CHAPTER III

EXPERIMENT

3.1 Raw materials

This experiment used anthracite and palm-oil shell for the production of activated carbon by zinc chloride activation. The palm-oil shell was crushed and sieve to four different particle sizes of 0.25-0.50, 0.50-1.18, 1.18-2.36, 2.36-4.75 mm. Anthracite particle size used was 0.8-0.9 mm only. The pictures of anthracite and palm-oil shell are shown in **Figure 3.1** and **Figure 3.2**, respectively.

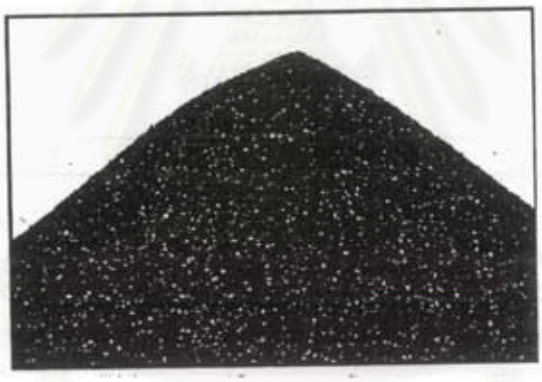


Figure 3.1 Anthracite.

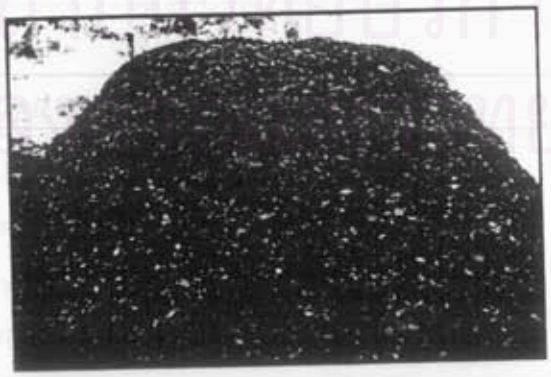


Figure 3.2 Palm-oil shell.

3.2 Chemicals

1. Zinc Chloride, ($ZnCl_2$) : Carlo Erba
2. Hydrochloric acid, concentrated (HCl) : Merck
3. Iodine, (I_2) : AJAX
4. Potassium iodide, (KI) : BDH
5. Potassium iodate, (KIO_3) : May & Baker
6. Sodium thiosulfate, ($Na_2S_2O_3 \cdot 5H_2O$) : Univar
7. Sodium carbonate, (Na_2CO_3) : Merck
8. Starch, soluble potato
9. Potassium phosphate,(KH_2PO_4) : Merck
10. Methylene blue, ($C_{16}H_{18}N_3SCl \cdot 3H_2O$) : Merck
11. Sodium phosphate, (Na_2HPO_4) : Univar

3.3 Apparatus

1. Carbonizer:

   The fixed bed reactor was a stainless steel tube of 158 mm inside diameter and 1150 mm in length. The fixed bed was heated by an electric element (2000 watt) and the bed temperature was measured by type K thermocouple. The carbonizer was used for this work and schematics of the experimental setup are shown in **Figures 3.3-3.4.**
2. Ball mill.
3. Laboratory test sieve: s/steel, sizes 0.25, 0.50, 1.18, 2.36 and 4.75 mm, Endecotts, England.
4. Sieve shaker: EFL1 mk3, Endecotts, England.
5. Muffle furnace: type ESF 12/23 (0-1,200°C), Carbolite, England.
6. Oven: 0-250°C, WT binder, Germany.
7. Tube furnace: type 21100 (0-1,200°C), Thermolyne Corporation, USA.

8. Mortor grinder: type RM100, Retsch, Germany. Shown in **Figure 3.5**.
9. Shaker.
10. Tube furnace: type MTF 12/25/250 (0-1,200°C), Carbolite England, shown in **Figure 3.6**.
11. Bubble flow meter.
12. Balance: type 1702 MP8, Sartorius, Germany.
13. Ultra-high centrifugal: Model KC-25, Kubota, Japan.
14. Spectrophotometer: Spectronic 21 (320-1,000 nm), Miltonroy company, USA.
15. X-ray spectrometer: Model PW 2400, Philips, Netherlands, shown in **Figure 3.7**.
16. Surface area analyzer: Model Flow Sorb II 2300, Micromeritics Instrument Corporation, USA, shown in **Figure 3.8**.

