P/P_0<0.9$ indicating uniform mesoporous characteristics at CSAC7 as presented in Table 2. The tail at high-pressure P/P0>0.9 shows the presence of macropores, but the addition of the physical activation temperature up to 900°C in CSAC9 showed a different N_2 isotherm absorption compared to CSAC7. Similar results were further confirmed through the N_2 gas absorption profile presented in Figure 3. The KOH reaction at a carbonization temperature of 700 °C showed the highest reduction in pellet density of 29.85% indicating that the CSAC7 sample has abundant pores for high performance of supercapacitor electrodes. The N_2 adsorption-desorption curve of CSAC9 shows a relatively linearly

increasing adsorption which is not too high at low pressure, thereby indicating the presence of low micropores. In addition, the H4 type hysteresis loop is not formed completely at a relatively short pressure range of P/P_0 = 0.6 to 0.95 which is mainly due to the pores growing towards the larger one and is dominated by narrow bottle neck-like types with a larger inner surface [37] as shown in Table 2.

Table 2. Specific surface area, pore volume and average pores size of CSAC7 and CSAC9

Sample	S_{BET} ($m^2\ g^{-1}$)	S_{micro} ($m^2\ g^{-1}$)	S_{meso} ($m^2\ g^{-1}$)	V_{tot} ($cm^3\ g^{-1}$)	V_{micro} ($cm^3\ g^{-1}$)	V_{meso} ($cm^3 g^{-1}$)	D_{aver} (nm)
CSAC7	1096.951	531.087	565.864	1.115	0.314	0.801	4.06
CSAC9	744.818	423.360	321.458	0.9126	0.4476	0.465	3.15

The pore size distribution as presented in Figure 4 show that both samples have a combination of micropores and macropores. This result is relatively consistent with SEM which also confirms the hierarchical porous structure. CSAC7 has a structure of hierarchically connected 3D pores leading to the presence of mesopores with a peak at 3.89 nm. Furthermore, the chemical impregnation of KOH at 700°C high-temperature pyrolysis allows the precursor to produce a good combination of micropores and mesopores to improve the performance of the base electrode.

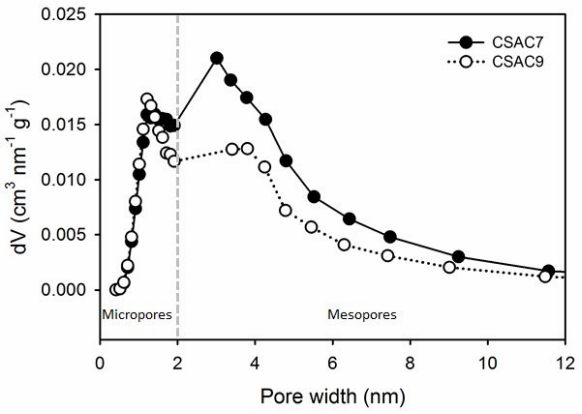

Figure 4. Pore sizes profile of CSAC7 and CSAC9

The microporous and mesoporous percentages of CSAC7 were 48.41% and 51.59% with a high surface area of 1096.951 m^2 g^{-1}. The high surface area coupled with the combination of the hierarchically connected pores is believed to improve the electrochemical properties of supercapacitors [38]. In addition, the high mesoporous volume fraction potentially facilitates rapid ion transport and act as an electrolyte ion reservoir, thereby reducing the ion diffusion distance to the carbon surface, ensuring high rate capability and high power density. Moreover, CSAC9 displays a relatively similar pore size distribution with CSAC7 but is dominated by micropores with a percentage of 56.84%. KOH impregnation at higher temperatures up to 900°C allows degradation of carbon aggregation and erodes the main pore walls, thereby creating tubular and nanofiber structures. This simultaneously reduces the specific surface area of CSAC9 to 744.818 m^2 g^{-1} with an average diameter of 3.15 nm as shown in Table 2. Nevertheless, the developed porosity properties of carbon materials are important for electrolyte ions' rapid transport and penetration [39]. These results are consistent with the SEM and density data.

The main parameters used to evaluate the electrochemical properties of hierarchical porous activated carbon include high specific capacitance, increased energy and fixed power density, as well as retention capacitance. Based on the N_2 gas absorption analysis, the corn silk-based activated carbon has a combination of micropores and mesopores that is able to accommodate a relatively high charge of active ions and facilitate diffusion without obstruction at the electrode/electrolyte interface. The electrochemical performance of CSAC6, CSAC7, CSAC8, and CSAC9 based on different physical activation temperatures was evaluated with a two-electrode configuration in an aqueous electrolyte using cyclic voltammetry and galvanostatic charge-discharge techniques. Cyclic voltammetry (CV) curves at 600, 700, 800, and 900 °C are shown in Figure 5. The CV profile exhibits a quasi-

rectangular shape, indicating the typical behavior of electrochemical double-layer type supercapacitors (EDLC) in an aqueous electrolytic system at a scan rate of 1 mV s^{-1}.

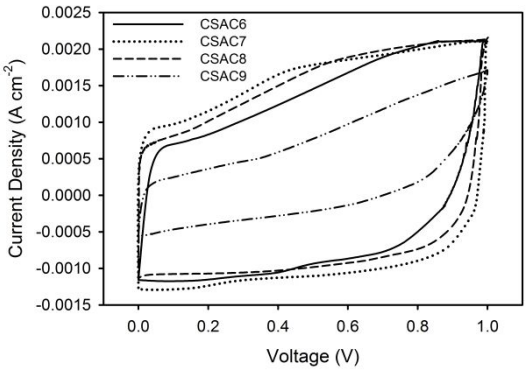

Figure 5. CV curve of CSAC6, CSAC7, CSAC8, and CSAC9

In addition, the pseudocapacitance property was found to be relatively low as indicated by a spike in current density of the voltage ranging from 0.3-0.6V. This property is a contribution of the heteroatom and wettability properties of elemental oxygen in the sample. In general, all samples exhibited similar electrochemical properties. Using standard equations, the specific capacitances of CSAC6, CSAC7, CSAC8, and CSAC9 was evaluated to be 184, 211, 194, and 143 F g^{-1}, respectively. An increase in the physical activation temperature improved the capacitive properties of the supercapacitor by 211 F g^{-1}. This is because the higher temperature of CSAC7 showed a relatively rich surface structure of mesopores and macropores, which provides a high surface area and diffusion path of ionic charge in all directions, thereby facilitating the high capacitive properties. This analysis is consistent with SEM results and N_2 gas absorption in Figures 2 and 3. At 800 °C, the surface structure of the capacitive properties was reduced to 194 F g^{-1}. This is because the increase in the physical activation temperature in the CSAC8 sample shows a surface morphology filled with macropores with a relatively large size less than 300 nm, as shown in Figure 2e. In addition, the morphology of the clustered tubular rod-like structure allows for fractures that narrow the ion transport pathways on the electrode surface. A further temperature is increased up to 900 °C for CSAC9 allows the erosion and breakdown of the particle walls of carbon aggregation

and rod-like structures to cover the existing pores, this reduced the capacitive properties to 143 F g^{-1}. Although the pore structure displays relatively dense nanofibers, as shown in Figure 2h, it is unable to maintain the 3D hierarchical pore distribution of micropores and mesopores thereby reducing the electrochemical properties.

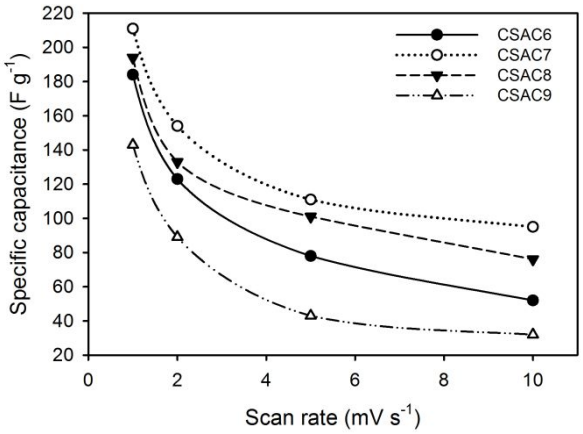

Figure 6. The curve of specific capacitance vs. scan rate of CSAC6, CSAC7, CSAC8, and CSAC9

The specific capacitances of CSAC6, CSAC7, CSAC8, and CSAC9 were also evaluated at different scan rates from 1 mV s^{-1} to 10 mV s^{-1}, as shown in Figure 6. The activated carbon which maintained the highest specific capacitance of 45% was CSAC7, followed by CSAC8, CSAC6, and CSAC9 with values of 39%, 28%, and 23%, respectively. The galvanostatic charge-discharge (GCD) curves of symmetric cells CSAC6, CSAC7, CSAC8, and CSAC9 at a current density of 1 A g^{-1} are shown in Figure 7a. In general, the GCD curve displays a normal profile for the EDLC type with a slightly faint iR drop. According to the GCD profile, the specific capacitances produced at CSAC6, CSAC7, CSAC8, and CSAC9 were 152, 237, 174 and 124 F g^{-1}, respectively, in the electrolyte of 1 M H_2SO_4. Higher activation temperature from 600°C to 700°C confirmed micro-mesopores combinations of 48.41% and 51.59% displayed the highest electrochemical properties from 152 F g^{-1} to 237 F g^{-1}. These results correlate with SEM, N_2 gas adsorption/desorption, and CV analysis. The high activation temperature of from 700°C to 900°C decreased the capacitive value of the carbon

electrode up to 124 F g^{-1} for the CSAC9 sample. This is associated with the collapse of the pore walls and the erosion of the carbon framework structure to cover the active site of the ionic charge below [37].

