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Activated Carbon from the Renewable Agricultural Residues Using Single Step Physical Activation: A Preliminary Analysis

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Abstract

The feasibility of preparing activated carbon from single step carbon dioxide activation was studied. The optimization of the activated carbons were carried out to study the effects of precursors (coconut fiber, rice husk, coconut shell, palm kernel shell and palm mesocarp fiber), particle size (250-1000 μm), CO_2 flow rate (100-300 cm^3/min), heating rate (5-25 $^\circ\text{C}/\text{min}$), reaction temperature (500-900 $^\circ\text{C}$) and residence time (15-90 mins) on the physical properties of the activated carbon. For the process parameter optimization, L_{25} orthogonal array and “the-larger-the-better” response were employed to determine the optimum yield of the produced activated carbon. In addition, the proximate analysis, chemical compositions and the surface morphology of the raw materials and activated carbons were also discussed. The utilization of the agricultural residues as the activated carbon may help abating the environmental problems caused by irresponsible disposal of agro-wastes.

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Keywords: Activated carbon, Direct CO_2 activation, Taguchi orthogonal arrays

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1. Introduction

At present, the abundance of the agro-wastes in Malaysia poses serious threats to both environment and society. Though agro-wastes are not classified as hazardous materials, but still they constitute a huge volume of total waste materials [1]. Besides, it is estimated that five million tons of agro-wastes have been produced annually in Malaysia [2]. Common practice of biomass disposal is often achieved by using two main methods – dumping in the landfill and open burning which will worsen the existing air pollution and results in a non-aesthetic view. Therefore, biomass upgrading to the value-added product such as the activated carbon is indeed significant to minimize these disposal problems. The urge of using the agro-waste as the raw materials for the activation carbon production is due its sustainability, carbon neutral and potential to decrease the production cost.

The activation process of these precursors can be divided into two main groups; physical and chemical activation. Despite of the benefits provided by the chemical activation process, the chemical consumption led to the secondary pollution problem during the disposal stage. For that reason, the physical activation consists of two-stage carbonization and activation using steam or carbon dioxide (CO₂) as the activation agent is the most commonly used. Yet, since single stage CO₂ activation has received lack of scientific research and there is still limited publication data in the literature. In view of that, further research work on this area is deemed very significant. Through recent research work done by Yang *et al.* [3], one stage activation process on the coconut shell produce comparable results as the two step approaches. According to the findings, the optimal condition of the process was at temperature of 900°C, heating rate of 10°C/min, CO₂ flow rate of 200 cm³/min and 140 mins holding time produced the porous carbon with BET surface area of 1667 m²/g and micropore volume of 0.8949 cm³/g. Meanwhile, Lua and Guo in their research work proved that the single step activation is able to produce carbon adsorbent with excellence properties – BET and micropore surface area of 1410 and 942 m²/g, respectively [4]. Thus, due to the high micropore surface area, the carbon sorbent was further utilized to capture nitrogen dioxide, NO₂ and it is found that the adsorption capacity can reach up to 250 mg NO₂/mg carbon. Besides, Rodriguez-Reinoso *et al.* [5] also conducting the similar research work on plum and peach stone and proves that the process yield significant results on the microporosity development of both materials at process condition of 800-850°C with holding time of 8 or 16 hrs. The benefits of utilizing the single stage activation are due to lower operating time and the energy consumption.

In this research work, Taguchi orthogonal array is used to optimize the production of activated carbon. Unlike the complex conventional approach which normally involves large number of experiments and long duration time, the Taguchi methods apply systematic design of orthogonal arrays to study the entire parameter within smaller number of experiments. Experimental results are then converted into signal to noise (S/N) ratio which can be divided into three classes; the-lower-the-better, the-higher-the-better and nominal-the-better. The main objective of this present research study is to evaluate the physical properties of the raw materials and the activated carbon prepared from different types of agro-wastes with respect to particle sizes, heating rate, CO₂ flow rate, activation temperature and residence times. Influence of these key variables on the yield of the activated carbon will be thoroughly studied. In addition, maximizing yield is crucial in the activated carbon production and thus, S/N ratio of the-higher-the-better response is utilized.

2. Materials and methods

2.1. Materials

Five carbonaceous materials used in this study i.e. palm kernel shell, palm mesocarp fiber, rice husk, coconut shell and coconut fiber were collected from the local market. As-received materials were oven-dried

for a day at 110°C to eliminate the moisture content. Subsequently, these starting materials were ground and sieved for 15 mins to obtain different particle size fraction using the cutting mill (Pulverisette 25/19; Fritsch GmbH, Germany) and sieve shakers (CISA Cedaceria Industrial, Spain), respectively. These samples were then stored in the dessicator for further use.

