# Chapter 4
# Equipment and Experimental Method on Classification of Ocimum Seed Powder

## 4.1 Equipment and Experimental Conditions

According to the standard and specification of ocimum bulk laxative drug, it should contain only the seed particles of 125-840 μm or 20-120 mesh size. The in-process product of ground ocimum seed powder, however, composes of particles with size range 0-840 μm, particles which are smaller than 125 μm should therefore be all removed in the step of classification. In addition, the aim of this experiment was also conducted to investigate the characteristics of elutriation and classification of ocimum seed powder.

Based on the knowledge of elutriation, superficial gas velocity and freeboard height are considered to be the most essential factors. Thus construction of equipment and experimental criteria were set up to evaluate these effects.

### 4.1.1 Range of superficial gas velocity

In general, the operating superficial gas velocity should fall between minimum fluidized velocity and terminal velocity of finest particles in the system. This concept is generally applied to minimize elutriation of fine (11). In this experiment, however, the study was designed to investigate the elutriation phenomena. Since elutriation will start to occur when gas velocity exceeds the particle terminal velocity as shown in Table 4.1, removal of particles smaller than 136.5 μm class size should be operated at gas velocity lower than 0.75 m/s. It was found in preliminary study that this system could readily be operated only at the gas velocity of 1.0-1.5 m/s. This may be due to the effects of particle shape and/or

Table 4.1 Terminal velocity of each particles cut size, calculated from equation 2.18-2.20

| mesh class | mean diameter (μm) | terminal velocity (m/s) |
|---|---|---|
| 20-40 | 601.04 | 3.2916 |
| 40-60 | 325.96 | 1.7851 |
| 60-80 | 212.13 | 1.1617 |
| 80-100 | 163.77 | 0.8969 |
| 100-120 | 136.47 | 0.7474 |
| 120-140 | 114.45 | 0.6274 |
| 140-200 | 88.15 | 0.4827 |
| 200-325 | 57.54 | 0.3146 |
| 325- | 22.00 | 0.0199 |

surface characteristics, believed to be involved in the process and have never been systematically investigated (5). Moreover, as generally accepted that the predicted value in area of fluidization may deviate up to 50 %. As a result, gas velocity of 1.0-1.3 m/s (Table 4.2) was selected for this experiment, because when operation was carried out off his limit, almost all of particles were elutriated from the system.

#### 4.1.2 Range of freeboard height

Height of bed used in this investigation were based on TDH that could be estimated from equation 2.25 as proposed by Wen and Chen;

$$\begin{aligned} TDH &= \left(\frac{1}{a}\right) \ln \left\{\frac{F_o - F_c}{0.01Fe}\right\} \\ &= \left(\frac{1}{5}\right) \ln \left\{\frac{(47.553 - 2.3826)}{(0.01)\ (2.3826)}\right\} \\ &= 1.5 \quad m \end{aligned}$$

Value of $F_o$ was determined from equation 2.9 and $F_c$ was the sum of elutriation rate of all particle classes ($F_{ci}$). Details of example calculation and the predicted values of $F_o$ and $F_{ci}$ used for this estimation are shown in Appendix C and Appendix B2 respectively. As suggested by Wen and Chen, the constant a varies from 3.5-6.4 $m^{-1}$, thus TDH of this system may be in the interval of 1.1-2.1 m. The heights of bed used in this experiment are shown in Table 4.3. Heights of 0.515 and 0.757 m were also performed in this study to investigate the elutriation rate under TDH.

#### 4.1.3 Construction of Fluidization Unit

Figure 4.1 is the picture of equipment used in this experiment. A 2 hp variable speed motor was used as generating blower which could supply system superficial gas velocity of 0.5-2 m/s or 0.02-0.10 $m^3/s$, the range obtained from

Table 4.2 Average superficial gas velocity, calculated from column center velocity

| | column center gas velocity (m/s) | correction factor | average gas velocity (m/s) |
|---|---|---|---|
| U3 | 1.4 | 0.963 | 1.304 |
| U4 | 1.3 | 0.949 | 1.234 |
| U5 | 1.2 | 0.932 | 1.155 |

Table 4.3 Height of column used in this experiment

| | column height (m) |
|---|---|
| H1 | 0.515 |
| H2 | 0.757 |
| H3 | 1.040 |
| H4 | 1.271 |
| H5 | 1.543 |

