



## CHAPTER 3

### EXPERIMENTAL CONSIDERATIONS, MATERIALS AND PROCEDURE

The experiment was separated into two parts ; one for rice hull pyrolysis and the other for heat capacity measurement. The details of each part are described as follows :

#### 3.1 Rice Hull Pyrolysis

##### 3.1.1 Experimental Apparatus

The sketch of the cylindrical furnace is shown in Figure 3.1 and the details are as follows : the stainless steel inner tube is surrounded by a 5 KVA heater coiled around it to sustain the pyrolysis and sand is filled between outer and inner tubes. The material to be pyrolysed is placed in the chamber of the inner tube. At mid-height of the heating zone, a small hole was drilled for inserting a thermocouple. The bottom of the inner tube is closed by iron sheets consist of two disks.

A 3 5/8 inch diameter screen was placed on the top of the inner tube. So that water and volatile matter released from the sample be able to exit the furnace during pyrolysis. On top of this screen was placed a sheet of asbestos insulator containing three small holes drilled for temperature measurements

at distances of 1.6 and 3.2 cm from the edge and at the center of the inner tube. The three thermocouples were then inserted parallel to the axis of the cylinder. The tip of the thermocouples were in the middle of the rice hull bed as shown in Figure 3.1.

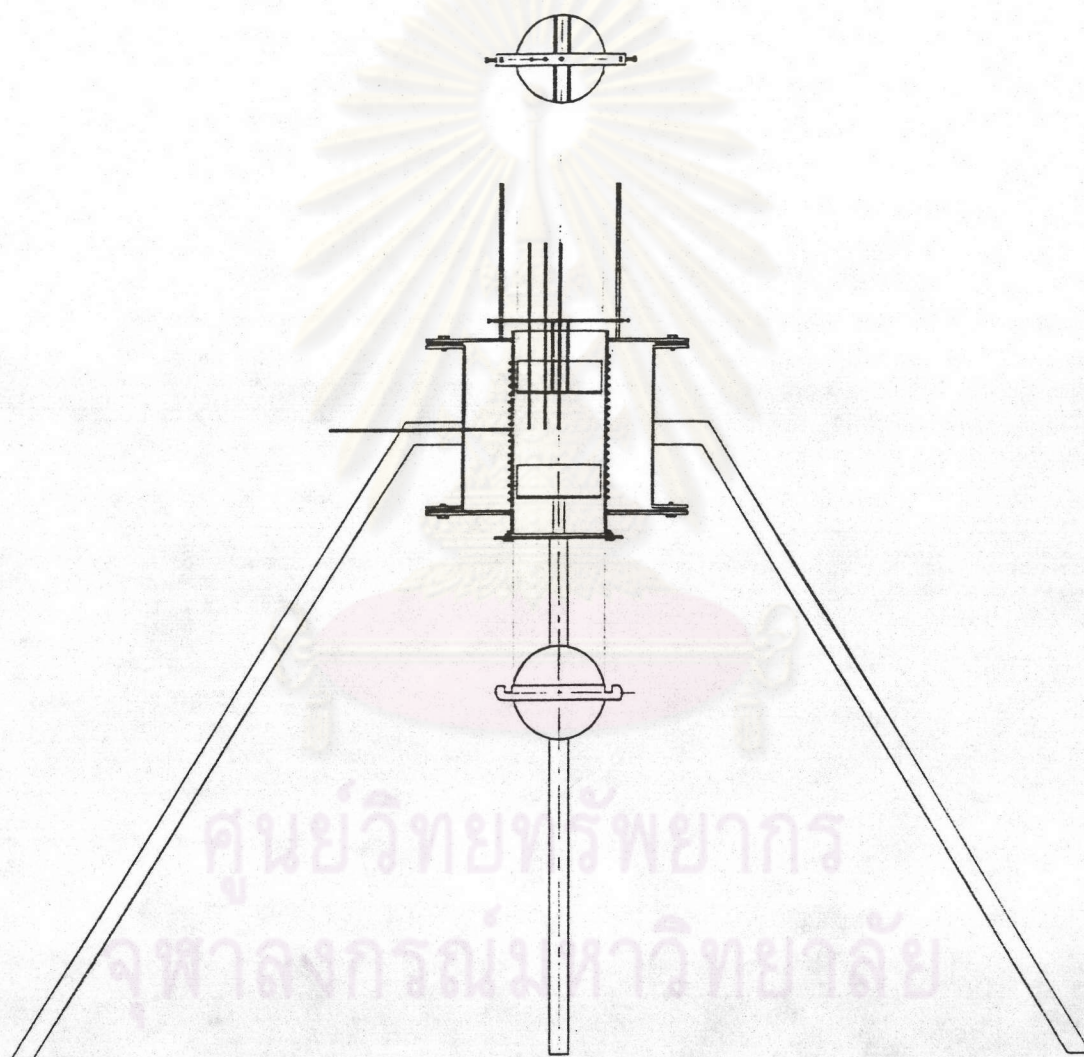


Figure 3.1 Sketch of the Cylindrical Furnace

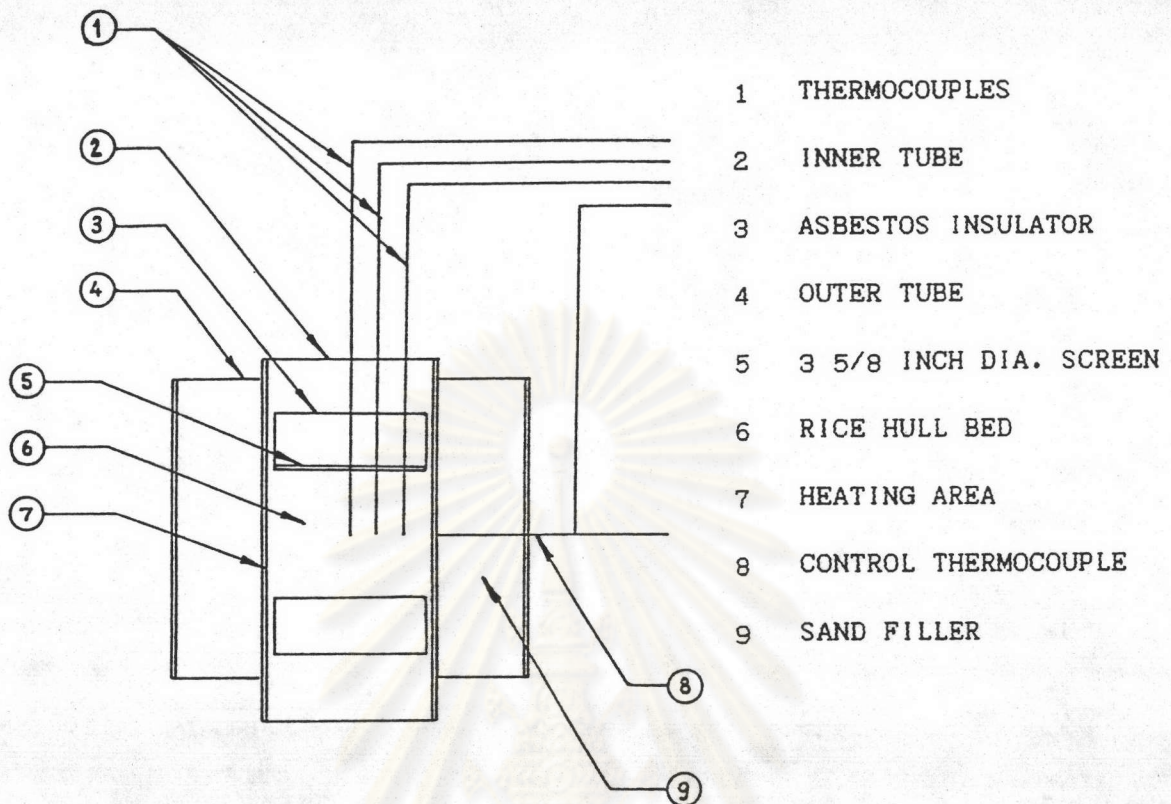


Figure 3.2 Schematic Diagram of the Pyrolysis Apparatus

