Production of Rubber Seed Pericarp Based Activated Carbon Using Microwave-Induced Different Chemical Activating Agent

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Abstract- Microwave assisted activated carbon from rubber seed pericarp (RSP) was successfully produced by using three different chemical activating agents of $ZnCl_2$, KOH, and H_3PO_4. The process was carried out at 600W of irradiation power for 20 min. The absorption study was applied for these modified activated carbon samples under cationic methylene blue (MB) dye. The Carbonisation of RSP using $ZnCl_2$ (RSP-ZnCl) as activating agent was found the best as compared among the rest which high BET surface area, total pore volume and iodine number were 1689.00 m²/g, 0.9321cc/g and 883.49 mg/g respectively. The adsorption capacity of RSP-ZnCl under MB was *ca.* 297.24 mg g^{-1}. This finding showed the feasibility of preparing activated carbon from RSP using $ZnCl_2$ activating agent via by microwave carbonisation technique.

Index Terms- Rubber seed Pericarp, Activated Carbon, Chemical Activation, Microwave irradiation, Methylene Blue,

I. INTRODUCTION

A ctivated carbon is one of the most important materials that have been widely used in many industrial fields of applications due to its excellent pore structure, high surface area and temperature stability [1, 2]. Yang et al. [3] state that the quality of the resulting activated carbon influenced by the raw material characteristics. Many research and study have been done in the production of activated carbon by using variety of type of starting material. Raw materials for the production of activated carbon include number of carbonaceous material such as wood, peat, brown coal, bituminous coal, lignite, coconut shells, almond shells, pits from peaches and other fruit. Activated carbon from rice husk for example was studied by Kalderis et al. [4], Kumagai et al. [5], Chen et al. [6], and Yan et al. [7]. Various types of nut shell such as pistachio nut shell, almond nut shell, Syzygium jambolanum nut shell and macadamia nut shell were also being used as precursor for activated carbon. The best of our knowledge, no investigations have reported by using Rubber Seed Pericarp (RSP) as precursor to produce activated carbon.

Rubber seed pericarp (RSP) is a biomass waste and currently has no commercial value [8]. This carbonaceous material is highly potential to be converted into useful, high-value adsorbent. According to Eka [9], Rubber tree (Havea brasiliensis) starts to bear fruits at four years of age. Normally, each fruit have about three or four seed, which fall to the ground when the fruit ripens and splits. Each tree yields about 800 seeds (1.3 kg) twice a year. A rubber plantation is estimated to be able produced about 800 - 1200 kg rubber seed per ha per year, and these are normally regarded as waste. Malaysia is known as a one of the main rubber producing country that has 1.7 million hectares of rubber plantation. Therefore, based on estimate average of 1000 kg seed per ha/yr, annually production of rubber seeds in Malaysia would be 1.7 million metric tons. Usually, the pip of rubber seed is sent to oil-mills, but a huge amount of rubber seed pericarp leave as agricultural waste and becomes an environmental problem. Therefore, one of the solutions for this situation is to reuse this waste to produce activated carbon.

Currently, high quality activated carbon from biomass is produced by using the conventional slow heating pyrolysis technique. This technique however, consumed much energy and took more than 6 hours to complete [10]. Another new promising technique that can produce equally high quality activated carbon that utilizes relatively less energy in shorter time is by using microwave irradiation. Microwave irradiation uses electromagnetic waves that are directly absorbed within the molecules of a material causing them to vibrate and agitate rapidly that result to an increase in temperature [11]. This microwave irradiation involves 'volumetric heating' since it is possible for the entire volume of bulk material to be heated rapidly [10-12]. Preparation of activated carbons under microwave radiation from materials such as pitch, tobacco stem, waste tea, wood and cotton stalk have been reported in previous studies [13-17] which has evidenced that the microwave heating has the advantages over conventional heat treatment method such as high heating rate easy control of the heating process, and no direct contact between the heating source and the materials. Microwave radiation can heat or cause arcing in many objects, powdered samples can absorb such radiation and be heated efficiently. The main advantage of using microwave heating is that the treatment time can be considerably reduced, which in many cases represents a reduction in the energy consumption. In addition, the consumption of gases (N_2) used in the treatment can also be reduced toward 40 – 50 % if calculate based on the heating and cooling time during the reaction. In view of the possible advantages associated with the use of microwave heating systems, it was therefore we propose to study the effect of microwave heating combine with three different chemical activating agents i.e. $ZnCl_2$, KOH, and H_3PO_4 treatment in more

detail using a carbon precursor selected (*Rubber Seed Pericarp*) with a better defined microstructure than that used previously.

