CHAPTER III

EXPERIMENT

3.1 Raw material

The raw material was prepared from used tires by cutting to the size of 5 x 5 x 5 mm. The pictures of used tires and used tires size of 5 x 5 x 5 mm are shown in **Figure 3.1 (a,b).**

Figure 3.1 (a) used tires and **(b)** used tires size of 5 x 5 x 5 mm.

3.2 Chemicals

1. Hydrochloric acid, concentrated (HCl) : AJAX
2. Sodium thiosulfate, ($Na_2S_2O_3 \cdot 5H_2O$) : Fluka
3. Iodine, (I_2) : AJAX
4. Potassium iodide, (KI) : BDH
5. Potassium iodate, (KIO_3) : May & Baker
6. Sodium carbonate, (Na_2CO_3) : Fluka
7. Starch, soluble potato
8. Methylene blue ($C_{16}H_{18}N_3SCl \cdot 3H_2O$) : Carlo Erba
9. Sodium phosphate, (Na_2HPO_3) : Carlo Erba
10. Potassium phosphate, (KH_2PO_3) : Merck

3.3 Apparatus

1. Muffle furnace: type ESF 12/23 (0-1,200°C), Carbolite, England.
2. Oven: 0-250°C, WT binder, Germany.
3. Tube furnace: type 21100 (0-1,200°C) Thermolyne Corporation, USA.
4. Ball mill.
5. Hammer mill.
6. Laboratory test sieve: s/steel, sizes 0.25, 0.60, 1.18, 2.36 and 4.75 mm, Endecotts, England.
7. Sieve shaker: EFL1 mk3, Endecotts, England.
8. Spectrophotometer: Spectronic21(320-1,000 nm), Miltonroy company, USA.
9. Ultra-high centrifugal: Model KC-25, Kubota, Japan.
10. Shaker.
11. Boiler: Model M1 00X-30 serial. No. L 75240, Cleaver brook.
12. Air pump.

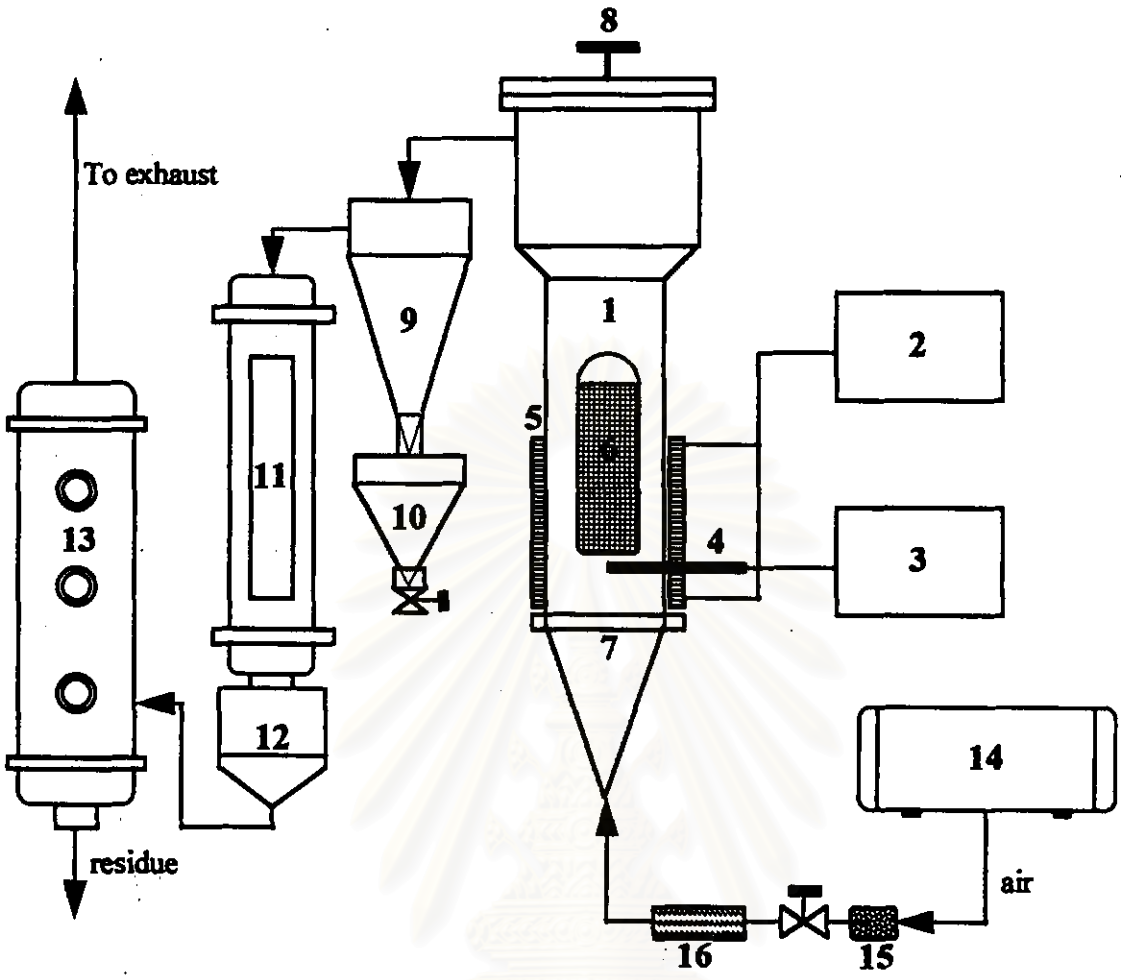
13. AC.ARC welder: Model LN300, Makito, Japan.
14. Surface area analyzer: ASAP 2000, Micromeritics Instrument Corporation, analysis program: run20.com.
15. Canbonizer:

