Malaysian Journal of Chemical Engineering & Technology 3 (1) (2020) 35–43

Malaysian Journal of Chemical Engineering & Technology

Journal Homepage: http://myjms.mohe.gov.my/index.php/mjcet

Conversion of automotive paint sludge to activated carbon via microwave pyrolysis technique for supercapacitor application

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Abstract

Conversion of waste to wealth has been one of the ways to reduce the volume of industrial waste to disposal site, hence reducing the impact to the environment. In this work, paint sludge from an automotive industry (APS) was converted into activated carbon through chemical activation (potassium hydroxide (KOH)) using microwave pyrolysis technique. The effect of power and radiation time on the produced activated carbon were investigated and characterised (carbon content, surface area, and pore volume) to identify the possibility of application as a supercapacitor. Potassium hydroxide activation of the APS char via microwave pyrolysis has shown that power level and radiation time has influenced the yield of the APS activated carbon. A longer radiation time and higher power supply has produced activated carbon having higher carbon contents, lower impurities, higher surface area and higher pore volume. Thus, the APS activated carbon obtained via microwave pyrolysis at power supply 1000 W and 45 minutes radiation time had produced the highest surface area and total pore volume of 434.3 m^2/g and 0.2901 cm^3/g, respectively. However, the produced activated carbon is not suitable for the supercapacitor application as the minimum surface area requirement must be more than 1000 m^2/g. The pore size of the activated APS char produced in this study was in the range of mesopores size which was also considered very poor for supercapacitor application. The outcome of this research has shown that the produced activated carbon could otherwise be used for other application than a supercapacitor.

Article Info

Article history:

Received date: 30 March 2020
Accepted date: 23 October 2020

Keywords:

Activated carbon
Automotive paint sludge (APS) char
Chemical activation
Supercapacitor
Microwave pyrolysis

1.0 Introduction

The automotive industry is among the largest contributor of waste generation. Automotive paint sludge (APS) is a waste product as a result of the painting process of vehicle bodies took place in a series of large enclosures known as paint spray booths (Ruffino & Zanetti, 2010). Several research have been done in order to overcome the problems from the wastes generated in the automotive industry (Burande, 2017; Januri et al., 2014; Salihoglu & Salihoglu, 2016). Automotive paint sludge can be converted to fuel through a thermochemical conversion technique such as a pyrolysis process (Kim et al., 1997). In the course of producing liquid fuel, the process also produces a considerable amount of solid fraction (char) which shall be disposed of properly at the disposal site (incineration). On the other hand, it is beneficial to utilise the solid fraction as it has high potential to be recovered to a more valuable product especially for energy applications.

The solid char produced from the pyrolysis process is the product from either a full or an incomplete recovery of organic materials. The operating conditions shall be adjusted based on the desirable product to be produced. Januri et al. (2015) has reported that, approximately 9–13 wt. % of APS char was recovered from microwave pyrolysis of APS, operating at an optimum condition for maximising production of pyrolytic oil. Just like other type of chars, APS char can also be used as an activated carbon. The charcoal can be activated by physical and chemical activation through a pyrolysis (carbonisation) process. Recently, microwave assisted pyrolysis has been discovered as one of the thermochemical conversion techniques for conversion of wastes. However, the

source of raw materials to produce activated carbon varies, so do the methods of conversion and activating techniques as shown in Table 1.

Microwave assisted pyrolysis technique has been actively investigated for converting waste materials such as automotive paint sludge (Syed Abdul Rahman et al., 2015), sludge of biological waste water treatment plant (Lin et al., 2020), sewage sludge (Domínguez et al., 2006) and many other. The use of microwave in processing high moisture waste materials are feasible for the fact that water, being a polar molecule interacts well with the electromagnetic wave (microwave). The interaction causing dipole moment being converted to heat hence inducing pyrolysis of the waste materials. The use of microwave technique in the production of activated carbon has been reported as successful and effective by several authors (Liew et al., 2019; Liu et al., 2010). The potential utilisation of APS char as activated carbon has been reported by Ruffino and Zanetti (2010). Previously, Kim et al. (1995) had investigated on producing activated carbon from automotive paint sludge for application in the wastewater treatment.

