CHAPTER III

EXPERIMENT

3.1 Raw material

This experiment used palm-oil shells for the production of activated carbon by pyrolysis and steam activation in one stage. The palm-oil shells were crushed and sieved to six particle sizes of < 0.60, 0.60-1.18, 1.18-2.36, 2.36-4.75, 4.75-6.00 and 6.00-8.00 mm before being treated. The proximate analysis of the raw palm-oil shells is shown in **Table 4.1**. The pictures of palm-oil shells and activated carbon from palm-oil shells are shown in **Figure 3.1**.

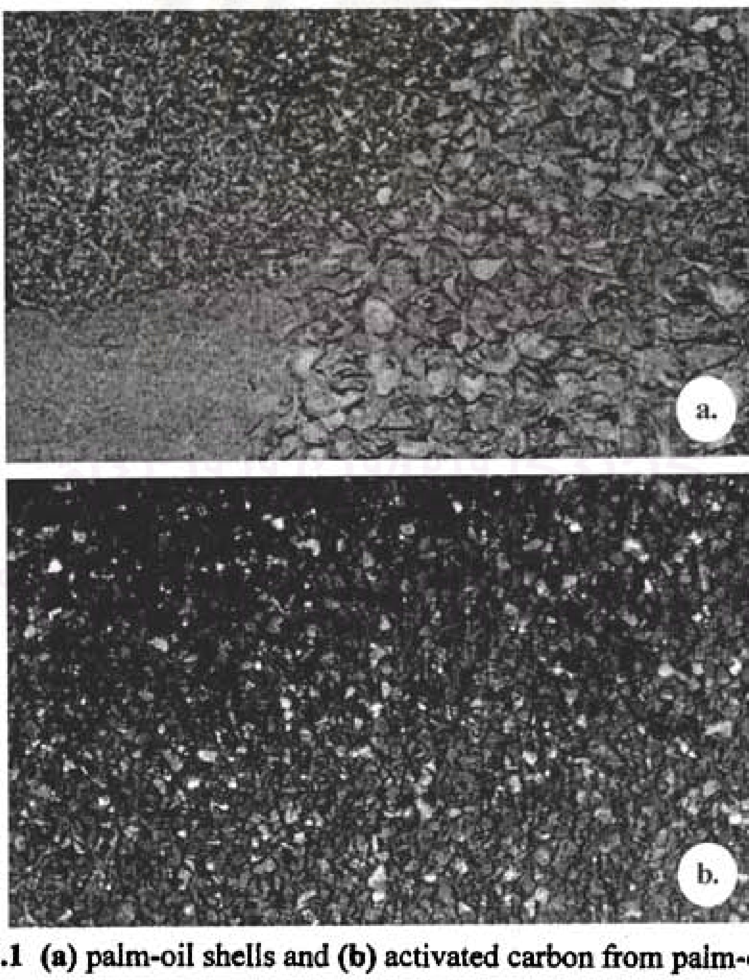


Figure 3.1 **(a)** palm-oil shells and **(b)** activated carbon from palm-oil shells.

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. Pyrolysis and steam activator:

    The fixed bed reactor was a stainless steel tube of 100 mm inside diameter, 450 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 flew into the bed which was controlled by using a rotameter. Steam was preheated by burner before flowing into the bed. The fixed bed pyrolysis and steam activator which was used in this work and a schematic of the experimental setup are shown in **Figures 3.2-3.3**.

