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ACTIVATED CARBON FROM FRUIT-BASED BIOMASS FOR SUPERCAPACITOR – MINI REVIEW

Norha Abdul Hadi*, Mawar Hasyikin Abu Seman, Madhiyah Yahaya Bermakai

[1]*Faculty of Applied Sciences, University Teknologi MARA (UiTM), Perlis Branch, Arau Campus,*
02600 Arau, Perlis, Malaysia

**Corresponding author:* norha.hadi@uitm.edu.my

Abstract

Derivation of activated carbon from biomass wastes for energy storage applications such as fuel cells and supercapacitors are attracting wide attractions as the world is now demand for other sustainable energy that can help to explore new technologies especially for energy conversion and storage. This is important because the world now is facing a rapid depletion of fossil energy. In this review, an outline of recent trends towards biomass-derived specifically from fruit-based biomass wastes is explained in a holistic manner. Thanks to their high carbon content, high specific surface area and developed porous structure, biomass-derived chars can be treated and converted into carbon. The performance of activated carbon in terms of Brunette Emmet Teller (BET) surface area, micropore volume, total pore volume and specific capacitance has been reported. This review showed that higher BET surface will contribute to higher pore volume in the activated carbon that makes them good candidates for the fabrication of electrodes in supercapacitor applications. This study was focused on providing a detailed comparison of published studies that utilized different physical and chemical routes and their effect of modification such as various activation temperatures and the ratio of activating agents towards the performance of the activated carbon under different parameters. Implementing chemical routes with an ideal 600°C – 850°C and inclusion ratio might be effective to produce high performance activated carbon.

Keywords : activated carbon, biomass wastes, chemical activation, supercapacitor

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Introduction

Since the rapid depletion of fossil energy, the world is now in demand for other sustainable energy that can help to explore new technologies, especially for energy conversion and storage (Li et al., 2019). Thus, in order to meet the new demand globally, developing high-performance, low-cost and environment friendly energy and storage techniques are strictly important (Hu et al., 2019). Global bioenergy statistics 2019 reported that residues originated from the agriculture sector were one of the promising sectors that contributed to the development of bioenergy production. Data showed that approximately 4.3 billion to 9.4 billion tons of residues had been produced annually from all major crops worldwide including Malaysia produced at least 168 million tons of biomass residues.

All these tons of residue products has puts the country in good condition to promote biomass as an energy storage material because the cheaper the renewable energy sources, the least impact on our mother nature (Hiremath et al., 2019). The managing director of Global Green Synergy, Datuk Joseph Lim Heng Ee saw an opportunity to make Malaysia one of the countries that maximize the usage of "clean and green" renewable energy. This initiative came as he noticed that millions of tons of biomass palm residues and empty fruit peel wastes are often left to rot. This biomass wastes will harm the environment as they release the greenhouse gas methane.

Looking that this problem has become severe day by day, the Malaysia's government has taken the initiative by launching the National Biomass Strategy 2020 back in the year 2013. This strategy is to develop new industries that exploit biomass wastes for high quality products in creative and innovative ways. The industries were encouraged to reduce the dependency on natural resources by transforming the biomass wastes to become valuable resources, either by converting to power generation or other products (Ghafar et al., 2017).

Green technologies have been introduced by the industries. For instance, supercapacitors. Supercapacitors are considered one of the promising energy storage devices for many industrial applications due to their low manufacturing cost, excellent round trip efficiency, and long-life cycle (Shanmuga et al., 2020). Supercapacitors are high performance devices that can be charged and discharged in seconds or minutes.

The energy storage mechanism in a supercapacitor is based on an accumulation of charges at the interface between electrode material and an ionically conductive electrolyte, where the electric double layer is formed (Yakaboylum et al., 2019). This double layer formation has caused adsorption and desorption process of the electrolyte ions occurred rapidly at the interface since non-faradaic process involved at this stage.