Depleting natural resources of energy is a major concern of the world nowadays. According to Kalyani & Anitha (2013), the energy demand of the world in the year of 2050 is expected to be twice the current demand, along with the non-stop usage of fossil fuel. Fossil fuel remains as the best choice of energy for consumption despite being non-environmental friendly as the combustion of such a fuel constantly emitted pollutants to the environment. Thus, urgent needs are increasing to search for a greener technology which will provide the source of energy that has low environmental impact and sustaining natural resources of energy (Kalyani & Anita, 2013). Renewable energy for electricity generation can be obtained from hydropower, geothermal energy, solar energy, and many more. However, in harnessing the intermittent electricity being generated from these renewable sources, the major requirement is an efficient energy storage system (Abioye & Ani, 2015). Thus, supercapacitors in which are good for energy conversion and storage, have been attracting many research in this area (Jin et al., 2014(a); Nitnithiphrut et al., 2017; Shen et al., 2012). Electrical storage device is in a serious demand throughout the world with the increasing demand on energy. With development of technology in every field, the use of storage device for electricity has taken an important part in this development. There has been great interest in developing and refining more efficient energy storage devices (Halper & Ellenbogen, 2006).

Table 1. Production of activated carbon from various materials

Sample Material	Method of production
Coconut shell (Iqbaldin et al., 2013)	50% KOH as the activating agent. Microwave oven activation 600 W for various times.
Distiller dried grains (Jin et al., 2014a)	KOH to biochar ratio of 0.075 mol/g activated at 950 °C to 1050 °C with 5 °C/min heating rate for 3 hours.
Corn stover (Jin et al., 2014b)	Microwave and slow pyrolysis using KOH and NaOH as activation reagent.
Non-metallic printed circuit board waste (Rajagopal et al., 2016)	Physical activation by using CO_2 as the activation gas. Heated at temperature 650, 750, and 850 °C for time intervals 3 h and 5 h.
Waste tea (Inal et al., 2015)	H_3PO_4 undergo 30 s microwave pre-treatment while the K_2CO_3 undergo impregnated at 110 °C for 24 h. Both samples undergo carbonization at 450 °C and 650 °C, respectively for 60 mins.
Coffee ground waste (Wang et al., 2016)	Activation by using equal weight of KOH as activation agent and undergo pyrolysis (carbonisation) at 600, 700, and 800 °C for 2 h.

A supercapacitor is an activated capacitor with very high surface areas. It is known with the names such as electric double-layer capacitors (EDLC) and ultracapacitor (Lehtimäki et al., 2014). Li et al. (2017) fabricated three-dimensional hierarchical porous carbon (HPC) material from oily waste sludge for the application of supercapacitor. The waste sludge was converted to carbon rich material by carbonisation and the produced carbon was de-ashed using hydrogen fluoride (HF) prior to impregnation with activating agent. Potassium hydroxide (KOH) solution was used to activate the produced carbon materials. The work had proved that the activated carbon produced had high surface area and rational pore distribution, suitable for the use as a supercapacitor.

Limited work is available in utilising APS char (activated) for supercapacitor application. The fact that the APS itself contains high percentage of titanium dioxide (TiO_2) (ca. >50 wt. %) along with other inorganic materials makes it interesting to study its application for supercapacitor (Rahman et al., 2016). This is because titanium dioxide (TiO_2) has been found to be an alternative oxide for supercapacitor (pseudo capacitor) because of its good dielectric materials and faradaic capacitance (Nitnithiphrut et al., 2017).

Thus far, limited studies on chemical activation of APS char using KOH as an activation agent and

carbonisation via microwave assisted irradiation system have been reported. Therefore, this forms the basis of the current study to characterise the activated carbon derived from APS char using microwave irradiation pyrolysis technique. The properties of the material produced such as carbon content, surface area, and pore size were analysed to study the potential of the material for supercapacitor electrode. For this application, an ideal surface area of >1000 m^2/g is desirable (Sevilla & Mokaya, 2014).

2.0 Methodology

2.1 Materials

Automotive paint sludge char was obtained from the previous research on the optimization of the liquid product of the automotive paint sludge (Januri et al., 2014). The sample was obtained when APS was subjected to microwave pyrolysis process at 1000 W for 30 minutes. The resulting char sample had a very hard structure and size reduction was achieved by crushing using marble mortar to a size around 1 mm. The crushed APS char was then sieved by using a 1 mm mesh to separate the coarser particles from the fine particles of the APS char.