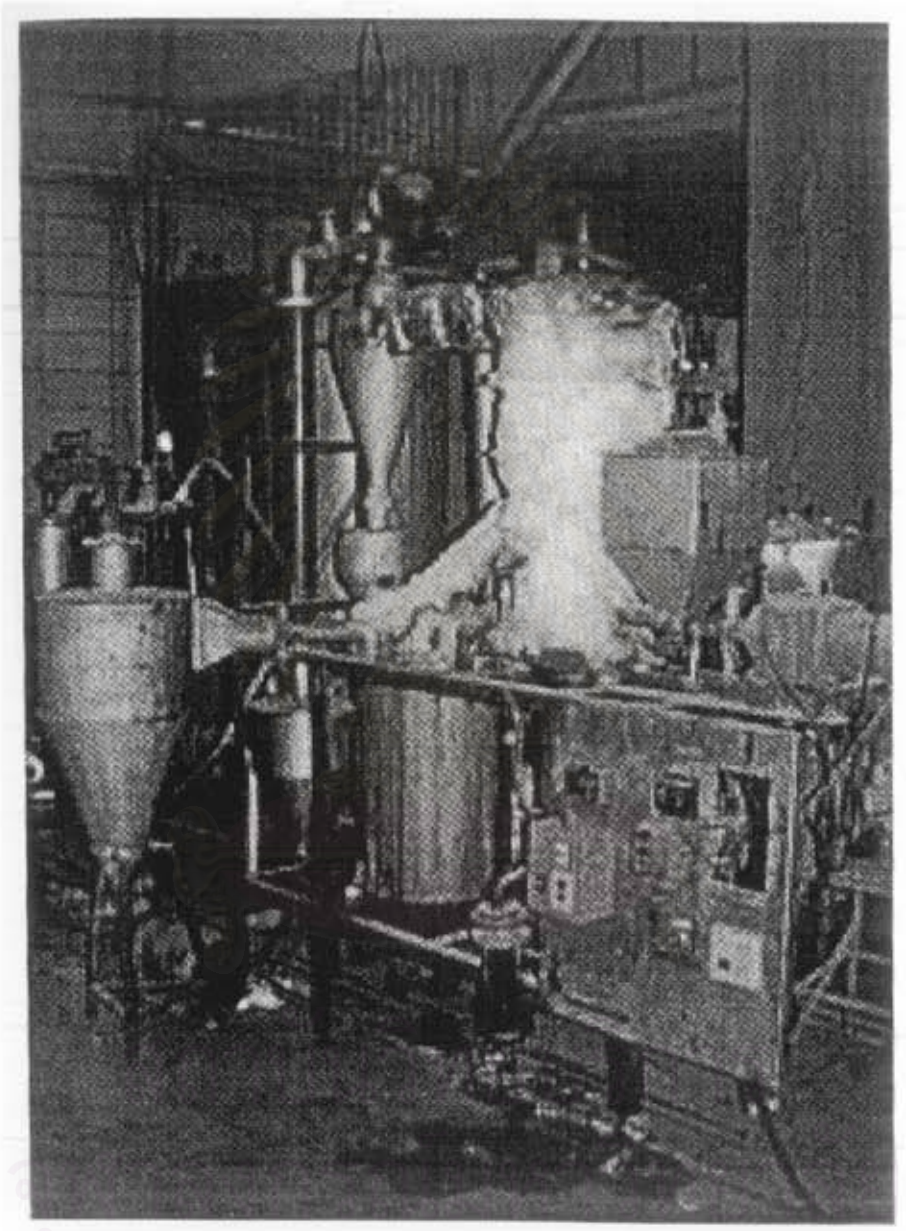
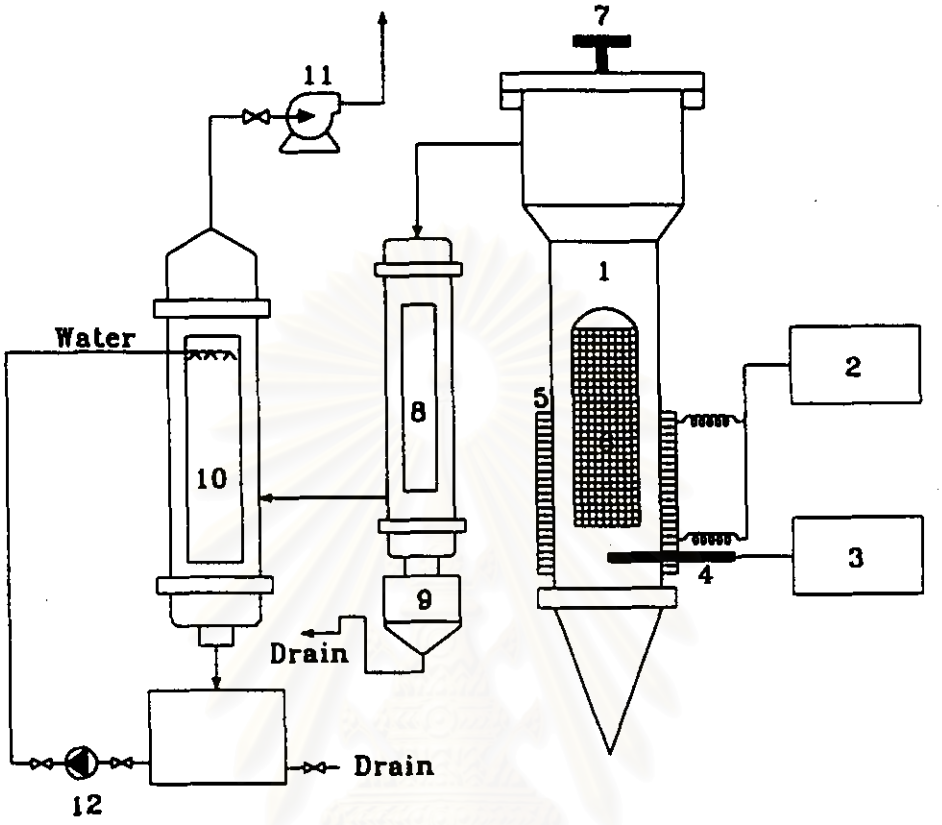


