

HEAT TRANSFER INFLUENCE ON FRACTIONATION IN FLOODED PACKED COLUMNS

SIMONA POPA^a, SORINA BORAN^a, GIANNIN MOSOARCA^{a,*},
COSMIN VANCEA^a

ABSTRACT. In packed fractionation, the liquid phase can be forced to become continuous, and the vapor phase dispersed, realizing emulsification regime. The paper presents the efficiency comparing results of packed fractionation with respect to the fractionation in an empty flooded column, as well as the influence of the heat flux and of the temperature inside the column's jacket. Experiments for determining the boiling heat transfer of the different methanol – ethanol concentration mixtures were performed. The variation of the boiling heat transfer coefficient at different concentrations of methanol-ethanol mixtures has a minimum, corresponding to the maximum of the difference between concentrations of vapor phase in equilibrium with the liquid phase.

Keywords: *fractionation, flooded columns, boiling heat transfer*

INTRODUCTION

In chemical industry columns are apparatus used in many different technologies, such as fractionation [1-3], polymerization [4-7] or esterification [8, 9]. The main separating process in chemical industry represents fractionation, and it is very important to find means to improve the process or to make it more economic efficient [10] or safety [11].

One of the main reasons of the low efficiency in operating packed columns, under ordinary hydrodynamic conditions, is the non-uniform distribution of the liquid phase over the packing surface. The liquid phase preferred channels when flowing through the packing, which determines a deep decrease of the interfacial area, a growth of the flowing liquid layer thickness and the heat and mass transfer deteriorate.

^a Polytechnic University of Timisoara, Victoriei Square, No.2, 30006-Timisoara, Romania
* Corresponding author: giannin.mosoarca@upt.ro

When operating a packed column with a large diameter, the liquid can't be uniform distributed on the entire packing surface, causing a decrease of the column's performance, with respect to a smaller diameter packed column, so the height equivalent to a theoretical plate (HETP) is also small [12].

The optimum operating conditions for a packed column are situated in the emulsification regime, which can be observed only in a narrow range of velocities of the liquid and gaseous phases. The upper limit is the column's flooding and the lower limit is when the emulsification disappears. During the emulsification regime, the HETP increases abruptly and the column operates at constant flow velocity. That is why the emulsification regime must be stabilized.

The literature [13-16] presents models for determining the flooding point for different packing. For several decades, the Sherwood-Leva-Eckert correlation chart had been the standard of the industry for predicting flood points and pressure drops for fractionation packed columns. The gas – liquid interaction [13] influences the pressure drop and the flooding, being connected to the maximum of velocity. The point limited by the maximum superficial gas mass flow rate and the superficial liquid mass flow rate is the "limit operating point", and the linking of all these points in the Sherwood-Leva-Eckert diagram represents the "limit operating line" – "flooding line". Many researchers [3, 17-21] tried to establish optimal conditions for fractionating columns.

The boiling heat transfer coefficients are used routinely in calculations and design of industrial boiling equipment. Many researchers calculate boiling heat transfer coefficients using different types of heat surface [22-28]. The literature also presents aspects regarding the control performances improvements [29, 30]. Some of the papers deal with the improvement and optimization of the separation process [30].

Although the heat transfer under flooding conditions and measurements for the holdup are important, only few researchers published in this area [31, 32].

To the best of our knowledge fractionation in flooded packed columns with the study of the influence of the liquid stopper and the calculation of the heat transfer at boiling was not yet been proposed. This is why the present paper proposed to study the separation efficiency when using a flooded column with or without packing material and to evaluate the partial boiling heat transfer coefficient (α) during the process as well as the dependence of boiling heat transfer coefficient on heat flux (q).

RESULTS AND DISCUSSION

In the first part of the experiments, when the efficiency of the flooded column was studied, it was established that, for the same heat flux, the jacket temperature influences the process performance, determining a greater or a

smaller liquid reflux ratio [27]. Empiric equations for the proposed dependences are presented in Table 1. It can be seen that with the increase of the temperature inside the jacket, the concentration of the distillate decreases and the distillate flow rate increases.

Using Fenske relations the height equivalent of a theoretical plate (HETP) was established. The results are presented in Table 2. At 47 and 62 °C, the results are similar to the ones reported in literature [27].