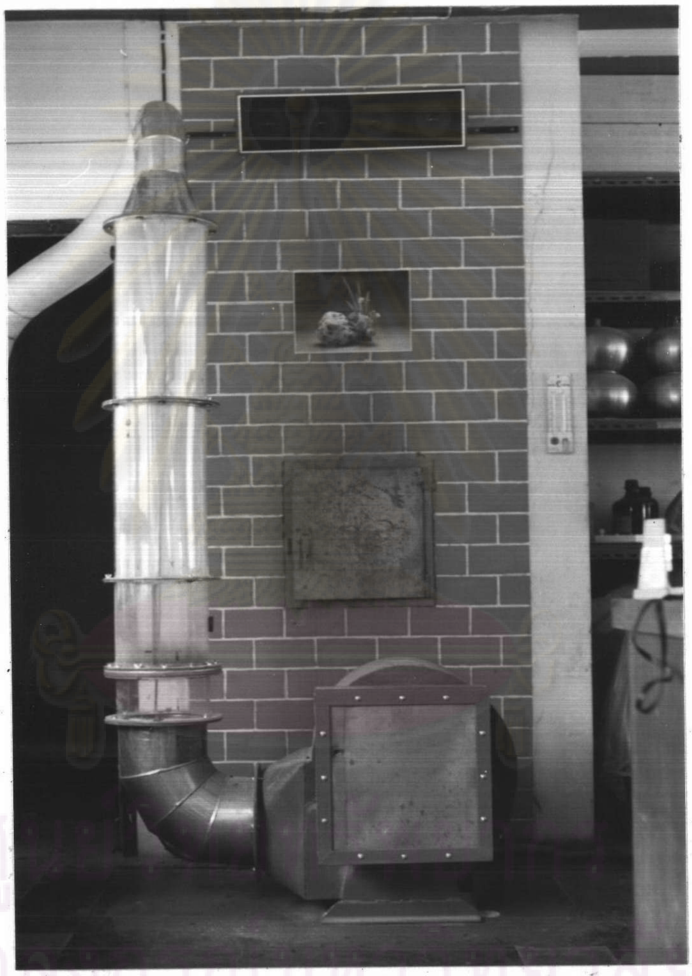


Figure 4.1 Photograph of fluidization unit

preliminary study. Inlet air was passed through the fabric filter of 20 μm in opening to prevent system from contamination of environmental fine particles.

Distributor was the perforated type since it was one which frequently used in previous experiments (12). Because the system powder composed of high content of fine particles, a number 120 wire mesh screen was covered on distributor to prevent loss of fine since it was proved to be sufficient.

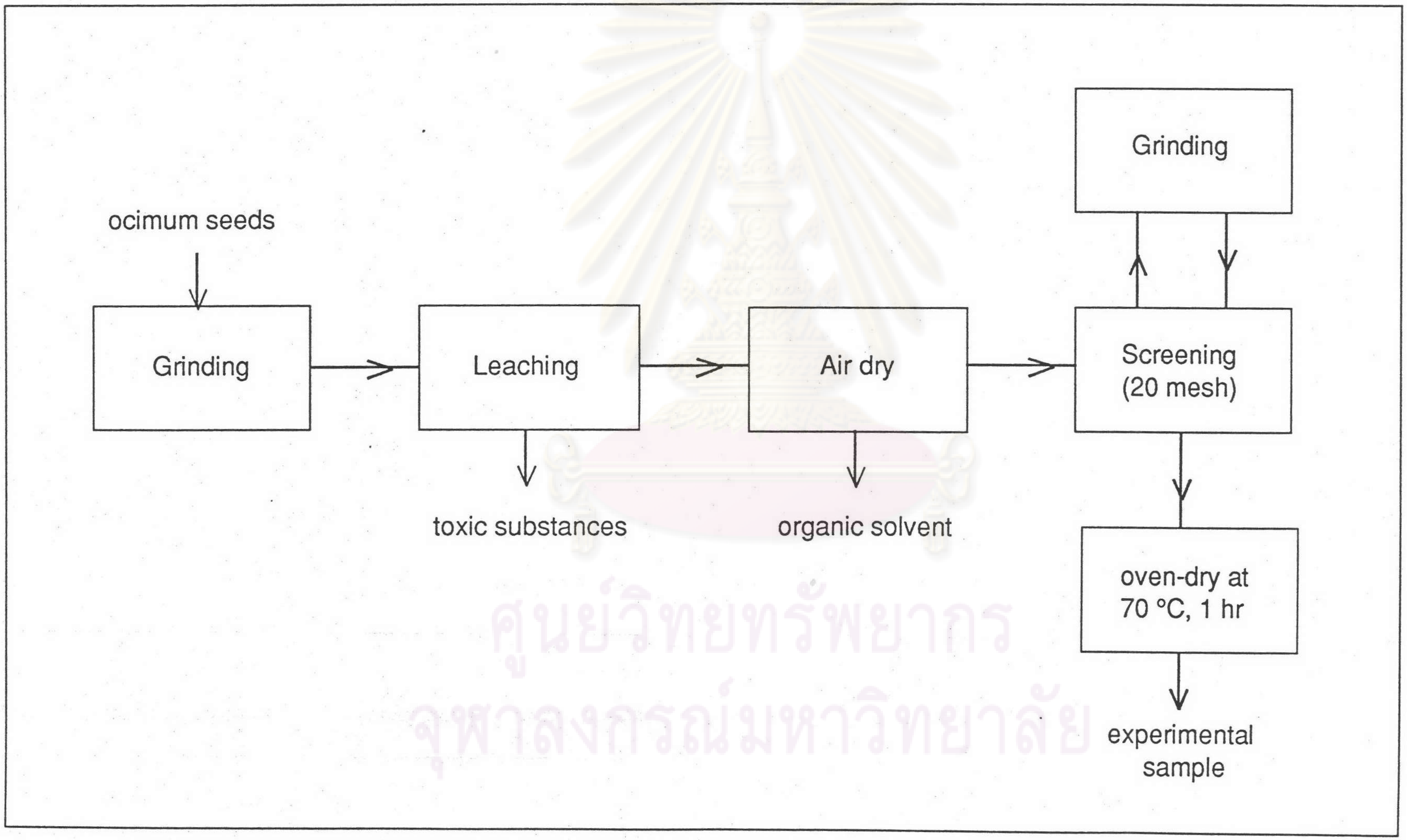
Material used for column body was transparent PVC type plastic because direct visual observation could be done. According to Lewis *et al* (31) entrainment rate becomes independent of vessel diameter when it is greater than 0.1 m. In addition, several reports on this area were performed in bed of diameter ranging from 0.05-0.6 m (11,12). Hence, in this experiment the diameter of column body of 10" or 0.254 m was chosen. Column body was composed of separable pieces of either 0.25 or 0.5 m in height and equal diameter. This would allow flexible arrangement in assembling the column body to any desire height. Outlet opening was fitted with a tube of 6" or 0.15 m in diameter to allow elutriated powder freely leaving from system. A small hole was bored on the column wall in which the anemometer probe was inserted to measure system gas velocity.

