CHAPTER III

EXPERIMENTAL PROCEDURE

This research was experimental in laboratory to find out condition in producing activated carbon from coconut shells by carbonization activation by carbon dioxide and superheated steam activation in fixed bed for study about qualification and potential of the activated carbon. This was used as information for producing or using in other cases and as data in large scale.

3.1 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: type21100 (0-1,200 °C) Thermolyne Corporation, USA.
4. Bubble flow.
5. Desicator.
6. Laboratory test sieve: s/steel, sizes 0.35,0.6, 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. Crucible.
13. Scanning Electron Microscope (SEM): Model JSM-6400
14. Surface area analyzer: ASAP 2000, Micromeritics Instrument Corporation, analysis program: run 20 com.

15. Activator:

The fixed bed reactor was a vertical retort heater. There was a stainless tube of 0.02 m inside diameter and 0.4 m in length for sample feed and removal port. The bed could be operated maximum 1,200 °C. Air flew into the bed, which was controlled by using a rotameter. The fixed bed pyrolysis and steam activator, which was used in this work and a schematic of the experimental setup, are shown in **Figures 3.1-3.2.**

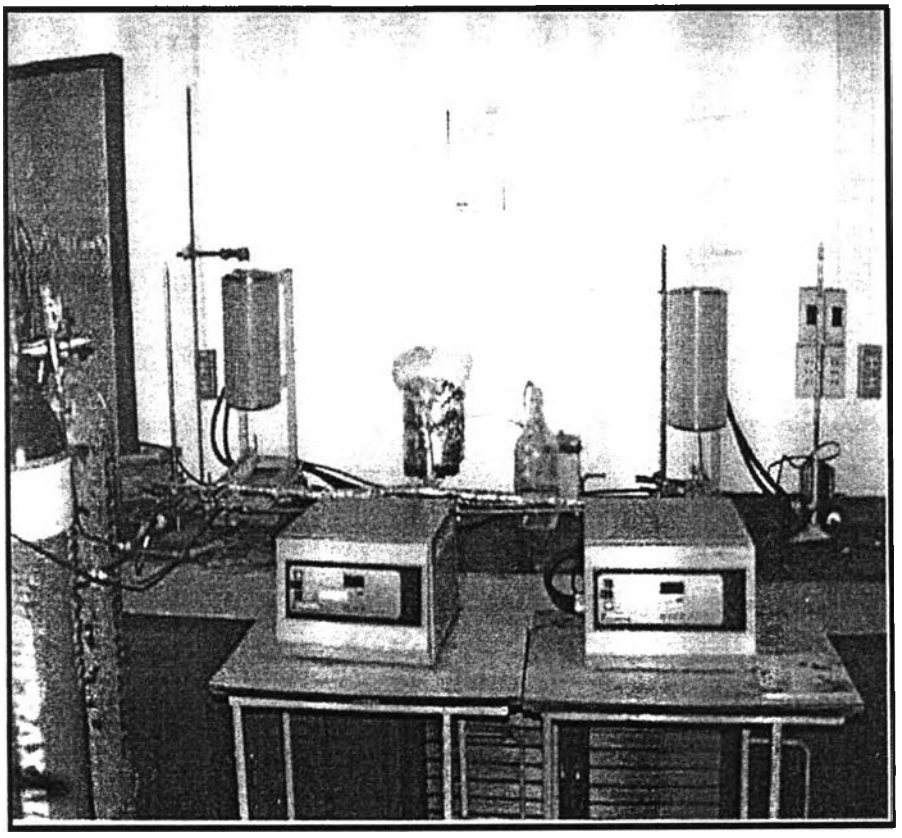


Figure 3.1 Activator.

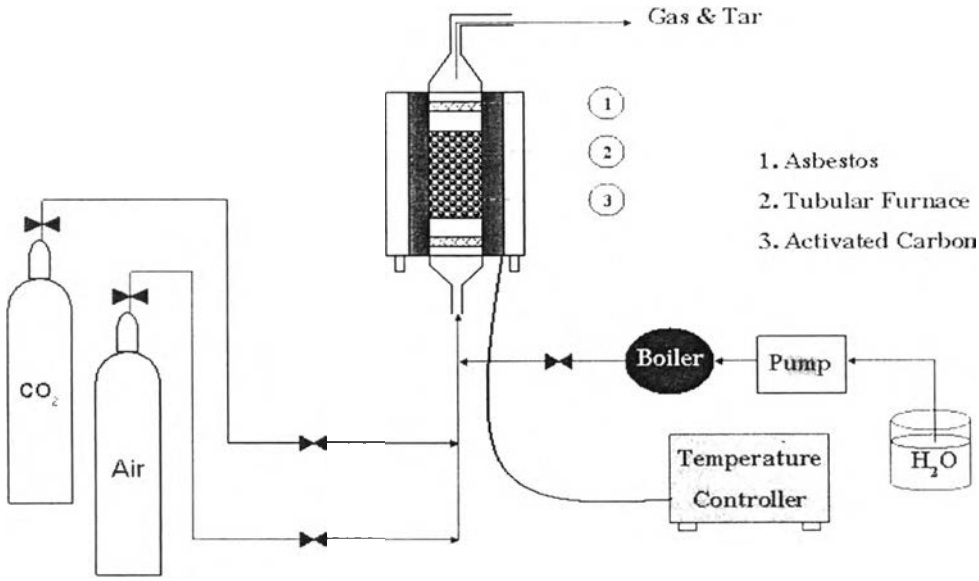


Figure 3.2 A schematic of the fixed bed activator experimental setup.

