Preparation of Activated Carbon by Reduction of SO_2 Adsorbed on Palm Oil with Microwave Energy

Rungsan Lawanna
Faculty of Science and Technology, Assumption University
Bangkok, Thailand

Abstract

The preparation of activated carbon from palm oil shell by activation with microwave energy was divided into three steps: carbonization, sulfur dioxide adsorption, and microwave activation. The first step - carbonization - was carried out at 400°C for 60 min. The characteristics of char were: yield 31.72%, volatile matter 18.87±0.06%, fixed carbon 69.41± 0.09%, and BET surface area 152±4 $m^2 g^{-1}$.

Sulfur dioxide, 2,000 ppm, was adsorbed for 420 min by the prepared char until reaching the equilibrium at room temperature in the reactor, diameter of 5 cm, and height of 30 cm.

The activation with microwave energy was carried out in Pyrex tube (U-shaped) reactor, diameter of 28 mm, length of 150 mm, and height of 100 mm. The reactor was set up in a microwave oven. The variables studied were: activation time (range of 30-180 min), activation energy (range of 225-500 watt), and number of cycles of adsorption and activation from 1 to 4 times. The optimum conditions from experimental results were: activation time of 90 min, activation energy of 450 watt, and second cycle of adsorption and activation. The characteristics of activated carbon produced at the optimum condition were: iodine adsorption number 1385± mg g^{-1}, methylene blue adsorption number 235±6 mg g^{-1}, bulk density 0.659 g cm^{-3}, pH 8.11, surface area 1172±49 m^2 g^{-1}.

Keywords: *Activated carbon, palm oil shell, microwave energy, carbonization, sulfur dioxide adsorption, microwave activation, reactor, carbonizer.*

Introduction

Physical activation used oxidizing gases such as H_2O, CO_2, NO_2, O_2, and SO_2 gases with applied energy. The effective activation factors were oxidizing concentration, temperature, time, type, and quantity of the raw material (Hassler 1974). The influence of oxidizing gas such as steam by gasification produces low adsorption surface area on charcoal because many macropores were formed (Rodriguez-Reinoso and Lanires-Solano 1965). The reaction is shown in Equation (1):

$$C + H_2O \longrightarrow H_2 + CO \quad \text{…………(1)}$$

The activation of carbon dioxide on charcoal was studied while temperature was set within 800-900°C. The endothermic reaction which illustrates the reaction between C on charcoal and carbon dioxide (Ergun and Mentser 1965) is shown in Equation (2):

$$C + CO_2 \longrightarrow 2CO \quad \text{…………...……..(2)}$$

The exothermic activation of oxygen formed pores easier on the surface area of charcoal rather than using steam and carbon dioxide. This is shown in Equations (3) and (4):

$$C + O_2 \longrightarrow CO_2 \quad \text{...…………...……(3)}$$
$$2C + O_2 \longrightarrow 2CO \quad \text{...……………..……(4)}$$

The activation of nitrogen dioxide, which is an exothermic reaction, reacts faster than any oxidizing gases and many more pores on charcoal were formed (Suzuki 1990), as seen in Equation (5):

$$3NO_2 + 2C \longrightarrow 3NO + CO + CO_2 \quad \ldots\ldots (5)$$

While a high temperature was employed, NO_2 gas was decreased at the surface area of charcoal, as seen in Equation (6):

$$2NO_2 \longrightarrow 2NO + O_2 \quad \ldots\ldots\ldots\ldots\ldots\ldots (6)$$

Microwave energy was applied so that nitrogen monoxide was decreased because the adsorption on surface of char was increased (Loren 1996) (Equations (7) and (8):

$$2NO + 2C \longrightarrow N_2 + 2CO \quad \ldots\ldots\ldots\ldots (7)$$
$$2NO + C \longrightarrow N_2 + CO_2 \quad \ldots\ldots\ldots\ldots (8)$$

The hot spots on the surface were formed when a small amount of nitrogen monoxide reacted at a low temperature (Equation (9):

$$2NO + 2CO \longrightarrow 2CO_2 \quad \ldots\ldots\ldots\ldots\ldots (9)$$

At 316^0C, the reaction is shown in Equation (10):

$$CO_2 + C \longrightarrow 2CO \quad \ldots\ldots\ldots\ldots\ldots\ldots (10)$$

However, the temperature within the range of 350-450^{o}C has effect on the reaction between nitric gas and carbon on charcoal particles that produced a small amount of water vapor. This water vapor produced the hot-spot adsorption area in charcoal (Kong 1996), as shown in Equation (11):

$$4HNO_3 + 3C \longrightarrow 2H_2O + 2NO + 2NO_2 + CO_2 + 2CO \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (11)$$

The Activation of Sulfur Dioxide with Microwave Energy

At highly temperature, micropores on charcoal were caused by the reaction between carbon and sulfur dioxide (Kong and Cha 1996) as shown in Equations (12) and (13):

$$C + SO_2 \longrightarrow CO_2 + S \quad \ldots\ldots\ldots\ldots\ldots (12)$$
$$2C + SO_2 \longrightarrow 2CO + S \quad \ldots\ldots\ldots\ldots\ldots (13)$$

Experimental

The palm oil sample was analyzed by determining the volatile matter (VM), fixed carbon (FC), ash (A), and moisture (M). Then the sample was carbonized by the carbonizer. This was done by placing 500 g of the sample inside the carbonizer, set it at 400^{o}C for 60 min. Then the charcoal was analyzed by preparing 2,000 ppm of SO_2 in 5cm diameter and 30 cm high. In this way, SO_2 was adsorbed on charcoal particles of 100 g of 1.18-2.36 mm size in the reactors for 420 min. Activate the passing charcoal sample by microwave energy. Place 40 g of charcoal sample in a U-shaped tube to the microwave by varying energy and time. Energy levels were varied for 225, 250, 450, 475, and 500 watt; varied time periods were 30, 60 90, 120, and 180 min. Analyze activated carbon by determining moisture (M), volatile matter (VM), ash (A), bulk density, pH, iodine adsorption number, methylene blue adsorption number, and surface area.

