EXPERIMENT 6
BINARY DISTILLATION

I. INTRODUCTION

Distillation is a separation method based on the difference in the volatility of the components in solution. It is one of the most important methods in chemical industry.

Distillation takes place in packed column or tray column. During the process, vapor moves upward in contact with liquid move downward. Since vapor is hotter than liquid, heat is transferred from vapor to liquid on trays (tray column) or on the surface of packing (packed column). Heat transfer leads to partial vaporization of liquid (mostly high volatile component) and partial condensation of vapor (mostly low volatile component). The vaporization and condensation repeat along the column making low volatile (heavy) component concentrate at the bottom and high volatile (light) component concentrate at the top of the column.

The distillation process may run continuously or in the batch form. Hence, distillation system is categorized into continuous and batch ones.

The efficiency of a (segment of) tray column is defined as the ratio between the number of theoretical plates (stages) and (real) plates: $\eta = \frac{N_{lt}}{N_{tt}}$

1.1. Theoretical plates

A theoretical plate is an ideal segment of the column where the vapor flow out/in is in equilibrium with the liquid flow in/out (Fig. 1).

1.2. McCabe – Thiele method

Calculating the number of theoretical plates requires vapor – liquid equilibrium (VLE) curve – which is a property of the solution, unrelated to the distillation process – and the operating lines of striping and rectifying sections.

Assumptions made during formulation of operating lines (Fig. 2):

- No separation occurs in the condenser (total condensing): $y_p = x_p$;
- No separation occurs in the reboiler: $y_w = x_w$;

- Molar flowrates of vapor and liquid flows do not change along each section, which means heats of vaporization of two components are equal so condensation of 1 mol heavy component will evaporate 1 mol light component: $r_A = r_B$.

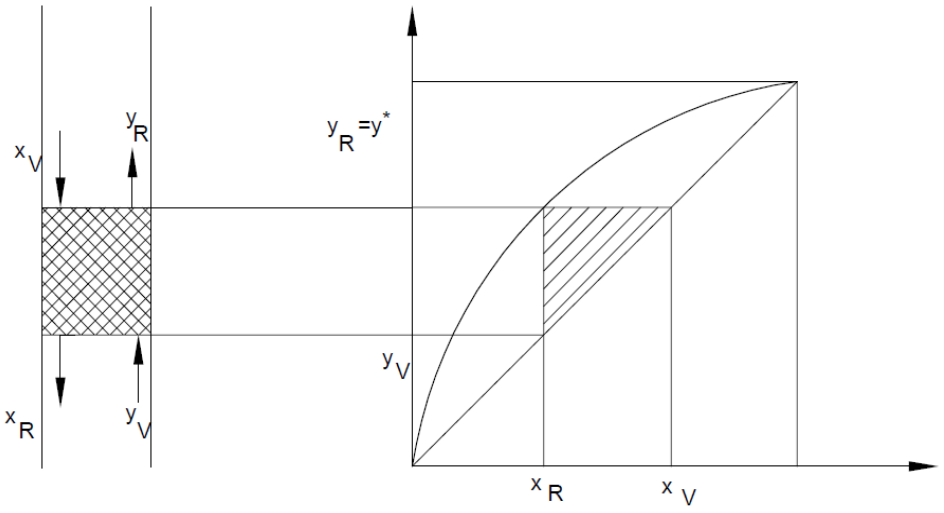

Figure 1: *Demonstration of theoretical plate on x-y coordinator*
x – molar fraction of light component in liquid
y – molar fraction of light component in vapor