The electrodes materials of the supercapacitor were fabricated with a large surface area of carbon, preferably activated carbon. As described by the International Union of Pure and Applied Chemistry (IUPAC), activated carbon is a porous carbon material that has been exposed to gaseous reactions, often with the addition of chemicals to strengthen its adsorption properties prior, after or during carbonization. Activated carbon has a well-developed porous structure and chemical surface functionality to increase interaction between polar and non-polar adsorbents (Oginni et al., 2019). Li et al. (2019) in their study, mentioned that due to their high specific surface area, excellent conductivity, cycling stability and good capacitive performance, porous carbons had attracted widespread interest as supercapacitor electrodes.

The activated carbon's physical and chemical characteristics vary according to the level of activation, biomass sources, the activation temperature and an oxidizing agent. In recent research, starting material of activated carbon was most likely to use agriculture wastes as there are abundant sources and they are also high content in carbon. It is also can help to reduce environmental problems.

This mini review focused on the difference’s activation methods, such as physical or chemical activation, activation temperatures, and activation agent impregnation ratios carbon materials derived from the fruit-based biomass done by previous researchers that affected the development of porous activated carbon as electrode materials for supercapacitor applications.

Methods

Biomass Wastes

Compared to other possible electrode materials such as graphene and carbon nanotubes, deriving carbonaceous materials from biomass wastes offered more benefits because they are low-cost, available in large quantities, renewable and presence naturally porous in the structure formation (Sesuk et al., 2019). The high porosity of porous carbon derived from biomass can provide a channel for ion transport, while at the same time providing a contact surface for gas adsorption, which is the secret to making high performance electrode materials for supercapacitors (Hu et al., 2019). There are four main groups of biomass materials which are plant-based, fruit-based, animal-based and finally microorganisms-based. Fruit-based and plant-based biomass wastes were the two most commonly biomass materials that the researchers have used for the application of EDLC. As a result of high percentage of lignin, cellulose and hemicellulose content in those two of biomass wastes, it has contribute to the carbon yield (Thomas et al., 2019). Figure 1 shows the biomass wastes’ conversion into activated carbons that have been widely used in energy storage devices (Vijayakumar et al., 2018).

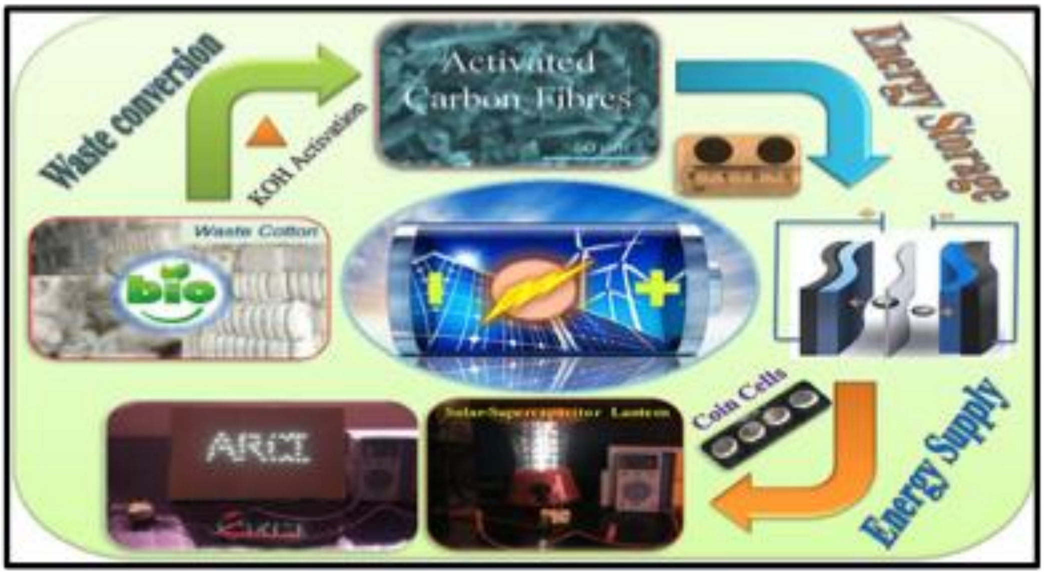

Figure 1. Schematic representation of turning biomass waste conversion into green energy storage (Vijayakumar et al., 2018).