Matos et al. [18] claimed that the activation process is the most important part in the production of activated carbon. This process creates or increases porosity on the surface carbon materials. Generally, activated carbon can be prepared by one of the following two methods of activation which are by physical or chemical activation. Physical activation is a two-step process and relatively slow. It involves carbonization of a carbonaceous material followed by the activation of the resulting char at elevated temperature in the presence of suitable oxidizing gases such as carbon dioxide, steam, air or their mixtures [19, 20]. In chemical activation carbonization is done after the addition of substances which restrict the formation of tar. This is a single operation of activated carbon preparation. In this method, an activating agent like H_3PO_4, $ZnCl_2$, and KOH are applied in large quantities and has to be removed in order to reveal the porous structure and to make the activated carbon to be accessible [21, 22]. Among the available choices of impregnating activating agents, KOH that has been reported to be more suitable for coal based precursors, while $ZnCl_2$ and H_3PO_4 are widely used for biomass based lignocellulosic precursors [23, 24].

Although large varieties of precursors have been used to produce activated carbon, the work related to the utilization of rubber seed pericarp as precursor has not been reported in literature. Hence, our study focused on the production of RSP activated carbon using different chemical activating agent. The activated carbon produced was evaluated based on product yield, iodine number, methylene blue adsorption capacity, and surface area development. These findings will be of useful in producing activated carbon from RSP for solid filtration or liquid/gas adsorbent material.

II. MATERIALS AND EXPERIMENTAL METHODS

A. Sample preparation

RSP were collected from the local rubber estate located in Jitra, Kedah, Malaysia. RSP were washed with distilled water to remove foreign materials and dried in an oven at 100 °C for 24 hours. The dried RSP were pulverised to a fine powder using grinder and sieved through progressively finer screen to obtain particle size of < 2.0 mm. Sieving were accomplished by shaking the RSP powder in an Endecotts Shaker Model EFL2 for about 30 mins. The fined RSP were stored in an airtight container for further used. The proximate and ultimate analyses of powdered RSP and rubber seed pericarps activated carbon (RSPAC) were measured using thermal gravimetric analyser (TGA) and elemental analyser (EA) respectively [25].

B. Microwave-Carbonization Processes

A weight amount (10.0 ± 0.01g) of RSP sample was mixed with 67 ml of different activating agents (KOH, $ZnCl_2$ and H_3PO_4) solution at various impregnation ratios (0.4 to 2.0 g g^{-1}) for 24 hr at room temperature. The impregnated sample was carbonized using a microwave oven at 600W for 20 min of irradiation time in the presence of nitrogen gas. Fig. 1 shows the schematic diagram for microwave irradiation treatment process. The irradiated samples were cooled at room temperature and washed with 0.1M HCl and left for overnight to isolate the residual activator. The samples were then washed with distilled water to remove residual organic matter, and neutralised with base until the pH of filtrate reached 6.5 - 7, and dried in an oven at 110 °C for 24 hours.
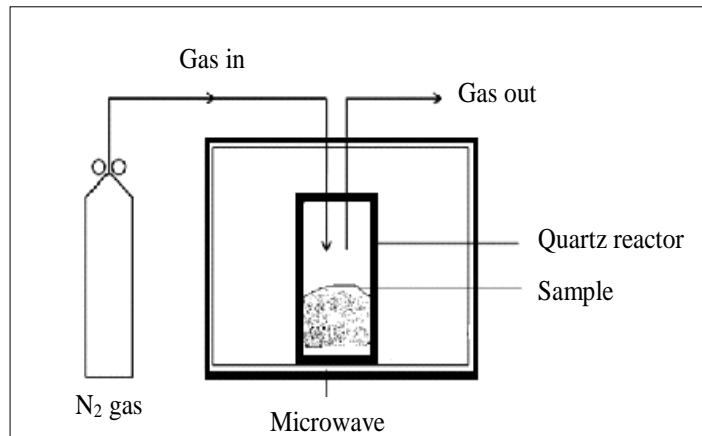

Fig. 1. Schematic diagrams of microwave unit for the preparation of RSPAC.