Preparation of 2,000 ppm Sulfur Dioxide Process

From the carbonization process at 400^{o}C for one hour, the charcoal sample was seized and was adsorbed by SO_2. The 2,000 ppm SO_2 adsorption on charcoal was prepared by using 2.50 L min^{-1} of dry air for 420 min.

Result

General Characteristics of Palm Oil

The general characteristics of palm oil are strength, stickiness and color appearance. It was found that when it was carbonized the strength was reduced; the stickiness was also reduced to become brittle; and the color turned from brown to black.

The properties of palm oil which were obtained from the experiment were:

Moisture	7.49$\pm$0.005%
Ash	6.65$\pm$0.01%
Volatile matter	79.0.15$\pm$0.15%
Fixed carbon	4.12$\pm$0.21%
pH	4.99

Carbonization Process

Carbonization was the first step of the experiment. It was found that the optimum condition for carbonization process was 400^{o}C for 60 min. The properties of charcoal were:

Yield 31.72%
Moisture 1.83$\pm$0.03%
Ash of 9.89$\pm$0.06%
Volatile matter 18.87$\pm$0.14%, and
Fixed carbon 69.41$\pm$0.09%.

Variation of Activation, Adsorption and Cycles

Charcoal was activated at 225 watt by varying the time ranges from 30-180 min. The result is shown in Table 1.

The result of activation of saturated SO_2 on charcoal within 30 min by varying energy is shown in Table 2.

The result of activation of saturated SO_2 on charcoal at 450 watt within 90 min by varying cycles is shown in Table 3.

Discussion

Activation and Adsorption with Microwave Energy by Varying Energy and Time

From Table 1, it was found that 90 min resulted in the highest surface area (302$\pm$10 m^{2} g^{-1}) and iodine adsorption number (367$\pm$8 g cm^{-3}). From Table 2, when charcoal was activated for 30 min by varying energy, ranging from 225-500 watt to reach the appropriate energy. The results showed that 450 watt were effective energy in the process. Thence 450 watt for 90 min of activation was an effective condition.

Finally the experiment was fixed at 450 watt for 90 min, varying the cycles of activation and adsorption from 1-4 times. It was found that the second cycle was the best one, with the highest surface area of 1172$\pm$49 m^{2} g^{-1}, and the highest iodine adsorption number of 1385$\pm$11 g cm^{-2}. Table 3 reveals that mostly high and effective surface area of activated carbon used to remove SO_2 toxic gas in the atmosphere from industries, vehicles, and any oxidization.

Either microwave energy activation or the adsorption of SO_2 on charcoal affected the formation of large amount of micropores. This results in the removal of a lot of toxic gases.

Conclusion

The advantages of this method for the preparation of activated carbon were that sulfur dioxide adsorption on charcoal using in the process was supplied from the flue gas in electrical power plant which used coal as raw material. By using microwave energy activation, the SO_2 adsorbed charcoal or carbon turns out to have a high surface area (1172$\pm$49 m^{2}g^{-1}) which was obtained from experimental results which were: activation time of 90 min, activation energy of 450 watt, and second cycle of adsorption and activation.

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Table 1. Activation of saturated SO_2 on charcoal at 225 watt by varying time.

Condition of activation: Energy level (watt)	Time (min)	Iodine number (g/cm^3)	Methylene blue number (g/cm^3)	Surface area (m^2/g)
0	0	212±4	291±6	152±4
225	30	274±4	283±7	211±8
225	60	326±6	240±6	272±14
225	**90**	**367±8**	**234±3**	**302±10**
225	120	360±8	237±5	301±8
225	180	325±6	240±6	280±9

Table 2. Activation of saturated SO_2 on charcoal within 30 min by varying energy.

Condition of activation: Energy level (watt)	Time (min)	Iodine number (g/cm^3)	Methylele blue number (g/cm^3)	Surface area (m^2/g)
0	0	212±4	291±6	152±4
225	30	274±4	283±7	211±8
250	30	303±6	239±5	242±8
450	**30**	**340±7**	**234±5**	**279±11**
475	30	308±6	243±6	264±8
500	30	283±5	236±6	185±6

Table 3. Activation of saturated SO_2 on charcoal at 450 watt within 90 min by varying cycles.

Cycle	Iodine number (g/cm^3)	Methylene blue number (g/cm^3)	Surface area (m^2/g)
0	212±4	291±6	152±4
1	418±10	235±5	351±17
2	**1385±11**	**235±6**	**1172±49**
3	1184±6	239±6	984±38
4	390±9	249±5	489±19