Table 1. Proposed empiric equations for the dependences of the distillate concentration and distillate flow rate on the jacket temperature for methanol-ethanol mixture in the packed column

Empiric equation of the distillate concentration on the jacket temperature	Empiric equation of the distillate flow rate on the jacket temperature
$y = -1.057x + 136.36$	$y = 1.8876x - 71.6$

Table 2. The height equivalent to a theoretical plate (HETP) at different temperatures

Temperature, °C	HETP, cm
47	7.7
50	7
55	6.4
60	5.2
62	4.7

In the second part of experiments, the efficiency in the flooded column with no packing material was calculated based on Fenske relation. The results are presented in Table 3. It can be observed that the efficiency of a flooded empty column is better than that of a packed column of the same diameter. When the height of the liquid stopper is bigger, the efficiency of the column is also better.

Table 3. Separation efficiency in flooded column with two heights of the liquid stopper for the methanol-ethanol mixture

Height of the liquid stopper (cm)	HETP (cm)
5.5	5.5
7	5.1
10	4.9
14	4.5

When the density of the heat flux (q) at the bottom of the column was increased, the separation efficiency in the flooded column with the liquid stopper of 14 cm increased as well. The performance of the column in this case is presented in Figure 1. It can be seen once more, that the separation process is more efficient by intensifying the heat transfer at the bottom of the fractionating column.

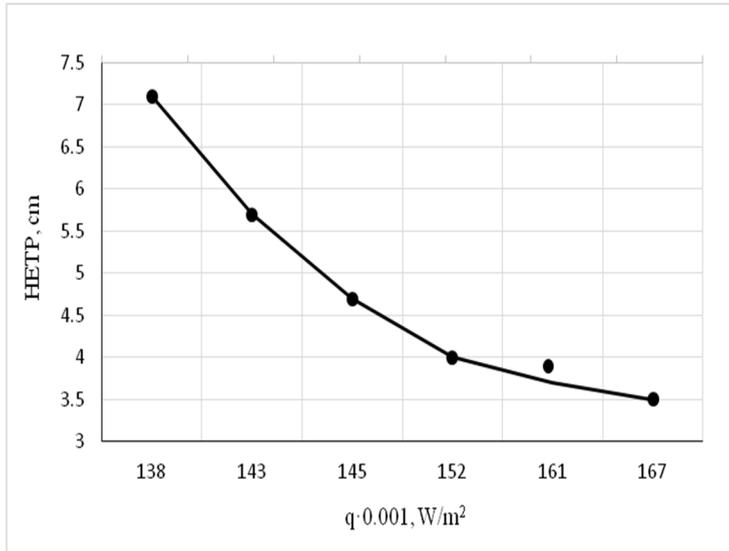


Figure 1. Dependence of the efficiency of a flooded column with the liquid stopper of 14 cm on the thermal flux (q) at separation methanol-ethanol mixture

In order to correlate the efficiency of the column, not only with the heat flux (q), but also with the partial boiling heat transfer coefficient (α), experiments for establishing the variation of α with the heat flux were performed. The experimental results for different concentrations of methanol-ethanol mixtures are presented in Figure 2.

Table 4. Dependent equations between α and q

C, % mol	$\alpha = a \cdot q^{0.56}$
0	$\alpha = 13.68 \cdot q^{0.56}$
42	$\alpha = 12.06 \cdot q^{0.56}$
55	$\alpha = 10.40 \cdot q^{0.56}$
65	$\alpha = 11.13 \cdot q^{0.56}$
100	$\alpha = 13.27 \cdot q^{0.56}$

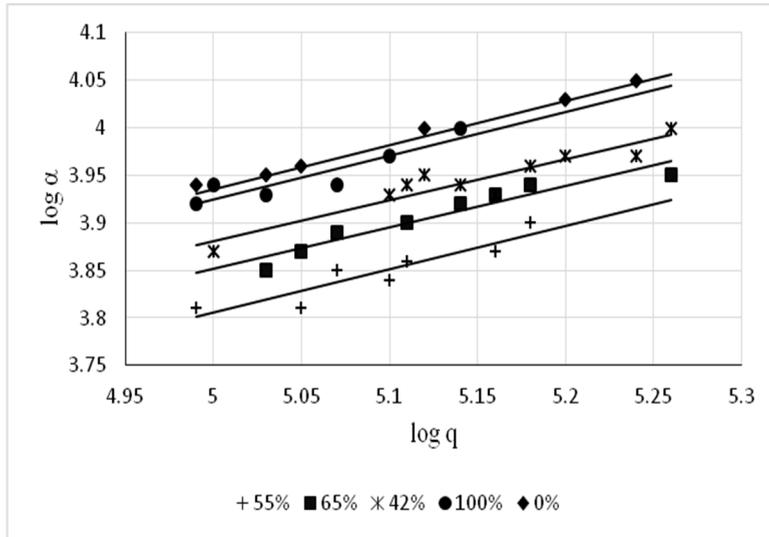


Figure 2. Dependence of boiling heat transfer coefficient (α) on heat flux (q)

The obtained dependences (Table 4) have the general equation:

$$\alpha = a \cdot q^{0.56} \quad (1)$$

where: a is concentration depending parameter.