2.2 Preparation of Activated APS Char

The prepared APS char of mass 30 g was added to chemical reagent 5 M potassium hydroxide solution in the ratio of 1:3 (mass), respectively and impregnated overnight in room temperature. The impregnated sample was then carbonised directly, without having to remove any moisture prior to using a microwave pyrolysis technique at various power level (600, 700, and 1000 W) at various irradiation times (10, 15, and 30 minutes) with the nitrogen gas as protection (100 ml/min). The samples are labelled as S1–S10 according to its process conditions as shown in Table 2. After completion, the sample was cooled to room temperature in the microwave oven with nitrogen gas protection. In order to dispose of excess KOH, the sample was mixed with the 0.1 mol/L sulphuric acid. Finally, the sample was washed with deionised water until the pH value of the water-sample mixture was about 7. Lastly, the leached product was dried in an oven at 105 °C, overnight (Jin et al., 2014a). The setup of the experiment is as illustrated in Fig. 1.

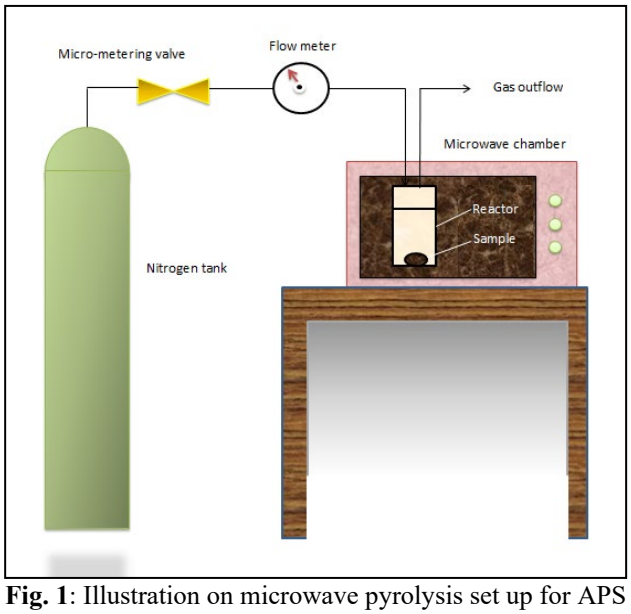

Fig. 1: Illustration on microwave pyrolysis set up for APS char activation

The char yield and volatile yield are calculated by Eq. (1) and (2);

$$Char\ Yield\ (\%) = \frac{mass\ final\ (m_f)}{mass\ initial\ (m_i)} \times 100 \quad (1)$$

$$Volatile\ Yield\ (\%) = 100 - \mathrm{CY} \quad (2)$$

2.3 Characterisation of Activated APS Char

Carbon content of the activated APS char was characterised by using a CHNS elemental analyser, model Thermo Finnigan Flash EA 1112. The crystallinity was characterised by using XRD (X-Ray Diffractometer) model Rigaku with voltage of 40 kV, current of 40 mA, with a starting angle at 10° and an end angle at 60° at the speed of 2°/min. Surface area and total pore volume were characterised by using surface area analyser BET (Brunauer-Emmett-Teller), 3 Flex Micromeritic. Nitrogen adsorption was done at −196.2 °C using an automatic volumetric sorption analyser and by immersion calorimetry at 19.9 °C (Iqbaldin et al., 2013). The BET surface area measured

Table 2: Samples notation according to process parameters

Sample	Power (W)	Exposure time (min)
S0	-	-
S1	600	10
S2	600	15
S3	600	30
S4	700	10
S5	700	15
S6	700	30
S7	1000	10
S8	1000	15
S9	1000	30
S10	1000	45