Figure 3.3 The fixed bed carbonizer.



1. Reactor
2. A.C. Arc welder
3. Temperature controller
4. Thermocouple
5. Heating coil
6. Sample support
7. Sample feed and removal port
8. Condenser
9. Separator
10. Scrubble
11. Brower
12. Circulate pump

Figure 3.4 A schematic of the fixed bed carbonizer experimental setup.

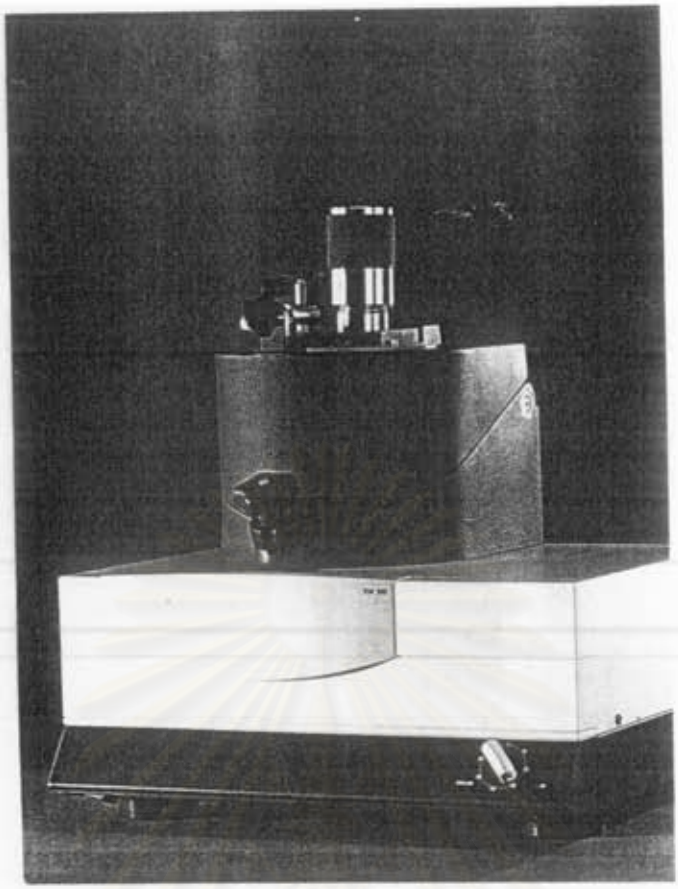


Figure 3.5 Mortor grinder: type RM100.