Biochar Conversion Technique

The preparation and conversion of biochar resulted in the production of activated carbon. Usually, the conversion process consists of carbonization and followed by an activation process that can be done separately in two stage processes or combined in a single process (Abioye et al., 2015). However, two stage process most likely the well-known technique among the researchers as activated carbon that produced from separate carbonization and activation showed the increasing of the micropores (about 90%) compared to activated carbon that produced through single step, larger pore diameters with about 90% of it is mesopores have been reported (Li et al., 2019).

Activation is a method of converting carbonaceous materials into activated carbon using a regulated atmosphere and heat through thermal decomposition in a furnace (conventional heating) or a microwave. In general, the activation cycle is applied to further alter or improve the biochar's surface region, pore structure and functional surface groups. The aim is to achieve a final activated carbon product with a well-organized pore structure, a large volume of pores and a high specific surface area, all of which usually contribute to significantly improved electrochemical performance (Yakaboylu et al., 2019). The activation process has two types, chemical activation and physical activation.

According to Oginni et al. (2019) chemical activation requires precursor to be soaked with a chemical activating agent, followed by the impregnated precursor is triggered at high temperatures, while physical activation is a two-stage process involving the thermochemical conversion of the precursor to the chars using motionless or inert atmospheric carbonisation reaction and followed by the activation of the char using an oxidizing gas such as carbon dioxide (CO_2), steam or high temperature air.

Activation Temperature

In general, different techniques of suitability and appropriate activation could be changed, and material characteristics could be varied. Although physical activation is easier and more environmentally friendly than chemical activation, the physical activation process was typically performed at high temperatures. Thomas et al. (2019) stated that it is a well-known fact that chemical activation requires low activation time and temperature compared to physical activation. The chemical activation process embodies the thermal treatment of biomass carbon precursor and the activating agent at a temperature range of 450°C–900°C.

Activator Ratio

The impregnation ratio is defined as the percentage of the chemical activation agent's weight to the starting material (Yahya et al., 2015). As discussed in the previous section, the ideal range of activation temperature is between 500°C to 600°C. X. Li et al and I. A. W. Tan et al added that the pore size distribution of activated carbon could be further regulated by careful selection of various activation parameters such as impregnation method, activation temperature and precursor ratio. Some of the published works that took biomass wastes such as fruit-based and plant-based and varied their activator ratio.

Results and Discussion

Biomass Wastes from Fruit-Based

Yagmur et al. (2020) have prepared the activated carbon (AC) from oleaster fruit using different fruit fractions. The purpose of the study was to compare the effects of fruit fractions with different composition of lignin, cellulose and hemicellulose on the properties of the AC. AC derived from flesh fraction have recorded the highest BET surface area, micropore volume and total pore volume as high as 1816 m^2/g, 0.735 c^2/g and 1.293 c^2/g, while AC from seed fraction reported the lowest BET surface area, micropore volume and total pore volume.

Table 1. Effects using different fruit fraction of Oleaster fruit towards the performance of AC (Yagmur et al., 2020)

Samples	Lignin (%)	Cellulose (%)	Hemicellulose (%)	Carbon (%)	BET (m^2/g)	Micropore volume (c^2/g)	Total pore volume (c^2/g)
O-peel	53.03	13.24	11.20	52.11	1305	0.580	1.179
O-flesh	16.83	36.91	4.67	42.88	1816	0.735	1.293
O-seed	45.05	22.42	10.37	46.99	1149	0.536	1.176

These results showed the significance of the chemical composition towards the effect on the surface area and the derived of AC's pore structure. Other proposed works from Saha et al. (2018) have also prepared AC from fruit-based biomass using different types of fruit and different fruit fraction through a simple chemical activation process. This study used walnut shell, orange peel and apricot seed. They found out that from all these three samples, orange peel has reported the highest BET surface area, 1636 m^2/g followed by the walnut shell, 1129 m^2/g and apricot seed 727 m^2/g.

