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## Low temperature preparation of chicken bone-based porous carbon for lithium ion capacitor (LIC) cathode

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**Abstract.** As a hybrid supercapacitor, the performance of lithium ion capacitor (LIC) relies on the porosity profile of the carbonaceous cathode as well as the lithiated anode. In this research, chicken bone waste (CBW) was used as a carbon source for LIC cathode. Here in, the CBW was impregnated using either 1.5 M KOH or 1.5 M CH<sub>3</sub>COOH at 150°C for 24 h, followed by carbonization at 500°C and 650°C for 1 h. The pore characteristics and morphology of the obtained carbon were examined using N<sub>2</sub>-physisorption and electron microscope techniques. The carbon samples were then mixed with Super P conductive carbon and polyvinylidene fluoride (PVDF) using the weight ratio of LIC: Super P carbon: PVDF is 85:5:10 to get the homogeneous cathode mixtures. These cathodes were then assembled incorporation with commercial lithium hexafluorophosphate (LiPF<sub>6</sub>) electrolyte and lithium titanate oxide (LTO) anode to obtain full-cell LIC. The performance of full-cell LIC was evaluated using cyclic voltammetry (CV) as well as the calculation of specific capacitance. The results showed that impregnation using CH<sub>3</sub>COOH, followed by carbonization at 650°C produced porous carbon with good pore characteristics. Furthermore, this kind of porous carbon stimulates good performance of the obtained full-cell LIC.

### 1. Introduction

Chicken bone waste (CBW) is an abundant organic waste due to consumption of chicken meat continues to increase up to 27.13% and the presentation menu of chicken meat that more instant without bone [1]. In the previous study, CBW was utilized to be activated carbon for adsorbent of heavy metal content [2]. CBW has undergone carbonization process as much as 52.42% [3], and can be improved with the activation process. The pore size generated from CBW's activated carbon based on the previous study was 212Å [2]. However, the use of CBW as energy storage has never been developed.

So far, the most widely developed energy storage device is lithium ion capacitor (LIC). As LIC is an energy storage device that combines the working mechanism between lithium ion battery (LIB) and electric double layer capacitor (EDLC) as shown in figure 1 [4-6]. LIB has a low power density, but has a high energy density. This is because in the process of charge and discharge, the process of



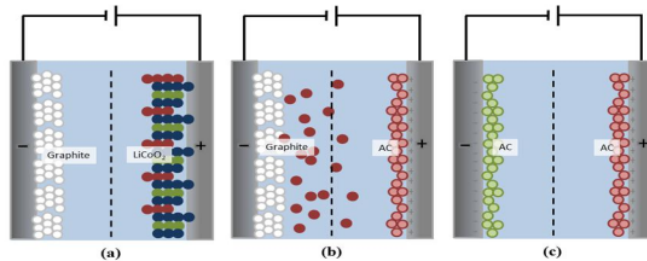


Figure 1. cell configuration for (a) LIB, (b) LIC and (c) EDLC [8]

reduction and oxidation reactions that requires a long time relative [6]. Unlike the EDLC which utilizes the activated carbon shaft to store energy and does not degrade chemical composition, so the resulting high power density but low energy density. In addition, the time required for charge and discharge is very short [6, 7].

The LIC component consists of cathode, anode, separator, and electrolyte. As a hybrid supercapacitor, the performance of LIC is highly dependent on the porosity of the activated carbon of the cathode used [6]. According to the International Union of Pure and Applied Chemistry (IUPAC), pores are divided into micropores (>2 nm), mesopores (2-50 nm) and macropores (<50 nm) [8]. The most suitable pore type for the LIC cathode is mesoporous because the charges that fill the pores are easier to move [9, 10].

The performance of a LIC supercapacitor can be determined from its ability to store energy. This can be seen from the magnitude of the surface area possessed by activated carbon as a cathode of LIC supercapacitor [8, 9]. The greater the surface area of the LIC's supercapacitor cathode, the resulting capacitance will also increase. In this study, we would like to investigate the relation between surface area and capacitance of activated carbon from chicken bone waste (CBW) as a cathode of LIC supercapacitor.

## 2. Experimental Methods

### 2.1. Carbonization of CBW

Chicken bone waste (CBW) type of broiler that has not been cooked, cleaned by distilled water and cut small-sized 1 cm then dried to reduce the water content. After that, CBW was impregnate as shown in figure 2, with stainless autoclave using activators  $\text{CH}_3\text{COOH}$  1.5 M and  $\text{KOH}$  1.5 M with the composition ratio of 9:20 (wt: vol) at  $150^\circ\text{C}$  for 24 h to open pores on the CBW surface and remove the tar or impurities.