## 4.2 Experimental Method

### 4.2.1. Sample preparation

Figure 4.2 is the flow diagram of the ocimum seed powder preparation. The seeds were ground, toxic substances and oil content in seed core were extracted with organic solvent and air-dried for a period of time to eliminates trace of solvent. The dry powder was ground again and passed through sieve number 20. The capacity of this preparation was 3 kgs per day. About 100 kgs of sample

Figure 4.2 Flow diagram showing method of ocimum seed powder preparation



were collected and then mixed together in a rotating cube mixer. Each particle cut size content was analyzed as present in Tables 3.4 and 3.5.

### 4.2.2 Measurement of superficial gas velocity

To measure experimental superficial gas velocity, anemometer probe was inserted into the empty column with its tip at the center of column. The experiment was started and when the indicator become stable, gas velocity was then recorded. The average velocity across the bed column could be calculated by multiplying the column center velocity with correction factor. Following the recommended procedure in the "Operating Manual of Anemometer" (DATAMETRIC ™, see Appendix A), correction factor was determined and calculated.

### 4.2.3 Experimental procedure

The bottom piece of column was connected to the distributor, Figure 4.3, to be the bed container of powder for each run. The amount of ocimum seed powder elutriated from fluidized bed was detected from different weight of the powder in the column before and after run provided the bed container being tared. Ground ocimum seed powder used throughout this study was oven-dried at 70 °C for 1 hour before used in fluidization. The experimental procedure was carried out as follows;

a) perforated plate and the covered wire mesh screen were cleaned up to ensure that there was no blockage of fine particles on openings

b) having assembled the fluidized unit to the desired column height, the gas velocity in empty column was then measured at center of the column and adjusted to the test velocity

c) the lower part of empty column, to be used as bed container, was taken apart from the unit and weighed to tare the balance

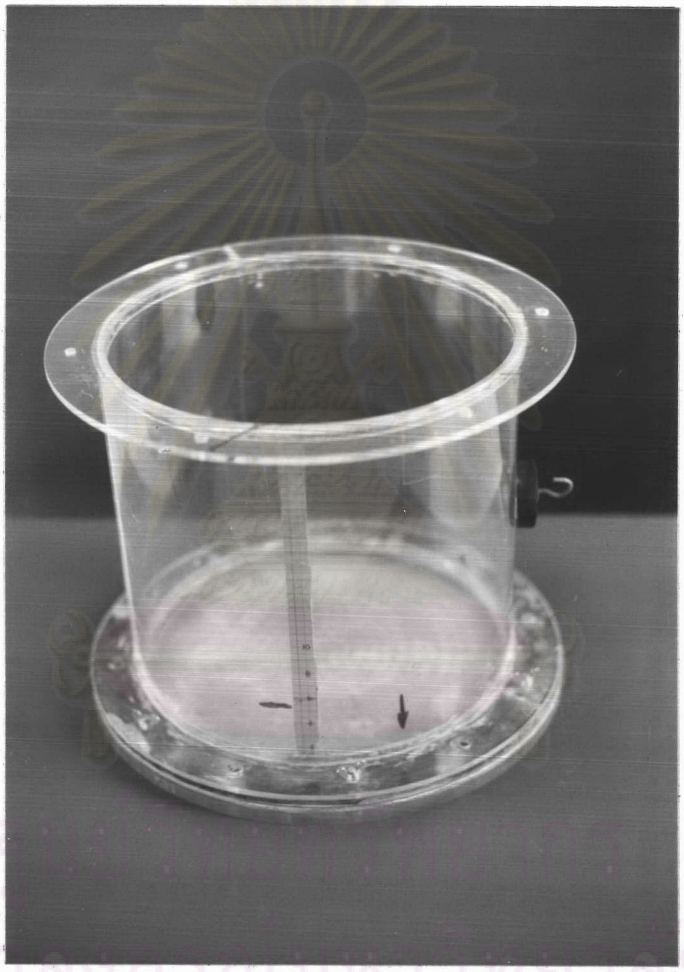


Figure 4.3 Bed container, assembled of distributor and bottom piece of bed column

d) ocimum seed powder, which had been freshly oven-dried and shaken as stated under the method of analyzing bulk density, was gently poured into the container until 0.5 kg additional weight was achieved

e) with ocimum seed powder in it, the bed container was reassembled to the fluidization equipment and the system was run for a specified time

f) bed container with the remaining powder was removed from equipment and weighed, about 100-200 g sample of the remaining powder was then collected for further particles size analysis as in chapter 3.