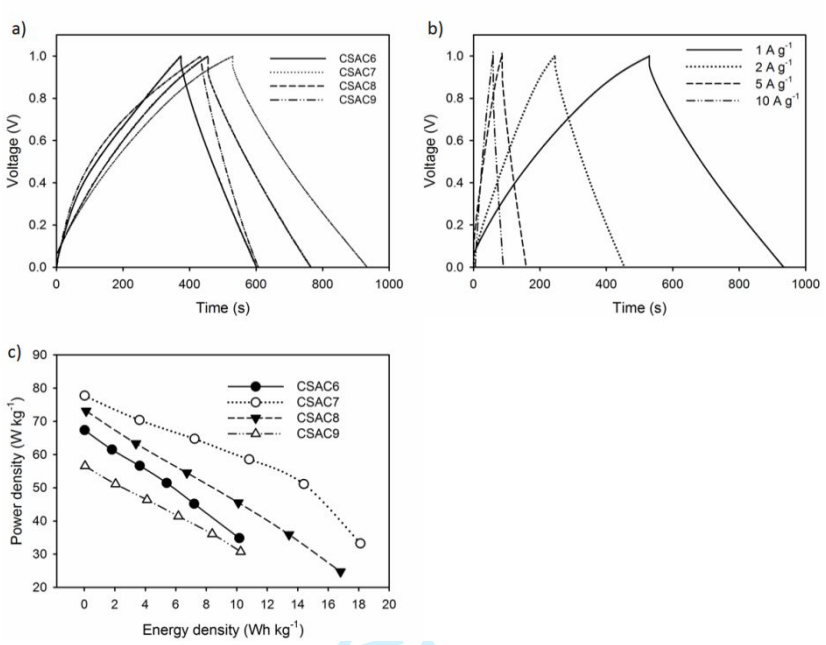

Figure 7. (a) GCD curve of CSAC6, CSAC7, CSAC8, and CSAC9, (b) GCD curve at different current density of CSAC7, and (c) Ragone plot of CSAC6, CSAC7, CSAC8, and CSAC9 in 1 M H_2SO_4 electrolyte

Furthermore, the resistance in CSAC7 was lower at 0.0025Ω due to the relatively high dominance of the mesopores which improved the diffusion of ionic charges in all directions compared to CSAC6, CSAC8, and CSAC9 which has a higher resistance value of 0.012, 0.27, and 0.034 Ω, respectively. In addition, the GCD profile of CSAC7 was reviewed at different current density of 1, 2, 5, and 10 A g^{-1}, as shown in Figure 7b. It can be noted that the GCD curves of CSAC7 still maintain primary symmetric triangle shape at 10 A g^{-1} suggesting stable double-layer capacitor behaviors, fast ion response, and low internal resistance in EDL processes [42]. At 1 A g^{-1}, CSAC7 sample delivers a specific capacitance of 237 F g^{-1} and it still maintains 210 F g^{-1} at 10 A g^{-1}, initiating a high-rate performance of 88.6%. To compare the performance of both samples in-depth, Ragone plots were used to

evaluate the energy and power density which are calculated using standard formulas as shown in Figure 7c. Symmetrical supercapacitors CSAC6, CSAC7, CSAC8 and CSAC9 assembled with an aqueous electrolyte of 1M H_2SO_4 in an organic separator had energy densities of 10.1712, 18.1081, 16.8049 and 10.2596 Wh kg^{-1} as well as power densities of 67.3793, 77.74, 73.1373 and 56.5421 W kg^{-1}, respectively. The high electrochemical capability of CSAC7 is due to the 3D hierarchical porous structure and the specific surface area which provides a short and accessible ion transfer area in all directions for ionic charges to diffuse at the electrode/electrolyte interface.

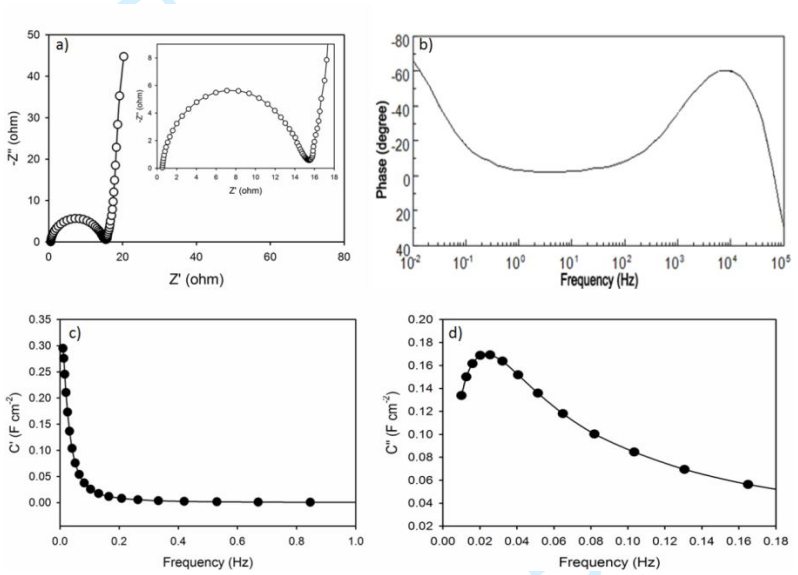

Figure 8. (a) Nyquist plot, (b) Bode phase angle plot, (c) real capacitance (C') vs. frequency plot, and (d) imaginer capacitance (C") vs. frequency plot of CSAC7 symmetrical devices

The high electrochemical confirmation of CSAC7 was reviewed in depth via a Nyquist Plot with a limited diffusion area in the frequency range of 0.01 Hz to 100,000 Hz, as shown in Figure 8a. Horizontal translation along the Z' axis confirms a relatively low Rs 0.87 Ωcm^2 indicating a good ions adsorption and desorption process at the electrode-electrolyte interface. Furthermore, the RCT (12.3 Ωcm^2) interpreted at the semicircular position confirmed the fast charge transfer kinetically as a result of the current passing through the

electrode forming an electric layer. This is due to the hierarchical pores structure with a combination of micro, meso, and macropores initiates the active site which is followed by the ability of ion diffusion in all directions without any obstacles. In addition, the straight line that tends to be perpendicular at low frequencies confirms the pure capacitive and high ion transfer rate at the CSAC7 electrode. The Bode phase angle plot of CSAC7 was also evaluated as shown in Figure 8b. The phase angle at low frequencies reveals specific information about the ability of the electrode to generate an electric double layer. CSAC7 electrode displays a phase angle close to -90° indicating that the normal dielectric layer of the EDLC version is accompanied by impaired ion degradation due to self-heteroatom doping. In addition, the Bode phase angle plot provides further information for the characteristics of the conductive material through analysis of the real capacitance (C') and the imaginary capacitance (C”) with respect to frequency, as shown in Figures 8b, and 8c. The C' values are weak in the lower frequency and higher frequency regions, the C' values show less frequency dependence, indicating a purely resistive behavior in CSAC7. Moreover, the value of the imaginary capacitance C” at the peak frequency confirms a relaxation time of 9.25 seconds, indicating that the symmetrical CSAC7 device acts as a pure capacitor. This value indicates a measure of how quickly the stored energy of the device can be efficiently distributed. For comparison, the obtained carbon nanofiber-based on corn silk waste showed higher electrochemical properties than the carbon nanofiber-based on bacterial cellulose and tofu dregs obtained through chemical activation of KOH and H_3PO_4 [40,43]. In addition, although polymer-based carbon nanofibers exhibit superior specific capacitance, our study was able to produce higher energy densities than the previously reported as confirmed in referee [42]. This is due to the combination of nanofiber and micro-mesoporous structures allowing the provision of high active sites and ion-transport channels. Moreover, the binder-free electrode design supports the high electrochemical performance of the supercapacitor.

4. Conclusion

Based on the results, hierarchically porous carbon nanofiber was successfully obtained from corn silk using a low-cost, simple, and sustainable technique without a binding material for a high energy density of symmetric supercapacitors. KOH activation using one-step integrated pyrolysis possessed excellent material properties including framework structure, porosity, wettability, and amorphousibilty. Furthermore, different physical activation temperatures display distinctive pores structure with individual advantages. At 700 ^{o}C, a 3D hierarchical pore structure was displayed followed by a high surface area leading to the formation of an ideal combination of micro and mesopores for enhancing the high performance of the electrode material. Using a two-electrode binder-free system, CSAC7 showed the best electrochemical properties with a specific capacitance of 237 F g^{-1} in a 1 M H_2SO_4 aqueous electrolyte. Hence, the maximum energy density of 18.19 Wh kg^{-1} and power density of 77.74 W kg^{-1} at current density of 1 A g^{-1}. This result shows that the approach applied in this study confirmed the potential of corn silk as an activated carbon based material with high properties.

Acknowledgement

This study was funded by the collaborative grants between universities, State Islamic University of Sultan Syarif Kasim Riau with contract No. 873/Un.04/L. 1/TL.01/03/2022.

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Gmail **Rika Taslim <rikataslim@gmail.com>**

[ANSN-2022-0119] - Decision on Manuscript: MAJOR REVISION

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NQL Editor-in-Chief <onbehalfof@manuscriptcentral.com> Sat, Aug 6, 2022 at 8:17 PM
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06-Aug-2022

Dear Dr. Taslim:

Manuscript ID ANSN-2022-0119 entitled "Agricultural Bio-waste-derived Hierarchical Porous Carbon Nanofiber for High-performance Supercapacitors" which you submitted to the Advances in Natural Sciences: Nanoscience and Nanotechnology (ANSN), has been reviewed. The comments of the Associate Editor and Reviewers are included at the bottom of this letter.