C. Analysis of Percent Yield and Iodine Number

The efficiency of activated carbon produced from RSP is evaluated based on product yield and iodine number [26]. The percent yield and iodine number of prepared activated carbon were calculated as follows:

$$\text{Yield (\%)} = \frac{Wf}{Wo} \times 100 \tag{1}$$

$$X/M = [A - (DF)(B)(S)] / M \tag{2}$$

Where Wf and Wo are the weight of activated carbon and dried rubber seed pericarp respectively (g). X/M is the iodine absorbed per gram of carbon (mg g^{-1}), A is the normality of standard iodine with 12693.0, B is the normality of sodium thiosulfate with 126.93, DF is the dilution factor, S is volume of sodium thiosulfate (mL) and M are the mass of carbon used (g). Iodine number is use to determine the adsorption capacity of activated carbon and it's indicate the porosity of the activated carbon [27]. Iodine number provides an approximation for the surface area and microporosity of actived carbon with good precision. The analysis of iodine was done based on the standard test method ASTM D4607.

D. Batch Adsorption studies

MB is a strong adsorption onto solid used in a wide variety of application. It was known as a common substances used in dying process due to its potential risk towards the survival aquatic compartment, ecosystem and environmental pollution [28]. Thus, it was chosen in this study. MB is a heterocyclic aromatic chemical component with a chemical formula of $C_{16}H_{18}N_3$ and a molecular weight of 319.85g mol^{-1} [29] Fig. 2 shows the chemical structure for MB. MB adsorption experiments were conducted to determine the optimum quality of the adsorbent for

maximum adsorption [30]. The MB solution for adsorption was prepared by dissolving MB in deionised water without pH adjustment. The adsorption test were carried out in a set of 250 ml conical flask containing 0.2 g of adsorbent and 150 mL dyes solutions with various initial concentrations range of 50-500 mg L^{-1}. The flask was placed on a shaker setting of 120 rpm for 24 hours to reach equilibrium. Then the samples were filtered to remove carbon and the residual concentration of MB in the filtrate was analyzed using Spectrophotometer at 664 nm wavelength. The MB uptake at equilibrium q_e (mg g^{-1}) was computed using equation 3.

$$q_e = \frac{(C_o - C_e)V}{w} \qquad (3)$$

Where C_o and C_e are the initial and equilibrium concentrations of dye (mg L^{-1}) in the solution, V is the volume of the solution (L), and W is mass of the adsorbent used (g). While, equation 4 was used to calculate the percentage of MB adsorption based on the initial and equilibrium MB concentration.

$$Adsorption\ (\%) = \frac{C_o - C_e}{C_o} \times 100 \qquad (4)$$

The pore size and structure of the activated carbon was determined using N_2 adsorption Brunauer-Emmett-Teller (BET) and scanning electron microscope (SEM) respectively.
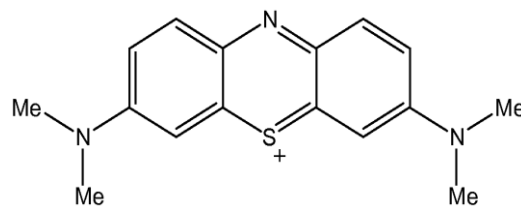

Fig. 2. Chemical Structure for Methylene Blue (MB)

III. RESULT AND DISCUSSION

Table 1 showed that waste RSP material is a carbonaceous material that contained high composition of carbon and high potential to become activated carbon. The percentage of carbon significantly increased after carbonization and activation process due to release of volatile matter during the activation as proven by decreased of volatile content in RSPAC. However, with reference to table 1, the RSPAC treated with $ZnCl_2$ produced the lowest amount of ash at 4.08% with high percent of fixed carbon at 45.06% in comparison to RSPAC treated with KOH and H_3PO_4, respectively.

