EVALUATION OF THE NUMBER OF TRANSFER UNITS(NTU) AND THE COLUMN HEIGHT BY USING ON-LINE TEMPERATURE MEASUREMENTS FOR A PILOT SCALE PACKED BATCH DISTILLATION COLUMN

Adnan Aldemir, Suna Ertunç, Hale Hapoğlu, Mustafa Alpbaz

Ankara University, Faculty of Engineering, Department of Chemical Engineering, 06100 Ankara, Turkey

aldemir@eng.ankara.edu.tr, ertunc@eng.ankara.edu.tr, hapoglu@eng.ankara.edu.tr, alpbaz@eng.ankara.edu.tr

Abstract: There have been relatively few experimental attempts at evaluating the column height and comparing experimental results obtained from a pilot scale packed batch distillation column with theoretical ones.

In the present study, the pilot plant packed batch distillation column was used to distillate the binary methanol-water mixture, and to obtain on-line temperature values. The top temperature changes with time were observed at steady-state and dynamic conditions. The column was operated initially for approximately one hour at the total reflux. In this case, There were no feed and product flows. Temperature samples were taken on-line from the top and bottom of the column. Temperature profiles observed on the computer were recorded. Refractive index of the samples were determined. When the temperatures were constant, the system was at a steady-state condition for total reflux. After the system reached the steady-state condition the reflux ratio was adjusted to a certain level. From experimental top and bottom temperature values, two models were obtained between mol fraction and temperature. The mass transfer coefficient was calculated, and the final mol fraction of reboiler was determined. The column height was calculated and compared with the real packed height. It is noted that the equation used for mass transfer coefficient is considerable well.

Keywords: Batch distillation, packed column, packed height, mass transfer coefficient

INTRODUCTION

The investigation of distillation columns is mainly based on two different approaches: fundamental studies, for example concerning mass transfer behaviour of packed sections, etc. and alternatively, integral studies on columns provided with trays or packing (Elgue, Prat, Cabassud, Lann and Cezerac, 2004; Zuiderweg, 1999). There has been many articles to produce a certain amount of product at desired composition either in minimal production time or with maximal economic profit (Betlem, Krijnsen and Huijnen, 1998; Muddu, Narang and Patwardhan, 2010). The investigation of column performance can be identified by studying the liquid composition profile.

The batch distillation have been widely used in the separation processes because of its easy operation and maintenance. Noda, Kato, Chida, Hasebe and Hashimoto (2001) discussed the optimal structure and operation of a batch distillation column separating ternary mixture from the viewpoint of energy conservation, and derived the optimal reflux operation for three types of batch distillation columns; rectifying, stripping and total reflux columns. They noted that the energy consumption of the total reflux column is reduced when the optimal operation is used. Kim and Han (1999) showed that the dynamic model gives an accurate design for the operation of a batch distillation column. The variations of reflux ratio and top product composition are obtained.

Sadeghifar and Kadri (2011) developed a general and accurate method for efficiency calculation of the distillation columns packed with structured packings. They noted that for distillation columns with a large number of components (except for total reflux conditions), it is too difficult to obtain the realistic value of the experimental efficiency. An approximate estimation of the experimental efficiency is also sometimes impossible. Senol (2001) suggested that the increased amount of effectives area in randomly packed distillation column, as compared with the wetted one, is due to tendency toward rippling, wave and droplet formation in the falling liquid film. It is shown that the observed and predicted behaviors of the relative proportion of effective interfacial area are overly sensitive to the vapor and liquid loads, as well as to the packing properties.

Rejl et. al. (2006) measured volumetric mass transfer coefficients in liquid an vapour phases in distillation column by the method consisting of a fitting of the concentration profile of liquid phase along the column obtained by the integration of a differential model to the experimental one. It is noted that the concentration profiles obtained by the integration of the differential model of the distillation column using the coefficients from absorption correlation are differed from the experimental profiles. Liu, Yu, Yuan and Liu (2009) proposed a numerical method for modeling the distillation process in a randomly packed column. They showed that the predicted height equivalent of theoritical plate of the distillation column concerned is in satisfactory agreement with the reported experimental data.