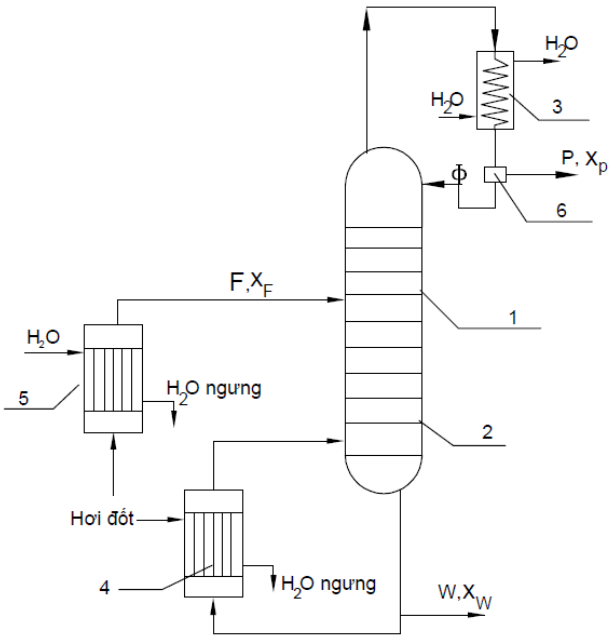

Figure 2. *Continuous binary distillation column*

1 – Rectifying section
2 – Stripping section
3 – Condenser
4 – Reboiler
5 – Preheater
6 – Reflux flowrate regulator

These assumptions make operating lines straight, their intersections with the angle bisector of the 1st quarter are points A and C on Fig. 3.

The operating line equation of rectifying section: $y_L = \frac{R}{R+1}x + \frac{x_p}{R+1}$

The operating line equation of stripping section: $y_C = \frac{R+F/P}{R+1}x + \frac{1-F/P}{R+1}x_w$

F – Feed flowrate, kmol/s P – Distillate flowrate, kmol/s

$R = \frac{\Phi}{P}$ – Reflux ratio x_P, x_W – Distillate and bottom molar fractions

When feed enters column in liquid state at boiling temperature, the operating lines intersect at point B(x_F, y_F) on Fig 3.