Essential parameters, i.e., gas velocity, column height, and time were strictly and precisely monitored throughout each run using above procedure, especially cleansing of the wire mesh screen. Height of dense phase was directly measured from operating bed.

Experimental data were recorded in the **input form**, see detail in Appendix B1, then typed and stored in microcomputer for further treatment.

## 4.3 Method of Data Assessment

### 4.3.1 Prediction of elutriation rate from proposed model

A computer program was developed under microcomputer for assessment and evaluating experimental data. This software consists of three parts;

- experimental data management
- particle size analysis, and
- calculation of entrainment and elutriation rate following Wen and Chen's model.

The program flowcharts and listings are shown in Figure 4.4 and Appendix C. Program for data management, named DATA.prg, and particle size analysis, named PCLEANAL.prg, were developed under dBASEIII (32), because of its

Figure 4.4a  Flowchart of DATA.prg

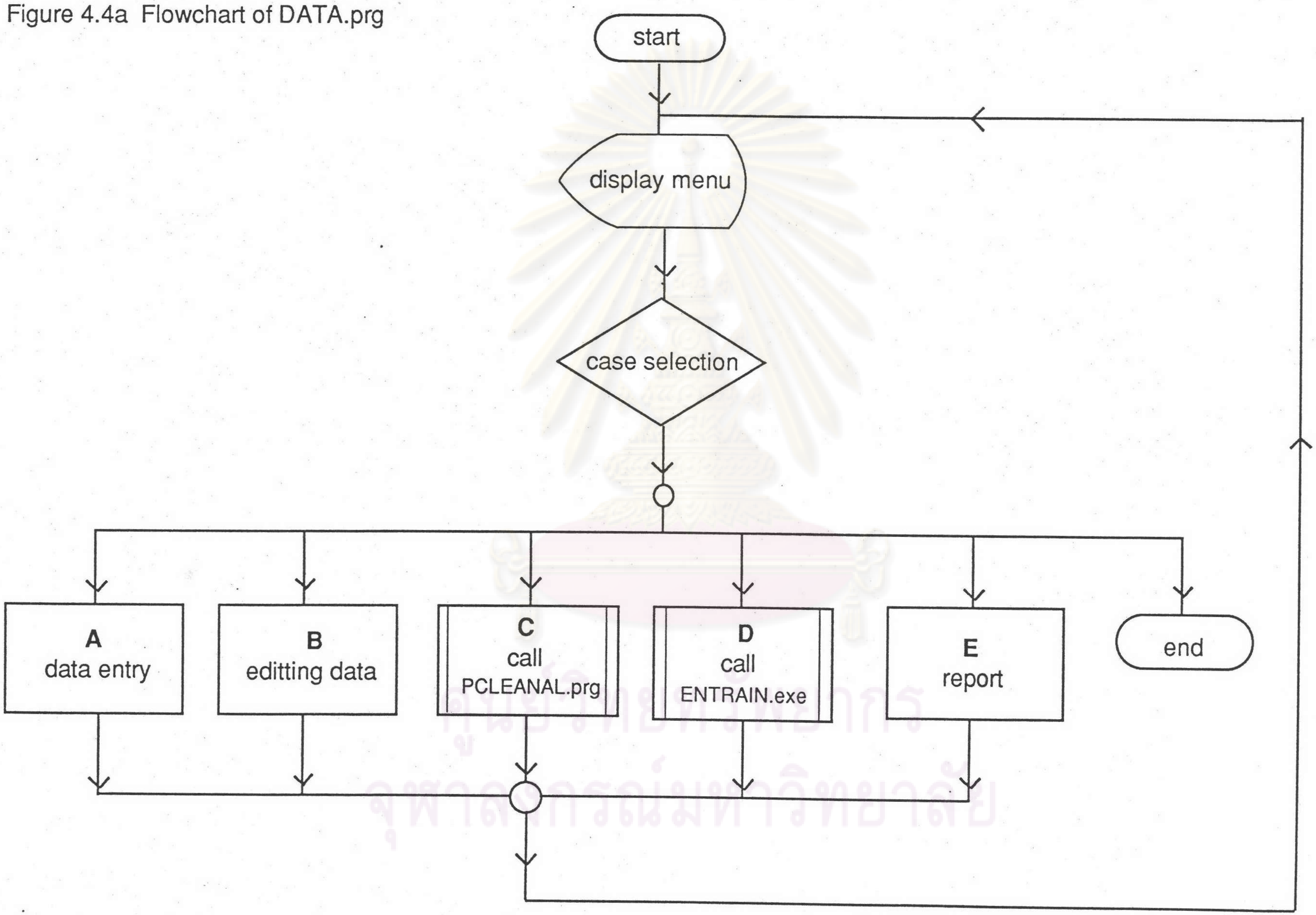


Figure 4.4b Flowchart of PCLEANAL.prg

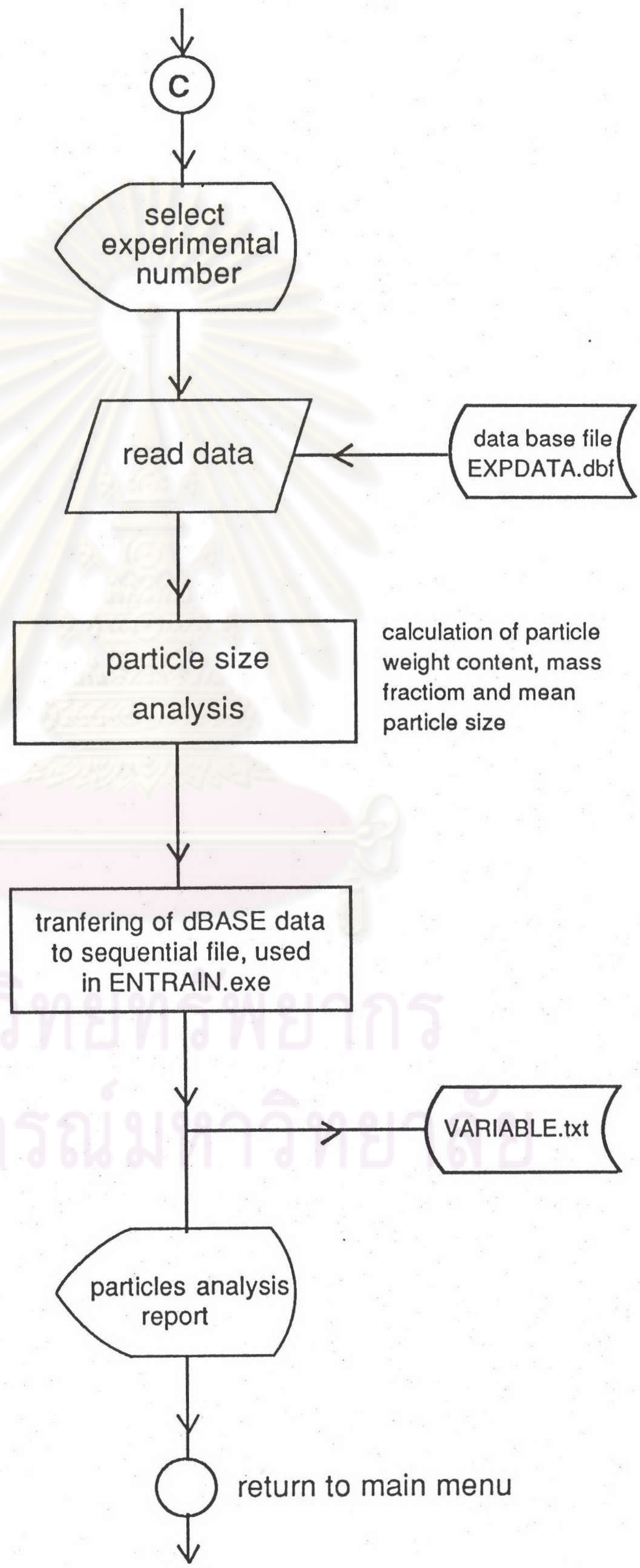


Figure 4.4c Flowchart of ENTRAIN.exe(bas)