### 3.1.2 Sample Preparation

The samples used for this experiment were prepared at 0.11, 8.49, 16.35 and 24.04% moisture content\* by means of drying and adding water.

Initial samples (fresh rice hull) were at 8.49% moisture content. The samples were conditioned to higher moisture

---

\* All moisture contents are in percent wet basis unless otherwise specified.

contents by adding water to fresh samples. These samples were placed in plastic bags and allowed to equilibrate for two weeks and occasionally mixed several times for a more uniform moisture distribution.

To lower the moisture content of the samples, the samples were dried using a drying oven. The moisture content of the samples were determined while conducting the experimental tests.

### 3.1.3 Experimental Procedure

The operating procedure was to weigh a certain amount of rice hulls, seal the furnace bottom, place the rice hulls in the furnace then place the screen on the top of the furnace, then set the controller at the desired temperature, and open the electrical transformer for power supply for heating until the desired time. Then close the power supply, measure the bed height quickly, and remove pyrolysed rice hulls through the bottom of the furnace to a tub. Close the tub and weigh the rice hull char as soon as cooled. This procedure was repeated by changing the temperature, the percent moisture content and weight of rice hull. During test periods of 60 min, the various temperatures were recorded as a function of time.

### 3.1.4 Experimental Variables and Conditions

The experimental variables and conditions were

as follows :

temperature, °C	= 350, 400, 450, 500
moisture content, %	= 0.11, 8.49, 16.35, 24.04
bulk density, kg/m <sup>3</sup>	= 113.5, 120.9, 130.2, 141.4
and time, min	= 10, 20, 30, 40, 50, 60

### 3.2 Heat Capacity Measurement

The basis for the method of determining the heat capacity of rice hull was that of a modified calorimetric heat capacity determination. A simplified method of measurement was used. In the method of measurement a know amount of sample at known temperature and moisture content was placed in a thermos bottle and mixed with a know amount of water at known temperature and then the mixture was allowed to attain an equilibrium temperature. The energy balance describing this experiment is as follows (Kazarian and Hall, 1965) :

$$C_{pw} W (\Delta T_c - \theta R_c) = C_{pw} W (\Delta T_w + \theta R_c) - H_c (\Delta T_f - \theta R_c) \quad (3.1)$$

The heat capacity constant,  $H_c$ , was determined by the following equation :

$$H_c = \frac{C_{pw} W \Delta T_c - C_{pw} W \Delta T_w}{\Delta T_f} \quad (3.2)$$

### 3.2.1 Experimental Apparatus

The heat capacity calorimeter was an ordinary flask with space in between the bottle and the tin container. The plastic cap of the thermos bottle was replaced by a piece of cork. A small hole was bored to pass thermocouple wires for measuring the temperature of the sample inside. The leads of the thermocouple were directly connected to the thermocouple temperature indicator. In Figure 3.3 details of the apparatus are shown.