In the present study, it is shown that mass transfer coefficient and the column height can be predicted much more simply using experimental top and bottom temperature data while being able to achieve the required separation in a pilot scale packed batch distillation column.

METHODS AND PROCEDURES

Distillation system consists of a glass flask (100 L), a heating mantle (2000 W), a packed 80 mm internal diameter column filled with 1 in Rasching rings, a valve adjusting reflux ratio at the top of the column and a condenser. System has two thermocouples to measure the temperature of top product and the boiler (See Figure 1).

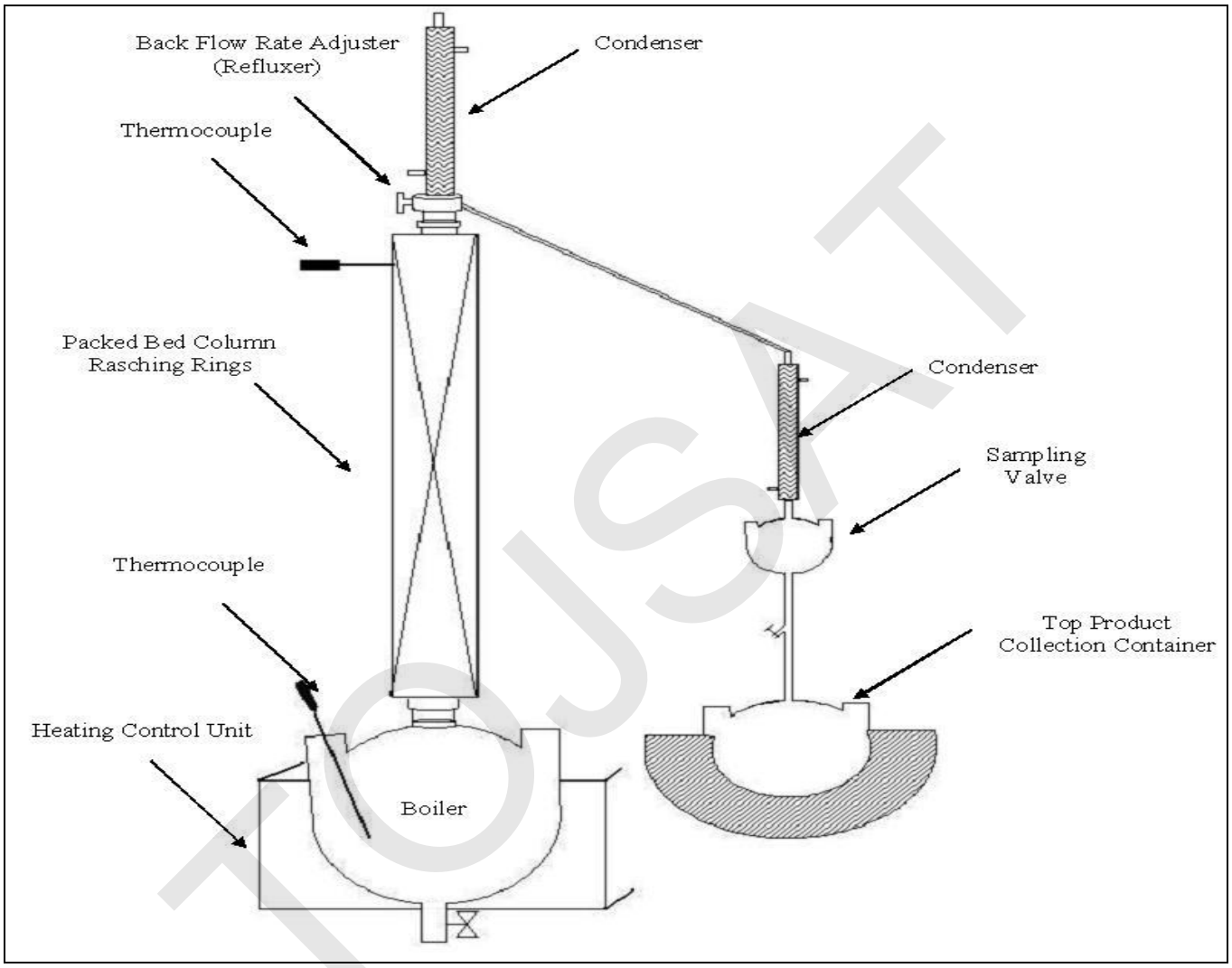


Figure 1. Experimental system

To operate the experimental system, certain volume and concentration of methanol-water mixture is filled into boiler. Cooling water inlet valve opening is provided for condenser. The button on the distillation column control panel that