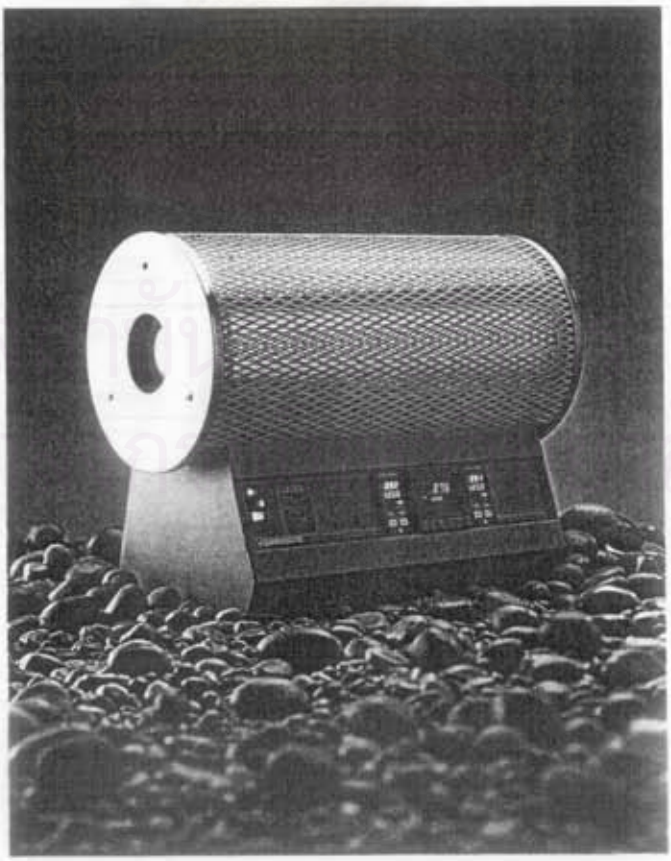
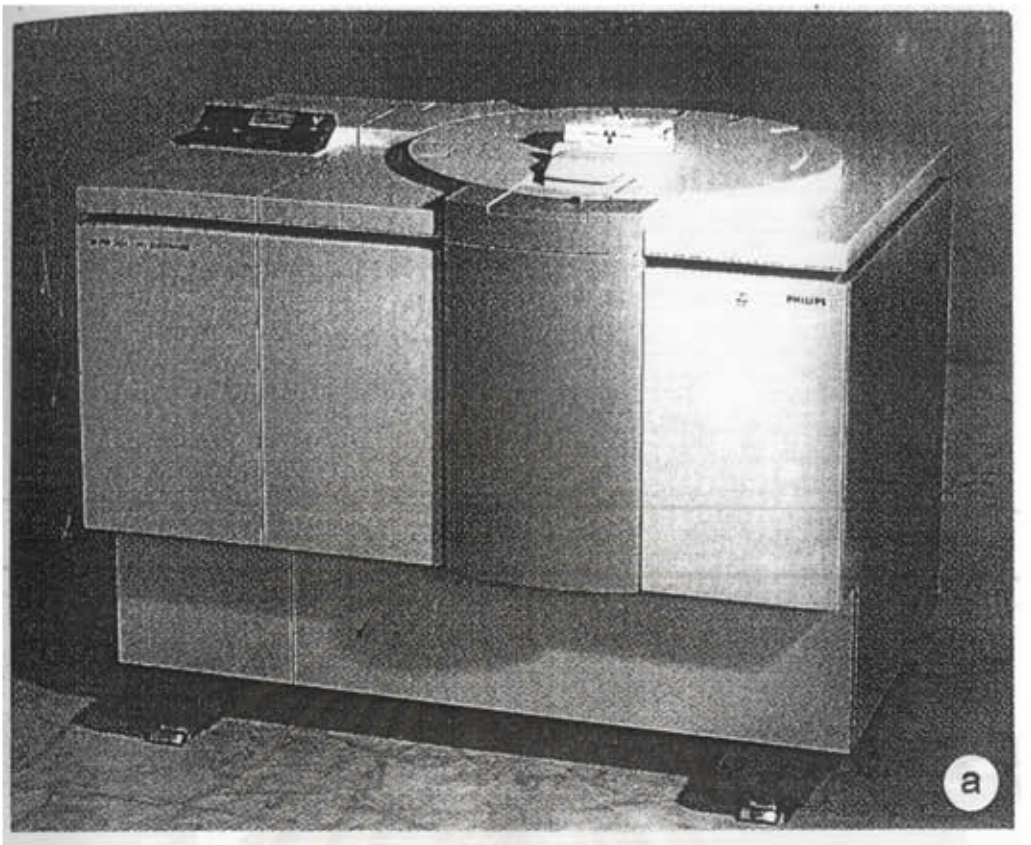