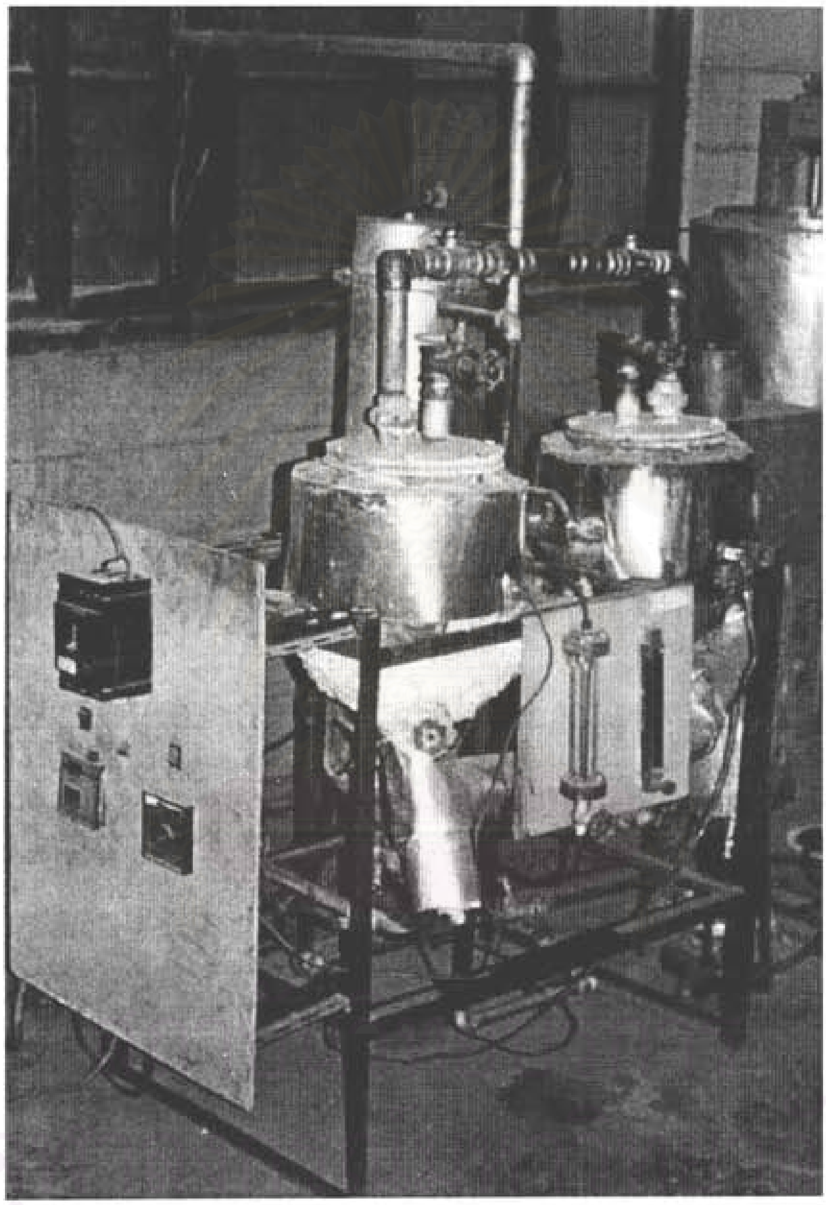
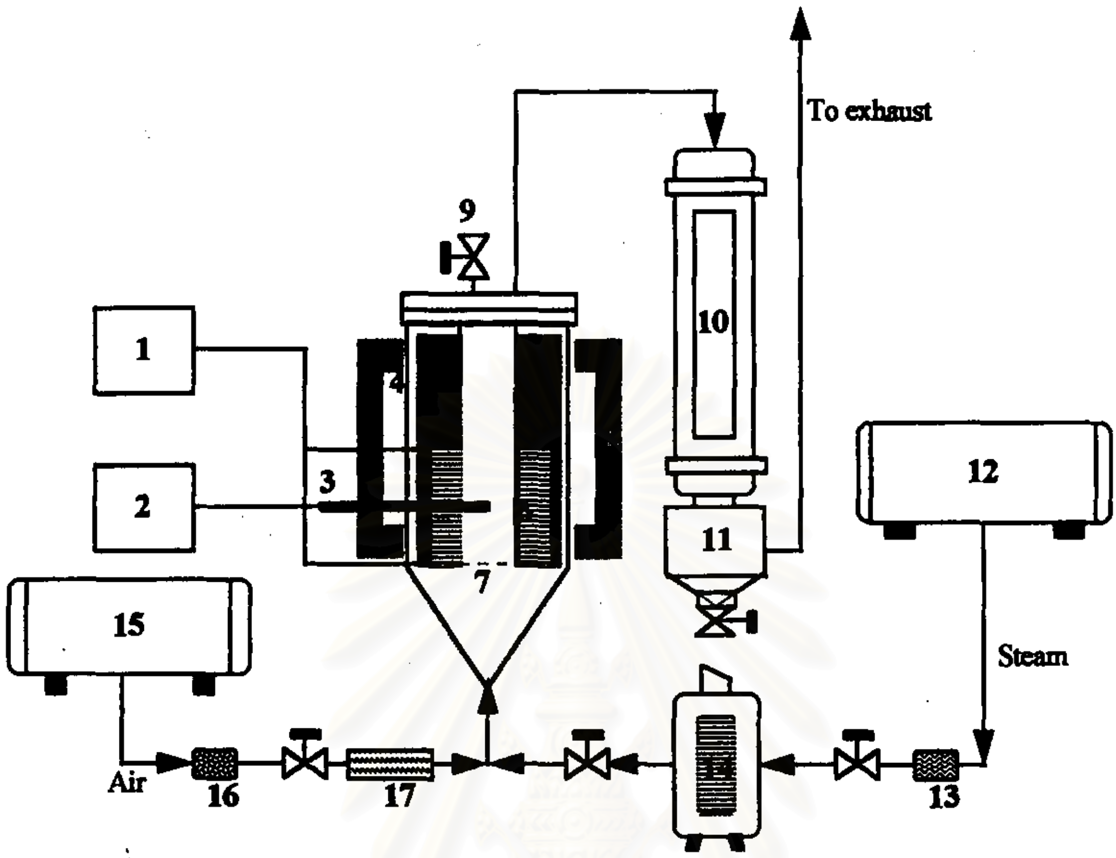