2.2. Preparation methods of activated carbon

Physical activation method using CO₂ (99.8% purity) as the activation agent was employed in the research work. The precursors with known amounts were transferred into the sample holder and placed into the vertical pyrolysis reactor. The samples were heated from room temperature ($\approx 28^\circ\text{C}$) to the desired temperature of 500-900°C at various heating rate of 5-25°C/min, under the gas flow of 100-300 cm³/min for various holding time (15-90 mins). The activated carbons then were cooled to room temperature under the constant stream of nitrogen flow (99.99% purity). Taguchi method of L₂₅ orthogonal array was developed and MINITAB 16.1.1 software was used in this study for the process optimization purposes. All of the experiments were performed in duplicates.

Table 1. Response table for average S/N ratio for activated carbon yield

Parameter	Parameter level				
	1	2	3	4	5
Precursors	Palm mesocarp fiber	Palm kernel shell	Rice husk	Coconut shell	Coconut fiber
Particle size (μm)	250	355	500	710	1000
Heating rate ($^\circ\text{C}/\text{min}$)	5	10	15	20	25
CO ₂ flow rate (cm ³ /min)	100	150	200	250	300
Temperature ($^\circ\text{C}$)	500	600	700	800	900
Holding time (min)	15	30	45	60	90

2.3. Analysis

The ultimate analyses of C/H/N/S of the carbonaceous materials were performed using the LECO CHNS-932 elemental analyzer. The oxygen content was calculated by mass difference. Also, the elemental compositions of the samples were evaluated using the Energy Dispersive X-ray (EDX). The proximate analysis of these samples was recorded using the thermal analyzer (EXSTAR TG/DTA 6300), as recommended by Elder [6]. Further, the surface morphology of these samples was studied using the Field Emission Scanning Electron Microscope (FESEM, Zeiss Supra55VP). FESEM analysis of both the raw precursors and activated carbons assisted in determining the changes in the material surface, before and after the activation process. Besides, yield of the activated carbon which was used as the indicator of process efficiency were calculated using Eqn. (1).

$$\text{Yield} = [\text{Mass after activation (g)} / \text{Original mass (g)}] \times 100\% \quad (1)$$

3. Result and discussion

3.1. Elemental analysis of carbonaceous materials and activated carbon

Typical chemical composition of lignocellulosic materials is 48% C, 6% H and 45% O, while the remaining are inorganic matters [7]. Based on Table 2, high carbon contents of the raw precursors i.e >40% make them a promising material to be utilized as activated carbon precursors. Besides, the low sulphur content (<0.10%) of these materials may decrease the possibility of acid rain caused by sulphur dioxide emission. In general, the typical sulphur contents may vary from 0.01% (for woody types) to 0.2% (for herbaceous types) [8].

Table 2. Average elemental composition of the raw carbonaceous materials (in dry basis)

Precursors	CHNS analysis					EDX analysis		
	C	H	N	S	O	C	O	Inorganic content
Palm kernel shell	50.49	5.83	0.77	0.06	42.85	55.27	43.38	1.35
Coconut shell	49.20	5.20	0.46	0.00	45.14	53.72	46.09	0.19
Rice husk	41.11	4.87	0.80	0.04	53.17	28.51	50.91	20.58
Coconut fiber	44.89	5.19	0.86	0.03	48.64	45.13	45.73	9.14
Palm mesocarp fiber	44.91	5.57	1.34	0.08	48.10	52.00	42.79	5.21

Apart from the CHNS analysis, the chemical composition (i.e. carbon, oxygen and inorganic elements) was also supported by the EDX analysis. The EDX studies provided the quantitative analysis on the elemental composition by weight percent and atomic percent. On average, lignocellulosic materials compose of 30-60 wt% carbon, 30-40 wt% oxygen, 5-6 wt% hydrogen and approximately 1 wt% of inorganic contents [9]. The inorganic elements may compose of nitrogen, sulphur and chlorine. In certain biomass, the inorganic content might exceeds 1% dry matters such as grasses and straws that have silica content of 10-15% [9]. Bakker and Elbersan reported that the inorganic constituents of dry biomass matter were as follows: 0.5-15% silica, 1-2% potassium, 0.1-5% calcium, 0.1-0.5% sulfur and 0.2-2% chlorine [10]. Depending on the degree of physical activation, the carbon contents vary and can reach up to 90 wt%.

