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by Rika Taslim

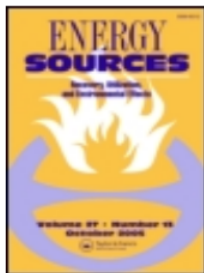
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Biomass-based activated carbon monolith from *Tectona grandis* leaf as supercapacitor electrode materials

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ABSTRACT

The complete study on the synthesis of biomass waste-based activated carbon monolith from *Tectona grandis* leaf for supercapacitor electrode materials has been successfully studied. This study was performed through a combination of chemical and CO₂ activation methods. The KOH was used as an activating agent in the chemical activation process. The pyrolysis process includes carbonization and CO₂ activation used integrated systems were conducted at temperature of 600°C under N₂ gas atmosphere, and CO₂ activation at different temperatures, specifically 750, 800, 850, and 900°C. The *Tectona grandis* leaf-based activated carbon (TL-AC) samples show different structures such as carbon rod (750°C), carbon sheet (800°C), and carbon fiber (900°C). The optimum-specific capacitance of the TL-AC samples as high as 168 F g⁻¹ in 1 M sulfuric acid solution using two-electrode configuration. This study provides an economic approach needed in the preparation of electrode materials with different structures for energy storage systems by abundant materials.

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Biomass; *Tectona grandis* leaf; activated carbon monolith; electrode materials; specific capacitance; supercapacitor

Introduction

The increased development of technology has caused a spike in energy consumption. Therefore, we need alternative energy sources to help satisfy the urgent demand. The energy storage devices were needed to be expected to efficiently save, have longer lifetime rather than batteries, store large quantities of power, and eco-friendly. One of the innovative energy storage technology products is the supercapacitor. Supercapacitors have been regarded as one of the most attracting research, due to their various applications in pulsing technique (Liu et al. 2016), digital devices (Dyatkin et al. 2013; Wu et al. 2019; Zhao et al. 2019), electrical vehicles (Choi et al. 2012; Wang et al. 2020), telecommunications (Cheng et al. 2021), aerospace and military (González et al. 2016), etc. This is due to superior pulse charge–discharge rate (Wang, Song, and Xia 2016), the high durability, excellent cyclic reliability and fast charging–discharging mechanism (Bose et al. 2012; Winter and Brodd 2004), high power density (Bose et al. 2012; Jayalakshmi and Balasubramanian 2008), and relatively simple cell configuration (Zhu et al. 2018). With their excellent performance, carbon materials have attracted attention in the field of energy conversion and storage studies (Ma et al. 2017). Various carbon materials such as carbon nanotubes (CNTs), single-walled carbon nanotubes (SWNTs), and graphene are often used in supercapacitor devices. The study on CNT and graphene-based supercapacitors in the past few years is stagnant; this is due to the high cost of the electrode material preparation process. Now, the concern has shifted toward carbon materials derived from biomass waste (Divyashree et al. 2016; Zhang et al. 2015).

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Biomass materials are one of the alternative energy sources with very abundant availability. Biomass materials can be classified into various types, namely, (i) wood and woody, (ii) herbaceous, (iii) aquatic, (iv) animal and humans waste, and (v) biomass mixtures (Tursi 2019). These materials have high compositions of cellulose, hemicellulose, and lignin. With the high composition of cellulose, hemicellulose, and lignin, biomass materials can be converted into activated carbon for various applications, one of the most is as supercapacitor electrode materials.

Waste management has been tagged as a huge challenge for some big cities, one of them is Pekanbaru city, Indonesia. The provision of waste separation plants in these cities helps segregate organic and inorganic waste. This was unsuccessfully performed due to increased number of carelessly disposed household, agricultural, factory, and industrial wastes. Most of these wastes are rich in carbon content, despite the presence of other elements with different portions, depending on the waste type and source. Natural wastes are usually burnt and produced of harmful gas pollution.