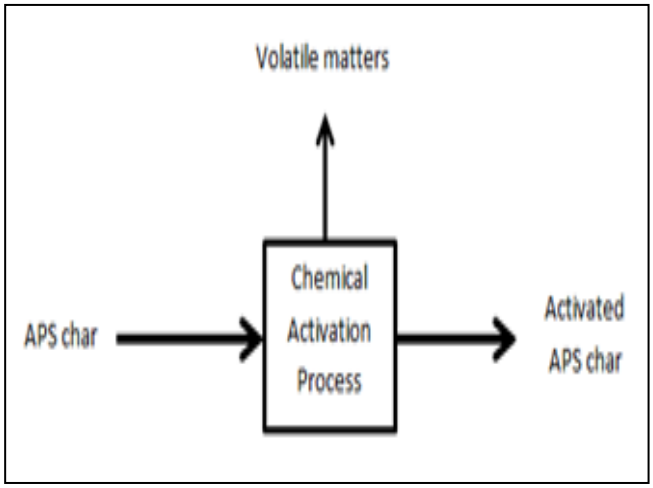

Fig. 2: Mass balance on activation process of APS char via microwave

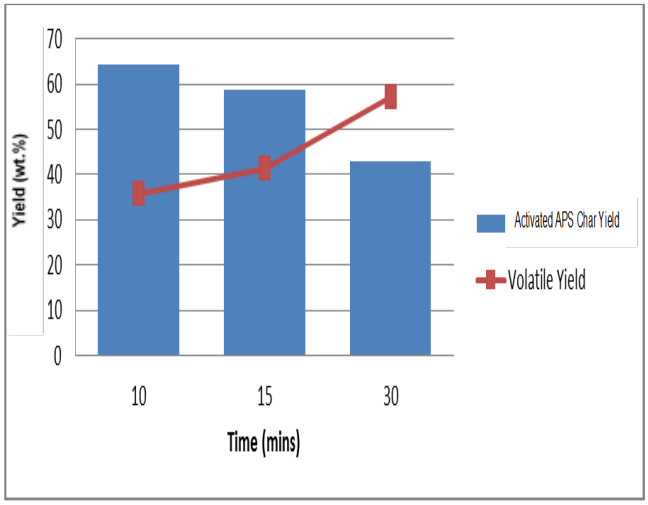

Fig. 3: Yield of activated charcoal against radiation time at 600 W power level

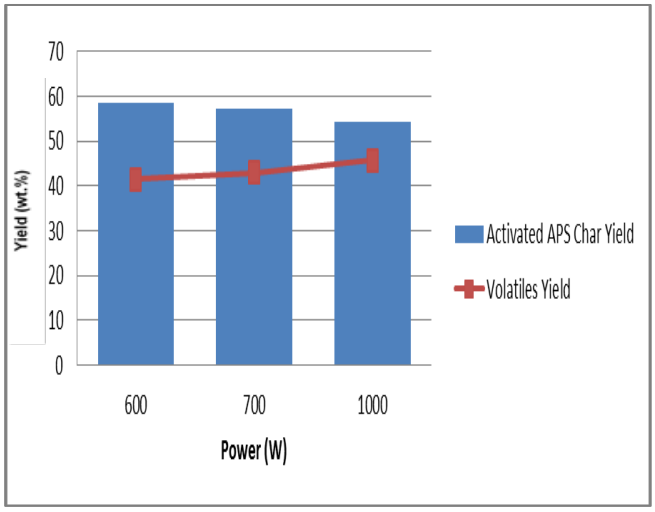

Fig. 4: Yield of activated charcoal against power level

by means of standard BET equation applied with relative pressure (P/P_o) ranging from 0.1 to 0.3. Total pore volume was determined at relative pressure P/P_o=0.99.

3.0 Result and Discussion

3.1 Yield of Activated APS Char

The activation process of APS char has affected the mass of the materials with the changes in irradiation time as well as irradiation power supply for the process. The illustration on mass balance is shown in Fig. 2.

Fig. 3 shows the effect of exposure time on the product yield with a constant power supply of 600 W. The yield was decreasing with the increasing time of microwave pyrolysis. This means that the increasing time allows more volatile components to be removed from the material and resulting in the decreasing of mass of the APS char. Besides that, the longer time also allows for the process to achieve the temperature in which desirable for the complete microwave pyrolysis process. According to research by Huang et al. (2015), the maximum temperature in microwave pyrolysis of the sewage sludge was reached within approximately 10 minutes and thus, longer time of 20 minutes should be sufficient for sewage sludge torrefaction to produce biochar. From observation, the samples which were treated for 30 and 45 minutes irradiation time have shown better results in the carbonisation process and thus might as well resulted in achieving the activation process for the sample to generate the high porosity of the carbon.