The boiling heat transfer coefficient values decrease at the beginning, but then increase when the concentration increase. This phenomenon was observed even at the different concentrations of boiling ethanol-water mixture [36].

The explanation may be linked to the boiling mechanism. On the basis of the equilibrium data for the methanol-ethanol system, it was established that the variation of the concentration difference between vapor phase in equilibrium to the liquid phase, $(y-x)$, as a function of concentration, has a maximum where the value of the boiling heat transfer coefficient (α) has a minimum. These are represented in Figure 3 and in Figure 4.

It is known that the value for boiling heat transfer coefficient (α) depends on the limit surface phenomena. Thus, if the heat transfer from the boiling surface to the liquid next to it determines a concentration in the low volatility liquid, which is greater if the difference $(y-x)$ increases, the effect is a decrease in the value of α until $(y-x)$ reaches the maximum value, afterwards a new increase of the α -value appears, when $(y-x)$ decreases.

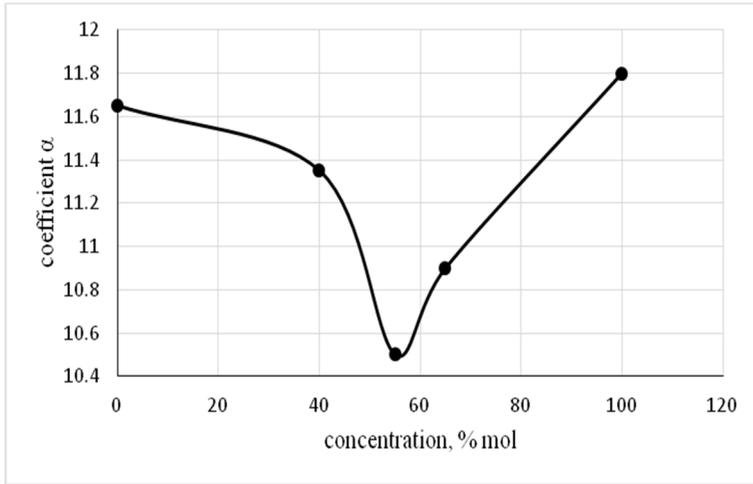


Figure 3. Variation of coefficient α with the concentration

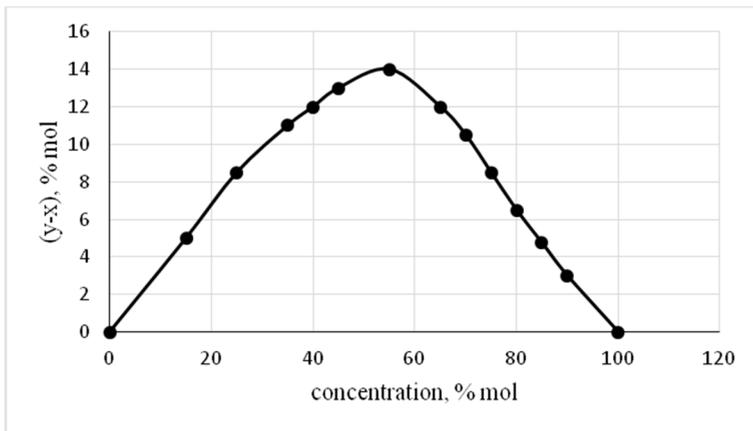


Figure 4. Variation of (y-x) with the concentration

CONCLUSIONS

Experiments show that, during separation of a methanol-ethanol mixture, by raising temperature inside the jacket of a packed flooded column, at the same heat flux, separation efficiency increases. Attempts were made to separate the same mixture of methanol-ethanol into a column with a liquid stopper of different heights, showing an increase in fractional efficiency compared to the filler column.

At the same time, by increasing the thermal flow in the column flap it was once again proved the beneficial influence of the increase of the boiling heat transfer on the performance of the fractionation columns.

Empirical equations for correlating the process parameters are proposed.