Table 2. Effects using different types of fruit with different fruit fraction towards the performance of AC (Saha et al., 2018)

Samples	Carbon yield (g)	BET (m^2/g)	Micropore volume (cm^3/g)	Total pore volume (cm^3/g)
Walnut shell	0.80	1129	0.52	0.60
Orange peel	0.31	1636	0.74	0.85
Apricot seed	0.60	727	0.32	0.34

Composition of chemicals in each fraction in various fruits depend on the geographical condition and the fruit sources (Saidur et al., 2011). Kim et al. (2019) commented that there is no ultimate analysis report that stated which fraction of the fruit will contribute to better performance of AC but if taking the chemical nature of each fraction of fruit into consideration, high content of lignin will lowering the BET surface area and the total pore volume of AC.

Although hemicellulose and cellulose are less stable and contribute to low carbon contents, those two components contribute to the porosity of the biochar. For these reasons, to decide on starting materials from biomass wastes, it is important to look for materials that lower lignin contents but high in cellulose contents to realize which biochar will give good conductivity and controllable defects for energy storage application (Cagnon et al., 2009).

Effect of Chemical Activation Towards Derived Activated Carbon

Many scientific studies have shown that chemical activation is a more promising technique compared to physical activation because it allows activated carbon to be synthesized with adjustable porosity that consists of micropores and mesopores, lower activation temperature and better BET surface area (Yakaboylu et al., 2019).

Research conducted by Dai et al, is to report a model in order to describe the effect of pore structure on the EDLC. This work studied the comparison between the activated and non-activated biochar derived from the chestnut shell. Plus, all the activated samples were impregnated with chemical activation process using different types of activating agents such as zinc chloride ($ZnCl_2$), potassium hydroxide (KOH), potassium carbonate (K_2CO_3) and phosphoric acid (H_3PO_4). The geometric parameters in activated and non-activated biochar showed a big difference while AC activated by potassium hydroxide reported the highest BET surface area and total pore volume compared to other activators.

Table 3. Derived AC from chestnut shell at different activating agents (Dai et al., 2020).

Samples	**Activator**	**BET_{Total} (m^2/g)**	**BET_{Micro}(m^2/g)**	**V_{Total} (cm^3/g)**	**V_{Micro} (cm^3/g)**
RBC	-	345.15	300.6	0.33	0.2
ABC	H_3PO_4	208.02	167.24	0.3	0.15
ABC	$ZnCl_2$	273.85	221.76	0.24	0.15
ABC	K_2CO_3	504.42	483.68	0.35	0.33
ABC	KOH	1347.93	1102.58	1.09	0.65

Reported by Boujibar et al. (2019). Several research showed that KOH or NaOH activation method as the most common manufactory route to produce micropore-rich, high pore volume, and large surface area activated carbon. To support that, Guan et al. (2019) also mentioned in their study that biomass is converted into carbon materials by the activation effect of KOH at elevated temperature, which usually exists in a particulate form with a high specific surface area and rich porosity.

In another published work by Suarez et al. (2020), grape seeds have been used as precursors of activated carbon for the application of supercapacitors. This study aims to look at either carbon biochar activated through physical activation or activated through chemical activation that will give better performance. High porosity developed in the samples activated by KOH activator was reported to have about 1500 to 2000 m^2/g. According to results recorded in other works such as Guardia et al. (2018) and Olivares-Marín et al. (2009), the fact that the chemical activation process has produced carbons with a highly developed porous network made almost exclusively of micropores has also been confirmed.

Table 4. Comparison between physical and chemical activation of the AC derived from grape seeds (Suarez et al., 2020).

Activation Technique			
Physical Activation		**Chemical Activation**	
Carbon	**BET (m^2/g)**	**Carbon**	**BET (m^2/g)**
SAC	819	SAK	1512
SHAC	950	SHAK	1791
SA900C	825	SA900K	1351
SA927C	965	SHA900K	1117

Sesuk et al. (2019) had conducted an interesting study when they used coconut coir-pitch as starting materials and they combined both chemical and physical activation process and looked for the effectiveness at different ratio of chemical activation, in this case NaOH. The microstructure and morphology of the activated carbon samples observed by SEM (Figure 2, a–f) show increased amount of porosity from AC-1 to AC-3.