gives energy to the system is turned on. Thus, heating mantle begins to heat the mixture in the vessel. Batch distillation is operated at the total reflux until the steady state condition occurs. When the steady-state condition is reached in the column, reflux valve is adjusted to a desired value and the top product is obtained by batch distillation. Experimental data for the distillation column is given in Table 1.

Table 1 Experimental data for distillation column

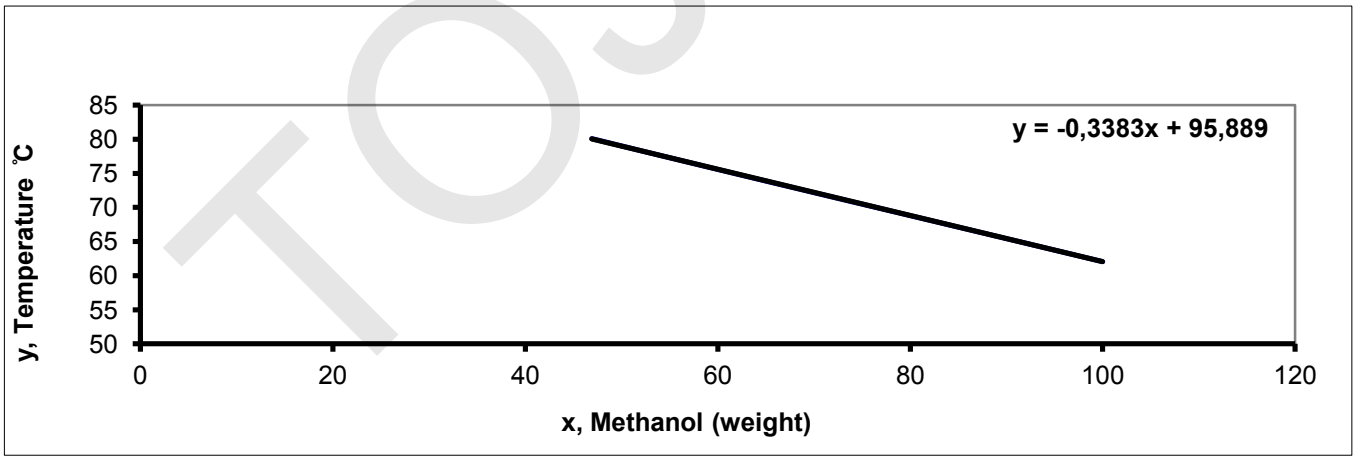
Initial reboiler amount (F)	55 L
Initial reboiler methanol mol fraction (Xs_1)	0.222
Reboiler temperature	81.7 °C
Top product flow rate (D)	0.14 mL/s
Total top product amount (d)	2.726 L
Vapour flow rate (V)	0.24 mL/s
Liquid flow rate (L)	0.10 mL/s
Average top product temperature	67.8 °C
Average top product density (ρ_{ort})	910.407 kg / m^3
Average top product molecular weight (M_w)	21.559 kg / kmol
Reflux ratio	0.682