The raw materials for production of supercapacitor carbon electrodes can be made from several leaves waste that has been reported such as phoenix (Ma et al. 2017), tea (Peng et al. 2013), neem and ashoka (Biswal et al. 2013), willow (Liu et al. 2016), pineapple (Sodtipinta, Amornsakchai, and Pakawatpanurut 2017; Sodtipinta et al. 2017), ginkgo biloba (Hao et al. 2017; Zhu et al. 2018), *Terminalia catappa* (Taer et al. 2018a), etc. The preparation of electrode made from phoenix tree leaves waste was performed using two steps, namely, hydrothermal carbonization method and chemical activation with KOH produce specific capacitance 367 F g^{-1} and 240 F g^{-1} , respectively (Ma et al. 2017). These materials show promising potentials to serve as raw materials in production, based on availability and low cost of materials.

This study aims to report the potential use of *Tectona grandis* leaf (TL) waste for the production of activated carbon monoliths as electrode materials for supercapacitor. For the production of supercapacitor electrodes, generally use the addition of several materials such as conductive materials, adhesives materials, metal oxide, etc. In this study, the electrodes were prepared in monolith form due to their good mechanical stability and with the 3D pore structure that leads to high electrical conductivity and good accessibility of the electrolyte ion so that it can improve the supercapacitor performance (Moreno-Fernandez et al. 2017; Wang et al. 2019). The production of the electrode was performed through the chemical activation and followed by carbonization and CO_2 activation at different activation temperatures, that is, 750, 800, 850, and 900°C under an integrated system. The influence of various activation temperature on the physical and capacitive properties will be discussed in this study.

Experimental investigations

Sample preparation

Tectona grandis leaf waste were obtained from Riau university campus area, Pekanbaru. Indonesia. The initial step of the sample preparation is drying and pre-carbonization at 250°C for 2.5 h (Taer et al. 2018b, 2018c; Taslim, Agustino, and Taer 2018). The rarefaction of samples was carried out with a ball mill and then sieved to get a powder size $\leq 53 \mu\text{m}$ (Taer et al. 2018d). This sample powders were chemically activated using 0.4 M KOH followed by the process of molding into pellet forms using 8 tons of pressure. The pellets were carbonized at 600°C under N_2 gas atmosphere, followed by CO_2 activation at various temperatures, that is, 750, 800, 850, and 900°C. Then, the samples washed in distilled water to a pH neutral (pH = 7). Finally, the *Tectona grandis* leaf (TL-AC) pellets were polished to 0.20 mm thickness. The samples were coded TL-AC750, TL-AC800, TL-AC850, and TL-AC900, based on CO_2 activation temperature variations.

Characterization

The physical properties characterization of the TL-AC samples are (i) density (55) morphological structure and elemental compositions, (iii) crystallographic information, and (iv) specific surface area and pore size distributions. The morphological structure and elemental compositions of samples were observed from the SEM-EDX test (JEOL-JSM 6510LA). The BET-specific surface area and BJH pore size distribution of electrodes were observed using Quantachrome TouchWin v1.2. The crystallographic information was measured by the XRD (X-Pert powder panalytical). The interlayer spacing (d_{hkl}) was calculated by the Bragg equation.

$$n\lambda = 2d \sin \theta \quad (1)$$

The microcrystallite dimensions (L_c and L_a), were calculated by the Scherer in equation 2 (Cullity 1978; Lu et al. 2001; Serafin et al. 2019).

$$L_{c,a} = \frac{K\lambda}{\beta \cos \theta} \quad (2)$$

where L_c and L_a are stack height and width (nm), K is a constant depending on the reflection plane, λ represents wavelength (nm), β = bandwidth ($^\circ$), θ is the diffraction angle.

Capacitive properties measurement

Supercapacitor cells consist of electrodes from *Tectona grandis* leaf activated carbon, separator from duck eggshell membrane, current collectors from stainless steel plates type 316 L, and sulfuric acid electrolytes. The production of supercapacitor cells began by immersing TL-AC samples into a 1 M H_2SO_4 electrolyte for 2×24 h. Furthermore, all supercapacitor cell components were then arranged in sandwich structure and capacitive properties tested using the cyclic voltammetry method. This test was performed at scan rate 40 $mV s^{-1}$ and potential of 0–1 V in two-electrode configuration. The specific capacitance (C_{sp}), energy density (E), and power density (P) were calculated based on equations (Chang et al. 2013; Hao et al. 2017):

$$C_{sp} = \frac{2I}{sm} \quad (3)$$

$$E = \frac{45}{2} V^2 \cdot \frac{1}{3.6} \quad (4)$$

$$P = \frac{E}{\Delta t} 3600 \quad (5)$$

where I , s , m , V , Δt are current (A), scan rate ($mV s^{-1}$), mass of the TL-AC samples (g), cell voltage (V), and the discharge time (s).