Figure 3.6 Tube furnace: type MTF 12/25/250.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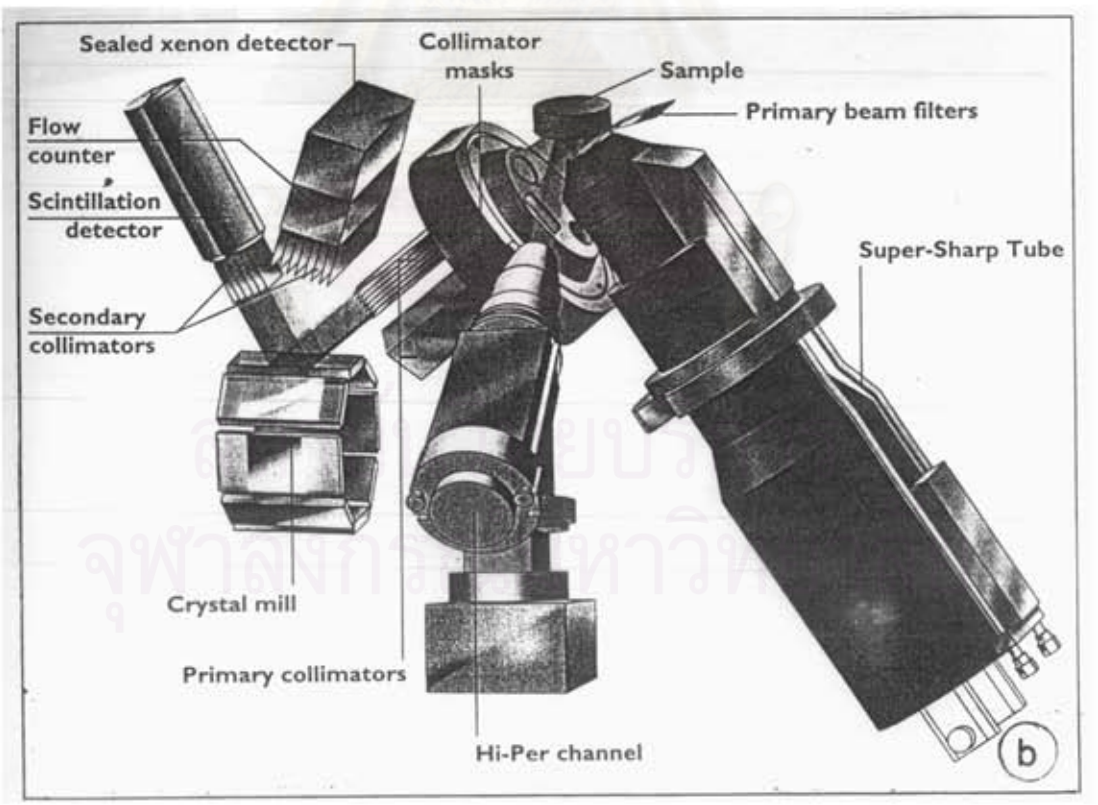
Sealed xenon detector
Collimator masks
Sample
Primary beam filters
Flow counter
Scintillation detector
Super-Sharp Tube
Secondary collimators
Crystal mill
Primary collimators
Hi-Per channel
b