RESULTS

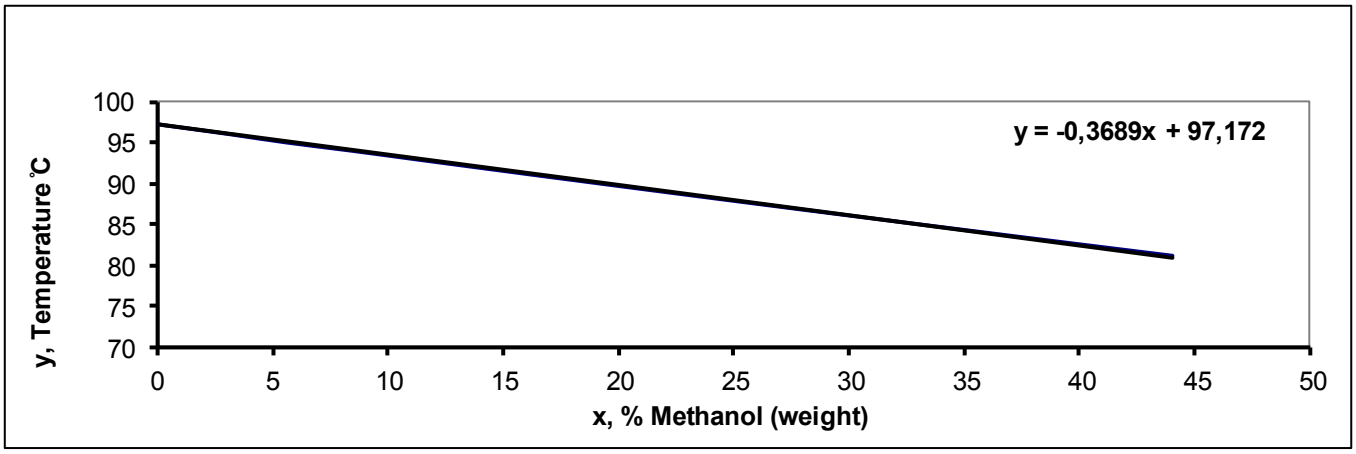
55 L mixture with 22% methanol concentration is fed to boiler. The temperature change is monitored during the distillation (Table 2). Samples are taken from the product collection container at a certain time intervals (10 min). The sample concentrations are determined by reading the indices of refraction from refractometer (see Figure 2 and Table 2). At the same time the top product is read on the temperature indicator.

Table 2 Experimental temperature data and calculated methanol mole fractions

Time (min)	Top Product Temperature (°C)	Reboiler Temperature (°C)	Calculated Top Product Methanol Mole Fractions X_D (See Figure2-b)	Intercept: $\frac{X_D}{R+1}$	Calculated Reboiler Methanol Mole Fractions X_S (See Figure2-a)	$\frac{1}{X_D - X_S}$
0	60.4	81.7	0,994	0,591	0,530	2,155
10	60.9	82.0	0,971	0,577	0,400	1,751
20	63.2	82.4	0,868	0,516	0,220	1,543
30	66.3	82.5	0,743	0,442	0,150	1,686
40	71.9	82.6	0,550	0,327	0,080	2,128
50	72.6	82.7	0,529	0,315	0,070	2,179
60	73.0	82.7	0,517	0,307	0,065	2,212



(a)



(b)

Figure 2 . Calibration plots a) for reboiler b) for top product

At the end of a certain operating time, to measure the flow rate of steam, refluxer is fully opened and the top of the product volume collected per unit time is measured in container. The concentration of the bottom product is determined by reading reflactive index. Total top product volume is measured at the end of the distillation time.

Equations used to calculate top product and reboiler methanol mole fractions (X_D and X_S) are given as follows:

Calibration Equation for Top Product **:** Y = -0.3689*X + 97.172

(3.1)

$$X_D = \frac{(X_A/M_A)}{\left(\frac{X_A}{M_A}\right) + \left(\frac{(100 - X_A)}{M_B}\right)}$$

(3.2)

Calibration Equation for Reboiler : Y = -0.3383*X + 95.889

(3.3)

$$X_S = \frac{(X_A/M_A)}{\left(\frac{X_A}{M_A}\right) + \left(\frac{(100 - X_A)}{M_B}\right)}$$

(3.4)

Initial methanol amount (S_1) in the reboiler before the distillation is determined as:

t = 0 $\rho_{methanol}$ = 721.6 kg/m^3 , ρ_{water} = 970.2 kg/m^3

Average Molecular Weight (Mw) = 0.283 * 32.04 + 0.717 * 18.02 = 21.986 kg / kmol

(3.5)