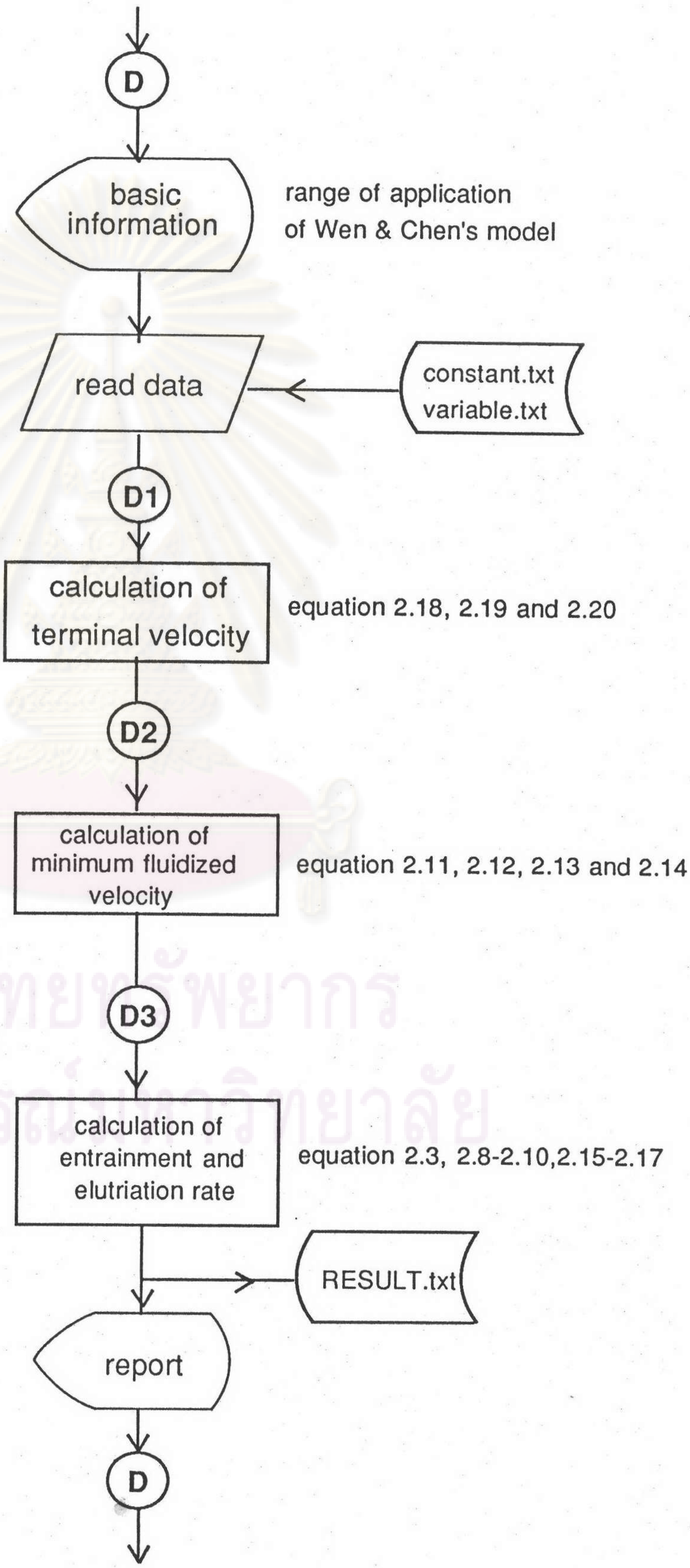


Figure 4.4c (continue)