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https://doi.org/10.1149/MA2020-01271893mtgabs
However, total number of the references in a regular article published in the ANSN should be less than 40. Therefore, only reasonably appropriate references should be selected.

Reviewers' Comments to Author:

Reviewer: 1

Comments to the Author
The authors here prepared a kind of biomass-derived carbon (biocarbon) [Ref. 1-2] and studied its application in EDLC. The method and application here referred to are routine, however, the carbon precursor (corn silk) and the activation agent (KOH/CO2 compared to single KOH) as well as the eggshell inner membrane separator is relatively new and of some interest [Ref. 3-9], which may add a member into the biocarbon family for EDLC application. Thus, after addressing the following concerns it may be published in the Journal.
1) The title may be improved to be specific by including the carbon precursor "corn silk", and the claimed "Hierarchical Porous Carbon Nanofiber" may not be verified by the characterizations, please consider improvement/alteration.
2) About the "duck eggshell membrane", please give some content on the advantages of this kind of separator. (https://www.exploratorium.edu/cooking/eggs/eggcomposition.html)
3) It is depicted that "dried corn silks powder was dissolved with a 0.5 m/L KOH solution," "The sample was precipitated and dried," is it more appropriate if replaced by "dispersed" rather than "dissolved", and by "precipitated and filtered", please specify it.
4) The structures in Fig 2 by SEM seem broken and powdered, any reasons? And why are there some nanofibers in the biocarbon, e.g., Fig 2h, please further discuss it?
5) Why the corn silk calcinated at 700 oC showed the highest capacitance (Fig. 7), please give some further explanations.
6) Cycling performance (and coulombic efficiency) of the EDLC may be provided for better understanding, e.g., in Fig 7.
7) Some grammatical and format issues, e.g., "15Kv", "002 reflection plane", "100 reflection plane","P/P0>0.9".
8) Some more related references may be cited for better understanding.

Ref.
1. Resol–Vegetable Fibers Composites, Materials Research Foundations, Vol. 122, pp 154-198, 2022. DOI: https://doi.org/10.21741/9781644901854-7
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Reviewer: 2

Comments to the Author
In this paper, the author has obtained hierarchically porous carbon nanofiber successfully from corn silk using a low-cost, simple, and sustainable technique without a binding material for a high energy density of symmetric supercapacitors. When the pyrolysis temperature was 700 oC, a 3D hierarchical pore structure was displayed followed by a high surface area leading to the formation of an ideal combination of micro and mesopores for

enhancing the high performance of the electrode material. What's more, CSAC7 showed the best electrochemical properties with a specific capacitance of 237 F g-1 in a 1 M H2SO4 aqueous electrolyte. I would like to recommend it for publication after minor revisions. The detailed comments are as follows:
1. Since the material studied is porous carbon nanofiber, Raman characterization is necessary. Please add relevant characterization.
2. Please add the XRD spectrum of CSAC6 and CSAC8.
3. The descriptions of Fig. 3b and Fig. 3c appear in the paper, but relevant figures are missing. Please add these figures.
4. Please check the whole manuscript carefully, for example, the "physical activation at a higher temperature above 700 °C led to a better decomposition into the basic components including hemicellulose, cellulose, lignin compared to 600oC" on page 7 should be changed to "physical activation at a higher temperature above 700 °C led to a better decomposition into the basic components including hemicellulose, cellulose, lignin compared to 600 oC".
5. Some important papers should be cited in this paper:
Natl. Sci. Rev., 2020, 7, 305-314.
Small Methods, 2021, doi: 10.1002/smtd.202101070.
Angew. Chem. Int. Ed., 2021, 60, 25318.
EnergyChem, 2019, 1, 100016.

Reviewer: 3

Comments to the Author
This is an interesting article and worth publishable in Advances in Natural Sciences: Nanoscience and Nanotechnology. However, I suggest a revision addressing following queries prior to consideration for publication.
1. Four samples were obtained but only two samples were evaluated, where are the others samples?
2. Why physical activation and chemical activation were used simultaneously? As you know, only chemical activation can also give a large BET surface area, I can't find any necessary explanation.
3. The role of physical activation and chemical activation should be enhanced.
4. In Figure 2, only a few amount of Nanofiber were observed, i.e., morphology of samples is mainly irregular granular not the Nanofiber, so the title should be modified.
5. What is the mass loading in an electrode?
6. More structural characterization, like XPS, Raman, (HR)TEM etc are requied to understand the better electrochemical performance.
7. The energy densities have been overestimated significantly. For example, for a supercapacitor device with the cell voltage is 3.0 V, the specific capacitance is 200 F/g (the corresponding cell capacitance is 50 F/g), the energy density is calculated to be 62.5 Wh/kg. For your device, however, the energy density can reach to 316.77 Wh/kg with a narrow working potential window of 1 V. It seems the unit was not converted.
8. I recommend the authors to refer others reported electrode materials in aqueous/nonaqueous electrolyte especially in the last three years.
9. The use of porous carbon materials for energy devices represents a topic highly in vogue nowadays, these relevant publications in the field are worth to be discussed: Electrochimica Acta 379 (2021) 138170; Carbon 141 (2019) 40-49.

Reviewer: 4

Comments to the Author
The analyse for the structure-activity relationship is the key in the manuscript, however, only the samples at 700 and 900 ℃ were characterized, which is inadequate. Therefore, the samples at 600 and 800 ℃ should be characterized. In addition, the chemical environment on the surface of as-prepared carbon is very important for the capacitance properties, therefore, the FTIR or XPS analyse should be provided in this manuscript. Finally, the TEM images of carbon should be provided to observe the porous structure.
In conclusion, I recommend that the manuscript can not be considered because of the obvious defect in the characterization.

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Your manuscript, referenced above, has been reviewed for publication in the ANSN:
Comments from the reviewer(s) are included below.
We conclude that further review and revision may be necessary.

Reviewer 1 has recommended that your manuscript must be minor revised.
We request that you for carefully revising the manuscript. Then, send the revised manuscript with revisions highlighted along with the Response letter to the reviewer via email to us for further consideration by 13-Sept-2022.

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We are looking forwards to hearing from you.

Thanks and regards

Reviewer 1.
Comments to the author:
The manuscript improved a lot after modification according to the suggestions, it is now recommended for publication.
Some tiny format issues, such as Ref. 31, and the significant digits (pore holes structure in the 64-7214 nm range).

Sincerely,

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Comments from the reviewer(s) are included below.
We conclude that further review and revision may be necessary.

Reviewer 1 has recommended that your manuscript must be minor revised.

We request that you for carefully revising the manuscript. Then, send the revised manuscript with revisions highlighted along with the Response letter to the reviewer via email to us for further consideration by 13-Sept-2022.

If you have any questions, please feel free to contact us at thanhtd@ims.vast.ac.vn and/or journal@ans.vast.vn.
We are looking forwards to hearing from you.

Thanks and regards

Reviewer 1.
Comments to the author:
The manuscript improved a lot after modification according to the suggestions, it is now recommended for publication. Some tiny format issues, such as Ref. 31, and the significant digits (pore holes structure in the 64-7214 nm range).

Sincerely,

Prof. Thanh Tran Dang
thanhtd@ims.vast.ac.vn

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Rika Taslim <rikataslim@gmail.com> Wed, Sep 7, 2022 at 9:45 AM
To: thanhtd@ims.vast.ac.vn

07-Sept-2022

ANSN-2022-0119.R1 - Agricultural Bio-waste of Corn Silk derived Porous Carbon for High-performance Supercapacitors

Dear Prof. Thanh Tran Dang

Thank you for evaluating our manuscript in *Advances in Natural Sciences: Nanoscience and Nanotechnology* (ANSN).

We greatly appreciate the editor's consideration of this manuscript for publication and valuable review comments. We have made every attempt to respond to all comments and questions raised by the reviewer and have adjusted the manuscript accordingly. All revisions in the manuscript are given a yellow highlight background.

Furthermore, we have added the revised manuscript and author response letter to reviewer in attachment file.

We confirm that this manuscript was revised in accordance with the reviewers' comments in *Advances in Natural Sciences: Nanoscience and Nanotechnology* (ANSN). We hope this manuscript can be accepted and published in the *Advances in Natural Sciences: Nanoscience and Nanotechnology* (ANSN).

Best regards,

Rika Taslim
Corresponding Author

Authors' Responses to Editors' Reviewers' Comments on the Manuscript

Reviewer 1.
Comments to the author:
The manuscript improved a lot after modification according to the suggestions, it is now recommended for publication. Some tiny format issues, such as Ref. 31, and the significant digits (pore holes structure in the 64-7214 nm range).

Authors' Response:

We appreciate and thank the reviewer for the very constructive comment in this manuscript. We apologize for the mistakes. Accordingly, we have corrected the format issues in ref 31 and we also corrected the significant digits in sentences "pore holes structure in the 64-7214 nm range" This revision has been added to the manuscript with yellow highlight background. Hopefully, the revised manuscript is found to be more convenient to read.

Result and discussions part, page 8, with yellow highlight background

… Figure 2c shows the relatively dominant pore holes structure in the 64-214 nm range. The diverse pore…

References part, page 21, with yellow highlight background

[31] Ni W, Shi L and Resol 2022 Vegetable Fibers Composites *Mat Res. Foundations* **122** 154-198

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Respon author for reviewer ANSN 2nd.docx

On Tue, Sep 6, 2022 at 5:05 PM Thanh Tran Dang <onbehalfof@manuscriptcentral.com> wrote:

06-Sep-2022

ANSN-2022-0119.R1 - Agricultural Bio-waste of Corn Silk derived Porous Carbon for High-performance Supercapacitors

Dear Dr. Rika Taslim:

Your manuscript, referenced above, has been reviewed for publication in the ANSN:
Comments from the reviewer(s) are included below.
We conclude that further review and revision may be necessary.