3.2. Proximate analysis of activated carbon

Proximate analysis classifies carbonaceous materials in terms of moisture content, volatile matter, fixed carbon and ash content. The fixed carbon content was calculated by mass difference. The proximate analysis was recorded by measuring the weight loss as a function of temperature and time. Thermogram of the TGA analysis was as shown in Fig.1. Based on Fig.1, the initial part of the analysis shows the moisture and volatile matter of the samples. The moisture content reflects the physically bound water only. There are two basic types of moisture available – intrinsic and extrinsic moisture. The extrinsic moisture is influenced by the weather condition while intrinsic moisture is the moisture content of the materials itself [11]. The second stage of the process involves the release of the volatile matters and organic matters. The biomass components such as cellulose, lignin and hemicelluloses are degraded during this stage. Based on the literature, the hemicelluloses start to degrade at 220°C, cellulose decompose at temperatures greater than 300°C while lignin degrade slowly in wider temperature range [12]. Moreover, the finding was supported by Mahanim *et al.* [13] whom reported that degradation of hemicellulose started at 250-320°C, cellulose pyrolysis at temperature range of 320-380°C and while lignin slowly decomposed throughout the whole temperature range of room temperature up to 900°C. Besides that, the ash contents were the remaining inorganic materials left after the combustion process and the fixed carbon was calculated by difference.

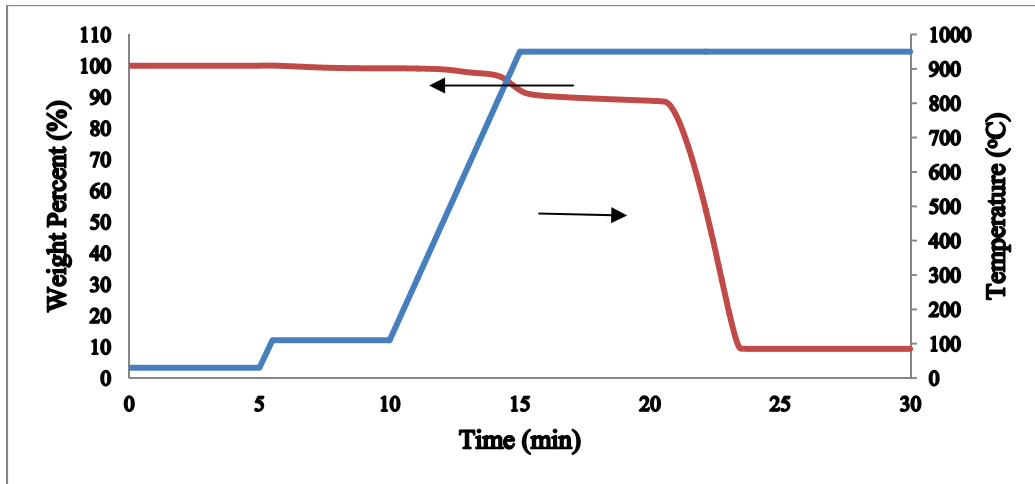


Fig.1. Proximate analysis of samples in TGA (Samples: 250 μm palm kernel shell; Temperature: 800°C; Heating rate: 10°C/min; Flow rate: 200 cm^3/min ; Holding time: 90 min)

Tables 3-5 summarizes the proximate analysis of the activated carbon samples in the study. Based on the findings, both temperature and the nature of precursors have pronounced effects on the development of the volatile matter, ash matters and fixed carbon, as shown in Tables 3-5. As the temperature keeps on increasing, the carbon contents are expected to increase and volatile matters to decrease since the devolatilization process is predominantly occur. As the reaction proceeds at elevated temperature, the volatile materials will slowly release as the gaseous products i.e. carbon monoxide. Also, the findings are similar with those reported by Lua *et al.* whom suggested that nature of starting materials and operating conditions may determine the proximate analysis of the activated carbon [14]. Overall, the results obtained in the present work were comparable with the work reported in literatures.