After impregnation, CBW carbonized using a furnace tube at temperature variation of  $500^\circ\text{C}$  and  $650^\circ\text{C}$  for 1 h, with a rate increase of  $20^\circ\text{C}/\text{min}$  to decompose the CBW content into carbon compounds. In this process, there are two stages of decomposition. The first stage, the decomposition occurs between the temperature of  $200^\circ\text{C}$  and  $400^\circ\text{C}$ . The second stage takes place over a temperature of  $400^\circ\text{C}$  which involves the process of forming an amorphous form of carbon compounds [11].

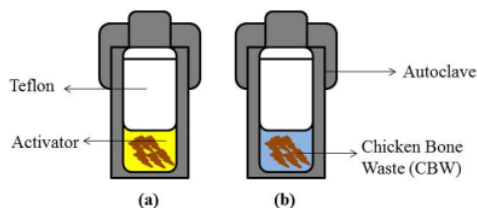


Figure 2. The impregnation of CBW using activators (a)  $\text{CH}_3\text{COOH}$  1.5 M and (b)  $\text{KOH}$  1.5 M.

Activated carbon of CBWs are washed and rinsed using aquadest to obtain neutral pH (pH 7). Then the activated carbon of CBWs were dried by an oven at 100°C for 5 h and put into 200 mesh.

2.2. Characterization of CBW activated carbon

Analysis of activated carbon of CBW's surface was performed by Brunauer Emmett Teller (BET) to saw the surface area, total of pore volume, pore diameter, pore distribution, and the isotherm of adsorption.

2.3. Coating and assembling process

To produce an LIC cathode, the activated carbon of CBW are mixed with a superconductive carbon P and polyvinylidene fluoride (PVDF) with a ratio of 85:5:10 dissolved into a Dimethyl Acetamid (DM) of 2.3 ml until we got the homogeneous slurry. The sample was printed with a thickness of 0.2 mm on 15×20 cm aluminum foil, and then dried by a drying box at 70°C for 30 minutes. The cathode sample is cut in a circular shape with a diameter of 16 mm. The cathode sample was assembled with anode Titanate Lithium (LTO) and separator on a lithium cell coin, and then given 60µL Lithium Hexafluorophosphate (LiPF6) electrolyte and punch.

2.4. Electrochemical characterization

The performance of supercapacitor LIC cathode was tested by cyclic voltammetry (CV) to determine the current, voltage and the cycle graph of it. The capacitance of supercapacitor LIC cathode was calculated by equation as followed [5]:

$$C_{sp} = \frac{Q \times I \times t}{m \times \Delta V} \quad (1)$$

where,  $C_{sp}$  is the specific capacitance (F/g) for single electrode,  $I$  represent the amount of charge flowing during the discharge (mA),  $t$  is the total discharge time (s),  $\Delta V$  denotes the discharge voltage (V) and  $m$  is the mass of the electrode cell (g) [5].

3. Results and Discussion

We produced supercapacitor LIC cathode by activated carbon of CBW with 4 samples, NC2, NK2, NC4 and NK4. In figure 3, the pore distribution graph shows that the pore size of activated carbon of CBW for samples of NC2, NK2, and NC4 are dominant into the mesoporous type based on the International Union of Pure and Applied Chemistry (IUPAC) [8]. With bigger mesoporosity level electrochemistry performance of carbon active will be better. This is because accessibility ion is faster and will improve the efficiency of the surface area of electrodes. Based on the results obtained, samples of NC2 and NC4 that impregnated by CH<sub>3</sub>COOH have better pore quality than other samples. It is also reinforced from table 1, the porosity data samples of NC2 and NC4 have higher surface area (S<sub>BET</sub>) of 71.35 m<sup>2</sup>/g and 63.38 m<sup>2</sup>/g. This is because the activator of CH<sub>3</sub>COOH is a type that can be decomposed in the low-temperature range between at 460°C to 595°C [12].