Reviewer 1 has recommended that your manuscript must be minor revised.
We request that you for carefully revising the manuscript. Then, send the revised manuscript with revisions highlighted along with the Response letter to the reviewer via email to us for further consideration by 13-Sept-2022.

If you have any questions, please feel free to contact us at thanhtd@ims.vast.ac.vn and/or journal@ans.vast.vn.
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Sincerely,

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Advances in Natural Sciences: Nanoscience and Nanotechnology (ANSN)
Jointly published by VAST (VN) and IOP (UK)
Abstracted in Web of Science (ESCI), Scopus, Chemical Abstracts,...
E-mail: journal@ans.vast.vn
Homepage: https://iopscience.iop.org/ansn; https://ans.vast.vn/
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Agricultural Bio-waste of Corn Silk derived Porous Carbon for High-performance Supercapacitors

Journal:	*Advances in Natural Sciences: Nanoscience and Nanotechnology*
Manuscript ID	ANSN-2022-0119.R1
Manuscript Type:	Original Article
Date Submitted by the Author:	n/a
Complete List of Authors:	Taslim, Rika; State Islamic University Sultan Syarif Kasim, Department of Industrial Engineering Halbi, Suryandri; State Islamic University Sultan Syarif Kasim, Department of Industrial Engineering Apriwandi, Apriwandi; University of Riau - Binawidja Campus, Department of Physics Taer, Erman; University of Riau - Bina Widya Campus, Department of physics
Keywords:	Nanofiber, Porous carbon, electrode material, Supercapacitor
Classification numbers:	5.16, 5.18

Agricultural Bio-waste of Corn Silk derived Porous Carbon for High-performance Supercapacitors

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Abstract. This study aims to develop a novel, simple, efficient, and low-cost method to prepare hierarchical porous carbon nanofiber derived from corn silks (CSAC) through a one-step carbonization-physical activation process. The carbon precursors were activated by KOH solution at a high pyrolysis temperature to prepare activated porous carbon as an electrode material for supercapacitors without using binders. This study focused on the effect of different activation temperatures of 600, 700, 800, and 900°C on the production of highly porous carbon nanofiber. An enhancement mechanism is proposed, which not only performed high nanofiber structures to possess the large specific active surface area to enhance energy density but also achieve micro-mesopores combination to realize fast ion-transport channels for boosting high power density. A maximum specific surface area of approximately 1096.951 m^2 g^{-1} was achieved by CSAC7. Furthermore, the electrochemical performance was evaluated using 1 M H_2SO_4 solution as an electrolyte through a novel two-electrode binder-free system. The electrode materials produced a maximum specific capacitance of 237 F g^{-1} at a current density of 1 A g^{-1}. These excellent characteristics show that the synthetic approach has a great potential for fabricating high-performance supercapacitors.

Keywords: Nanofiber, porous carbon, electrode material, supercapacitor.

Classification numbers: 5.16, 5.18

1. Introduction

Supercapacitors are globally considered as superior storage devices due to the higher energy and power density compared to batteries, capacitors, and fuel cells [1]. In addition,

they function as suitable materials for electrical components, pulse laser systems, and other electronic devices [2]. Supercapacitors are also directly utilized or combined with batteries for certain electrical facilities, such as electric braking, starters, vehicle, and generators [3]. The electric double-layer capacitors (EDLC) are considered to be the best type due to the variable and adaptable active materials, low cost, abundant availability, superior material properties, maximum electrolyte flow rate, high operating temperature and conductivity, compatible with a variety of low and high current components, environmentally friendly, and relatively safer [4]. However, supercapacitors still have various challenges such as the relatively low energy compared to the high power density, expensive active materials, and complex synthetic methods. In different studies, the energy density of supercapacitors has been increased with various approaches, but with a reduced power density. The main key to improving the performance of supercapacitors is the active electrode material, followed by the electrolyte and separator [5]. Three groups of active electrode materials have been reported including conduction polymers, metal oxides, and carbon [6]. Conducting polymer materials and metal oxides were used to successfully increase the energy density up to 200 Wh kg^{-1} [7]. This is several times greater than the result from other studies in the last decade. However, both materials require complex, complicated, corrosive, and toxic instruments, hence, they are not recommended for environmentally friendly mass production. Therefore, activated carbon from biomass and bio-organic waste is more promising as it has superior and attractive characteristics including abundant availability, renewability, easy fabrication, and low cost [8,9]. Activated carbon materials also have a good performance in increasing and maintaining energy density, although the energy density produced is still relatively low compared to metal oxides and conduction polymers [10]. Additionally, biomass precursors rich in structurally diverse porosity and significant heteroatoms provide a large number of active ion contacts and facilitate the transport of electrolytic ion charges in various pores

including micro-, meso-, and macro- leading to a high performance of energy and balanced power density [11]. Moreover, the 3D hierarchical pore structure obtained from the biomass-derived activated carbon has been shown to increase the energy density up to 5 times coupled with a high power density [12]. Hierarchical porous carbon consisting of micro-, meso-, and macropores are obtained from various biomass precursors such as onion peel [13], pineapple leaves [14], rice husk [15], ginger waste [16], peanut shells [17], bamboo stem [18], and jujube fruit [19]. The peanut shell agricultural biomass produced activated carbon having a 3D hierarchical pore structure as well as abundant micropores and mesopores with surface area=2014.6 m^2 g^{-1}. Activated carbon is prepared by a new and low-cost method using $ZnCl_2/CO_2$ activation which produced a specific capacitance of 310 F g^{-1} in a 3-electrode system [20]. Shang *et al.,* (2021) obtained activated carbon from chitin (*Portunus trituberculatus* Crab) waste [21], which was converted into hierarchical porous carbon through ZIF-8 nanoparticles bio-template followed by high-temperature carbonization, leading to a specific capacitance of 182 F g^{-1}. Similar results were also obtained from different biomass precursors such as cauliflower which produced a unique hierarchical pore structure using KOH activation at 700°C high-temperature simple which possessed a high surface area of 2061 m^2 g^{-1} [22]. However, the activated carbons obtained from the above precursors are prepared in powder form, hence, they require synthetic binders and insulators to test the electrochemical properties. Although the counter electrodes used are good conduction materials, it is still considered to reduce the efficiency. Also, the template/bio-template method often used to ascertain the 3D pore structure is a relatively risky technique because the final step requires more treatment to remove the by-products. On the other hand, corn silk agricultural biomass has high potential as a hierarchical porous carbon precursor. The basic fibrous structure allows the discovery of dense nanofibers on activated carbon, thereby adding to its renewability. Although the use of corn silk as a starting material for

activated carbon in supercapacitors has been previously reported, the method of preparation using binders through a relatively complicated technique is considered to limit its novelty. Therefore, this study aims to prepare binder-free activated carbon through a novel one-step activation of KOH/CO_2. The chosen strategy has been shown to produce higher capacitance compared to Mitravinda *et al.* (2018) [23] which used different methods and possesses higher energy density compared to a study by Zhou *et al.* (2020) [24]. In addition, we selected H_2SO_4 aqueous electrolytes because of their advantages including the highest ionic concentration, highest conductivity, and low viscosity compared to other aqueous electrolytes. In result, the optimum specific capacitance of activated carbon obtained is 237 F g^{-1} with a maximum energy density of 18.19 Wh kg^{-1} in an aqueous electrolyte of 1 M H_2SO_4.

2. Materials and methods

2.1. Materials

The corn silks (CS) were initially obtained from the traditional plantations of the Kampar community, Riau and then cut into small pieces within a range of 2 cm, cleaned, and sundried for 48 hours to obtain dry samples. Furthermore, the samples were pre-carbonized and crushed using a crusher machine to obtain dried corn silk powder. The KOH activating agent, HCl, and aqueous electrolyte H_2SO_4 were obtained from Merck KGaA, 4271 Darmstadt, Germany, and Panreac Quimica Sau, Espana, while deionized water (DI) as a material for neutralizing samples was made on a lab scale. The organic separator from the duck eggshell membrane was extracted with 1 M HCl solution.

2.2. Synthesis of carbon pellets

30 g of dried corn silks powder was dispersed with a 0.5 m/L KOH solution on a hotplate at 300 rpm for 2 hours. The sample was filtered and dried in an oven at 110 °C, while the corn silk powder was converted into pellets using a hydraulic press without using

any adhesive material, hence, the adhesive properties of the pure powder were derived originally from the sample. A total of 20 pellets samples were prepared for pyrolysis using carbonization in the N_2 gas environment and physical activation with CO_2 in the furnace tube. In addition, the pyrolysis process began from a temperature of 30° C to 600 °C in N_2 gas and then increased to 900 °C in a CO_2 environment. Four different carbonization temperatures were applied namely 600 °C, 700 °C, 800 °C, and 900 °C. Moreover, to facilitate data interpretation, samples were labeled CSAC-x, CSAC was corn silk activated carbon (CSAC), while x was the number 6, 7, 8, and 9 indicating carbonization at the four temperatures. The pelleted carbon samples were neutralized using DI water.