3.2 Chemicals

1. Hydrocholoric acid, concentrated (HCl)	AJAX
2. Sodium thiosufate, ($Na_2S_2O_3$ $5H_2O$)	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 ($C_{16}H_{18}N_3SCL$ $3H_2O$)	Carlo Erba
9. Sodium phosphate, (Na_2HPO_3)	Carlo Erba
10. Potassium phosphate, (KH_2PO_3)	Merck

3.3 Raw material

The raw material was prepared from coconut shell from Nakron-Pratom province which were left from agriculture. The raw material is shown in Figure 3.3.

Figure 3.3 Coconut shells.

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 ratio CO_2/H_2O and adding pyrolysis before activation in order to determine the optimum condition. Experiment is shown in Figure 3.4. The procedures are described as follows :

3.4.1 Carbonization

The optimum temperature and time for carbonization of coconut shells were studied at 300, 350, 400, 450 and 500^{0}C for the duration of 45, 60, 90 and 120 min of time. The procedures started by weighing sample about 10 mg in crucible. Next, muffle furnace were heated at the final temperature as 300, 350, 400, 450 and 500^{0}C and put the sample into the muffle furnace at 45, 60, 90 and 120 min, respectively. Finally, the %yield, %ash, bulk density, %volatile matter and %fixed carbon of products would be analyzed following ASTM.

3.4.2 Activation

The chars from the carbonization at 400°C for 60 min were produced to become the activated carbon. They were crushed and sieved to particle sizes of 0.33-0.60, 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

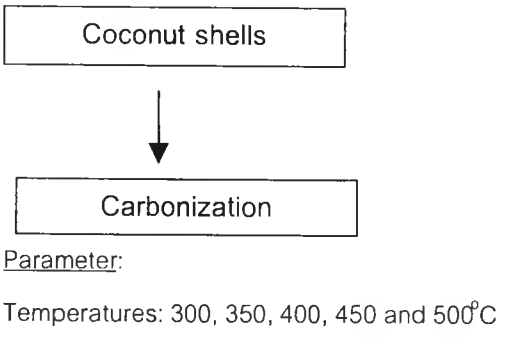
At 800, 850, 900, 950 and 1,000°C were studied the optimum temperature in activator and reached 60, 120, 180 and 240 min, respectively. Second, the 10g of 1.18-2.36 mm of the chars were charged into a stainless tube of 0.02 m inside diameter and 0.4 m in length with the air at a flow rate of 0.005 l/min. Then, the superheated steam and CO_2 at a flow rate of 0.01 l/min and 0.5 l/min were passed through the bed for 60, 120, 180 and 240 min. The products would be characterized as the %yields, % 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 0.33-0.60, 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 950°C. The 10 g of each size of chars were charged into it with the air at a flow rate of 0.005 l/min, the superheated steam and CO_2 at a flow rate of 0.01 l/min and 0.5 l/min were passed through the bed for 60 min. The products would be characterized as the %yield, %ash, bulk density, iodine number, methylene blue number and B.E.T. surface area.

3.4.2.3 The optimum composition of CO_2 for activation

The composition of CO_2 (by weight) at 0, 4.2, 6.5, 8, 14 and pure CO_2 with fixed air and superheated steam at a flow rate of 0.005 l/min and 10 g/min were studied for activation. In the beginning, the fixed bed activator was heated until the temperature in the bed reached 950^0C. The 10 g of 0.6-1.18 mm of the chars were charged into it with air, superheated steam each flow rate of CO_2 were passed through the bed for 60 min. Finally, The products were characterized as the %yield, %ash, bulk density, iodine number, methylene blue number and B.E.T. surface area.



Optimum temperature and time	Optimum size	Optimum Composition of CO_2
Parameters: Temperatures: 800, 850, 900, 950 and 1,000°C. Time: 60, 120, 180 and 240 min. Fixed: Size 1.18-2.36 mm. Air flow rate: 0.005 l/min. CO_2 flow rate: 0.5 l/min. H_2O flow rate: 0.01 l/min.	Parameters: Size: 0.3350.6, 0.6-1.18, 1.18-2.36 and 2.36-4.75 mm. Fixed: Temperatures: 950°C. Time: 60 min. Air flow rate: 0.005 l/min. CO_2 flow rate: 0.5 l/min. H_2O flow rate: 0.01 l/min.	Parameters: Composition of CO_2 (by weight) at 0, 4.2, 6.5, 8, 14 and pure CO_2. Fixed: Temperatures: 950°C Time: 60 min. Size 0.6-1.18 mm. Air flow rate: 0.005 l/min. H_2O flow rate: 0.01 l/min.

Figure 3.4 Experiment scheme of the production of activated carbon from coconut shells in an activator.