Figure 3.2 The fixed bed pyrolysis and steam activator.



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

Figure 3.3 A schematic of the fixed bed pyrolysis and steam activator experimental setup.

3.4 Procedures

The various parameters which had the effect in the production of the activated carbon: temperature, time, size of the raw material, flow rate of air and adding pyrolysis with air before steam activation were studied in order to determine the optimum condition. Experimental scheme is shown in **Figure 3.4**. The procedures are described as follows:

3.4.1 The optimum temperature and time for pyrolysis and steam activation

Four temperatures were studied in this work. They were 600, 650, 700 and 750°C. Each of these was studied for 1, 2 and 3 hr to get the optimum temperature and time for pyrolysis and steam activation. The fixed bed reactor was heated until the temperature in bed reached 600°C. The 200 g of 2.36-4.75 mm of the palm-oil shells were charged into it with air at a flow rate of 0.72 nl/min. Then, the temperature in the fixed bed reactor was raised and fixed at the final temperature 600, 650, 700 and 750 °C. The excess steam was continued on, passing up through the bed for 1, 2 and 3 hr. Finally, the products were characterized as % yield, % moisture, % ash, bulk density, iodine number and methylene blue number.

3.4.2 The optimum size for pyrolysis and steam activation

The sizes of the palm-oil shells <0.06, 0.06-1.18, 1.18-2.36, 2.36-4.75, 4.75-6.00 and 6.00-8.00 mm were studied to get the optimum size for production of activated carbon. The fixed bed reactor was heated until the temperature in bed reached 600°C. The 200 g of each size of the palm-oil shells were charged into it with air at a flow rate of 0.72 nl/min. Then, the temperature in the fixed bed reactor was raised and fixed at 750°C. The excess steam was continued on, passing up through the bed for 2 hr. The final products were characterized as % yield, % moisture, % ash, bulk density, iodine number, methylene blue number and B.E.T.surface area.

3.4.3 The optimum flow rate of air for pyrolysis and steam activation

The flow rates of air 0.20, 0.49, 0.72 and 0.97 nl/min were studied to get the optimum flow rate of air for production of activated carbon. The fixed bed reactor was heated until the temperature in bed reached 600°C. The 200 g of 1.18-2.36 mm of the palm-oil shells were charged into it with each flow rate of air. Then, the temperature in the fixed bed reactor was raised and fixed at 750°C. The excess steam was continued passing up through the bed for 2 hr. Finally, the products were characterized as % yield, % moisture, % ash, bulk density, iodine number and methylene blue number.

3.4.4 Pyrolysis with air 30 min before steam activation

This time, five temperatures were studied. They were 600, 650, 700, 750 and 800°C for 1, 2 and 3 hr. But each temperature for each specific time needed pyrolysis with air 30 min before steam activation in the production of the activated carbon. These experiments were studied to obtain the optimum condition for this work. The fixed bed reactor was heated until the temperature in bed reached 600°C, the 200 g of 1.18-2.36 mm of the palm-oil shells were charged into it with air at a flow rate of 0.72 nl/min. Then, the temperature in the fixed bed reactor was raised and fixed at the final temperature of 600, 650, 700, 750 and 800°C. The palm-oil shells were pyrolysed with air for 30 min before activation with steam for 1, 2 and 3 hr. In the end, the products were characterized as % yield, % moisture, % ash, bulk density, iodine number, methylene blue number and B.E.T. surface area.