2.3. Characterization of materials

The carbon pellets' density was evaluated based on changes in mass, thickness, and diameter during the pyrolysis process through standard equations. Furthermore, the amorphous properties of the samples were characterized using the XRD (X-ray diffraction) technique at an angle range of 5-60° in a Cu Kα radiation source (Phillips expert powder instrument). Surface morphology and variation of sample elements were also examined through the SEM-EDS (Scanning electron microscopy-energy dispersive spectroscopy) technique using the JEOL-JSM-LA-3600 instrument at a voltage of 15kV. Additionally, the specific surface area and pore diversity were determined through the N_2 gas absorption technique and evaluated using BET and BJH calculations.

2.4. Supercapacitor electrochemical performance

The electrochemical properties of the supercapacitor were evaluated in two-electrode configurations. The supercapacitor cells were made in the form of sandwich layers consisting of a cell body made of acrylic, stainless steel current collector, pelleted carbon electrodes, and an organic separator from the duck eggshell membrane. Interestingly, the semi-permeable duck eggshell membrane allows the electrolyte ions to diffuse optimally on the

surface of the carbon electrode [25]. The cyclic voltammetry (CV) and galvanostatic charge-discharge (GCD) methods are techniques commonly used in evaluating specific capacitance, as well as energy and power density. CV was evaluated using the CV-UR Rad-Er 1380 instrument at a constant voltage range of 0-1.0V at a scan rate of 1 mV s^{-1}, while GCD used the 2018 CD-UR Rad-Er at a fixed current of 1 A, both instruments were calibrated with a mean error of ± 6.01%.

3. Results and discussions

Crystal phase change properties of corn silk-based hierarchical porous carbon were reviewed using the powder X-ray diffraction (XRD) method. The XRD pattern with different physical activation temperatures particularly between 700 and 900 °C, are shown in Figure 1.

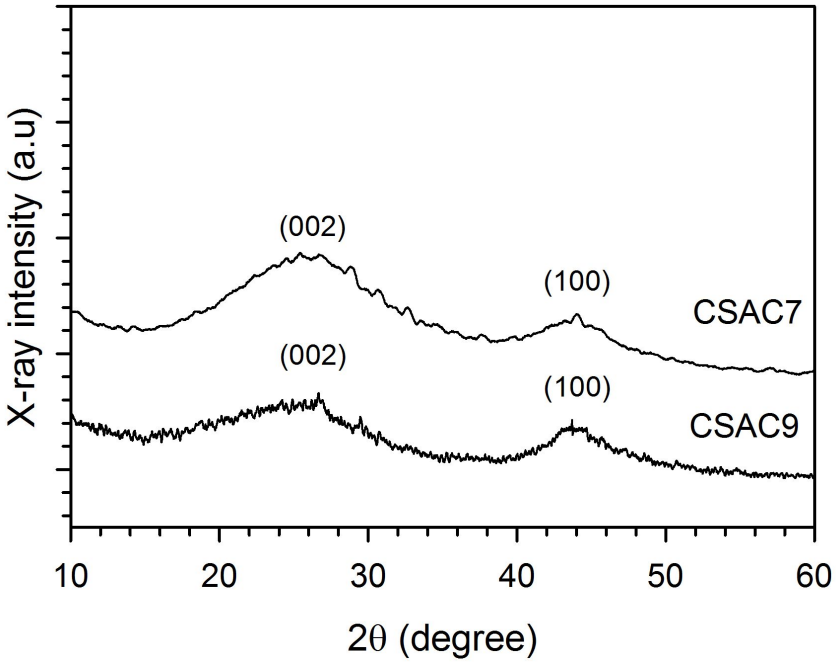

Figure 1. The XRD pattern of CSAC7 and CSAC9

The CSAC7 display two clearly confirmed broad peaks at 2θ angles 24.34° and 43.92° which correspond to the (002) and (100) scattering planes. These characteristics show that the structure of the turbostratic/disturbed carbon has a high amorphous nature [26]. This property is important for improving the good hierarchical pore structures in carbon samples. On the other hand, CSAC9 performed low broad peaks at angles of 2θ=24.86° and 44.13° indicating

the amorphous nature degraded towards graphitization due to high-temperature treatment. Furthermore, the most obvious broad peaks in the (002) reflection plane ranging between angle 24.34°-24.86° indicate a random aromatic sheet structure, while the (100) reflection plane from 43.92°- 44.13° confirms the type of carbon that is close to the graphite structure, in this case, the attenuation of the graphite structure towards the formation of a disturbed carbon structure [27].

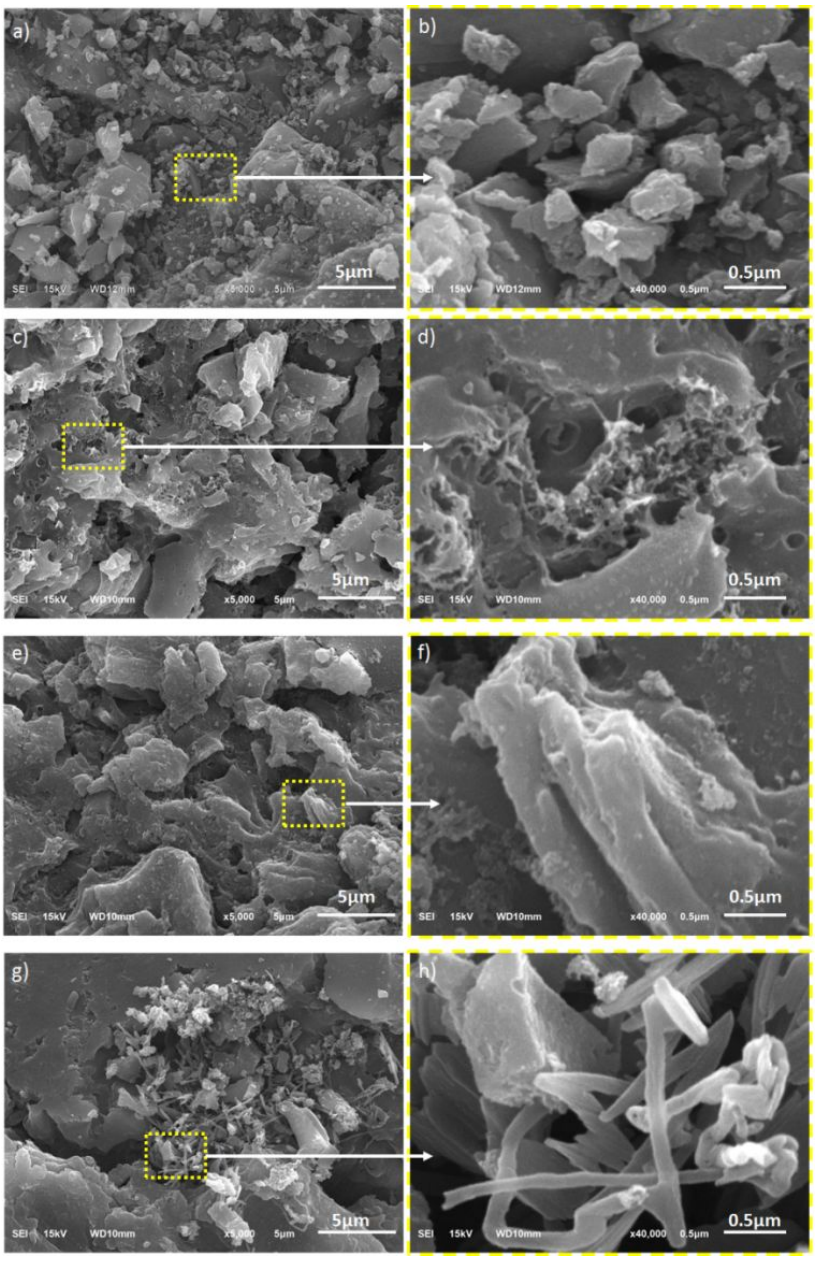

Figure 2. The SEM macrograph of CSAC6 in magnification of (a) 5000x, (b) 40000x, CSAC7 in magnification of (c) 5000x, (d) 40000x, CSAC8 in magnification of (e) 5000x, (f) 40000x, CSAC9 in magnification of (g) 5000x, (h) 40000x

The morphological structure of activated carbon-based corn silk waste was evaluated using the SEM method at a voltage of 15 kV. Figure 2 shows the morphological structure with different physical activation temperatures of 600, 700, 800, and 900 °C. In general, the surface structure of the sample is dominated by bulk materials, aggregation and clumps of large wrinkled particles, while some parts display a different inner surface structure for each temperature. At 600 °C (Figure 2a), the activated carbon consisted of particle aggregations with surface wrinkles. Additionally, a larger zoom in Figure 2b shows that the sample surface is relatively flat in each aggregation without any obvious pores. Furthermore, comparatively to 600 °C, physical activation at a higher temperature led to a better decomposition into the basic components such as hemicellulose, cellulose, and lignin. This indicates a relatively different surface morphology as shown in Figure 2c-d. Figure 2c shows the relatively dominant pore holes structure in the 64-7214 nm range. The diverse pore structure including mesopores and macropores were confirmed at a larger magnification area as shown in Figure 2d. Mesopores have a size ranging from 18-48 nm, while macropores are in the range of 73-136 nm. However, micropores structures were not found at this magnification. An increase in the physical activation temperature of 800 °C in the CSAC8 sample shows a surface morphology filled with macropores which have a relatively large size less than 300 nm as shown in Figure 2e. In addition, Figure 2f shows a distinct morphology in which clustered tubular-rod-like structures are found. This is because the higher temperature porosity allows the breakdown of the main carbon structure derived from cellulose and lignin drastically. The rod shape is contributed from the basic structure of cellulose while the outer tubular shape is attributed to lignin [28]. This analysis has been reported in previous studies with different biomass precursors such as cassava petiole [29] and cacao shells [30]. Moreover, the temperature of 900°C maximized the basic decomposition process of cellulose and lignin to form a clear surface morphology of carbon

fibers, as shown in Figure 2g-h. The diameter of the obtained nanofibers varied relatively in the range of 76 nm to 127 nm. The predominant nanofiber fiber allows well-connected pores in a relatively small size range [31]. The elemental composition of porous activated carbon samples at activation temperatures of 700 °C and 900 °C for CSAC7 and CSAC9 was examined using energy dispersive spectroscopy (EDS) techniques in the energy range of 0-20 keV. Table 1 shows the elemental composition of the CSAC7 and CSAC9 samples in detail. Elemental carbon (C) has the highest percentage of approximately 83.7485-85.2755% followed by oxygen 8.0333-8.6075%, silicon (Si) 3.2471-3.1535%, potassium (K) 1.6359-3.9428%, magnesium (Mg) 0.8317-0.7930%, and aluminum (Al) 0.4022-0.3290%.