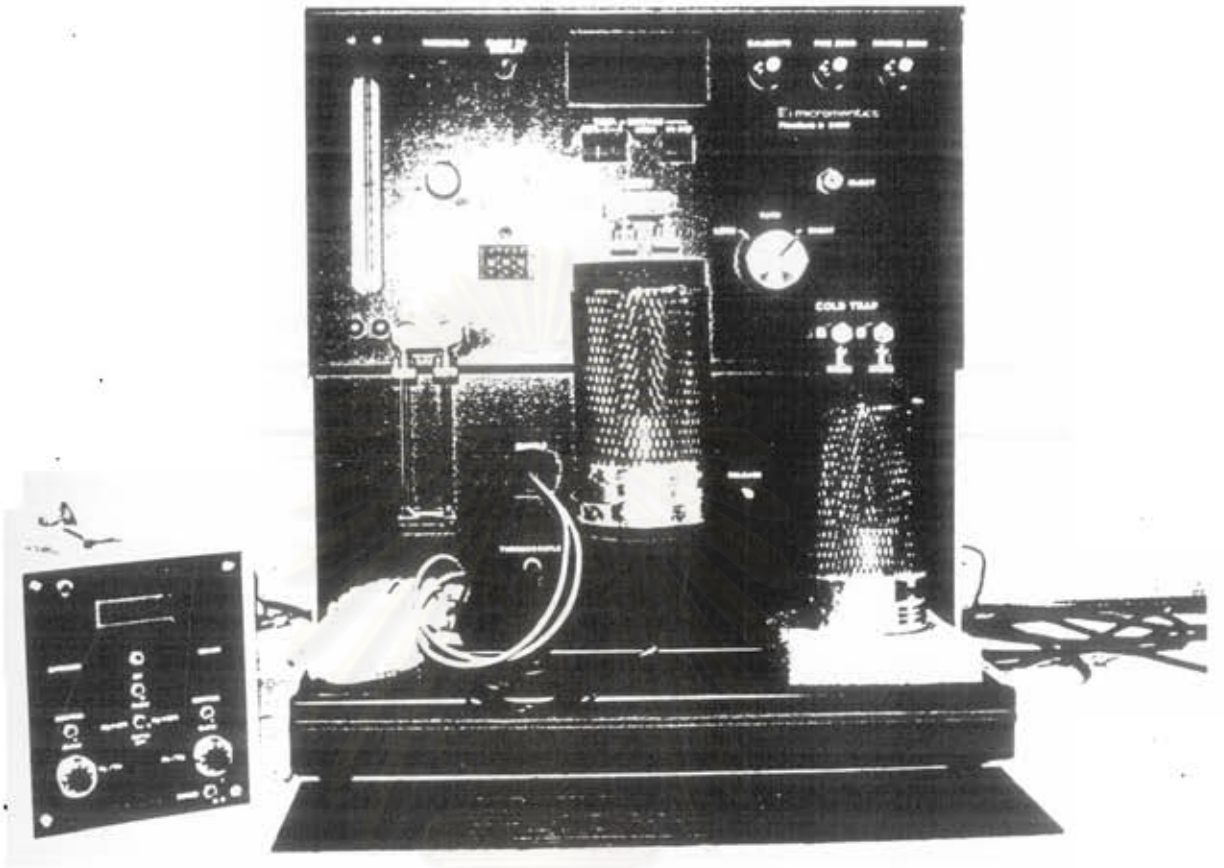

Figure 3.8 Surface area analyzer.
(Model: Flow Sorb II 2300)

3.4 Procedures

3.4.1 Carbonization of palm-oil shell

The optimum temperature and time for carbonization of palm-oil shell (from Patra's work) were fixed at 400°C, 60 min. Firstly, carbonizer was heated and fixed at 400°C. Next, the mass of 500 g of palm-oil shell was charged in the carbonizer. When the temperature of the bed reached at 400°C, it was carbonized for 60 min. The char from carbonization was crushed and sieved for four different particle sizes of 0.25-0.50, 0.50-1.18, 1.18-2.36 and 2.36-4.75 mm. Finally, the % yield, % ash, % moisture, % volatile matter, % fixed carbon and bulk density of the palm-oil shell char and anthracite were analyzed approximately.

3.4.2 Activation of anthracite and palm-oil shell char

The variables which have an effect on the activation such as : temperature, activation time, size of char and concentration of zinc chloride solution were studied in order to determine the optimum conditions. Experimental scheme of the production of activated carbon from anthracite and from palm-oil shell were shown in **Figure 3.9** and **Figure 3.10,** respectively. The procedures were described as follows:

3.4.2.1 The optimum temperature for activation

Five different temperatures were studied in this work for activation. They were 500, 600, 700, 750, 800°C. Firstly, the sample mass of 15 g of anthracite and palm-oil shell char with particle size of 1.18-2.36 mm were soaked in 40% zinc chloride solution, then dried and loaded into a stainless tubular reactor. Then the reactor was passed with nitrogen gas at the bottle. The nitrogen gas was then allowed to flow through from the bottom to the top at the flow rate of 100 nl/min. Secondly, the bed was heated to the temperature desired to vary at

500, 600, 700, 750 and 800°C. The nitrogen gas was continued on, passing up through the bed for each fixed temperature and for 3 hr of reaction time. Finally, the product was characterized by bulk density, iodine number, methylene blue number, B.E.T. surface area and determined of deposition of zinc in prepared activated carbon.

3.4.2.2 The optimum time for activation

Different reaction times of 1, 2, 3 and 4 hr were studied for activation. Firstly, the mass of 15 g of anthracite and palm-oil shell char with particle size of 1.18-2.36 mm were soaked in 40% zinc chloride solution, then dried and loaded into the reactor. The procedure of operation was the same as the former experiment namely, the reactor was connected to a nitrogen gas bottle. The nitrogen gas was then allowed to flow through the reactor from the bottom to the top. Secondly, the reactor was heated until the temperature of the bed increasing and being fixed at the temperature 750°C. The nitrogen gas was continued on, passing up through the bed for varying reaction time of 1, 2, 3 and 4 hr. Finally, each product was characterized by bulk density, iodine number, methylene blue number, B.E.T.surface area and determined of deposition of zinc in the activated carbon.

3.4.2.3 The optimum size of palm-oil shell char for activation

The sizes of palm-oil shell char of 0.25-0.50, 0.50-1.18, 1.18-2.36 and 2.36-4.75 mm were studied to get the optimum size for preparation of activated carbon. The mass of 15 g of palm-oil shell char was soaked in 40% zinc chloride solution, then dried and loaded into reactor. The nitrogen gas was then allowed to flow through the reactor from the bottom to the top. Then, the bed was heated until the temperature in the tube was raised and fixed at the final temperature 750°C. The nitrogen gas was continued on, passing up through the tube reactor for 3 hr. Finally, the product of each experiment was characterized by bulk density, iodine number,

methylene blue number, B.E.T.surface area and also determined the deposition of zinc in the activated carbon.