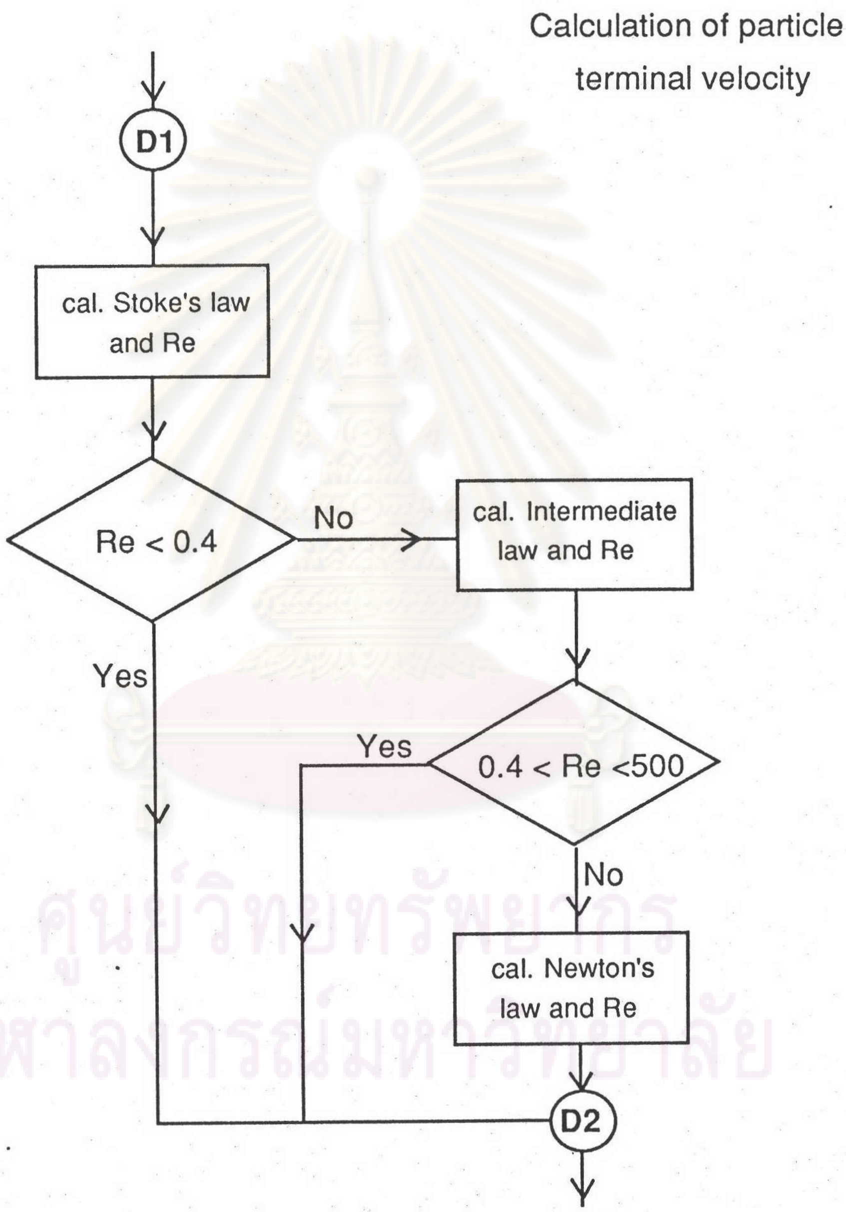
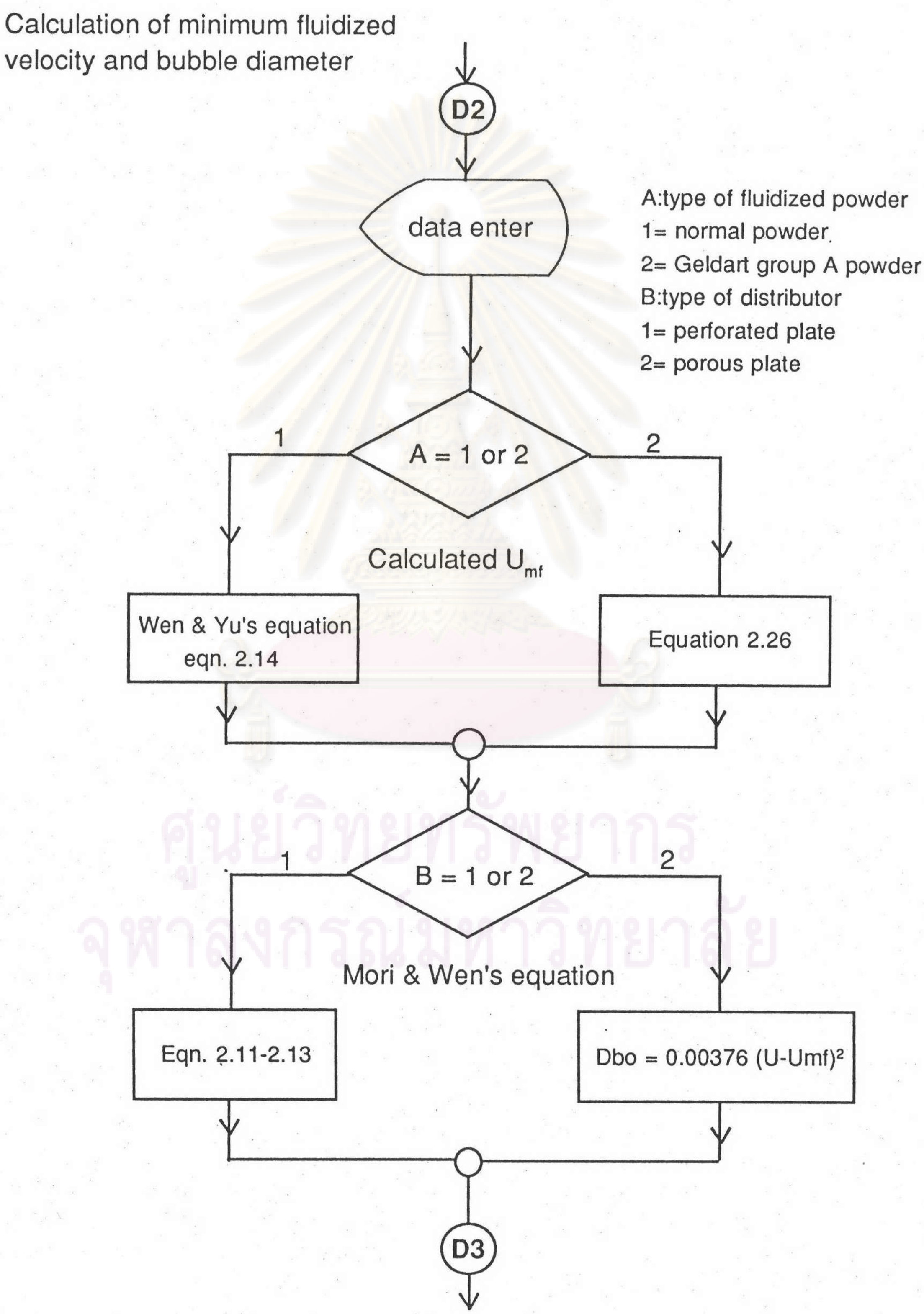


Figure 4.4c (continue)



powerful data managing capacity. According to Wen and Chen's model, calculation of entrainment and elutriation rate require high precision and may consume much computer time so that the program, ENTRAIN.exe, was written under Borland's Turbo Basic owing to its appropriate nature and capacity for this purpose (33,34).

#### *4.3.1.1 Program for data management (DATA.prg)*

This program is the main procedure, including experimental data entry, editing, storage and transferring information between different types of software used in analysis of data. PCLEANAL.prg and ENTRAIN.exe are called by this program via main menu. After experimental data was typed in, the editing screen on monitor displayed the same as **input form**, and after being analyzed, report was then printed out or shown on monitor via **report form**, as seen in Appendix B.

#### *4.3.1.2 Program for particle size analysis (PCLEANL.peg)*

Having edited the experimental data, this part of software will be called by DATA.prg for particle analysis according to the method stated in chapter 3. The results are then collected and transferred to sequential file, VARIABLE.txt, which is able to be performed by ENTRAIN.exe.