Results and discussion

The physical properties analysis

The decrease in density data of TL-AC samples (Figure 1), which is caused by removal of compounds other than carbon and formation of new pores. The highest decrease in density occurred at TL-AC800 sample with outcome equal to 42.22% followed by a 36.35 and 22.29% increase in temperature, to 850°C with 900°C, respectively. This elevation in density was attributed to the breaking of more carbon bonds, which resulted in atom rearrangement, to further facilitate a decline in porosity (Farma et al. 2013). However, higher density have been implicated in reduced electrode porosity, and consequently

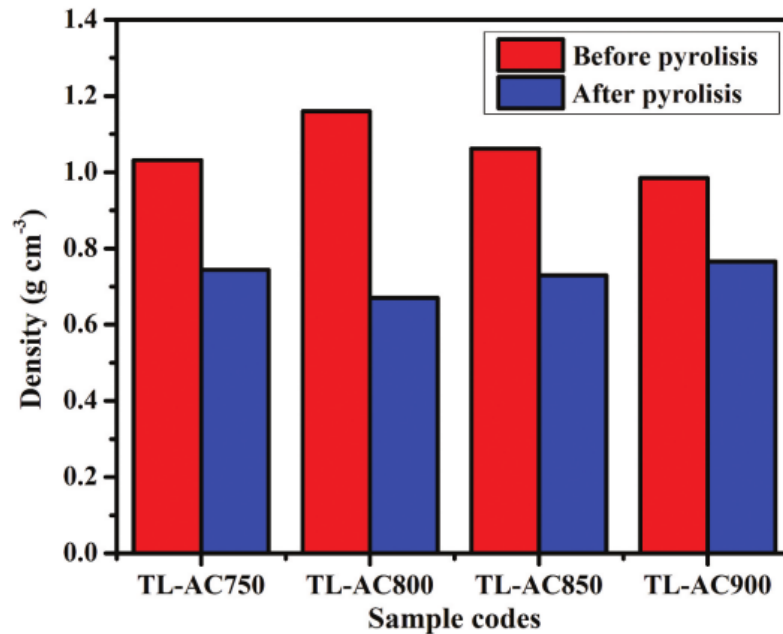


Figure 1. The density of TL-AC samples.

in decreased specific capacitance (Taer et al. 2019a). Based on obtained data, temperature of 800°C was determined to be the optimum level required for the formation process and development of pore structures. This was known to encourage the best outcome for specific capacitance. In addition, the changes in densities were similar to other biomass materials, especially oil palm empty fruit bunches (Deraman et al. 2010), oil palm empty fruit bunches fibers (Farma et al. 2011), and durian shell (Taer et al. 2019b).

Figure 2 displays the morphological structure of the TL-AC samples that were observed using SEM. The morphological structure of TL-AC750 sample (Figure 2 (a)) shows particles in the form of rods with particle sizes ranging 293–582 nm. The morphological structure of the TL-AC800 sample (Figure 2 (b)) shows the surface of the electrodes in the form of nanosheets with sizes in the range of 139 nm until 327 nm x 679 nm until 1679 nm. The TL-AC900 sample (Figure 2(c)) shows the presence of fiber with a clearer fiber shape with diameters ranging from 136 to 222 nm. The difference in the CO₂ activation temperature in TL-AC samples produces morphological structure with different structures, namely, rods, sheets, and fibers. The rod structure was obtained in the TL-AC750 because the lignin in the raw materials are not decomposed and still binds to cellulose. When the activation temperature was increased at the TL-AC800 sample, a sheet structure is obtained, where the lignin component begins to decompose due to heating and abandon little bond between lignin and cellulose. The highest activation temperature on the TL-AC900 sample results in the complete breakdown of the lignin and leaving only cellulose, which forms a single fiber structure.