3.4.2.4 The optimum concentration of zinc chloride for activation

The different concentrations of zinc chloride at 20%, 30%, 40%, 50%, and 60% by weight were studied to determine the optimum concentration for soaking for preparation of activated carbon. Firstly, the mass of 15 g of anthracite (0.80-0.90 mm of particle size) and palm-oil shell char (size of 1.18-2.36 mm) were soaked in different concentrations of zinc chloride solution, then dried and loaded into reactor. The procedure of operation was the same as the former experiment namely, the tube reactor was connected to a nitrogen gas bottle. The nitrogen gas was then allowed to flow from the bottom to the top of reactor. Secondly, the bed was heated until the temperature in the tube raising and being fixed at the final temperature 750°C. The nitrogen gas was continued on, passing up through the bed for 3 hr of each experiment. Finally, the product was characterized by bulk density, iodine number, methylene blue number, B.E.T. surface area and determined the deposition of zinc in activated carbon.

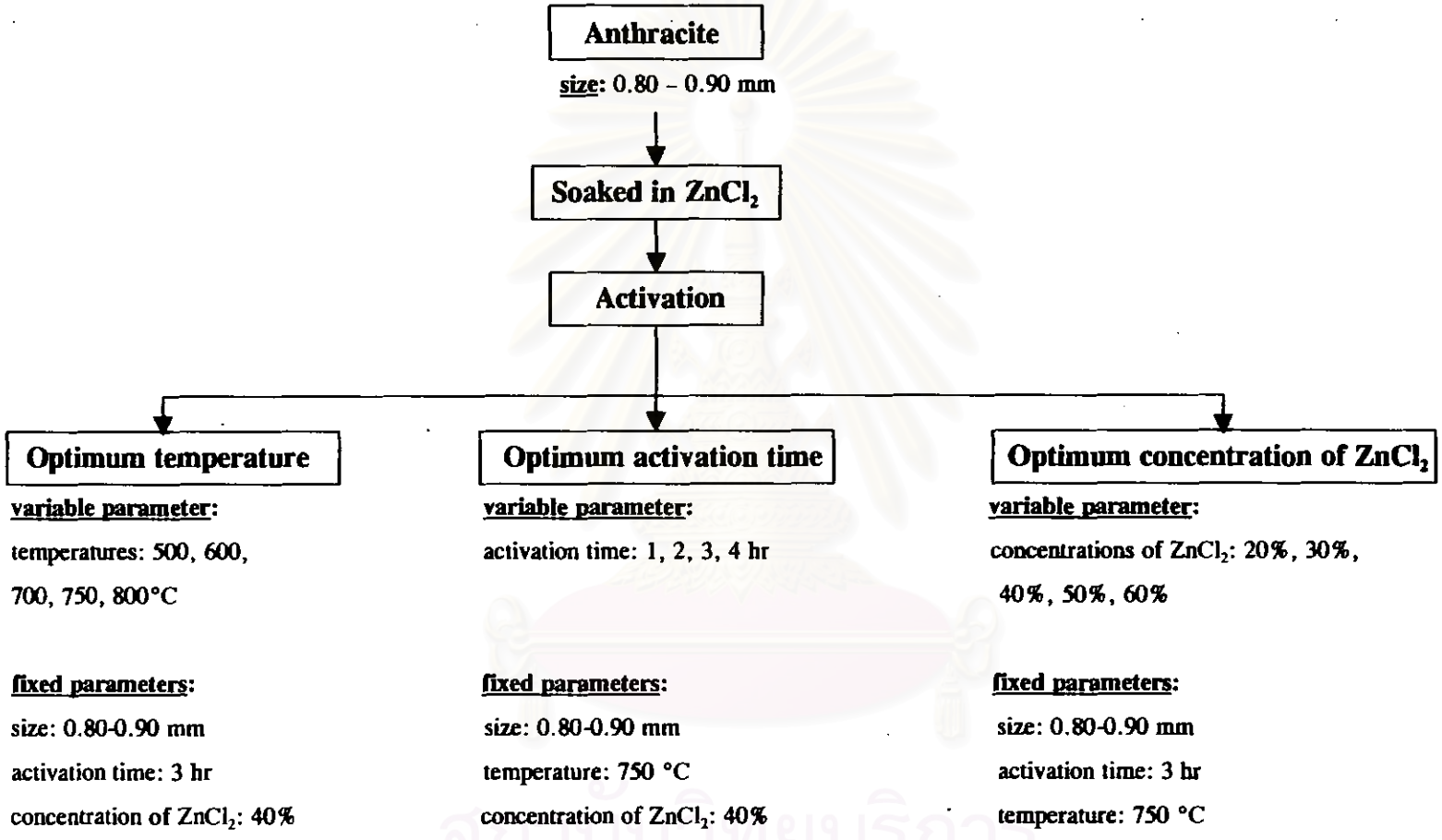

Figure 3.9 Experiment scheme of the production of activated carbon from anthracite by $ZnCl_2$ activation.