#### *4.3.1.3 Program for calculating Wen and Chen's model (ENTRAIN.exe)*

The function of this part of software is to predict the entrainment and elutriation rate of particles following Wen and Chen's model, equation 2.8-2.22. After calculation, data was then transferred to dBASE fashion and returned to the main menu. Example calculation following the instruction in this program is shown in Appendix D.

### 4.3.2. Determination of elutriation rate expression from experiment

Raw data from experiment , weight fraction ($W_i/W_{io}$) or mass fraction ($X_i/X_{io}$) versus time, will be plotted in log-normal scale and anticipated that it would be first order relation. Trying to correlate rate equation, Least square curve fitting method or regression analysis was employed to determine constants of equation.

In the first place, linear regression of transform first order rate correlation was performed, by applying logarithm to the result from integrating first order rate equation,

$$\ln\left(\frac{X_i}{X_{io}}\right) = -kt \qquad (4.1)$$

or

$$Y = kt \qquad (4.2)$$

This linearized form may be evaluated and plotted by a function key in Lotus software. However plotting of raw data revealed that correlation of ($W_i/W_{io}$) or ($X_i/X_{io}$) versus time seems apparently to be in the form of

$$Y = A + Be^{kt} \qquad (4.3)$$

To confirm whether this equation was sufficiently and satisfactorily described the rate characteristics, a non linear curve fitting equations were developed (35,36). From equation 4.3 one may write

$$Y = A + BZ^t \qquad (4.4)$$

Let z be an approximate value of Z, by Taylor's theorem;

$$\hat{Y} = a + bz^t = a + b(z^t) + b(Z-z)(tz^{t-1}) + \ldots\ldots\ldots \qquad (4.5)$$

where $\hat{Y}$ is the prediction value and if $c = b(Z-z)$, $X_0 = 1$, $X_1 = z^t$ and $X_2 = tz^{t-1}$, then

$$\hat{Y} = aX_0 + bX_1 + cX_2 \qquad (4.6)$$

$$a = \bar{Y} - b\bar{X}_1 - c\bar{X}_2 \qquad (4.7)$$

here $\bar{Y}$, $\bar{X}_1$ and $\bar{X}_2$ are the average value of Y, $X_i$ and $X_{io}$. Replace equation 4.6 with 4.7 gives

$$\hat{Y} = \bar{Y} - b\bar{X}_1 - c\bar{X}_2 + bX_1 + cX_2 \tag{4.8}$$

then

$$(Y_i - \hat{Y}_i) = (Y_i - \bar{Y}) + b(X_{1i} - \bar{X}_1) + c(X_{2i} - \bar{X}_2) \tag{4.9}$$

where $(Y_i - \hat{Y}_i)$ or $\delta$ is the deviation of experimental data point from prediction value. Let $y = Y_i - Y$, $x_1 = X_{1i} - X_1$ and $x_2 = X_{2i} - X_2$ ,then

$$\delta = y + bx_1 + cx_2 \tag{4.10}$$

Estimation of constants from least square method are obtained by choosing the value which minimize the sum of squares of these deviations;

$$S = \sum_{i=0}^{n} \delta^2 \tag{4.11}$$

By partial differentiated of S with respected to a and b, and setting the derivatives, $\frac{\partial S}{\partial a}$ and $\frac{\partial S}{\partial b}$, equal to zero, then solving the resulting equations, one could obtain the value of constants from;

$$b = \frac{(\Sigma x_2^2)(\Sigma x_1 y) - (\Sigma x_1 x_2)(\Sigma x_2 y)}{D} \tag{4.12}$$

$$c = \frac{(\Sigma x_1^2)(\Sigma x_2 y) - (\Sigma x_1 x_2)(\Sigma x_1 y)}{D} \tag{4.13}$$

$$D = (\Sigma x_1^2)(\Sigma x_2^2) - (\Sigma x_1 x_2)$$

The value of a is calculated from equation 4.7 and the coefficient of determination ($r^2$) is determined from;

$$r^2 = \frac{\sum_{i=1}^{n}(\hat{y}_i - \bar{y})^2}{\sum_{i=1}^{n}(y_i - \bar{y})^2} \tag{4.14}$$

Its value lies between 0 to 1 and the closer to unity the value is, the more degree of exists between the experimental values and the predicting equation.

Analytical procedure, using trial and error method, is as followed;

1. approximate the value of z, the suggested first approximation for 6 data points, may obtain from

$$z_1 = \frac{(4y_5+4y_4+2y_3-3y_2-7y_1)}{(4y_4+4y_3+2y_2-3y_1-7y_0)} \qquad (4.15)$$

2. determined value of a, b and c from equation 4.7, 4.12 and 4.13

3. observe the result of $r^2$ which obtained from equation 4.14

4. then make the second approximation from

$$z_2 = z_1 + \frac{c}{b} \qquad (4.16)$$

5. repeat, until the maximum value of $r^2$ is reached

By iterating approximation of z in equation 4.16, value of c will decrease and finally approach zero (Appendix E). This procedure was performed in Lotus™ worksheet by transform experimental data from dBASEIII.