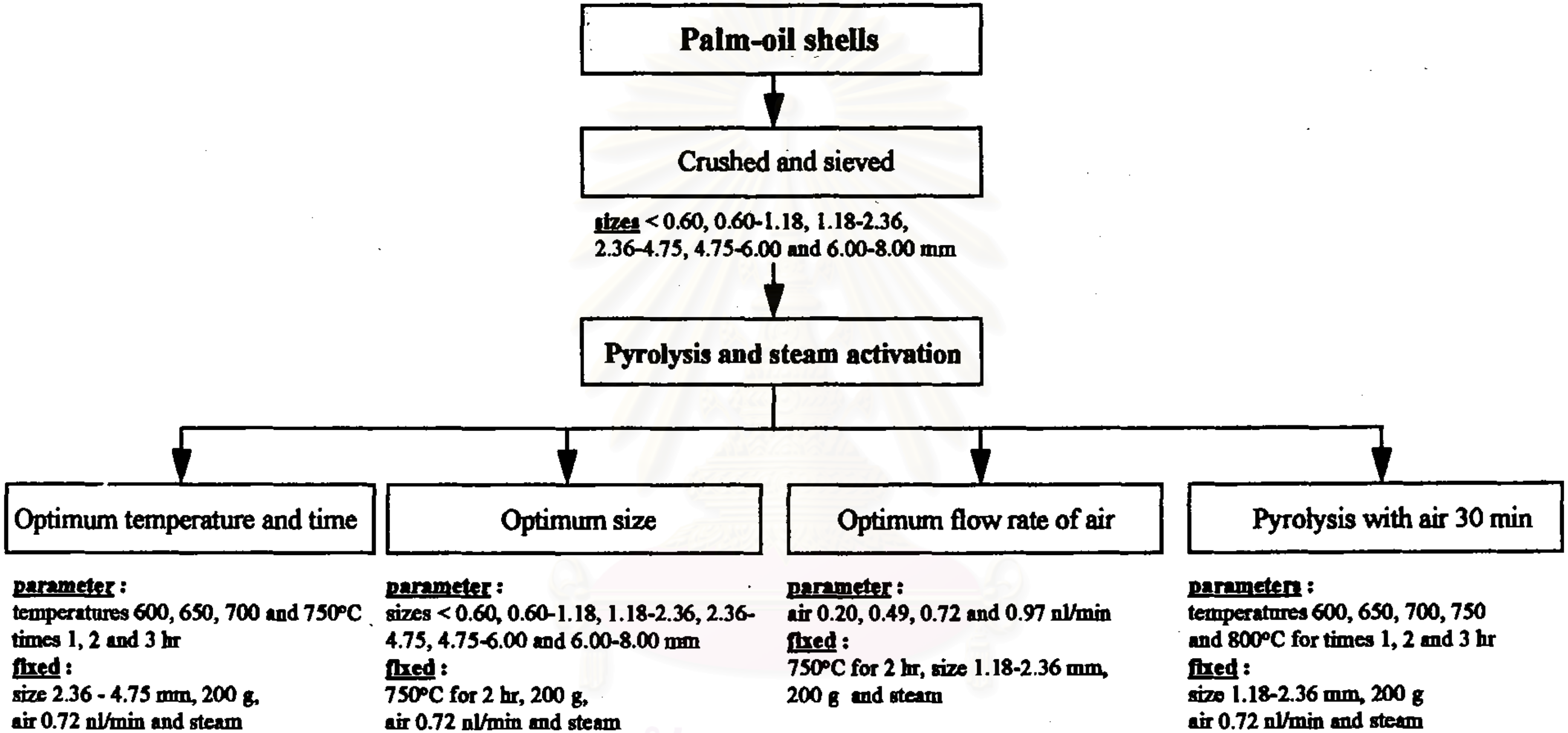


Figure 3.4 Experiment scheme of the production of activated carbon from palm-oil shells by pyrolysis and steam activation in a fixed bed reactor.