The fixed bed reactor was a stainless steel tube of 158 mm inside diameter and 1150 mm in length. The tube was fitted with a perforated stainless steel gas distributor. The distributor had 0.5 mm diameter holes on a triangular pitch. The fixed bed was heated by an electricity (2000 watt), and the bed temperature was measured by type K thermocouple. Air flew into the bed were controlled using a rotameters. The fixed bed carbonizer was used in this work and a schematic of the experimental setup are shown in **Figures 3.2-3.3.**

16. Activator:

The fixed bed reactor was a stainless steel tube of 100 mm inside diameter, 45 mm wall thickness and 300 mm in length. The tube was fitted with a perforated stainless steel gas distributor. The distributor had 0.5 mm diameter holes on a triangular pitch. The fixed bed was heated by an electricity (1000 watt), and the bed temperature was measured by type K thermocouple. The bed could be operated between ambient temperature and 900°C, and the temperature was controlled to an accuracy of ± 5°C by means of a temperature controller. Air and CO_2 flew into the bed which were controlled using a pair of rotameters. Superheated steam was preheated by burner before flowing into the bed. The fixed bed activator was used in this experiment and schematic of the experimental setup are shown in **Figures 3.4-3.5.**

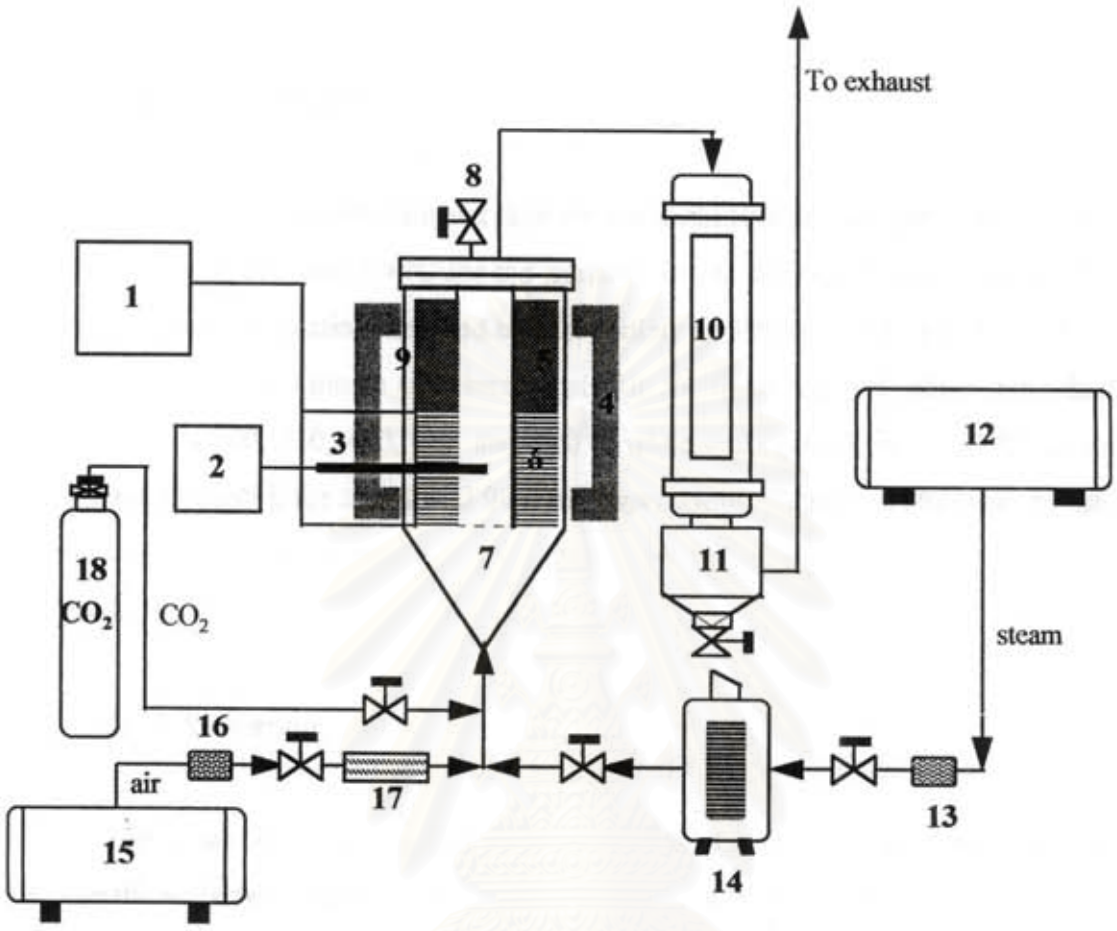
Figure 3.2 The fixed bed carbonizer.