Table 1. The elemental composition of CSAC7 and CSAC9

Element	CSCA7 (%)	CSAC9 (%)
Carbon (C)	85.2755	83.7485
Oxygen (O)	8.6075	8.0333
Silicon (Si)	3.2471	3.1535
Potassium (K)	1.6359	3.9428
Magnesium (Mg)	0.8317	0.7930
Aluminum (Al)	0.4022	0.3290

High-temperature pyrolysis of 700 °C for CSAC7 showed the highest carbon composition of 85.2755%. This property is considered to increase the capacitive properties of the electrode material. Furthermore, the highest elemental oxygen was also found in the CSAC7 sample which contributed to the wettability of the material thereby initiating the pseudocapacitance effect [32]. An increase in the activation temperature up to 900 °C for CSAC9 reduced the percentage of carbon and oxygen in the sample. This is because the activation of high temperatures evaporates the constituent elements of the material in the form of more H_2O and CO_2. Moreover, other elements such as Silicon, Potassium, Magnesium, and Aluminum are obtained in relatively low amounts from basic sources of biomass that have not been completely evaporated [33].

The analysis of N_2 adsorption/desorption isotherms of corn silk-based hierarchical porous activated carbon at different pyrolysis temperatures are needed to evaluate the impact of pore structure and porosity properties. Figure 3 shows the N_2 adsorption-desorption isotherm curve of both samples, CSAC7 had significant N_2 adsorption at a relative pressure of $0.0<P/P_0<0.2$, thereby confirming the relatively large number of micropores.

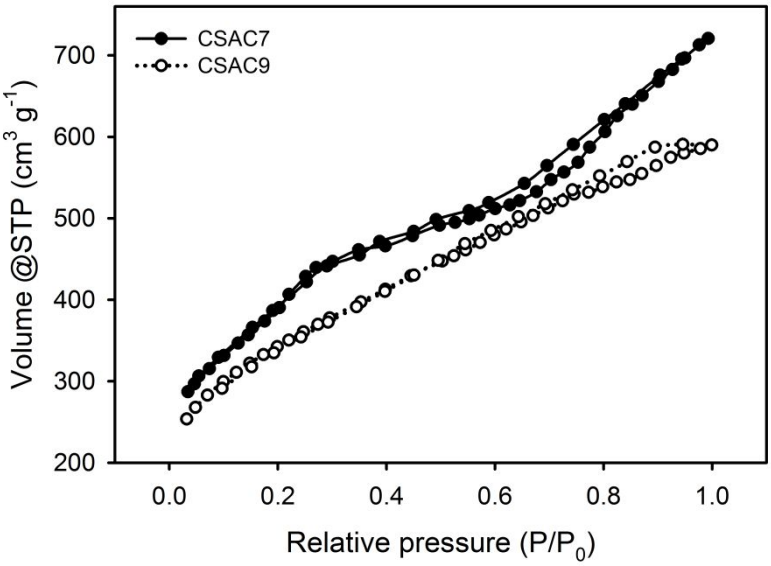

Figure 3. Nitrogen adsorption-desorption isotherms of CSAC7 and CSAC9

Table 2. Specific surface area, pore volume and average pores size of CSAC7 and CSAC9

Sample	S_{BET} ($m^2 g^{-1}$)	S_{micro} ($m^2 g^{-1}$)	S_{meso} ($m^2 g^{-1}$)	V_{tot} ($cm^3 g^{-1}$)	V_{micro} ($cm^3 g^{-1}$)	V_{meso} ($cm^3 g^{-1}$)	D_{aver} (nm)
CSAC7	1096.951	531.087	565.864	1.115	0.314	0.801	4.06
CSAC9	744.818	423.360	321.458	0.9126	0.4476	0.465	3.15

Furthermore, the H4 type hysteresis loop was clearly shown over a relatively long relative pressure range of $0.2<P/P_0<0.9$ indicating uniform mesoporous characteristics at CSAC7 as presented in Table 2. The tail at high-pressure $P/P_0>0.9$ shows the presence of macropores, but the addition of the physical activation temperature up to 900°C in CSAC9 showed a different N_2 isotherm absorption compared to CSAC7. The N_2 adsorption-desorption curve of CSAC9 shows a relatively linearly increasing adsorption which is not too high at low pressure, thereby indicating the presence of low micropores. In addition, the H4 type hysteresis loop is not formed completely at a relatively short pressure range of $P/P_0 = 0.6$ to 0.95 which is mainly due to the pores growing towards the larger one and is dominated by narrow bottle neck-like types with a larger inner surface [34] as shown in Table 2. The pore

size distribution as presented in Figure 4 show that both samples have a combination of micropores and macropores. This result is relatively consistent with SEM which also confirms the hierarchical porous structure. CSAC7 has a structure of hierarchically connected 3D pores leading to the presence of mesopores with a peak at 3.89 nm. Furthermore, the chemical impregnation of KOH at 700 °C high-temperature pyrolysis allows the precursor to produce a good combination of micropores and mesopores to improve the performance of the base electrode.

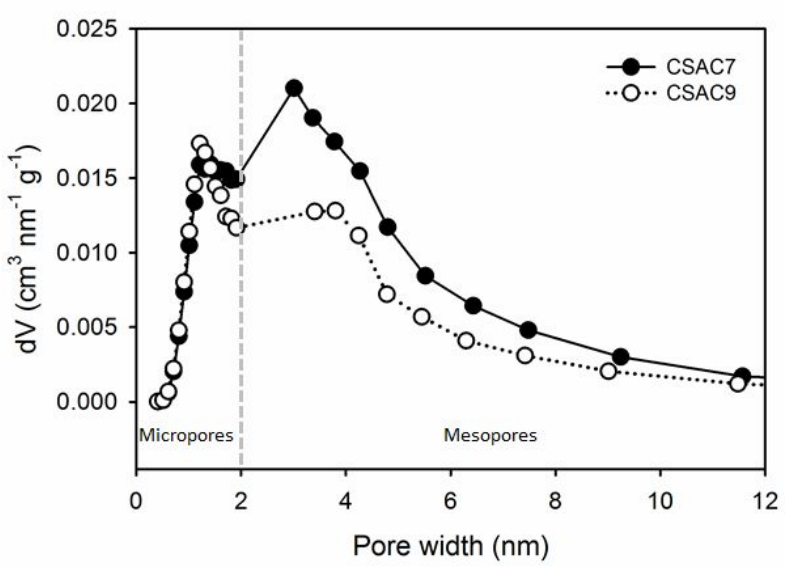

Figure 4. Pore sizes profile of CSAC7 and CSAC9

The microporous and mesoporous percentages of CSAC7 were 48.41% and 51.59% with a high surface area of 1096.951 m^2 g^{-1}. The high surface area coupled with the combination of the hierarchically connected pores is believed to improve the electrochemical properties of supercapacitors [35]. In addition, the high mesoporous volume fraction potentially facilitates rapid ion transport and act as an electrolyte ion reservoir, thereby reducing the ion diffusion distance to the carbon surface, ensuring high rate capability and high power density. Moreover, CSAC9 displays a relatively similar pore size distribution with CSAC7 but is dominated by micropores with a percentage of 56.84%. KOH impregnation at higher temperatures up to 900°C allows degradation of carbon aggregation and erodes the main pore walls, thereby creating tubular and nanofiber structures. This simultaneously reduces the

specific surface area of CSAC9 to 744.818 m^2 g^{-1} with an average diameter of 3.15 nm as shown in Table 2. Nevertheless, the developed porosity properties of carbon materials are important for electrolyte ions' rapid transport and penetration [36]. These results are consistent with the SEM and density data.

The main parameters used to evaluate the electrochemical properties of hierarchical porous activated carbon include high specific capacitance, increased energy and fixed power density, as well as retention capacitance. Based on the N_2 gas absorption analysis, the corn silk-based activated carbon has a combination of micropores and mesopores that is able to accommodate a relatively high charge of active ions and facilitate diffusion without obstruction at the electrode/electrolyte interface. The electrochemical performance of CSAC6, CSAC7, CSAC8, and CSAC9 based on different physical activation temperatures was evaluated with a two-electrode configuration in an aqueous electrolyte using cyclic voltammetry and galvanostatic charge-discharge techniques. Cyclic voltammetry (CV) curves at 600, 700, 800, and 900 °C are shown in Figure 5. The CV profile exhibits a quasi-rectangular shape, indicating the typical behavior of electrochemical double-layer type supercapacitors (EDLC) in an aqueous electrolytic system at a scan rate of 1 mV s^{-1}.