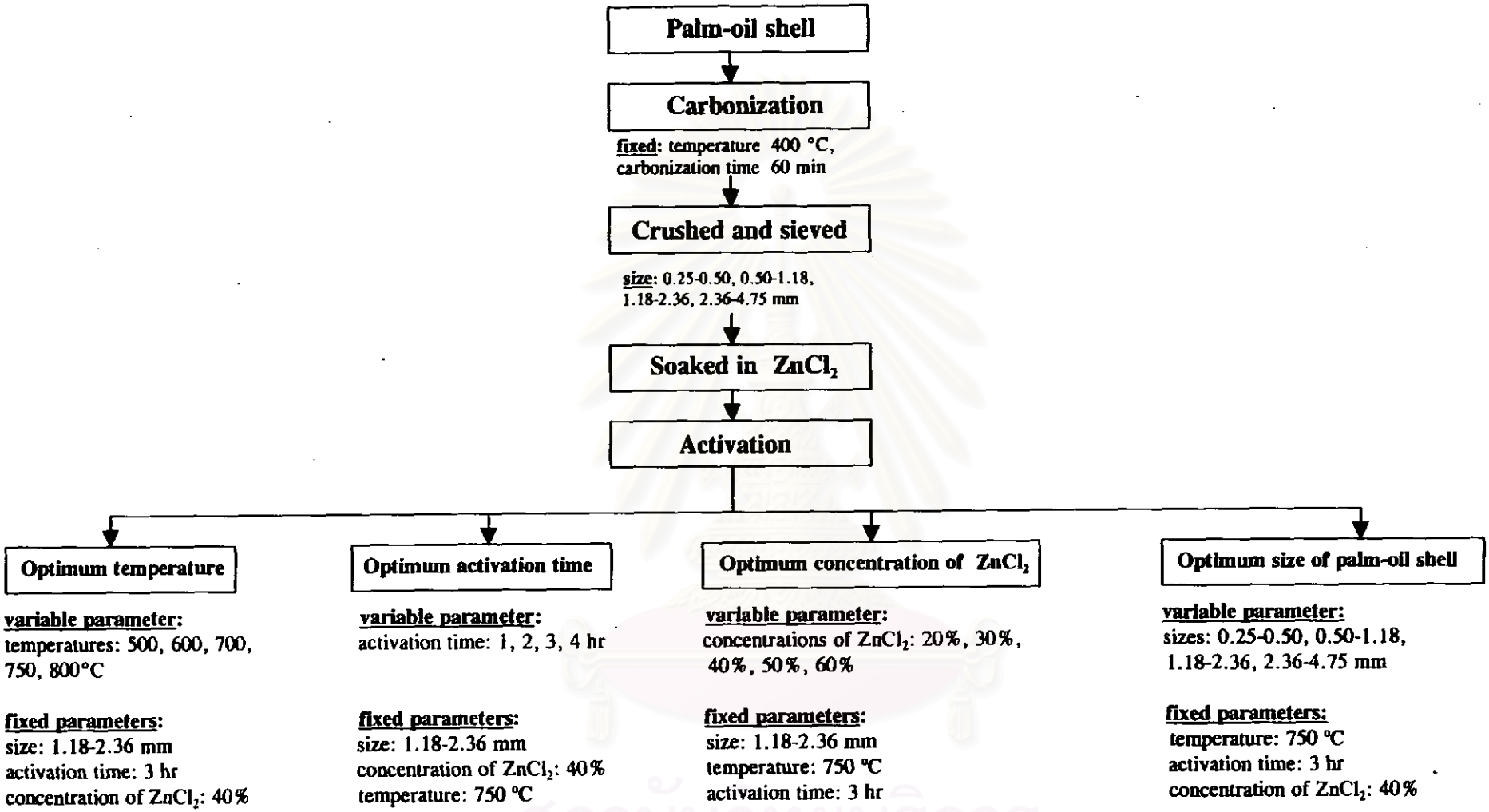

Figure 3.10 Experiment scheme of the production of activated carbon from palm-oil shell by $ZnCl_2$ activation.