The variation of the boiling heat transfer coefficient at different concentrations of methanol-ethanol mixtures has a minimum, corresponding to the maximum of the difference between concentrations of vapor phase in equilibrium with the liquid phase ($y-x$).

EXPERIMENTAL SECTION

In order to study the fractionation process in flooded columns, an installation, where most of the influencing parameters can be maintained constant was realized [27, 28, 30, 34] (Figure 5). This facilitates the obtaining of constant values for the gas flow rates, corresponding to the mixture concentration.

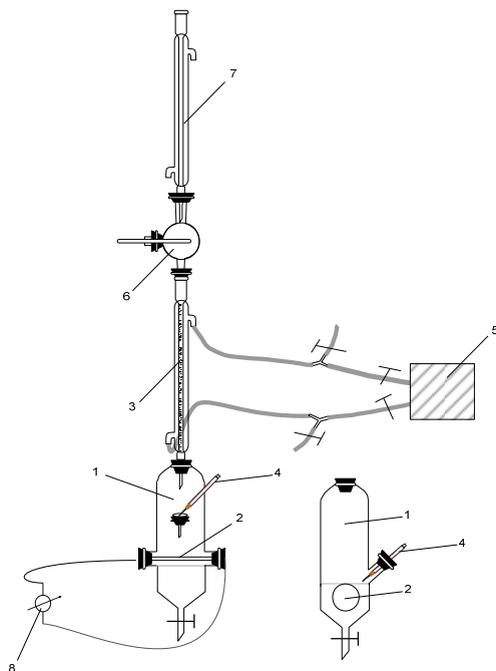


Figure 5. Experimental installation: 1-boiling vessel; 2-heat device; 3-column; 4-pipette; 5-thermostat; 6-dropper separator; 7-refrigerator; 8-current intensity measurement system

Experimental equipment includes a boiling vessel with an inside heater, a packed column provided with a refrigerator and a drops separator.

The boiler is heated by a heating system with internal source, made by a short circuit heating metal, where the heat flux can be measured and reproduced.

The packing material which was in glass Rashig rings ($\sigma = 776 \text{ m}^2/\text{m}^3$; $V_f = 0.71 \text{ m}^3/\text{m}^3$) having a height $H_p = 0.3 \text{ m}$.

The inside heater is an horizontal cylindrical stainless steel tube, having the external diameter of 0.01065 m, and the length of 0.0504 m. High amperage alternating current is supplied through thick copper connectors attached to the heating tube. The control of the thermal flux resulting from the Joule — Lenz effect is achieved by adjusting the amperage of the electrical current supplied.

In order to study the efficiency of the flooded column, it was necessary to compare the effectiveness of this column with one whose operation and parameters are established under identical operating conditions. So, at first, the column was performed at total reflux ratio with an ideal methanol-ethanol mixture [35], which has the relative volatility value close to one.

For determining of height equivalent to a theoretical plate (HETP) Fenske relations (1-3) were used [36]:

$$y^* = \frac{\alpha_r \cdot x}{1 + (\alpha_r - 1) \cdot x} \quad (2)$$

where: x is mole fraction of the more volatile component in the liquid phase, y^* is molar fraction of the more volatile component in the vapor phase in equilibrium with the liquid phase and α_r represent the coefficient of relative volatility.

$$N = \frac{\lg\left(\frac{x_D}{1-x_D} \cdot \frac{1-x_W}{x_W}\right)}{\lg(\alpha_{med})} - 1 \quad (3)$$

where: N is the minimum number of theoretical plates, x_D , x_W represent mole fraction of the more volatile component in the distillate, and in the still pot respectively and α_{med} is average volatility.

$$\alpha_{med} = \sqrt{\alpha_D \cdot \alpha_W} \quad (4)$$

where: α_D , α_W are relative volatilities in the distillate, and in the still pot respectively.

During the first part of the experiments the temperature ranged between 47 and 62°C.

In the second part, the experiments were performed at 47°C with an empty column of the same diameter, at an identical heat flux, where the liquid was maintained at flooding, working with the height of the liquid stopper between 5.5 cm and of 14 cm respectively.