The elemental compositions of TL-AC samples were characterized using energy dispersive X-ray analysis (Table 1). The carbon (C) element has the highest percentage compared to other elements in TL-AC samples. Furthermore, oxygen (O) is one of the elements present in biomass, besides existence of polymeric cellulose analysis causing end results of carbonization processes (Yahya, Al-Qodah, and Ngah 2015). Meanwhile, silica (Si) elements were obtained as a side effect of the product commonly obtained after activation processes. The purity level of this element is influenced by activation duration and heating process applied to samples (Promdee et al. 2017). Based on presence of potassium (K) element, the chemical activation process uses KOH activator and imperfect washing. The calcium (Ca)

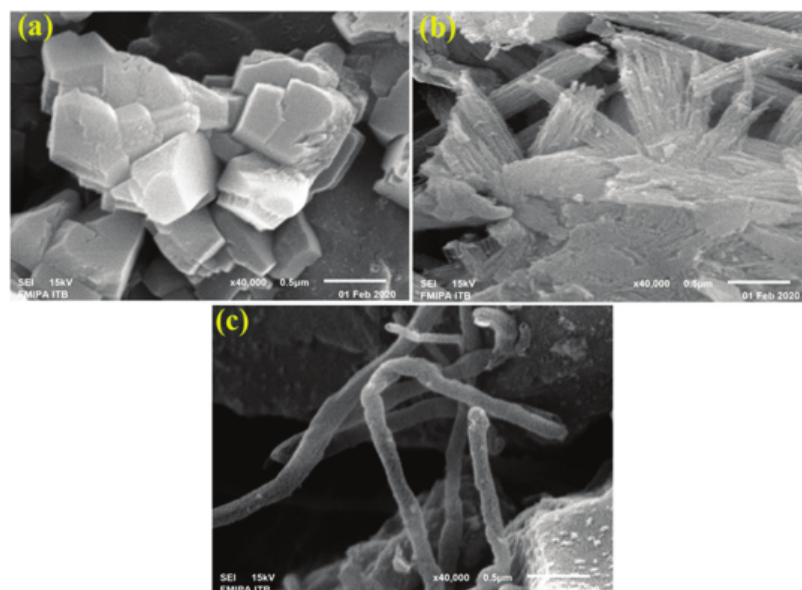


Figure 2. Morphological structure of TL-AC samples with magnifications 40,000 times.

Table 1. The elemental compositions of the TL-AC samples.

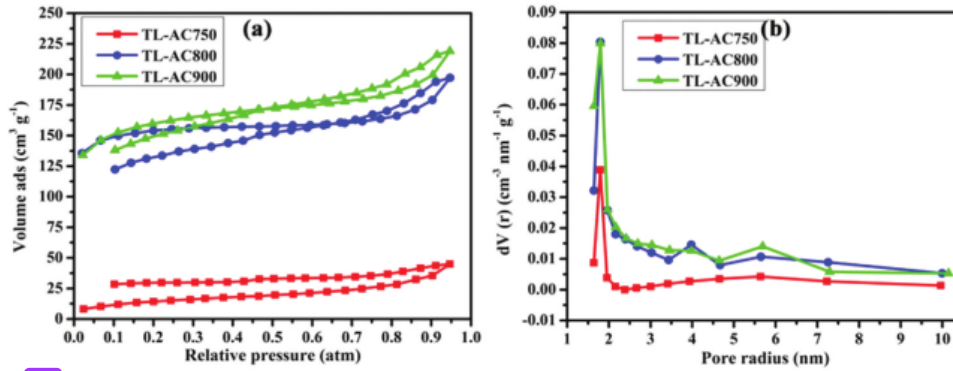
Elemental compositions	TL-AC750 Atomic (%)	TL-AC800 Atomic (%)	TL-AC900 Atomic (%)
C	81.34	83.30	83.52
O	14.50	14.40	13.79
Si	2.39	1.32	0.13
K	0.42	0.06	1.94
Ca	1.34	0.92	0.62
Total		100	

element is basically present in *Tectona grandis* leaf, although the mineral concentration depends on soil type.

The EDX results show an increase in activation temperature, which increases the number of carbon elements in activated carbon TL-AC. Therefore as the CO₂ activation temperature increases, slight impurities and organic matter are formed such as oxygen, silica, calcium, and several other materials with different molecular masses.