Fig. 4 shows the effect of power supplied during the microwave pyrolysis to the product yield with a constant radiation time of 15 minutes. The increase of power supply reduces the mass of the product. This is because the larger power supply helped in providing more energy in the form of heat for the process to take place more effectively in removing the available volatiles from the APS char during the activation process. The higher the microwave power, the more energy can be absorbed by the samples, and thus the pyrolysis reaction is more drastic at higher temperatures which results in more complete pyrolysis process (Song et al., 2017). Study on microwave pyrolysis of tire powder by Song et al. (2017) has shown similar effect of microwave power supply where the higher irradiation power has resulted in the decreasing of solid yield due to loss of volatiles from the sample.

In this study, the APS char has a high amount of carbon content as reported in previous study of APS and a little amount of volatiles. Thus, the significant loss in mass was due to the moisture contents from the impregnation process as well as the presence of

reactions. The carbon left reacted with the potassium hydroxide in order to form porous structures in the activation process (Iqbaldin et al., 2013). From this analysis and observation, 6 best samples (S3, S6, S7, S8, S9, and S) from the experiment were selected to be further analysed.

3.2 Carbon Content

CHNS elemental analyser was used to determine the amount of carbon inside the activated APS char. CHNS elemental analysis is a form of analysis for determination of only carbon, hydrogen, nitrogen and sulphur presence in a sample. From previous data, the amount of carbon was expected to increase for the activated carbon which has lower product yield due to the loss of volatile matter. The results of CHNS elemental analysis of the activated APS char is shown in Table 3.

The amount of carbon is increasing with increasing power level and radiation time of the microwave pyrolysis. The sample which undergoes higher microwave power irradiation had higher energy for the release of volatiles in the sample. Meanwhile, the sample with longer time had longer carbonisation process which subsequently removed more volatiles from the materials to obtain high carbon content product. As the chemical activation process involves simultaneous carbonisation and activation process, the carbonisation process conducted in this research has shown a good result for the development of the activated carbon.

From the result, it shows that highly carbonaceous materials property is produced from the chemical activation with KOH via microwave irradiation in which is similar with the activated carbon produced by Iqbaldin et al. (2013), where the amount of carbon content significantly increases to 80.13% after the activation process of the coconut shell, in which the initial carbon content was only 49.62%. Wang et al. (2015) reported similar finding which the higher amount of temperature has provided higher energy and subsequently increased the amount of carbon content of the activated carbon. In the study, the activated carbons produced from willow catkins had the carbon content increased to 74.63%, 80.82% and 87.04% after the activation process at 600, 700, and 800 °C, respectively. The same trend was also observed in the activated carbon from distiller dried grain with soluble biochar which showed increasing carbon content with increasing temperature from 89.190% at 950 °C to 90.803% at 1050 °C (Jin et al., 2014a).

3.3 Surface Area and Pore Volume

The results of the BET analysis are shown in the Table 4 for the surface area and total pore volume of the activated APS char samples. The surface area and total pore volume of the activated carbon produced are higher than the original APS char (S0). This is because the presence of activating agent has helped in the development of pores in the APS char. Hollow pits will form in the presence of KOH activating agent on the carbon material. KOH impregnates the material, subsequently producing pores and with the increasing temperature, the formation and deformation of pores will occur inside the material due to the activation reactions if there is still presence of the activating agent KOH.

With the pyrolysis process, carbonisation of the sample will produce high purity carbon as more volatiles are being removed from the material. This produces pure activated carbon with high porosity with respect to its surface area and total pore volume. Chemical activation by using potassium hydroxide increased the porosity up to 434.3369 m^2/g compared

Table 3: CHNS elemental analysis on activated APS char

Sample	Power (W)	Time (min)	C	H	N	S
			(wt. %)			
S0	-	-	50.20	4.29	10.5	0
S3	600	30	85.41	5.26	9.33	0
S6	700	30	86.31	4.61	9.08	0
S7	1000	10	82.13	11.07	6.80	0
S8	1000	15	84.73	6.56	8.71	0
S9	1000	30	86.97	4.86	8.17	0
S10	1000	45	89.00	3.50	7.50	0

Table 4: Results of BET analysis.