REFERENCES

1. S. Bhatia, A.L. Ahmad, A.R. Mohamed, S.Y. Chin, *Chem. Eng. Sci.*, **2006**, *61*(22), 7436.
2. Z. Olujić, M. Jödecke, A. Shilkin, G. Schuch, B. Kaibel, Equipment improvement trends in distillation, *Chem. Eng. Process.*, **2009**, *48*(6), 1089.
3. A.A. Kiss, Z. Olujić, *Chem. Eng. Process.*, **2014**, *86*, 125.
4. S. Popa, C. Csunderlik, S. Florea, V. Jascanu, N. Plesu, *Revista de Chimie*, **2002**, *53*(4), 259
5. S. Popa, C. Csunderlik, V. Jascanu, D. Jurcau, N. Plesu, *Materiale Plastice*, **2003**, *40*(4), 177-181.
6. S. Popa, V. Jascanu, D. Jurcau, N. Plesu, *Revista de Chimie*, **2003**, *54*(7), 595.
7. S. Popa, C. Csunderlik, V. Jascanu, D. Jurcau, N. Plesu, *Materiale Plastice*, **2004**, *41*(2), 62.
8. E. Özcan, S. Sargin, Y. Göksungur, *Biochem. Eng. J.*, **2014**, *92*, 9.
9. C.J. Stacy, C.A. Melick, R.A. Cairncross, *Fuel Process. Technol.*, **2014**, *124*, 70.
10. P.C. Wankat, *Chem. Eng. Sci.*, **2015**, *137*, 955.
11. L. Bodisz, M. Hahn, A. Rix, J. Schallenberg, *Chem. Eng. Res. Des.*, **2015**, *99*, 208.
12. C. Stratula, "Fractionation. Principles and calculating methods", Editura Tehnica, Bucharest, **1986**.
13. H.Z. Kister, D. R. Gill, *Chem. Eng. Prog.*, **1991**, *87*(2), 32.
14. A.K. Coker, *Chem. Eng. Prog.*, **1991**, *87*(11), 93.
15. M. Leva, *Chem. Eng. Prog.*, **1992**, *88*(1), 65.
16. A. Badea, "Basis of heat and mass transfer", Editura Academiei Romane, Bucharest, **2004**.
17. R.E. Strigle, *Chem. Eng. Prog.*, **1993**, *89*(8), 79.
18. M. Kotorá, J. Markos, V. Camaj, *Chem. Eng. Sci.*, **2007**, *62*, 5193.
19. L.L. Simon, H. Kencse, K. Hungerbühler, *Chem. Eng. Process.*, **2009**, *48*(4), 938.
20. Q. Qian, H. Wang, P. Bai, G. Yuan, *Chem. Eng. Res. Des.*, **2011**, *89*(12), 2560.
21. T. Chen, B. Zhang, Q. Chen, *Energy*, **2014**, *72*, 311.
22. Y.J. Chung, K.H. Bae, K.K. Kim, W.J. Lee, *Ann. Nucl. Eng.*, **2014**, *71*, 298.
23. E. Demir, T. Izci, A. S. Alagoz, T. Karabacak, A. Kosar, *Int. J. Therm. Sci.*, **2014**, *82*, 111.
24. G.M. Chen, V.P. Zhelezny, A.V. Melnyk, K.O. Shestopalov, *Int. J. Refrig.*, **2015**, *58*, 137.

25. S.J. Thiagarajan, R. Yang, C. King, S. Narumanchi, *Int. J. Heat Mass Transfer*, **2015**, 89, 1297.
26. X. Zheng, C.W. Park, *Appl. Therm. Eng.*, **2015**, 86, 14.
27. S. Popa, S. Boran, *Rev. Roum. Chim.*, **2016**, 61(11-12), 851.
28. S. Popa, S. Boran, *Thermal Science*, **2017**, 21(5), 2031.
29. S. Nanu, T. L. Dragomir, *Control Eng. Appl. Inf.*, **2001**, 3(2), 1.
30. S. Popa, S. Boran, *Rev. Roum. Chim.*, **2015**, 60(10), 991.
31. M. Asadollahzadeh, M. Torab-Mostaedi, R. Torkaman, *Chem. Eng. Process.*, **2016**, 109, 97.
32. P. Amani, J. Safdari, H. Abolghasemi, M.H. Mallah, A. Davari, *Int. J. Heat. Fluid Fl.*, **2017**, 65, 266.
33. T.L. Dragomir, I. Silea, S. Nanu, Control performances improving by interpolator controllers, *6th World Multiconference On Systemics, Cybernetics and Informatics*, Orlando, Florida, USA, **2002**, p.208.
34. D. Kohn, S. Popa, *Exp. Heat Transfer*, **1999**, 12(3), 193.
35. R.J.P. Brierley, *Chem. Eng. Progress*, **1994**, 90, 68.
36. I. Bratu, "Procese si aparate in industria chimica, vol. III. ", Editura Tehnica, Bucharest, **1985**.