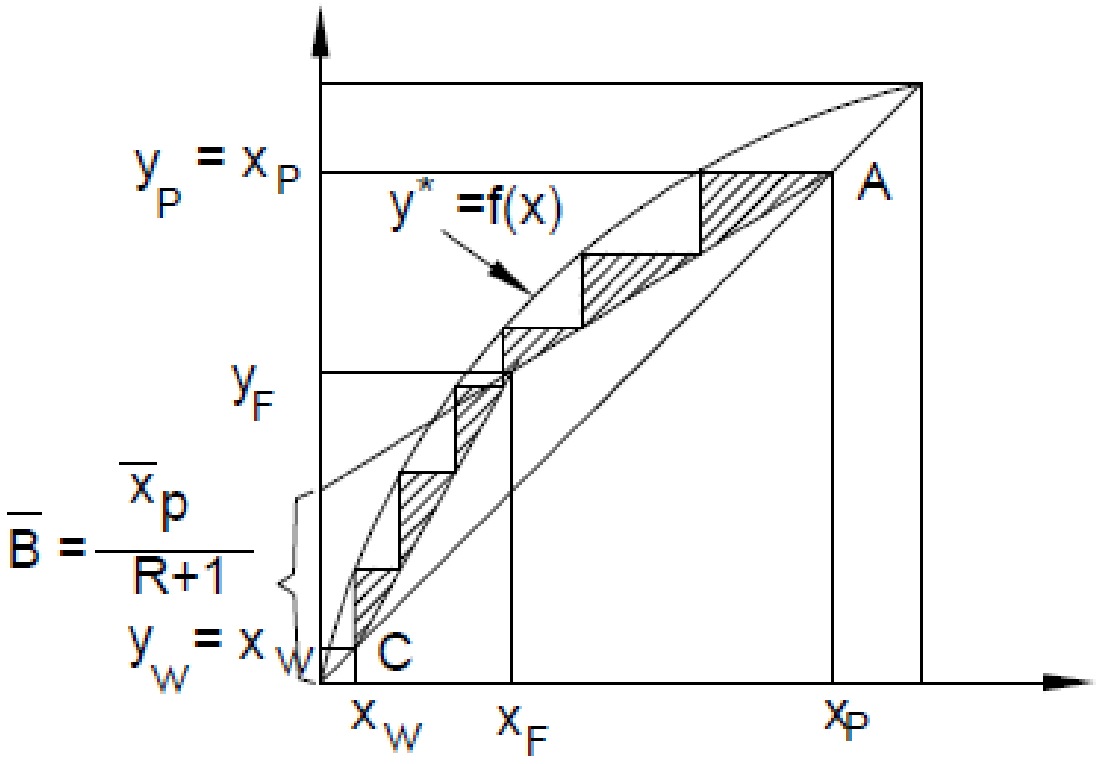

Figure 3. *Calculating number of theoretical plates by Mc Cabe–Thiele method*
AB – operating line of rectifying section
BC – operating line of stripping section

Draw triangles between equilibrium curve and operating lines as illustrated on Fig. 3. The number of triangles is the number of theoretical plates.

1.3. Calculating minimum number of theoretical plates by Mc Cabe-Thiele method

When reflux ratio R approach infinity, the column works at total reflux condition, no feed enters the column and no product leaves. Because $\lim_{R\to\infty}\frac{R}{R+1}=1$ the operating lines are on the angle bisector of the first quarter (Fig. 4) and the number of theoretical plates is minimum. The efficiency of the column in total reflux condition is: $\eta=\frac{N_{ltmin}}{N_{tt}}$