Table 3. Response table for average mean of volatile matters

Level	Precursors	Particle size (μm)	Heating rate ($^{\circ}\text{C}/\text{min}$)	Gas flow rate (cm^3/min)	Temperature ($^{\circ}\text{C}$)	Holding time (min)
1	18.42	15.22	15.53	15.92	28.53	16.76
2	16.96	11.69	13.76	13.41	19.91	16.28
3	9.95	16.14	12.36	14.19	11.43	13.15
4	11.98	13.97	16.66	12.25	7.17	12.80
5	14.69	14.97	13.68	16.20	4.95	12.98
Delta	8.47	4.45	4.30	3.95	23.59	3.96
Rank	2	3	4	6	1	5

Table 4. Response table for average mean of fixed carbon

Level	Precursors	Particle size (μm)	Heating rate ($^{\circ}\text{C}/\text{min}$)	Gas flow rate	Temperature ($^{\circ}\text{C}$)	Holding time (min)
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	(cm ³ /min)					
1	61.15	61.90	55.74	62.03	50.40	60.61
2	68.72	59.17	64.31	62.59	59.05	57.69
3	49.47	62.19	64.46	63.58	61.49	58.28
4	67.80	61.30	62.27	59.59	63.83	64.57
5	56.52	59.11	56.87	55.87	68.89	62.51
Delta	19.24	3.08	8.72	7.71	18.49	6.88
Rank	1	6	3	4	2	5

Table 5. Response table for average mean of ash content

Level	Precursors	Particle size (µm)	Heating rate (°C/min)	Gas flow rate (cm ³ /min)	Temperature (°C)	Holding time (min)
1	18.44	21.28	27.18	20.25	19.07	20.82
2	13.07	27.72	20.15	22.17	18.95	24.54
3	39.17	19.89	21.30	20.29	25.53	26.85
4	18.84	22.84	19.28	26.67	27.74	20.88
5	26.33	24.13	27.95	26.47	24.55	22.76
Delta	26.10	7.83	8.67	6.42	8.78	6.03
Rank	1	4	3	5	2	6

3.3. Yield analysis of activated carbon

In mass production of activated carbon, relatively high product yield is expected to ensure the economic viability. The S/N of higher-the-better ratio was utilized to determine which parameters have the significant impact on the performance characteristics. The S/N ratio is defined as follows [15]:

$$Y = -10 \log \left[\frac{1}{n} \left(\sum_{i=1}^n \frac{1}{y_i^2} \right) \right] \quad (2)$$

where n is the number of tests and y_i refers to the performance values in the i th experiment. The calculated S/N ratio of yield of activated carbon based on L_{25} matrix design is listed in Table 6. The last two rows in the table provide the delta values and rank of variables in the orthogonal array system. Delta value measures the size of effect by calculating the differences between the maximum and minimum characteristic average for each factor. Thus, the largest value of delta indicates that the factor has the greatest effect on that particular process. Meanwhile, 'rank' suggests the order factors having the greatest effect to the lowest effect (based on delta values) on the response characteristics [16-17]. In addition, analysis of response by calculating the means and S/N ratio was recommended since the analysis was simple [15]. Hence, according to the magnitude of delta as shown in Table 6, the order of the parameter effect was as follows: activation temperature > types of starting materials > particle sizes > heating rate > gas flow rate > holding time. The yield obtained in this study was within the range of 26-41%. The finding by far is comparable to the published results by Yang *et al.* [3] whom also investigated the performance of single step activation on coconut shell. Based on their findings, the yield was in range of 4 to 20% only, irrespective of the temperature, holding time and gas flow rate.

Meanwhile, Lua and Guo managed to obtain the yield of activated carbon in the range of 5-35% with respect to the holding time and temperature [4]. Moreover, the high yield obtained in this study exceeded the recommended minimum yield of activated carbon (20%) [18]. While the temperature and holding time was related to the release of volatile materials as gaseous products, flow rate and heating rate affected the chemical reaction between carbon and carbon dioxide to produce carbon monoxide.

Table 6. Response table for average S/N ratio of activated carbon yield

Level	1	2	3	4	5	Delta	Process influencing rank
Precursors	30.42	30.76	31.45	29.55	30.05	1.91	2
Particle size (μm)	30.08	30.36	30.43	30.19	31.16	1.08	3
Heating rate ($^{\circ}\text{C}/\text{min}$)	30.91	29.97	30.28	30.51	30.55	0.94	4
Gas flow rate (cm^3/min)	30.55	30.60	30.60	29.87	30.61	0.74	5
Temperature ($^{\circ}\text{C}$)	31.78	31.11	29.88	29.58	29.89	2.20	1
Holding time (min)	30.74	30.16	30.39	30.31	30.63	0.58	6