Table. 1. Proximate and ultimate analyses of raw rubber seed pericarp (RSP) and rubber seed pericarp activated carbon (RSPAC) with different activating agent

Analysis	RSP	RSPAC		
		KOH	H_3PO_4	$ZnCl_2$
Proximate				
Moisture	3.60	8.48	9.59	4.88
Volatile Matter	94.25	70.05	53.89	45.44
Fixed Carbon	0.03	14.19	22.56	45.60
Ash	2.12	7.27	13.96	4.08
Ultimate				
Carbon	52.93	89.2	66.1	93.5
Hydrogen	6.24	2.48	2.27	1.76
Nitrogen	0.00	0.41	0.53	0.55
Sulphur	0.08	0	0	0
Oxygen*	40.75	7.91	31.1	4.19

Notes : * calculated by difference

A. Textural Characteristics of RSPAC

Fig. 3 compares the SEM micrographs of RSP and RSPAC treated with three different activating agents. The SEM micrograph of RSP (Figure 3a) shows smooth surface with no pore development. Comparatively, the SEM micrographs of RSPAC treated with different activating agents show pores development as depicted in figures 3 (b), (c) and (d). This phenomenon is mainly due to the released of volatile matter and reaction between activating agent and the carbon atom in the precursor [26, 32]. The SEM micrograph for $ZnCl_2$ treated RSPAC, however reveals high distribution of pore development. Comparatively, the KOH and H_3PO_4 treated RSPAC exhibit some porosity with the latter showed development of hollow and rugged surfaces, respectively. Apparently, the mean pore diameter of all the treated RSPAC were about the same in the range of 2.4 – 2.9 nm.

In addition, the effect of pore development was also reflected on the surface area development. The $ZnCl_2$ treated RSPAC has the highest surface area of 1689 m^2 g^{-1} in comparison to KOH and H_3PO_4 treated RSPAC at 392 m^2 g^{-1} and 1589 m^2 g^{-1}, respectively. Therefore, the $ZnCl_2$ is found that to be an effective activating agent to produce activated carbon from RSP with high-surface area.
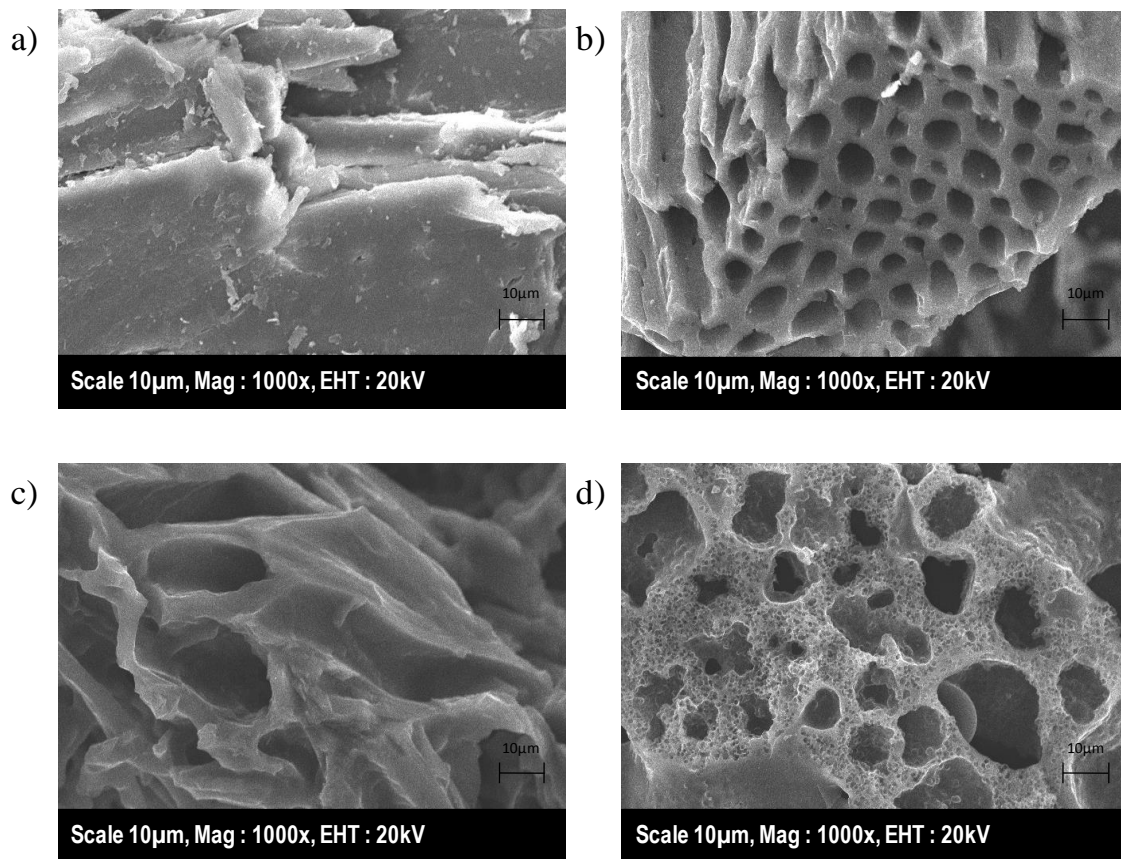