N_{ltmin} – the minimum number of theoretical plates

N_{tt} – the real number of theoretical plates

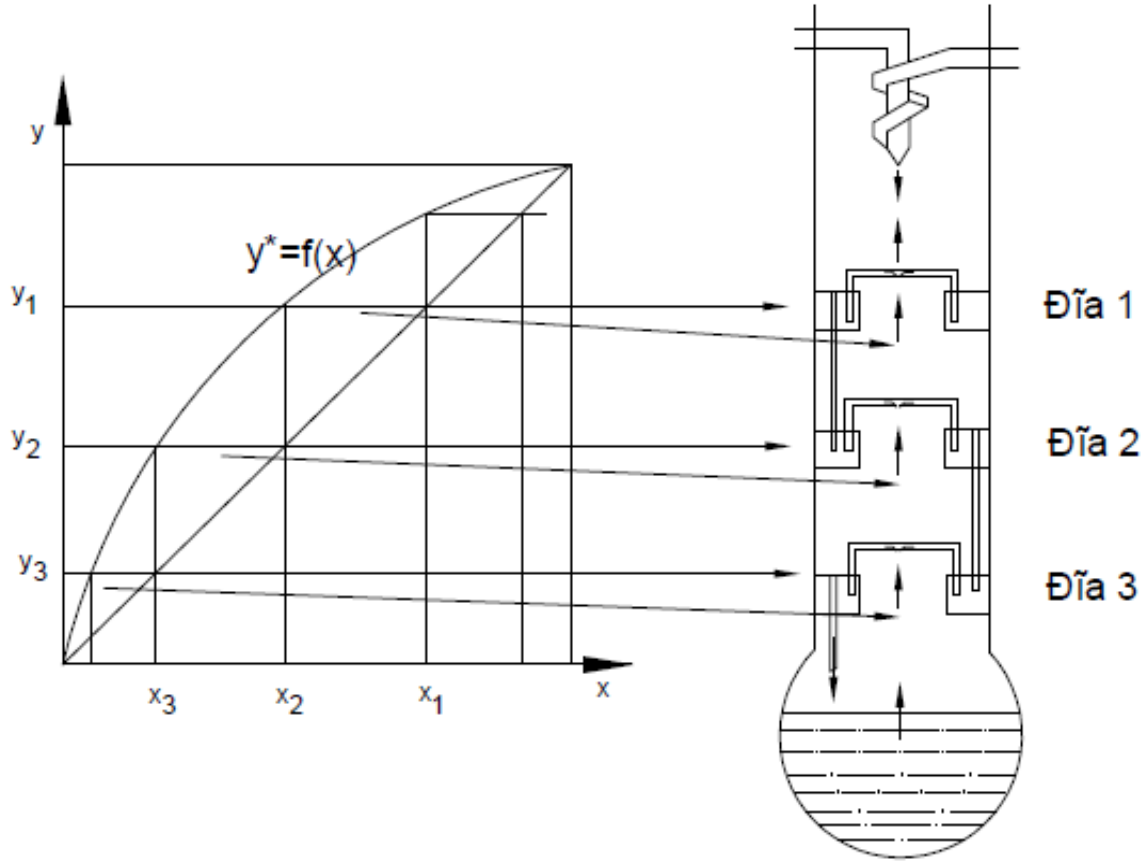

Figure 4. *Calculating minimum number of theoretical plates by Mc Cabe – Thiele method*

1.4. Calculating minimum number of theoretical plates by Fenske – Underwood equation

If the solution follows Raoult's law (ideal solution with constant relative volatility $\alpha=\frac{y^*/(1-y^*)}{x/(1-x)}\approx Const$), the minimum number of theoretical plates can be calculated by Fenske – Underwood equation.

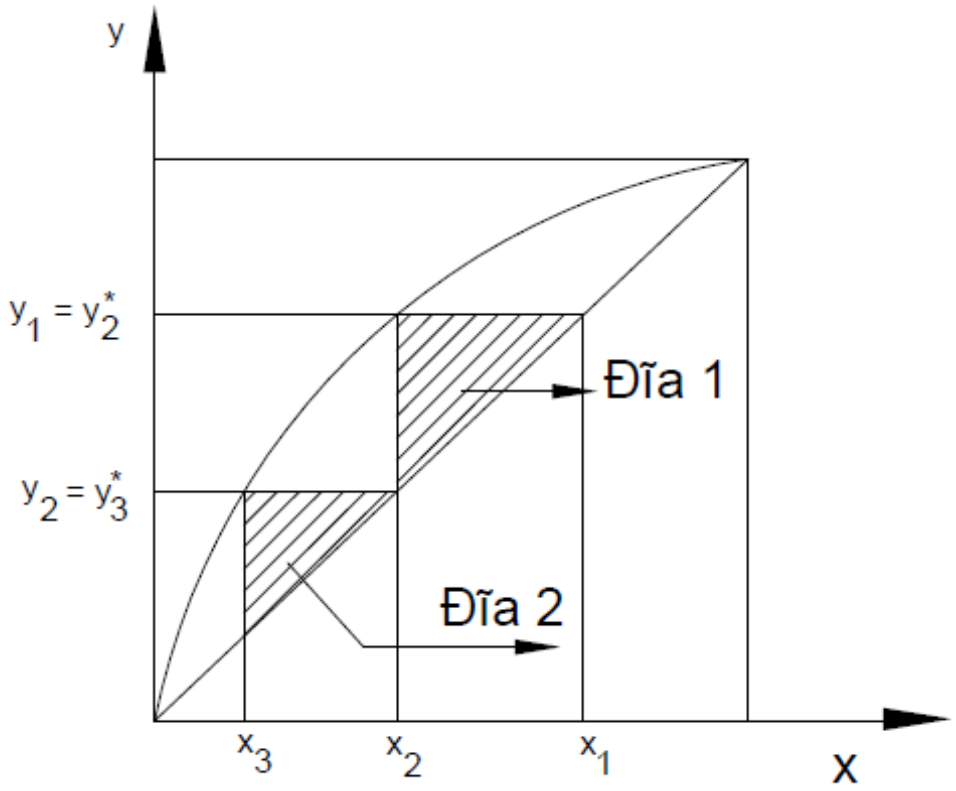

Figure 5. *Illustration of Fenske – Underwood equation derivation*

The relative volatility of the first stage (Fig. 5):

$$\frac{y_1}{1-y_1} = \frac{y_2^*}{1-y_2^*} = \alpha \frac{x_2}{1-x_2} \qquad \text{(a)}$$