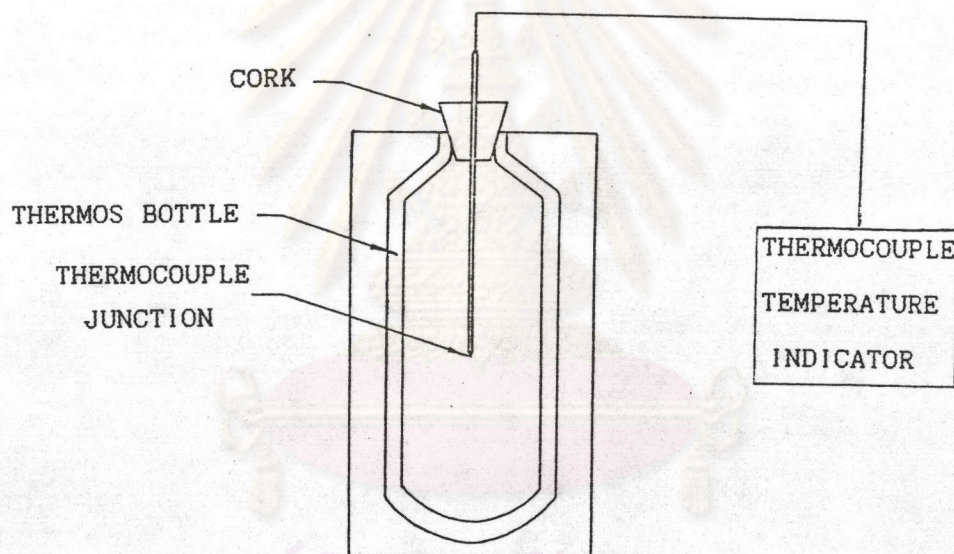


Figure 3.3 Heat Capacity Calorimeter System

### 3.2.2 Correction for Heat Losses

A heat loss curve was constructed to take care of conduction heat losses during the experiment. Since in most cases the temperature of the mixture inside the calorimeter was well above room temperature, therefore there was always heat loss

from the apparatus. The rate of temperature drop in degrees per minute was determined at various temperature difference levels close to those that were supposed to be encountered throughout the experiment. Water at elevated temperature was taken in the calorimeter flask and temperature drop was noted against time. The resulting curve is presented in Appendix C. Later, heat loss correction values were read directly from this curve for the computation of heat capacity.

### 3.2.3 Calibration

The calorimeter was calibrated by determining its heat capacity constant  $H_c$ . Heat capacity constant defines the amount of heat stored by the flask and attached auxiliary parts. The method for determining the heat capacity constant was the same as that for heat capacity except that water was used instead of the sample. Approximately 250 g of hot water at  $33^\circ\text{C}$  was poured in the calorimeter flask and allowed to reach equilibrium. Also approximately 300 g of cold water at  $17^\circ\text{C}$  was poured in another calorimeter flask and allowed to reach equilibrium.

Then cold water in one calorimeter flask was poured into the calorimeter flask containing hot water. Agitation was continued by shaking the calorimeter until the temperature equilibrium was reached. This procedure was repeated by changing the temperature of hot water and cold water. Then from equation (3.2) the heat capacity constant was computed. Since the equilibrium temperature was near room temperature hence no heat

gain or loss correction was necessary.

#### 3.2.4 Preparation of Samples and Procedure

The samples used for heat capacity determination were prepared at different moisture contents by means of drying and adding water. For temperature treatments an oven was used and set at desired temperatures (35, 45, 50 and 60 °C) before the samples were put in. For each experiment, about 53 g of sample was placed in small plastic bag. Sample weight was taken by means of an electrical balance up to a precision of 0.01 g.

The plastic bag containing the samples was put inside the oven and kept for 1 hour to allow for equilibrium to be reached with the pre-set oven temperature. The sample temperature was measured by a thermometer placed in it after that the sample was transferred to the calorimeter flask. When the sample reached equilibrium temperature, the test was done by pouring cold water into the sample calorimeter flask.

About 45 g of water cooled to a temperature of 6-17 °C was used in each test. Since the equilibrium temperature was approximately room temperature, the heat gain or loss term was negligible. Usually, 1.5 to 2 minutes were required to attain equilibrium. The equilibrium temperature was measured using the thermocouple placed inside the calorimeter (see Figure 3.3). Calculations were done using the heat capacity equation (3.1).