In contrast to KOH requiring high temperatures to decomposed, the possibility samples of NK2 and NK4 have a low surface area because the activator of KOH has not been maximally lost the tar or

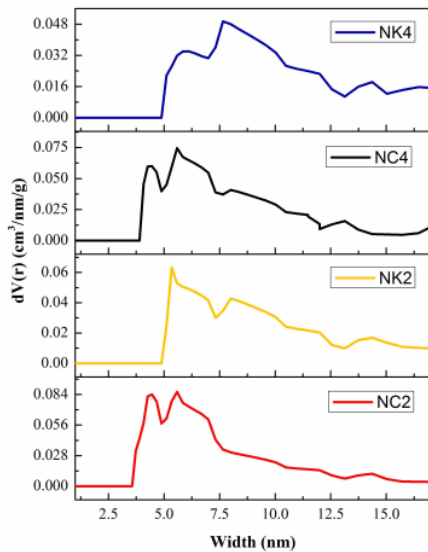
Table 1. Porosity data sample of CBW activated carbon.

	Activator	Carbonization Temperature (°C)	Pore size (nm)	Pore Volume (cm <sup>3</sup> /g)	S <sub>BET</sub> (m <sup>2</sup> /g)
NC2	CH <sub>3</sub> COOH	500	12.78	0.456	71.35
NK2	KOH		30.01	0.493	32.84
NC4	CH <sub>3</sub> COOH	650	14.16	0.449	63.38
NK4	KOH		82.24	1.525	37.09

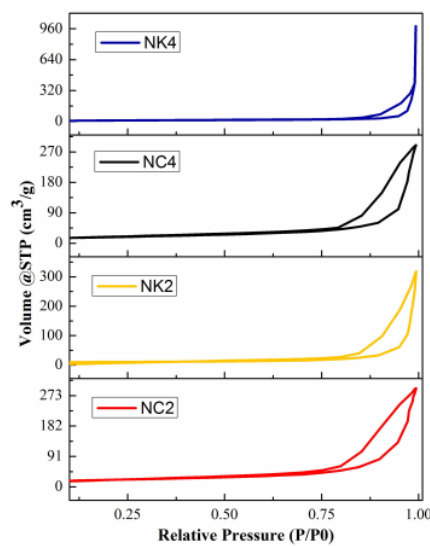
impurities on the activated carbon of CBW. In addition, activated carbon of CBW impregnated from activator of CH<sub>3</sub>COOH and followed carbonized at 650°C is physically black than other samples. In the process of carbonization, the temperature has an important role to form a material into carbon. In this research, we found that the resulting color obtained by activated carbon of CBW depends on the type of activator used. Samples impregnated with CH<sub>3</sub>COOH had a black color while the sample impregnated with KOH and followed by carbonization at 500°C (sample of NK2) had a dark gray color. The use of KOH activators as a strong base keeps the samples from burning by reacting with mineral so as not to form ash which causes a grayish color. However, in this case, sample of NK2 has not reached the maximum temperature to form carbon content. It is different with sample of NK4 which actually produces a darker color though not perfectly black.

Interestingly, the pore size samples of NK2 and NK4 were greater than the others although they have smaller surface area because the activator of KOH is a strong base compound. Therefore NK2 and NK4 have strong performance as well as the pore volume.

A unique case occurs in the NK4 sample, where the N<sub>2</sub> adsorption volume graphs at a glance are similar to those of other samples. However we found that, for the sample of NK4, it is classified into the macropore type because the resulted pore size was greater than 50 nm. This is supported by figure 4, where the isotherm graph of the sample of NK4 is very different than other samples where sample



**Figure 3.** The pore distribution of activated carbon CBW.



**Figure 4.** The relation of absorption volume for CBW activated carbon.

**Table 2.** Data of specific capacitance (single electrode) cathode LIC.

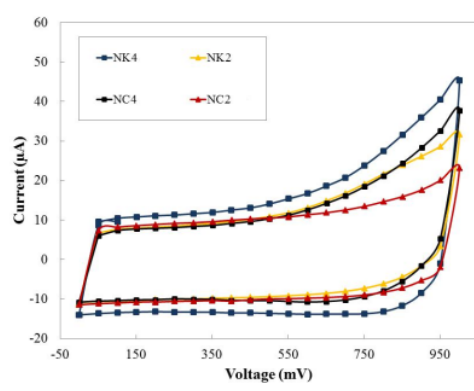
	$m_k$ (g)	I (A)	t (s)	V (v)	$C_{sp}$ (mF/g)
NC2	0.0241	0.00000803	109	0.481	33.7
NK2	0.0262	0.00000310	105	0.478	129
NC4	0.0225	0.00000289	105	0.478	157
NK4	0.0268	0.00000338	103	0.469	160

of NK4 included in type I based on the size of micropore, although charts distribution to figure 3 shows size of pore is dominant at mesopore. This pore size variation is influenced by the amount of pore formed on activated carbon. The more pores are formed the smaller the pore size, and vice versa. The NK4 sample had an 82.24 nm pore size. This result is very far from the pore size in the other sample. In addition, the size of the pore volume is very large, which is 1.525 cm<sup>3</sup>/g. Although the material formed has a large pore and volume size but the resulting surface area is small.