Figure 3 showed the BET-specific surface area (S_{BET}) and BJH pore size distribution of the TL-AC samples at three different activation temperatures. Figure 3 (a) demonstrates the volume ads-des @STP ($\text{cm}^3 \text{g}^{-1}$) versus relative pressure (atm). This was classified based on the IUPAC (International Union of Pure and Applied Chemistry) as a type IV isotherm, with H4 hysteresis loop type, which corresponds to the capillary condensation in mesopores for carbon materials (Biswal et al. 2013; Liu, Wang, and Teng 2005; Zhao et al. 2015). The TL-AC750 sample shows an imperfect hysteresis curve instigated by an incomplete desorption process, resulting from the presence of nitrogen trapped in pores (Qi et al. 2017). The shape of the ink-bottle neck triggers capillary condensation at the pore tissue to be blocked, due to narrowing, which slows down the desorption rate (Ayinla et al. 2019).

The BJH pore size distributions of the TL-AC samples are shown in Figure 3 (b). The average pore radius of the TL-AC samples increases from 2.718 nm to 1.258 nm with the application of high CO₂ activation temperatures from 750°C to 800°C, effectively. This change is attributed to the opening of more new pores, thus causing a shift in dominant size toward a relatively smaller direction, and



39 **Figure 3.** (a) Nitrogen adsorption–desorption isotherm and (b) BJH pore size distribution of the TL-AC samples.

a simultaneous increase in the pore volume from $0.069 \text{ cm}^3 \text{ g}^{-1}$ up to $0.306 \text{ cm}^3 \text{ g}^{-1}$. Moreover, the further elevation to 900°C causes a slight modification toward a greater size (1.320 nm). These data show an increase in pore volume from 0.069 up to $0.339 \text{ cm}^3 \text{ g}^{-1}$, which significantly elevates the specific surface area from $51 \text{ m}^2 \text{ g}^{-1}$ up to $512 \text{ m}^2 \text{ g}^{-1}$. The adjustments in terms of area provide more active sites in the electrolyte ions to diffuse on the electrode surface. The textural and structural parameters of the TL-AC samples are shown in Table 2.

Figure 4 displays the XRD pattern of the TL-AC samples. All the curves show the two wide peaks and can be indicated as an amorphous structure for carbon materials. These were recognized in biomass showing a random arrangement of atoms in sample. The scattering angle used was within the range of 10° – 60° , where 2θ was identified in the range of 24° – 45° for reflecting plane 002 and 100, respectively. Moreover, the sharp peaks observed indicate the presence of an element different from the carbon observed in the TL-AC samples, which was exhibited in the form of calcium carbonate (CaCO_3), at a 2θ angle of 29° (Taslim, Agustino, and Taer 2018). Table 3 shows the interlayer spacing (d_{hkl}) and microcrystallite dimensions (L_c and L_a) of the TL-AC samples.

Based on Table 3, the interlayer spacing (d_{hkl}), which calculated using equation (1), was found in the range of 3.49 nm up to 3.62 nm for d_{002} and 0.199 nm up to 0.204 nm for d_{100} , respectively. It showed the TL-AC samples structure that are higher than the graphite structure ($d_{002} = 0.335 \text{ nm}$); this indicates that the TL-AC samples have an amorphous structure (Ghosh et al. 2019). The microcrystalline dimension is calculated using equation (2) and obtained the L_c in the range of 0.899 nm up to 1.049 nm and 1.323 nm up to 4.779 nm for L_a , respectively.

The capacitive properties analysis

The CV curve of the TL-AC samples is shown in Figure 5. All the CV curves (Figure 5a) show the rectangular shape which is ideal for biomass-based activated carbon electrodes (González et al. 2016). The TL-AC800 sample presents a larger curve with a specific capacitance of 168 F g^{-1} and it was determined as optimum-specific capacitance, compared to other electrodes with specific capacitances of 135 F g^{-1} , 115 F g^{-1} , and 99 F g^{-1} for TL-AC750, TL-AC850, and TL-AC900 samples, respectively. This treatment was determined to possess the best CO_2 activation temperature required to produce

47 **Table 2.** The textural and structural parameter of the TL-AC samples.