1. Reactor
2. AC. Arc welder
3. Temperature controller
4. Thermocouple
5. Heating coil
6. Sample support
7. Distributor plate
8. Sample feed and removal port
9. Cyclone
10. Separator
11. Condenser
12. Separator
13. Scrubble
14. Air pump
15. Moisture separation
16. Rotameter

Figure 3.3 A schematic of the fixed bed carbonizer experimental setup.

Figure 3.4 The fixed bed activator.



1. AC. Arc welder
2. Temperature controller
3. Thermocouple
4. Insulator
5. Castable
6. Heating coil
7. Distributor plate
8. Sample feed and removal port
9. Stainless steel tube
10. Condenser
11. Separator
12. Boiler
13. Steam trap
14. Preheater
15. Air pump
16. Moisture separation
17. Rotameter
18. CO_2 cylinder

Figure 3.5 A schematic of the fixed bed activator experimental setup.

3.4 Procedures

3.4.1 Carbonization

The optimum temperature and time for carbonization of used tires were studied at 300, 350, 400, 450, and 500°C for the duration of 30, 60 and 90 min of time. The procedure started by heating the fixed bed carbonizer to 300°C. Next, the 500 g of cut pieces (about 5 x 5 x 5 mm in size) were heated in the fixed bed carbonizer at the final temperature as 300, 350, 400, 450 and 500°C while the air flow rate of 0.52 nl/min was passed through it for 30, 60 and 90 min, respectively. Finally, the % yield, % ash, bulk density, % volatile matter and % fixed carbon of the products would have been analyzed.

3.4.2 Activation

The chars from the carbonization at 350°C for 60 min were produced to become the activated carbon. They were crushed and sieved to particle sizes of 0.25-0.60, 0.60-1.18, 1.18-2.36 and 2.36-4.75 mm.

3.4.2.1 The optimum temperature and time for activation

At 750, 800, 850 and 900°C for time 30, 45 and 60 min were studied the optimum temperature and time for activation. First, the fixed bed activator was heated until the temperature in the bed reached 750, 800, 850 and 900°C, respectively. Second, the 200 g of 1.18-2.36 mm of the chars were charged into it with the air at flow rate of 0.27 nl/min. Then, the superheated steam and CO_2 at a flow rate of 5.0 nl/min were passed through the bed for 30, 45 and 60 min. The products would be characterized as % yield, % moisture, % ash, bulk density, iodine number, methylene blue number and B.E.T. surface area.

3.4.2.2 The optimum size for activation

The chars with the sizes of 0.25-0.60, 0.60-1.18, 1.18-2.36 and 2.36-4.75 mm were studied in this work. The fixed bed activator was heated until the temperature in the bed reached 900°C. The 200 g of each size of chars were charged into it with the air at a flow rate of 0.27 nl/min. The superheated steam and CO_2 at a flow rate of 5.0 nl/min were passed through the bed for 45 min. The products would be characterized as % yield, % moisture, % ash, bulk density, iodine number, methylene blue number and B.E.T. surface area.