When $R = \infty$ the operating line is on the angle bisector hence $y_1 = x_1$ (b)
Equation (c) is derived from equations (a) and (b):

$$\frac{x_1}{1-x_1} = \alpha \frac{x_2}{1-x_2} \qquad \text{(c)}$$

Similarly, equation for the second stage cab written:

$$\frac{y_2}{1-y_2} = \frac{y_3^*}{1-y_3^*} = \alpha \frac{x_3}{1-x_3} \qquad \text{(d)}$$

Since $y_2 = x_2$ equation (e) can be derived from equation (d):

$$\frac{x_2}{1-y_2} = \alpha \frac{x_3}{1-x_3} \qquad \text{(e)}$$

Combination of (c) and (e):

$$\frac{x_1}{1-x_1} = \alpha^2 \frac{x_3}{1-x_3} \qquad \text{(f)}$$

In general, when the number of theoretical stages is N_{LTMin}, the distillate fraction is the molar fraction of the first stage $x_1 = x_p$, the bottom fraction is the molar fraction of the last stage x_w, then:

$$\frac{x_P}{1-x_P} = \alpha^{N_{LTMin}} \frac{x_w}{1-x_w}$$

The Fenske – Underwood equation for calculating the number of theoretical plates:

$$N_{LTMin} = \frac{\ln \frac{x_p(1-x_w)}{(1-x_p)x_w}}{\ln \alpha}$$

The procedure for calculating the efficiency of tray column:

- Running column with a (nearly) ideal solution in total reflux condition;
- Measuring top and bottom molar fraction when the working condition is stable;
- Calculating relative volatility (if experiment data is N/A) using equation (the less difference between TA and TB, the more accuracy attained):

$$\log \alpha \approx 9 \frac{T_B - T_A}{T_B + T_A}$$

- Calculate the minimum number of theoretical plates using Fenske – Underwood equation;
- Calculating column efficiency in total reflux condition: $\eta = \frac{N_{ltmin}}{N_{tt}}$

II. EXPERIMENT

2.1. Purpose

1. Have knowledge of distillation system, its operating procedure and the structure of bubble-cap tray column.
2. Understand methods for conducting experiment, acquiring and processing data.
3. Calculating the number of theoretical plates and column efficiency in total reflux condition

2.2. Experiment setup diagram

(Students observe the setup and draw its diagram)

2.3. Illustration of one bubble-cap tray

(Students draw)

2.4. Illustration of flows of vapor and liquid through two consecutive trays with highlight on the contact of two phases.

(Students draw)

2.5. Water – methanol VLE at 760 mmHg

x (% mol)	0	5	10	20	30	40	50	60	70	80	90	100
y^* (% mol)	0	26,8	41,8	57,9	66,5	72,9	77,9	82,5	87	91,5	95,8	100
$t_{sôi}$, ^oC	100	92,3	87,7	81,7	78	75,3	73,1	71,2	69,3	67,6	66	64,5

2.6. Operating

The distillation system is monitored and operated via control board on the electrical cabinet (Fig. 6) and Human Machine Interface (HMI) on the computer.

On the HMI, which can be accessed via a web browser, user can monitor temperature along the column and reboiler power, start/stop recording these values and download the record as shown in Fig. 7.