Fig. 3. SEM micrographs (1000x) of a) RSP and RSPAC treated with b) $ZnCl_2$ c) KOH and d) H_3PO_4

Table 2. Optimum surface characteristics of rubber seed pericarp activated carbon (RSPAC) treated with different chemical activating agents

Analyses	RSPAC		
	KOH	H_3PO_4	$ZnCl_2$
Percent Yield (%)	18.56	36.09	30.69
Iodine Number (mg/g)	561.16	700.38	883.49
MB Adsorption (mg/g)	56.89	287.92	297.24
BET surface area (m^2/g)	392.00	1589.00	1689.00

B. Percent Yield and Iodine Number

The percent yield and iodine number of RSPAC prepared by different activating agents is shown in Fig. 4. Expectedly, the results showed that KOH produced the lowest percent yield of activated carbon followed by $ZnCl_2$ and H_3PO_4. RSP is a lignocellulosic material, with hemicelluloses, cellulose and lignin as the main components; and this type of material has been reported suitable to be treated with $ZnCl_2$ and H_3PO_4 activating agents [24]. It can be seen that the yields of activated carbon decrease with increasing impregnation ratios. The decrease in product yield might due to continuous release of volatile matters from the material creating more pores [26]. Further, it can be seen that the decrease in product yield correlates with increasing in iodine number, indicating an increasing in adsorption capacity of the materials.