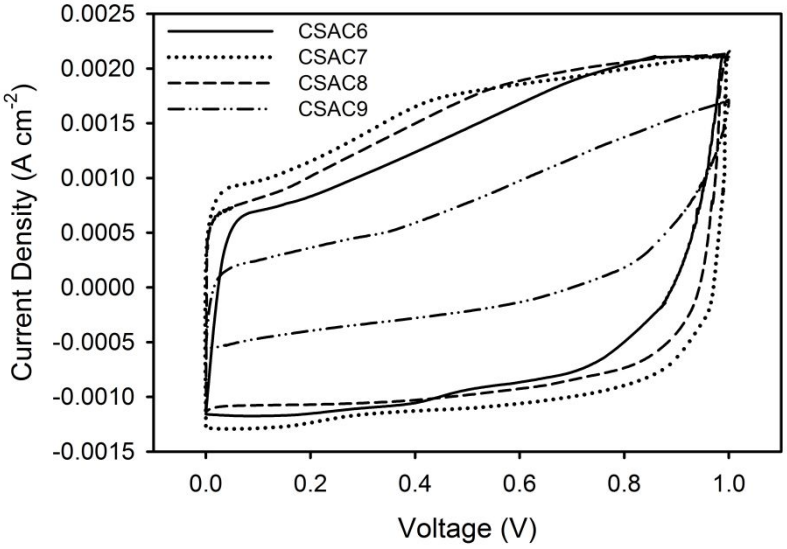

Figure 5. CV curve of CSAC6, CSAC7, CSAC8, and CSAC9

In addition, the pseudocapacitance property was found to be relatively low as indicated by a spike in current density of the voltage ranging from 0.3-0.6V. This property is a contribution of the heteroatom and wettability properties of elemental oxygen in the sample. In general, all samples exhibited similar electrochemical properties. By using standard equations, the specific capacitances of CSAC6, CSAC7, CSAC8, and CSAC9 was evaluated to be 184, 211, 194, and 143 F g^{-1}, respectively. An increase in the physical activation temperature improved the capacitive properties of the supercapacitor by 211 F g^{-1}. This is because the higher temperature of CSAC7 showed a relatively rich surface structure of mesopores and macropores, which provides a high surface area and diffusion path of ionic charge in all directions, thereby facilitating the high capacitive properties. This analysis is consistent with SEM results and N_2 gas absorption in Figures 2 and 3. At 800 °C, the surface structure of the capacitive properties was reduced to 194 F g^{-1}. This is because the increase in the physical activation temperature in the CSAC8 sample shows a surface morphology filled with macropores with a relatively large size less than 300 nm, as shown in Figure 2e. In addition, the morphology of the clustered tubular rod-like structure allows for fractures that narrow the ion transport pathways on the electrode surface.

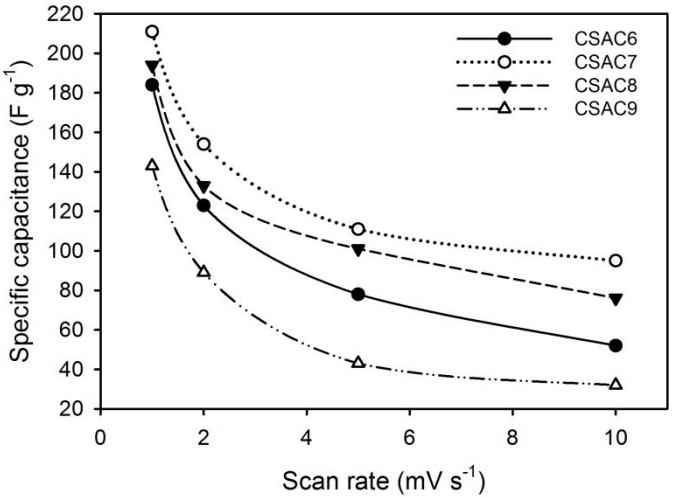

Figure 6. The curve of specific capacitance vs. scan rate of CSAC6, CSAC7, CSAC8, and CSAC9

A further temperature is increased up to 900 °C for CSAC9 allows the erosion and breakdown of the particle walls of carbon aggregation and rod-like structures to cover the existing pores, this reduced the capacitive properties to 143 F g^{-1}. Although the pore structure displays relatively dense nanofibers, as shown in Figure 2h, it is unable to maintain the 3D hierarchical pore distribution of micropores and mesopores thereby reducing the electrochemical properties. The specific capacitances of CSAC6, CSAC7, CSAC8, and CSAC9 were also evaluated at different scan rates from 1 mV s^{-1} to 10 mV s^{-1}, as shown in Figure 6. The activated carbon which maintained the highest specific capacitance of 45% was CSAC7, followed by CSAC8, CSAC6, and CSAC9 with values of 39%, 28%, and 23%, respectively. The galvanostatic charge-discharge (GCD) curves of symmetric cells CSAC6, CSAC7, CSAC8, and CSAC9 at a current density of 1 A g^{-1} are shown in Figure 7a. In general, the GCD curve displays a normal profile for the EDLC type with a slightly faint iR drop. According to the GCD profile, the specific capacitances produced at CSAC6, CSAC7, CSAC8, and CSAC9 were 152, 237, 174 and 124 F g^{-1}, respectively, in the electrolyte of 1 M H_2SO_4. Higher activation temperature from 600°C to 700°C confirmed micro-mesopores combinations of 48.41% and 51.59% displayed the highest electrochemical properties from 152 F g^{-1} to 237 F g^{-1}. These results correlate with SEM, N_2 gas adsorption/desorption, and CV analysis. The high activation temperature of from 700°C to 900°C decreased the capacitive value of the carbon electrode up to 124 F g^{-1} for the CSAC9 sample. This is associated with the collapse of the pore walls and the erosion of the carbon framework structure to cover the active site of the ionic charge below [34].

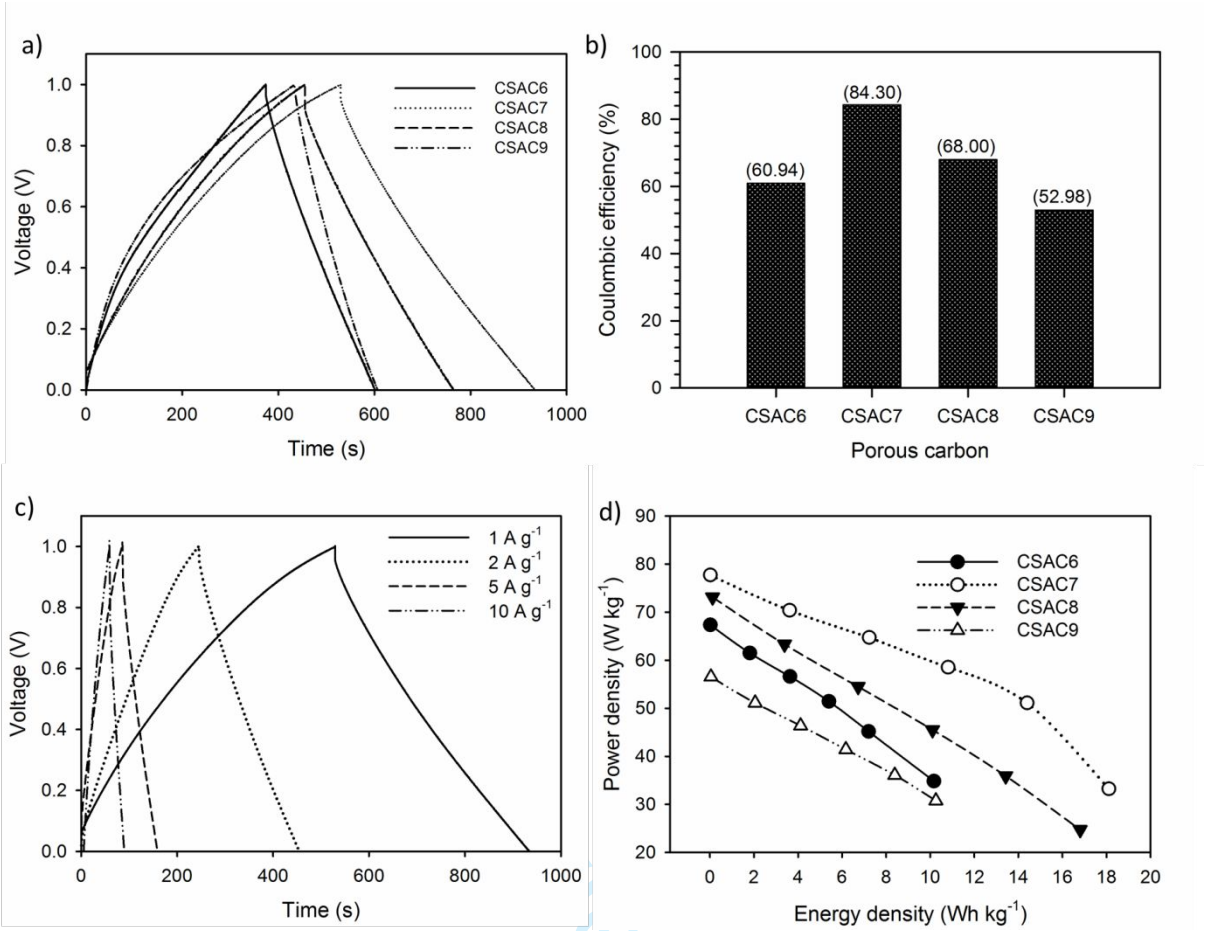

Figure 7. (a) GCD curve, (b) Coulombic efficiency of CSAC6, CSAC7, CSAC8, and CSAC9 (c) GCD curve at different current density of CSAC7, and (d) Ragone plot of CSAC6, CSAC7, CSAC8, and CSAC9 in 1 M H_2SO_4 electrolyte