***Figure 6**. Control board*

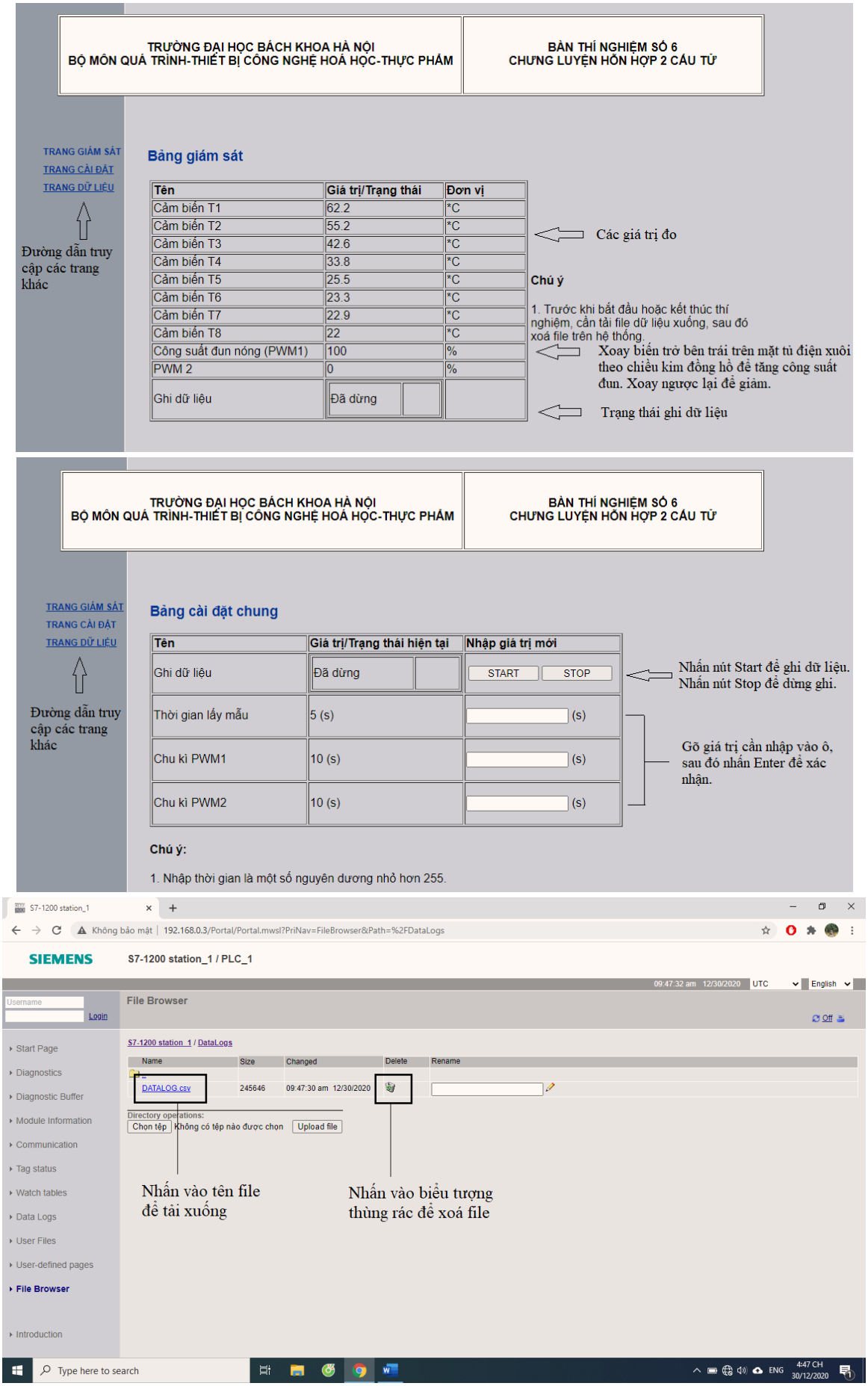

***Figure 7**. HMI on web browser*

EXPERIMENTAL RESULT

Liquid mixture: ……

Pressure p = ……

Molar fraction of mixture before experiment x_F = ...

Working condition R = ...

Time	Top temp., TT8	Tray 1 temp., TT7	Tray 3 temp., TT6	Tray 5 temp., TT5	Tray 7 temp., TT4	Tray 9 temp., TT3	Tray 11 temp., TT2	Bottom temp., TT1

Molar fraction on trays:

Tray	Top	Tray 1	Tray 3	Tray 5	Tray 7	Tray 9	Tray 11	Bottom
Temperature								
Molar fraction								

2.7. Experimental data processing

a. Estimate the number of theoretical plates by McCabe – Thiele method

b. Estimate the number of theoretical plates by Fenske – Underwood equation

c. Efficiency

$$\eta = \frac{N_{LTMin}}{N_{TT}}$$

2.8. Discussion

- Compare two method for estimating the number of theoretical plates (McCabe – Thiele and Fenske – Underwood)
- Comment on efficiency of bubble-cap tray column for distillation