Sample	Power (W)	Time (min)	Surface area (m^2/g)	Total pore volume (cm^3/g)
S0	-	-	1.082	0.008
S3	600	30	114.842	0.155
S6	700	30	126.151	0.152
S7	1000	10	9.426	0.037
S8	1000	15	10.575	0.039
S9	1000	30	152.306	0.157
S10	1000	45	434.337	0.290

to the raw material of APS char at only 1.0815 m^2/g. The total pore volume of the activated APS char also increased to 0.2901 cm^3/g compared to the initial 0.0077 cm^3/g. This indicates that the activation process has successfully increased the surface area and porosity of char derivative from carbonised APS char. This was similar to findings by Iqbaldin et al. (2013) where the surface area of the coconut shell activated carbon was higher up to 1768.8 m^2/g compared to only 36.5 m^2/g before it was chemically activated.

From Table 4, the effect of power level on the surface area and pore volume of the activated carbon of APS at 30 minutes radiation time (S3, S6, and S9) shows good development to produce the activated carbon with increasing power order. The highest power 1000 W (S9) was successful in producing a better activated APS char with higher porosity compared to lower power supply. The surface area was increases from 114.8415 to 152.3058 m^2/g with increasing power supply from 600, 700, and 1000 W, respectively. This is because the increase in power will increase the activation temperature. and resulted in increase of surface area due to the development of pores in samples during activation process (Rajagopal et al., 2016).

The activated carbon produced from non-metallic circuit board has shown an increasing surface area with the increase of temperature for the activation process from 316 m^2/g surface area for 650 °C temperature to 700 m^2/g surface area for 850 °C of the activation temperature. As for the effect of radiation time at a fixed power, in this case (1000W), the production of the activated carbon (represented by sample S7, S8, S9, and S10), the results show that the radiation time significantly affect the development of pores of the materials. The longer the time taken for irradiation, the higher the porosity of the activated APS char produced. The longer irradiation time has allowed the material to complete the carbonisation process and subsequently remove the volatile matters of the material and provided enough time for the activation process to take place for the development of pores.

Besides that, the irradiation time will increase the activation temperature in the reaction for the simultaneous formation and deformation of pore to occur. This trend is similar to that obtained by Iqbaldin et al. (2013), where the surface area of coconut shell activated carbon in the study increased with the radiation time.

It can be seen, with the highest power (1000 W) and irradiation time (45 minutes), sample S10 shows the surface area of 434.337 m^2/g and total pore volume of 0.2901 cm^3/g. Biagini et al. (2006) suggested that operating microwave process beyond 45 minutes is not efficient, hence this study considers operation of up to 45 minutes only. Even so, the produced activated carbon of APS had the lowest surface area and total pore volume as compared to the other activated carbon materials shown in Table 5. This is because current technique (i.e. activation using KOH at fixed molarity and ratio) and the combined process condition were not able to obtain the higher formation of pores as shown in Table 5.

The produced activated carbon was obtained from chemical activation method via microwave assisted pyrolysis with 5 M of potassium hydroxide solution with the ratio APS char: KOH was equal to 1:3. Good porosity of the activated carbon was successfully developed via chemical activation route as reported by Tamilselvi & Asaithambi (2015) whereby the activated

Table 5: Porosity characteristics of activated carbon from various materials

Sample Materials	Reference	Surface Area (m^2/g)	Total Pore Volume (cm^3/g)	Pore Size (nm)
APS char	This study	434.3	0.290	2.67
Coconut shell	Iqbaldin et al. (2013)	1768.8	-	2.7
Distiller dried grains	Jin et al. (2014a)	2959.0	1.520	-
Corn stover	Jin et al. (2014b)	1432.9	0.760	2.13
Non-metallic printed circuit board waste	Rajagopal et al. (2016)	700.0	0.0022	2.64
Waste tea	Inal et al. (2015)	1125.0	0.592	-
Willow catkins	Wang et al. (2015)	1586.0	0.780	1.06
Coffee ground waste	Wang et al. (2016)	1622.7	0.830	2.04

* (-) sign indicates data which is not presented in the literature

Table 6: Pore size of the activated APS char sample

Sample	Pore size (nm)
S0	28.64
S3	5.41
S6	4.82
S7	15.6
S8	14.8
S9	4.12
S10	2.67

carbon was chemically activated via microwave pyrolysis method resulted in higher surface area as compared to the conventional one. Thus, the chemical activation method might not be the reason for the poor formation of pores in this study. Besides that, according to Hsu & Teng (2000), the chemical activation process would also produce a higher yield of activated carbon as compared to the physical activation due to the burn-off process of char in physical activation. The only drawback regarding chemical activation method was that it produced a lower quality of activated carbon compared to physical activation method to be used in supercapacitor applications due to the porosity of the chemically activated carbon had lower performance in storing electrolyte ions. Since this study did not cover until the capacitance test, thus the performance of the chemically activated APS char was not able to be determined.