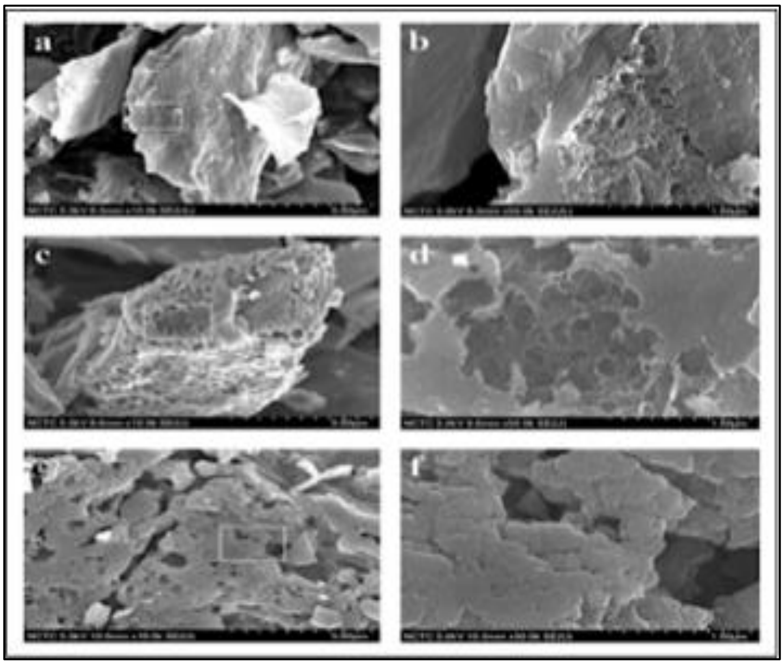

Figure 2. Scanning electron micrograph (SEM) from AC-1 to AC-3 (Sesuk et al., 2019)

The pores appeared to be shallow in AC-1, while in AC-2 pores were connected and some pore-widening was observed. Macropores and mesopores, which are known to facilitate the transport of electrolyte ions, were easily observed. In AC-3 (Figure 2, e–f), a rather dissimilar microstructure to AC-1 and AC-2 was observed, where the macropores appeared to be highly interconnected, while the surface of the carbon was rough and layered.

Effect of Activation Temperature on the Performance of Activated carbon

Many established works focus on understanding the effect of the activation temperature on the physical and chemical properties of the AC. Fu et al., (2020) took an initiative in synthesized AC derived from the walnut shell by the KOH activation process. This study reported the highest BET surface area at 800°C activation temperature. The WS-600 sample has the highest volume of micropores, microporous surface area and specific capacity. As the activation temperature increase from 500°C to 600°C, the micropores and specific capacitance increased however it decreased at 800°C activation temperature, which leads to a conclusion that 500°C to 600°C is the optimum activation temperature range since micropores provide a rapid ion transfer path.

Table 5. BET surface area and specific capacitance of AC from walnut shells (Fu et al., 2020)

Samples	BET surface area (m^2/g)	Specific capacitance at 0.5 A/g
WS-500	696.76	220.14
WS-600	1315.62	262.74
WS-700	1239.96	200.30
WS-800	1789.12	179.02

Serafin et al. (2019) have successfully obtained a activated carbons originated from *Trametesgibbosa* or known as a lumpy bracket. The focused of this study is to evaluate the electrochemical properties of biochars that are ranging from 600°C to 900°C activation temperature. For samples from 600°C to 850°C, the BET surface area continuously increased but decreased at 900°C. High carbonization temperature favoured pore development, but biomass oxidation was too intensive at 900°C and the porous structure was not well developed as at 850°C. Calculation of pore size distribution and pore volume for pores (0.3–1.1 nm) was using CO_2 adsorption isotherms.

The increase in carbonization temperature to 850°C resulted in an improvement in biochar's ability to absorb CO_2. It was related to the formation of pores in greater amounts. CO_2 adsorption at 900°C probably decreased as a result of damage to porous structure. Thus, the optimum carbonisation temperature combined with chemical activation for the most efficient CO_2 sorbent production was 850°C.