From fig. 4, it also shows that iodine number increase with increasing the impregnation ratio of activating agent used. This is due to electrolytic action terms as swelling in the molecular structure of cellulose [26]. However, according to Ahmed et al. [26] they claimed that high concentration of activator could also lead to excessive dehydration and destruction of micropores that form larger pores and reduce adsorption efficiency.
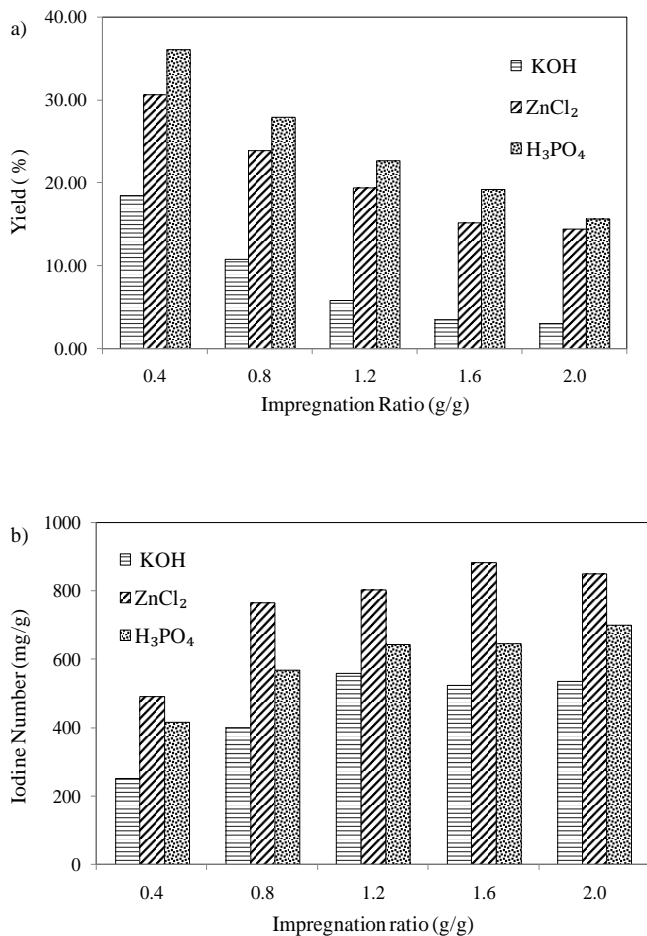
C. Methylene Blue (MB) Adsorption Analysis

MB is a relatively large molecule and often employed as adsorbates to evaluate the efficiency of activated carbon for removal of dyes. Fig. 5 compares the adsorption capacity of treated RSPAC with $ZnCl_2$, KOH and H_3PO_4. The MB adsorption capacity was conducted based on the approximation of the highest iodine number to give the highest amount of adsorption. Apparently, the MB adsorption capacity for RSPAC treated with $ZnCl_2$ produced the highest amount of adsorption capacity with comparison to KOH and H_3PO_4 treated RSPAC. The amount of MB adsorption increase from about 65.54 to 297.24 mg g^{-1} with increasing ratio of $ZnCl_2$ from 0.4 to 1.6, and decrease slightly at ratio of 2.0. Similar trend could be observed with KOH and H_3PO_4. The fact that by increasing the ratio of activating agent, more pores would be created, however as the ratio arrives at the optimum value, the pores would be widened and burnt off [14, 26]. Therefore, the adsorption capacity of activated carbon would increase to a certain value and then decreased with ratio of activating agent.
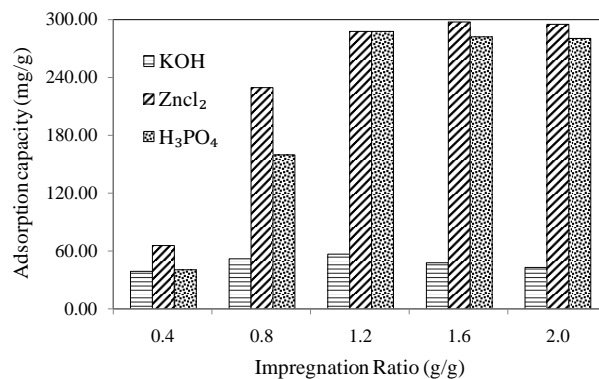

Fig. 5. MB adsorption capacity for RSPAC treated with $ZnCl_2$, KOH and H_3PO_4.

IV. CONCLUSION

A study of the textural and adsorption characteristic of the activated carbon reveals the feasibility to prepare activated carbon from rubber seed pericarp (RSP) via microwave irradiation method using $ZnCl_2$ as activating agent. The activation process was conducted for 20 mins at the operating power of 600W resulted in a yield of 30.69% and iodine number of 883.49 mg/g. While the BET surface area, total pore volume and adsorption capacity of MB were 1689.00 m²/g, 0.9321cc g^{-1} and 297.24mg g^{-1} respectively. This research would significantly contribute to new findings in term of activated carbon production from abandon biomass waste using new heating technique.

Fig. 4. a) Percent yield and b) Iodine number of treated RSPAC with different activating agent.

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