Average Density (ρ_{av}) = $0.283 * 721.6 + 0.717 * 970.2 = 899.871\ kg/m^3$

(3.6)

$$S_1 = \frac{\rho_{av} * V}{M_w} \Rightarrow S_1 = \frac{\left(899.871\ \frac{kg}{m^3}\right) * (55 * 10^{-3} m^3)}{(21.986\ kg/kmol)} = 2.251\ kmol$$

(3.7)

Methanol amount(S_2) in the reboiler after the distillation is evaluated as below:

Amount of mixture in the boiler ; V = F - D V = 55 – 2.726 = 52.274 L

(3.8)

t = ∞ $\rho_{methanol}$ = 741.3 kg/m^3 , ρ_{water} = 967.5 kg/m^3

Average Molecular Weight (Mw) = 0.2524 * 32.04 + 0.7476 * 18.02 = 21.559 kg / kmol

(3.9)

Average Density (ρ_{av}) = $0.252 * 741.3 + 0.748 * 967.5 = 910.407\ kg/m^3$

(3.10)

$$S_2 = \frac{\rho_{av} * V}{M_w} \Rightarrow S_2 = \frac{\left(910.407 \frac{kg}{m^3}\right) * (52.274 * 10^{-3} m^3)}{(21.559\ kg/kmol)} = 2.207\ kmol$$

(3.11)

$$V = 0.24 \frac{ml}{s} * 3600 \frac{s}{hr} * 910.407 \frac{kg}{m^3} * \frac{1 m^3}{10^6 ml} * \frac{1\ kmol}{21.559\ kg} = 0.037 \frac{kmol}{hr}$$

(3.12)

$$L = 0.10 \frac{ml}{s} * 3600 \frac{s}{hr} * 910.407 \frac{kg}{m^3} * \frac{1 m^3}{10^6 ml} * \frac{1\ kmol}{21.559\ kg} = 0.015 \frac{kmol}{hr}$$

(3.13)

D = V – L = 0.037 – 0.015 = 0.022 kmol / hr

(3.14)

Column Cross-sectional Area= S = $\pi * (0.08)^2 / 4 = 0{,}005\ m^2$

(3.15)

$$G_y = \frac{V}{S} = \frac{0.037}{0.005} = 7.4 \frac{kmol}{m^2.hr} \quad and \quad G_x = \frac{L}{S} = \frac{0.015}{0.005} = 3.0 \frac{kmol}{m^2.hr}$$

(3.16)

Mass transfer coefficient is written as follows (Sahay and Sharma,1973; Karacan, Hapoğlu, Cabbar, and Alpbaz, 1997)

$$K_y a = 1.28 * 10^{-5} * (V)^{0.64} * (L)^{0.48}$$

(3.17)

$$K_y a = 1.28 * 10^{-5} * (7.4 * 10^3)^{0.64} * (3.0 * 10^3)^{0.48} = 0.179 \frac{kmol}{m^2.hr}$$

(3.18)

To determine Height of Transfer Units (H_{Theo}) and Number of Transfer Units (N_{Theo}) for multistage batch distillation, the equations given below and Table 3 and 4 are utilized.

$$In \frac{S_1}{S_2} = \int_{X_{S2}}^{X_{S1}} \frac{dX_S}{X_D - X_S} = (1 | X_D - X_S)_{average} * (X_{S1} - X_{S2}) \Rightarrow 0.01974 = 1.54 * (0.222 - X_{S2})$$

(3.19)

$S_1 / S_2 = 0.014$, $X_{S1} = 0.222$, $X_{S2} = 0.209$

$$y_{average} = \frac{S_1 * X_{S1} - S_2 X_{S2}}{S_1 - S_2} \Rightarrow y_{average} = 0.874$$

(3.20)