Turmuzi *et al.* [19] observed that the activated yield decreased linearly with respect to increasing reaction time and temperature. This led to the higher oxidation rate at elevated temperature and duration time. Besides, temperature and holding time were responsible in influencing the porosity of the activated carbon. At low values, well-developed porosity might not be created due to insufficient reaction and meanwhile, at elevated values might results in negative effects towards the porosity development due to the sintering effect that cause widening of micropores to the macropores. Also, the greater availability of the CO_2 flow rate is preferred to assist the oxidation process. In the optimization analysis, the heating rate has the least effect towards the yield. In previous work, Tay *et al.* [20] reported that as long as the long residence time and temperature plays the predominant role, the effect of heating rate on production yield is minor. In this experimental study, it is observed that reaction temperature and heating rate have much greater impact on the yield production. As a result, the contribution of holding time is insignificant. Further, the particle size affects the reaction rates in which the smaller particle sizes will burn-out rapidly due to the absence of mass and heat transfer limitations and it is believed that smaller sizes contributes to higher surface area. The precursors also have a significant effect since the quality of activated carbon is depending on both physical and chemical properties of the raw materials. Furthermore, similar findings were reported by Tan *et al.* [21] who confirms that activation temperature has greater impact on the yield production while activation time and impregnation ratio are less significant.

3.4. FESEM characteristics of the activated carbon

FESEM analysis for the raw materials and the activated carbons depicted changes in the surface morphology. According to the FESEM analysis, it can be seen clearly that the structure of the activated carbons were smoothed and porous with many cavities. This was due to the devolatilization of carbonaceous matters that allows the release of reaction gases from the core of particles to outer surroundings [22]. In

contrast, the precursors were rough, dented and less of cracks and voids. This attributed to the agglomeration of inorganic matters which results in pore blockage to some extent. Likewise, Swaidan [23] proposed that the rough and irregular texture of the exterior surface is due to the presence of cellulose, hemicelluloses and lignin which results in absence of cracks. During the activation process, cellulose is converted to porous solid carbon. The conversion of cellulose material to solid led to the opening of pores and the spaces between the crystallites are cleared with the volatile matters and the carbonaceous compounds [24]. The different degrees of the physical activation process assisted in modifying the porosity and texture of the samples. Besides, the enhancement of the pore development in the activated carbon is crucial since these pores act as the active sites during the adsorption process.

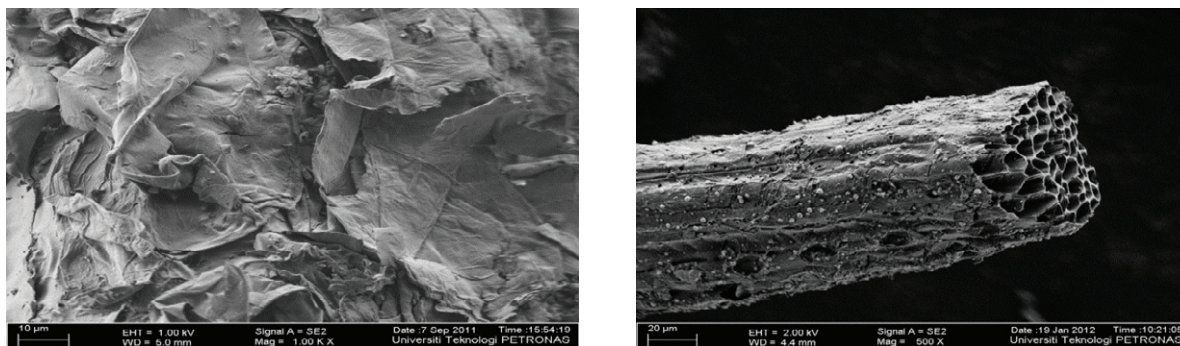


Fig. 2. (a) FESEM images of raw coconut fiber (b) FESEM images of activated coconut fiber at 500x magnification

4. Conclusion

The present work demonstrates the potential of the biomass as the starting materials as activated carbon. Based on findings, the physical characteristics of the prepared activated carbon depend on the activation degree, that is influenced by the key variables involved in the study – nature of the raw precursors, particle size, reaction temperature, heating rate, gas flow rate and residence time. Hence, it is concluded that there are significant changes in the elemental and proximate analysis as well as the surface morphology of both starting materials and resultant activated carbon due to variation in process condition. In conclusion, the activated carbon yield from the agricultural residues was acceptable by using the single step activation.

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