Samples	S_{BET} ($\text{m}^2 \text{ g}^{-1}$)	S_{BJH} ($\text{m}^2 \text{ g}^{-1}$)	r_{average} (nm)	V_{total} ($\text{cm}^3 \text{ g}^{-1}$)
TL-AC750	51	16	2.718	0.069
TL-AC800	486	61	1.258	0.306
TL-AC900	514	68	1.320	0.339

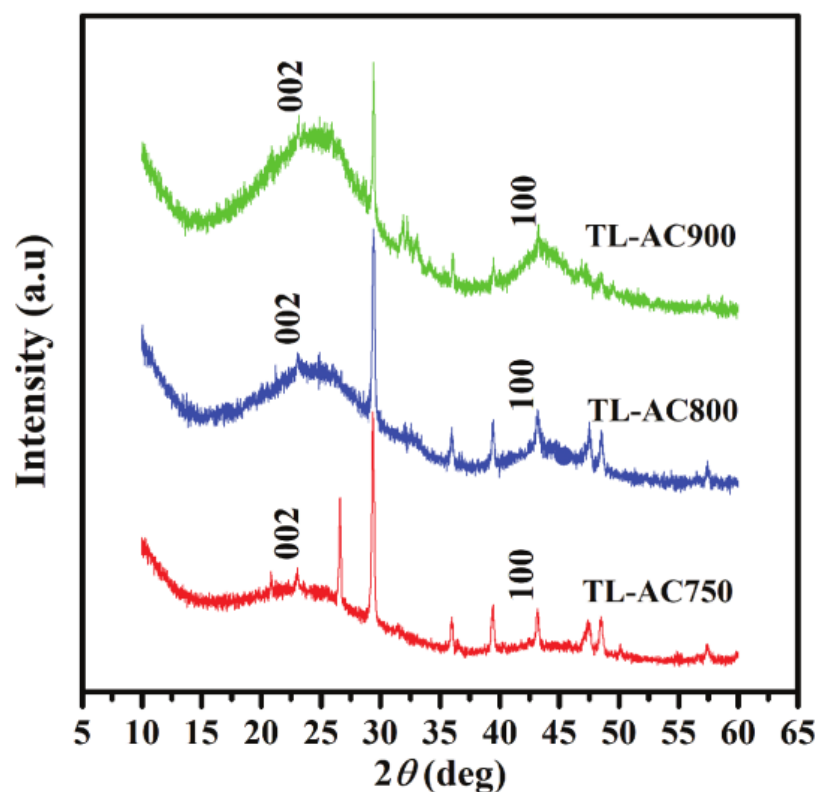


Figure 4. The X-ray diffractogram pattern of the TL-AC samples.

Table 3. The interlayer spacing and microcrystallite dimension of the TL-AC samples.

Samples	$2\theta_{002}$ (°)	$2\theta_{100}$ (°)	d_{002} (nm)	d_{100} (nm)	L_c (nm)	L_a (nm)
TL-AC750	25.462	45.306	0.349	0.200	1.007	1.323
TL-AC800	25.314	45.586	0.351	0.199	0.899	4.779
TL-AC900	24.555	44.389	0.362	0.204	1.049	3.135

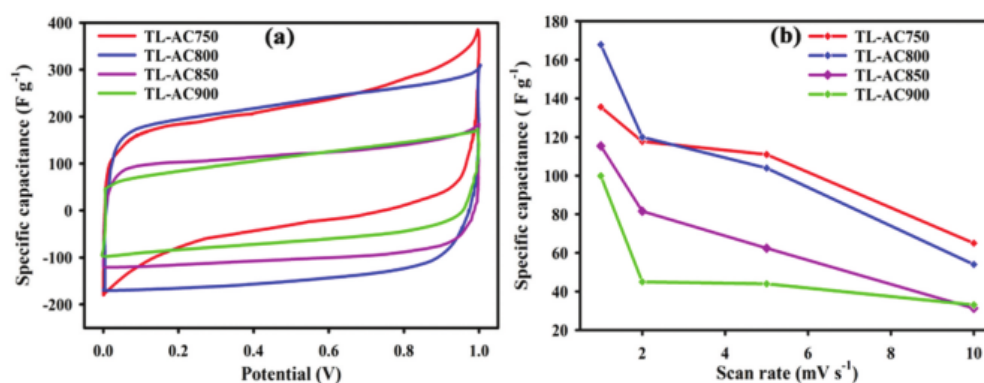


Figure 5. CV curve of the TL-AC samples (a) the specific capacitance vs. potential, and (b) the specific capacitance vs. scan rate.