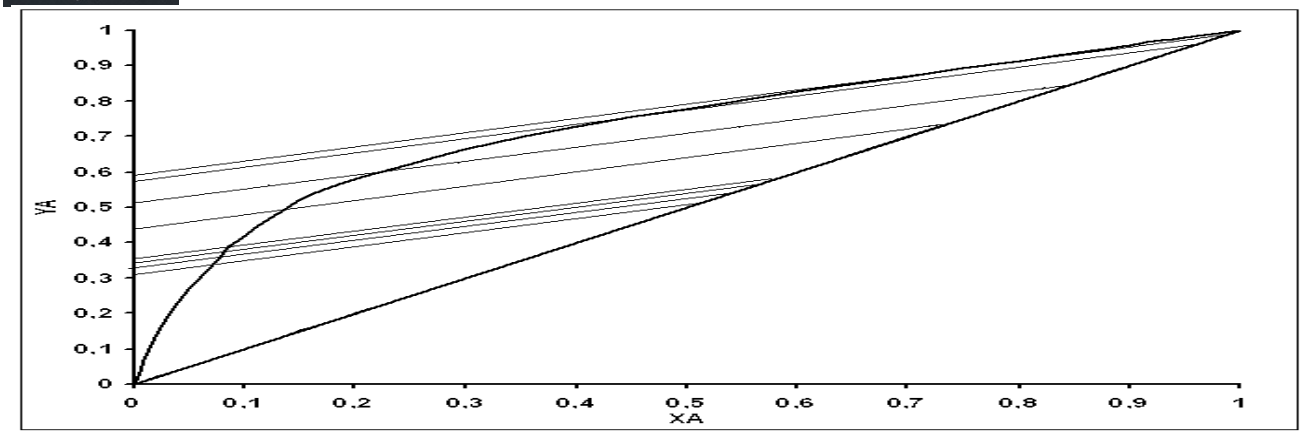


Figure 3. Equilibrium curve and Operation lines for the case studied

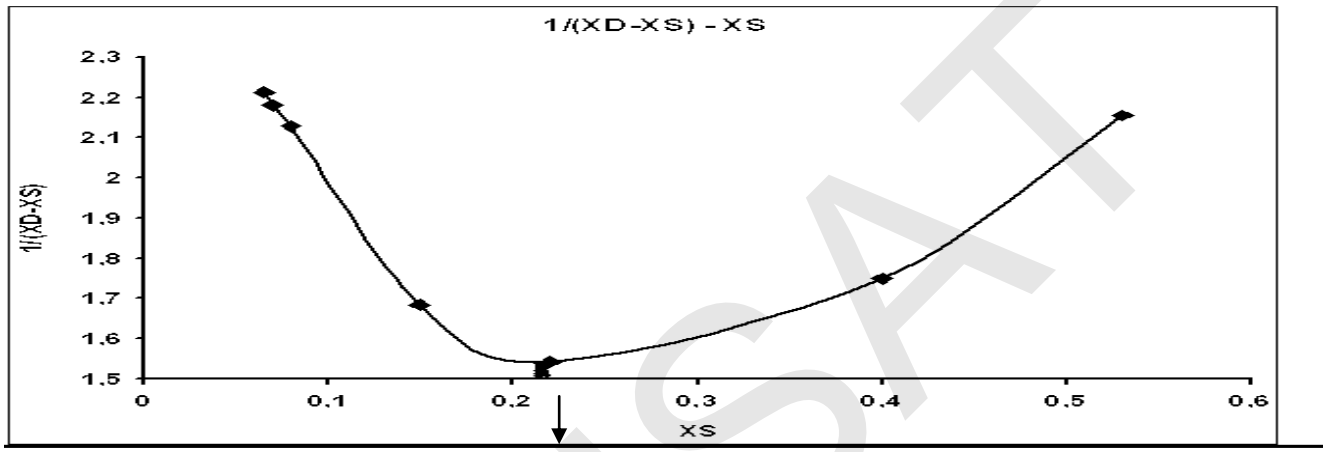


Figure 4. Relationship between $1/(X_D-X_S)$ and X_S values.