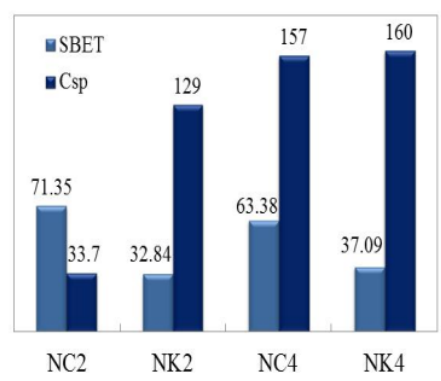
The CV curve is a graph that showed the relation between current and voltage. A CV curve exhibits hysteresis as shown in figure 5, where the wider hysteresis means the larger capacitance produced by electrodes supercapacitor. The CV graph shows the current generated in a certain time interval upon applied voltage. The size of the capacitance can be influenced by the sample size and the scan rate during CV testing. In this research, we use the scan rate of 5 mV/s to produce large current. This is caused by the reduction - oxidation reaction and quick diffusion of ions that enter into pores so as to produce great capacitance

Based on figure 5, NK4 possesses the best result, which is followed by NC4, NK2, and NC2. This can be seen by the magnitude of the charge current. The sample NC2 shows poor capacitance because the scan rate is high thus it is hard to diffuse into the ions pore. From the CV graph, we would saw performance of the cathode-specific capacitance (C<sub>sp</sub>) for single electrode of supercapacitor LIC cell. The larger current distance is formed in the cycle of greater the capacitance. So it can be concluded that samples of NK4 and NC4 have the greatest capacitance value. This is supported by the calculation data of the specific capacitance of supercapacitor LIC cathode from the activated carbon of CBW in table 2, which shows that the NK4 and NC4 samples have a cathode capacitance value of 160 mF/g and 157 mF/g.

The magnitude of the capacitance in a supercapacitor LIC can be affected primarily by the quality of activated carbon. Activated carbon that has large surface area can produce large capacitance value as well. Although with low surface area, if mesopores dominate the particle size, the capacitance can be enhanced. With the applied current, the collector will charge and induce the electrodes. Accessibility ions into the surface of electrodes will be affected by the diameter or indirectly in the form of the volume. If kept on a constant review from the relations of the value of of capacitance against of large volumes pores, then it obviously the value of of capacitance in the sample NK4 synchronous to the size of the volume of to which it belongs. However, in this case, the smallest cathode capacitance value is generated by NC2 which has the highest surface area as shown in figure 6. This is probably due to the small porous size of sample of NC2 so that the movement of charges that will fill the activated carbon becomes difficult. In addition, based on the CV results in table 2 shows the sample of NC2 has a very small current, so the resulted capacitance value is also quite small.



11 **Figure 5.** The pore distribution of activated carbon CBW.



11 **Figure 6.** The pore distribution of activated carbon CBW.

Similarly, the sample of NK4 yields the greatest capacitance value, although it has a small surface area, but the pore size produced is very large and is classified as macropore.

Supercapacitor performance is also influenced by the pore structure possessed by activated carbon as the cathode. The micropore type determines the charging stage and the magnitude of the capacitance value, whereas the macropore type determines the magnitude of the power density of the super-capacitor. The load that will enter the cathode surface will pass through the macropore as the starting gate, then enter to fill the smaller pore, in this case, mesopores. The more load that enters a pore occupies the pore the greater the specific capacitance it has. Because there are many factors of activated carbon for increasing the specific capacitance value of a supercapacitor, we believed that the process of making activated carbon itself becomes very important to understand. This indicates that the treatment of the material in the process of making activated carbon as the supercapacitor LIC cathode greatly affects the value of the resulting capacitance.

#### 4. Conclusions

LIC supercapacitor cathode has been produced from activated carbon of chicken bone waste (CBW). The result of BET test performed shows that the surface area is inversely proportional to the single electrode capacitance value resulted from the CV test. Based on 4 samples obtained, the sample of NC4 impregnated with CH<sub>3</sub>COOH and followed by carbonization at 650°C. Potential of CBW as porous activated carbon can be increased used metode and different treatments to produce a higher surface area. Furthermore, this porous carbon stimulates the better performance of the complete cell LIC obtained.

#### Acknowledgements

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