Tectona grandis leaf activated carbon electrodes. The elevation in pyrolysis temperature from 750°C to 800°C shows a significant increase in specific capacitance from 135 F g⁻¹ to 168 F g⁻¹. This characteristic is attributed to the ability for higher temperature to facilitate suitable pore distribution required to provide relatively more active sites needed for the formation of surface charge layers. This was confirmed by nitrogen adsorption–desorption analysis, which showed a significant increase in BET-specific surface area from 51 m² g⁻¹ to 486 m² g⁻¹. Conversely, the addition of activation temperatures of 850°C up to 900°C showed decrease dramatically in specific capacitance from 115 F g⁻¹ to 99 F g⁻¹. This is attributed to the shift in pore size distribution on the higher activation temperature, which interferes with a suitable pore combination to produce higher capacitance of TL-AC samples. The dominant pores size shifts in a direction to the higher activation temperature of 800°C to 900°C, it was confirmed on the nitrogen adsorption–desorption analysis although the specific surface area is slightly increased. This evaluation was also supported by density data, indicating an increase after the pyrolysis process.

Figure 5(b) shows the specific capacitance versus scan rate variation. The specific capacitance of the TL-AC samples decreases with increasing scan rate. This was due to less time required for the ions to diffuse into the carbon electrode pores. Conversely, the application of the low scan rate will produce more time for ions to diffusion completely in the carbon electrode pore (Awitdrus et al. 2016). Table 4 shows the capacitive properties of the TL-AC samples based on CV measurement.

Table 4. The capacitive properties of the TL-AC samples.

Samples	C_{sp} (F g ⁻¹)	E (Wh kg ⁻¹)	P (W kg ⁻¹)
TL-AC750	135	18.75	64.8
TL-AC800	168	23.19	83.56
TL-AC850	115	15.97	57.6
TL-AC900	99	13.75	50.4

Table 5 shows comparison of capacitive properties for *Tectona grandis* leaf and other leaf waste-based activated carbon electrodes and other leaf waste-based activated carbon electrodes previously reported. The TL-AC samples with different morphological structures show great potential for use as supercapacitor electrodes, due to the good capacitive properties.

Table 5. The comparison of capacitive properties for *Tectona grandis* leaf and other leaf waste-based activated carbon electrodes.

Materials	C_{sp} (F g ⁻¹)	E (Wh kg ⁻¹)	P (W kg ⁻¹)	References
Phoenix tree leaves	367	-	-	(Ma et al. 2017)
Tea leaves	330	-	-	(Peng et al. 2013)
Neem tree leaves	400	55	569	(Biswal et al. 2013)
Willow leaves	216	-	-	(Liu et al. 2016)
Pineapple leaf	131	-	-	(Sodtipinta, Amomsakchai, and Pakawatpanurut 2017)
Pineapple leaf	202	-	-	(Sodtipinta et al. 2017)
Ginkgo biloba leaves	364	16	50	(Hao et al. 2017)
Ginkgo biloba leaves	272	9.2	48	(Zhu et al. 2018)
<i>Terminalia catappa</i> leaf	54	-	-	(Taer et al. 2018a)
<i>Schefflera octophylla</i> leaves	336	47.5	71.9	(Yang et al. 2019)
<i>Eucalyptus</i> leaves	213	16.2	47.2	(Jain, Kanungo, and Tripathi 2020)
<i>Tectona grandis</i> leaf	168	23.19	83.56	This work

Conclusions

Based on all the analysis in the previously section, it can be concluded that:

- *Tectona grandis* leaf waste is a promising biomass with good potentials for use as a supercapacitor carbon electrode. This waste was successfully converted into activated carbon monolith for supercapacitor carbon electrodes achieved by applying various CO₂ activation temperatures.
- The density of TL-AC samples was a decline after the pyrolysis process. Based on SEM characterization, the TL-AC samples have different morphological structures such as rods, nanosheets, and fibers. The optimum-specific surface area and specific capacitance of the TL-AC samples is 514 m² g⁻¹ and 168 F g⁻¹.

For the future research, it can be applied using a various chemical-activating agent such as KOH, NaOH, ZnCl₂, H₃PO₄, etc. for increasing the supercapacitor electrodes performance made from *Tectona grandis* leaf waste.

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