Furthermore, the resistance in CSAC7 was lower at 0.0025Ω due to the relatively high dominance of the mesopores which improved the diffusion of ionic charges in all directions compared to CSAC6, CSAC8, and CSAC9 which has a higher resistance value of 0.012, 0.27, and 0.034 Ω, respectively. This is also contributed by the semi-permeable duck eggshell membrane which allows the electrolyte ions to diffuse optimally on the surface of the carbon electrode [25]. In addition, the charge and discharge times on the GCD profiles reflect the coulombic efficiency of GLACs, as shown in Figure 7b. CSAC7 had the highest coulombic efficiency of about 84.30%, followed by CSAC8, CSAC6 and CSAC9 68.00%, 60.94 and 52.98%, respectively which confirmed confirming the high collective interpenetration and

accessibility of the electrolytes. Moreover, the GCD profile of CSAC7 was reviewed at different current density of 1, 2, 5, and 10 A g^{-1}, as shown in Figure 7c. It can be noted that the GCD curves of CSAC7 still maintain primary symmetric triangle shape at 10 A g^{-1} suggesting stable double-layer capacitor behaviors, fast ion response, and low internal resistance in EDL processes [42]. At 1 A g^{-1}, CSAC7 sample delivers a specific capacitance of 237 F g^{-1} and it still maintains 210 F g^{-1} at 10 A g^{-1}, initiating a high-rate performance of 88.6%.

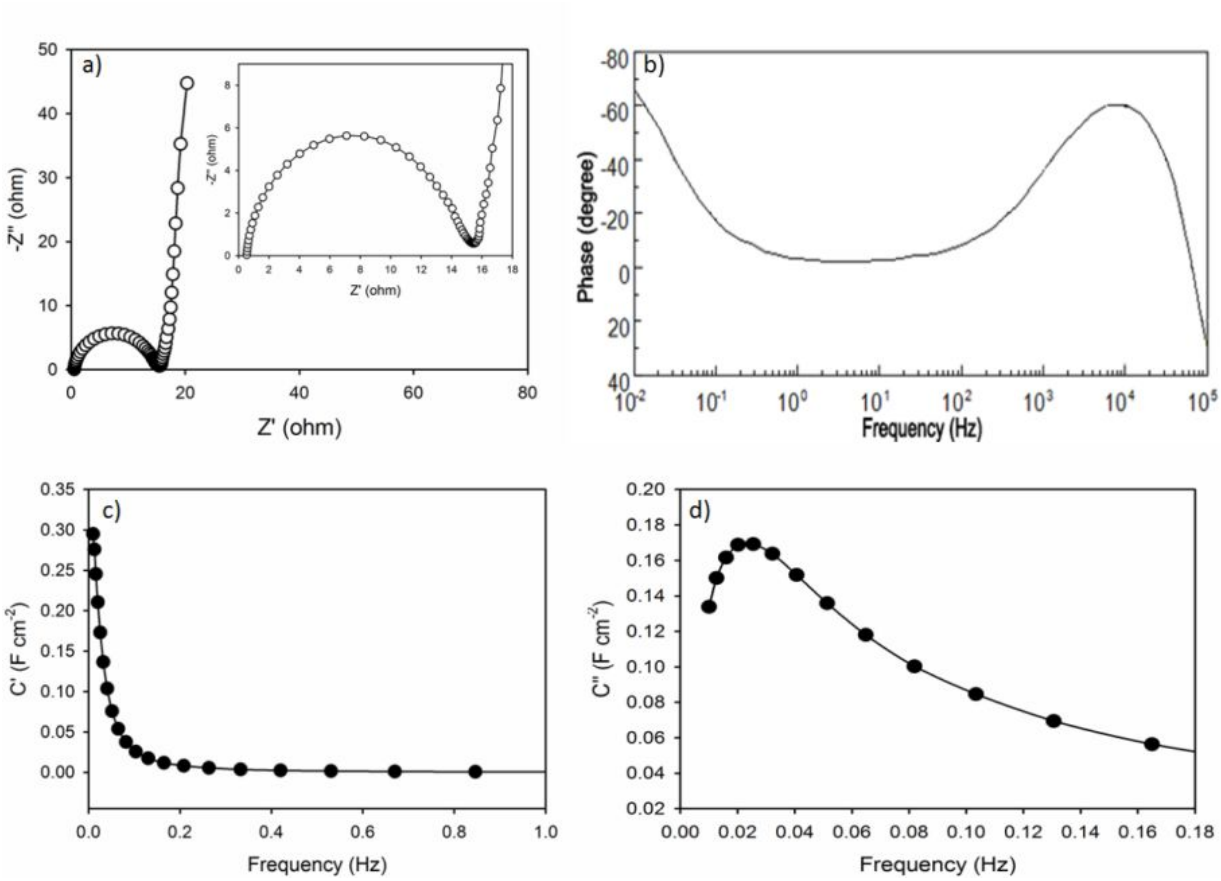

Figure 8. (a) Nyquist plot, (b) Bode phase angle plot, (c) real capacitance (C') vs. frequency plot, and (d) imaginer capacitance (C") vs. frequency plot of CSAC7 symmetrical devices

To compare the performance of both samples in-depth, Ragone plots were used to evaluate the energy and power density which are calculated using standard formulas as shown in Figure 7c. Symmetrical supercapacitors CSAC6, CSAC7, CSAC8 and CSAC9 assembled

with an aqueous electrolyte of 1M H_2SO_4 in an organic separator had energy densities of 10.1712, 18.1081, 16.8049 and 10.2596 Wh kg^{-1} as well as power densities of 67.3793, 77.74, 73.1373 and 56.5421 W kg^{-1}, respectively. The high electrochemical capability of CSAC7 is due to the 3D hierarchical porous structure and the specific surface area which provides a short and accessible ion transfer area in all directions for ionic charges to diffuse at the electrode/electrolyte interface.

The high electrochemical confirmation of CSAC7 was reviewed in depth via a Nyquist Plot with a limited diffusion area in the frequency range of 0.01 Hz to 100,000 Hz, as shown in Figure 8a. Horizontal translation along the Z' axis confirms a relatively low Rs 0.87 Ωcm^2 indicating a good ions adsorption and desorption process at the electrode-electrolyte interface. Furthermore, the RCT (12.3 Ωcm^2) interpreted at the semicircular position confirmed the fast charge transfer kinetically as a result of the current passing through the electrode forming an electric layer. This is due to the hierarchical pores structure with a combination of micro, meso, and macropores initiates the active site which is followed by the ability of ion diffusion in all directions without any obstacles. In addition, the straight line that tends to be perpendicular at low frequencies confirms the pure capacitive and high ion transfer rate at the CSAC7 electrode. The Bode phase angle plot of CSAC7 was also evaluated as shown in Figure 8b. The phase angle at low frequencies reveals specific information about the ability of the electrode to generate an electric double layer. CSAC7 electrode displays a phase angle close to -90° indicating that the normal dielectric layer of the EDLC version is accompanied by impaired ion degradation due to self-heteroatom doping. In addition, the Bode phase angle plot provides further information for the characteristics of the conductive material through analysis of the real capacitance (C') and the imaginary capacitance (C”) with respect to frequency, as shown in Figures 8b, and 8c. The C' values are weak in the lower frequency and higher frequency regions, the C' values show less frequency

dependence, indicating a purely resistive behavior in CSAC7. Moreover, the value of the imaginary capacitance C" at the peak frequency confirms a relaxation time of 9.25 seconds, indicating that the symmetrical CSAC7 device acts as a pure capacitor. This value indicates a measure of how quickly the stored energy of the device can be efficiently distributed. For comparison, the obtained carbon nanofiber-based on corn silk waste showed higher electrochemical properties than the carbon nanofiber-based on bacterial cellulose and tofu dregs obtained through chemical activation of KOH and H_3PO_4 [37,38]. In addition, although polymer-based carbon nanofibers exhibit superior specific capacitance, our study was able to produce higher energy densities than the previously reported as confirmed in referee [39]. This is due to the combination of nanofiber and micro-mesoporous structures allowing the provision of high active sites and ion-transport channels. Moreover, the binder-free electrode design supports the high electrochemical performance of the supercapacitor.

4. Conclusion

Based on the results, hierarchically porous carbon nanofiber was successfully obtained from corn silk using a low-cost, simple, and sustainable technique without a binding material for a high energy density of symmetric supercapacitors. In addition, different physical activation temperatures display distinctive pores structure with individual advantages. At 700 ^oC, a 3D hierarchical pore structure was displayed followed by a high surface area leading to the formation of an ideal combination of micro and mesopores for enhancing the high performance of the electrode material. Using a two-electrode binder-free system, CSAC7 showed the best electrochemical properties with a specific capacitance of 237 F g^{-1} in a 1 M H_2SO_4 aqueous electrolyte. Hence, the maximum energy density of 18.19 Wh kg^{-1} and power density of 77.74 W kg^{-1} at current density of 1 A g^{-1}. This result shows that the approach applied in this study confirmed the potential of corn silk as an activated carbon based material with high properties.

Acknowledgement

This study was funded by the collaborative grants between universities, State Islamic University of Sultan Syarif Kasim Riau with contract No. 873/Un.04/L. 1/TL.01/03/2022.

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