3.4.2.3 The optimum flow rate of CO_2 for activation

The flow rates of CO_2 at 0.0, 2.0, 5.0, 10.0 and 15.0 nl/min were studied for activation. In the beginning, the fixed bed activator was heated until the temperature in the bed reached 900°C. The 200 g of 0.60-1.18 mm of the chars were charged into it with air at a rate of 0.27 nl/min. The superheated steam and each flow rate of CO_2 were passed through the bed for 45 min. Finally, the products were characterized as % yield, % moisture, bulk density, iodine number, methylene blue number and B.E.T. surface area.

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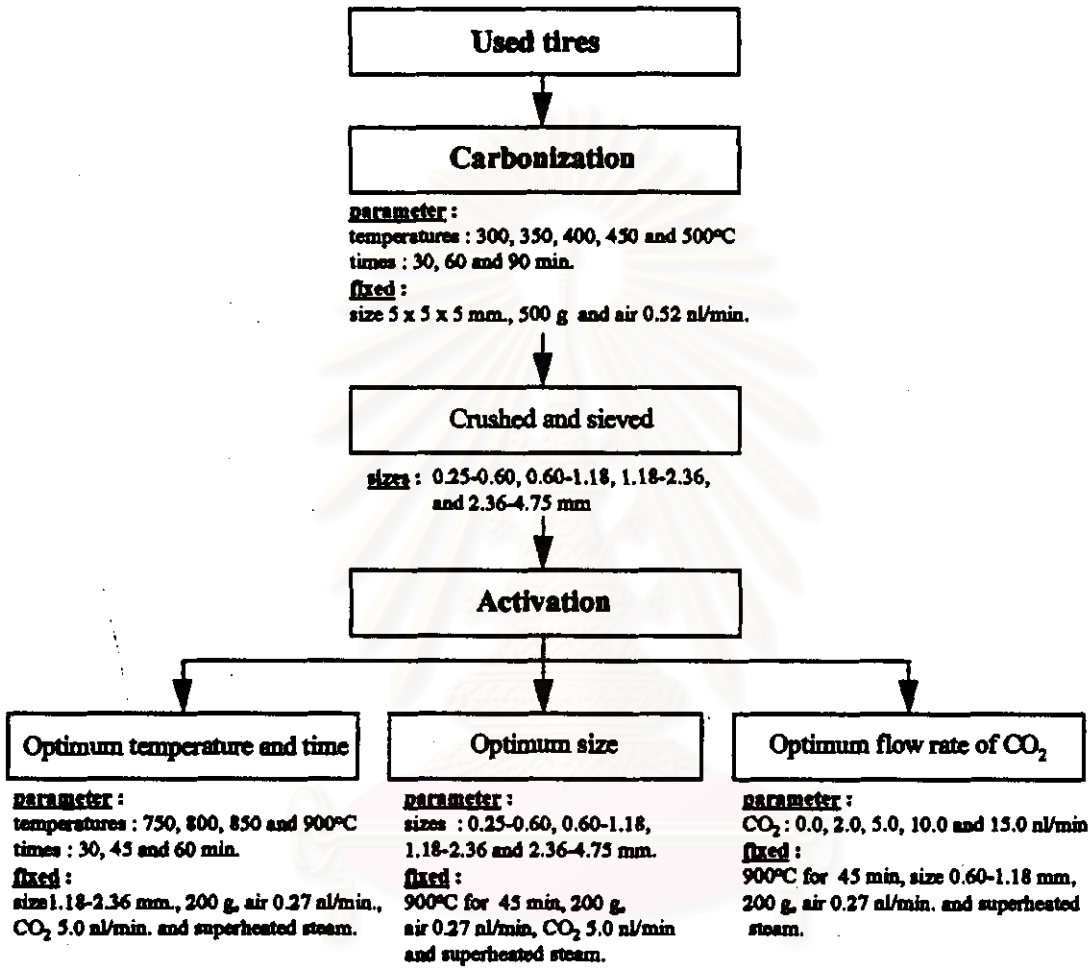

Figure 3.6 Experiment scheme of the production of activated carbon from used tires by superheated steam and carbon dioxide activation in a fixed bed reactor.