Table 6. Electrochemical properties of AC from *Trametesgibbosa* (Serafin et al., 2019)

Samples at different activation temperature	**BET surface area (m^2/g)**	**V_T (cm^3/g)**
AC-600	483	0.302
AC-650	559	0.268
AC-700	967	0.453
AC-750	1482	0.756
AC-800	1750	0.851
AC-850	1968	1.144
AC-900	1208	0.806

Effect of Activator Ratio on the Performance of Activated Carbon

Lin et al. (2020) extracted hemicellulose from the Pomelo peel and used a different zinc chloride ratio as an activating agent to activate the carbon materials. In this study, the highest BET surface area recorded was 1361 m^2/g at one $ZnCl_2$ ratio activated at 500°C, while the lowest BET surface is 399 m^2/g at two $ZnCl_2$ ratios.

A similar study reported by Samantray et al. (2020) derived from *Saccharum spontaneum*, a kind of wasteland weeds. The precursors were activated using $ZnCl_2$ at different ratio of activator (0.5-1). They noticed that the highest BET surface area contributed by AC was at one ratio of $ZnCl_2$ activated at 500°C, agreed with the Pomelo peel derived AC study. Temperature effect on surface area and volume of the pore, keeping the ratio of impregnation constant as 1. The total surface area and micropore area show the maximum value at 500°C and 60 min. $ZnCl_2$ concentration has a high effect on the pore structure and surface area derived from activated carbon. The pores are distributed in a microporous range with a chemical ratio of 0.5 to 0.75. Rising the ratio to 1 results in a certain mesoporous range. With the increment in chemical ratio, the volume of adsorption increases as it leads to a higher total volume of the pore.

Table 7. BET surface area of AC derived from wasteland weed (Samantray et al., 2020)

Samples	**Activator Ratio**	**Activation Temperature (°C)**	**S_{BET} (m^2/g)**
A1	0.5	400	304.08
A2	0.75	400	657.15
A3	1	400	1080.34
A4	0.5	500	496.89
A5	0.75	500	666.25
A6	1	500	1205.71
A7	0.5	600	634.76
A8	0.75	600	876.46
A9	1	600	1134.12

Other previous work from Moralı et al. (2018), activation temperature and the activating agent was also selected as main factors to study the performance of the AC derived from the sunflower seed. The activator usage ratio ranging from 2 to 4 with the activation temperature from 400°C to 600°C has resulted to highest BET surface area, up to 1534.93 m^2/g at 3:1 activator ratio. The pattern of BET surface area increased from 2:1 to 3:1 however, it started to decrease at 4:1.

Table 8. S_{BET} of AC derived from sunflower seed (Moralı et al., 2018)

Samples	Activation Temperature (°C)	S_{BET} (m^2/g)
AZ-2:1	400	28.13
AZ-3:1	400	631.43
AZ-4:1	400	548.04
AZ-2:1	500	1042.89
AZ-3:1	500	1430.11
AZ-4:1	500	1146.00
AZ-2:1	600	1241.81
AZ-3:1	600	1534.93
AZ-4:1	600	1418.93

The high amount of the triggering agent caused pore widening which resulted in lower values for the particular area of the surface and volume of micropores. Owing to the contribution of mesopores, the total volume of pores also increased. Micropore volume has been observed to decrease as the impregnation ratio increases from 2:1 to 3:1. The greater impregnation ratio has also influenced impregnated sample swelling, which encouraged mesopore formation. Besides, the rise in micropore and mesopore volumes increased the total carbon pore volume prepared at 4:1 impregnation ratio and 600°C activation temperature (Nayak et al., 2017).

Conclusion

Based on their structure, surface area, pore size, pore distribution and electrochemical behavior, numerous biomass wastes has been largely reviewed for activated carbon materials to enhance the existing energy and power density factors. Given the range of methods, physical and chemical criteria that need to be addressed, the precise use of activated carbon is a major factor in deciding the choice of starting material due to variations in the molecular composition of each biomass waste. In the long term, these carbon-derived electrode materials will be scaled up to produce highly flexible, stretchable and foldable electronic devices that are environmentally sustainable, non-toxic and cost efficient. Future efforts will be made to develop new synthetic methods to generate carbonaceous materials from biomass and to manufacture carbon-rich materials in energy storage devices into flexible electrodes to foster a greener environment.

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