The microwave pyrolysis method as the medium for carbonisation and activation process was usually used in order to reduce the time taken for the production of activated carbon (Foo & Hameed, 2011; Hoseinzadeh

Hesas et al., 2013). It is also more efficient with rapid heating technology compare to the conventional pyrolysis process. The results have shown that the increasing irradiation time had increased the resulted surface area and total pore volume of the activated APS char. This suggested that the poor pore formation was because of the irradiation time which was still not

enough for the pore formation of the carbon. The study by Iqbaldin et al. (2013) has shown that the irradiation time will increase the activation temperature for the activation reaction for the simultaneous phenomenon of pore formation and deformation to occur. Thus, the insufficient irradiation time has not able to achieve the activation temperature for the reactions and subsequently resulted in the activating chemical potassium hydroxide to not be fully utilised in the activation reaction (Januri, 2017).

The radiation time however, might not affect the pore structure in the case where the amount of activation chemical provided was not even sufficient in the first place as reported by Iqbaldin et al. (2013), where the activation reaction was over when activating agent presence was used up for the sample that go through longer irradiation time. This shows that the ratio of the material to the activating chemical is also important as it plays an important role in the pore formation. In chemical activation, all the chemical reagents used are dehydrating agents which influence pyrolytic decomposition which increases the carbon yield (Kalyani & Anitha, 2013). This is because the dehydrogenation properties, promote the formation of cross-links, leading to the formation of a rigid matrix, less prone to volatile loss and volume contraction upon carbonisation (Hsu & Teng, 2000). The activation chemical needs to be sufficient as the solid reaction of the potassium hydroxide and the carbon forms the porous structure to the carbon material. Presently, the produced activated APS char may not be suitable to be used in the supercapacitor applications because the electrode in supercapacitor has to be made of material which has high capacitance such as activated carbon in which has high surface area for charge storing mechanisms. An amount of 1 gram of activated carbon has a surface area of about one tenth the size of an American football field due to its high micro porosity (Pradhan, 2011). A standard activated carbon for supercapacitor application usually has the surface area of >1000 m^2/g in which the produced APS activated carbon in this study has failed to achieve (Sevilla & Mokaya, 2014). The produced activated carbon has the surface area of 434 m^2/g only. The low surface area might limit the ability of the supercapacitor to store charges and thus will have a low capacitance value. Besides, the resulted pore size was only 2.67 nm which may not be suitable for the storing of ions in the supercapacitor applications. Recently, few studies have found that micropore carbons with pore sizes in the range between 0.7 to 2 nm show high capacitance value (Inal et al., 2015). The activation process in this work has failed to form micropore structure of the activated carbon of the APS char. This was mainly due to the insufficient irradiation time because the deformation of the pores and its influences towards the increase and decrease of the average pore size depends totally on the activation time (Rajagopal et al., 2016).

The resulted pore size of all the activated APS char in this study as shown in Table 6 was in the mesopore size range. Micropores, mesopores and macropores have the porosity of <2.0 nm, 2.0–50.0 nm, and >50.0 nm, respectively (Halper and Ellenbogen, 2006). The smallest pore size diameter achieved in this work was only 2.67 nm. Thus, from this work, the activated carbon of APS has yet to achieve the desired characteristics of a material in constructing carbon electrodes in the supercapacitor applications although from the pore size it was decreasing compared to the original sample, S0.

4.0 Conclusion

Potassium hydroxide activation via microwave pyrolysis of the APS char at power supply 1000 W and 45 minutes radiation time had the highest surface area and a total pore volume of 434.34 m^2/g and 0.2901 cm^3/g, respectively. Unfortunately, the surface area and total pore volume of the activated carbon is not suitable for the supercapacitor application. Nevertheless, the outcome of this research has shown that the produced activated carbon could otherwise be used for other application than a supercapacitor, such as for wastewater treatment application. Further work will be carried out using KOH or other chemical combinations at varying concentration ratio in order to improve the porosity requirement.

Acknowledgment

The authors would like to thank Universiti Teknologi MARA (UiTM) for the facilities provided to conduct this study.

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