$$H_{Theo} = \frac{G_Y}{K_y a} = \frac{7.4}{0.179} = 41.37 \quad (3.21)$$

$$N_{Theo} = \int_{y_1}^{y_2} \frac{dy}{y - y^*} = \frac{y_2 - y_1}{\Delta y_L} = N_{Theo} = \frac{(y_w - y_F)}{\Delta y_L} = \frac{(0.209 - 0.222)}{-0.379} = 0.0343 \quad (3.22)$$

$$\Delta y_L = \frac{(y_2 - y_2^*) - (y_1 - y_1^*)}{In\frac{(y_2 - y_2^*)}{(y_1 - y_1^*)}} = \Delta y_L = \frac{(0.209 - 0.59) - (0.222 - 0.60)}{In\frac{(0.209 - 0.59)}{(0.222 - 0.60)}} = -0.38 \quad (3.23)$$

Column Height (Z) is determined as follows:

$$Z_{Theo} = H_{Theo} * N_{Theo} => \ Z_{Theo} = 41.37 * 0.0343 = 1.42\ m \quad (3.24)$$

The column height is evaluated as 1.42 m and by comparing with the real packed height of 1.25 m, correction factor (e) is calculated for the column studied as follows:

$$Z_{Real} = Z_{Theo} * e \ => \ e = \frac{Z_{Real}}{Z_{Theo}} = 0.88 \quad (3.25)$$

it is shown that the equation used for mass transfer coefficient can be written as follows:

$$K_y a = e^{-1} * 1.28 * 10^{-5} * (V)^{0.64}(L)^{0.48}$$
(3.26)

CONCLUSION

The evaluated packed distillation column height is in satisfactory agreement with the real one. The separation efficiency of packed column is generally expressed in terms of height equivalent of theoritical plate (HETP). For distillation at total reflux, HETP may be calculated by the following equation:

$$HETP = H_{Theo} * e = (Z_{Theo} * e)/N_{Theo} = Z_{Real}/N_{Theo}$$
(3.27)

This work provides a new tool for those concerned with the design or batch performance of packed distillation columns. It gives a simple method to assess the sensitivity of a packed bed to batch distillation.

REFERENCES

Betlem B.H.L., Krijnsen H.C. and Huijnen H. (1998). Optimal batch distillation control based on spesific measures. *Chemical engineering journal* 71 111-126.

Elgue S., Prat L., Cabassud M., Lann J.M.Le., Cezerac J. (2004). Dynamic models for start-up operations of batch distillation columns with experimental validation. *Computers and chemical engineering* 28 2735-2747.

Karacan S., Hapoğlu H., Cabbar Y., Alpbaz M. (1997). Pole placement self tuning control for packed distillation column *Chemical engineering and processing* 36 309-315.

Kim Y. Han (1999). Optimal design and operation of a multi-product batch distillation column using dynamic model. *Chemical engineering and processing* 38 61-72.

Liu G.B., Yu K.T., Yuan X. G. and Liu C.J. (2009). A numerical method for predicting the performance of a randomly packed distillation column. *International journal of heat and mass transfer* 52 5330-5338.

Muddu M., Narang A., Patwardhan S.C. (2010). Reparametrized ARX models for predictive control of staged and packed bed distillation columns. *Control engineering practice*. 18 114-130.

Noda M., Kato A., Chida T., Hasebe S., Hashimoto I. (2001). Optimal structure and on-line optimal operation of batch distillation column. *Computers and chemical engineering*. 25 109-117.

Rejl J.F., Linek V., MouchaT., Prokopova E., Valenz L. and Hovarka F. (2006). Vapour and Liquid side volumetric mass transfer coefficients measured in distillation column.Comparison with data calculated from absorption correlations. *Chemical engineering science*. 61 6096-6108.

Sadeghifar H., Kordi A.A.S. (2011). A new and applicable method to calculate mass and heat transfer coefficients and efficiency of industrial distillation columns containing structured packings. *Energy*. 36 1415-1423.

Sahay B.N. and Sharma M.M. (1973). Effective interfacial area and liquid and gas side mass transfer coefficients in a packed column. *Chemical engineering science* 28, 41-47.

Senol A. (2001). Mass transfer efficiency of randomly-packed column: modeling considerations. *Chemical engineering and processing*. 40 41-48.

Zuiderweg F.J. (1999). Distillation composition profiles –What do they tell us? *Institution of chemical engineers, Trans IChemE*. 77 (A) 475-481.