Mixture separation in distillation column with semi-cylindrical structured packing

N I Pecherkin¹, A N Pavlenko¹, V E Zhukov¹, A D Nazarov¹, G Meski² and P Houghton²

¹ Kutateladze Institute of Thermophysics SB RAS, 1 Lavrentiev ave., Novosibirsk, 630090, Russia
² Air Products, Inc., 7201 Hamilton Boulevard, Allentown, PA 18195-1501, USA

E-mail: pecherkin@itp.nsc.ru

Abstract. This paper presents investigation results on separation of a binary mixture in distillation columns of semi-cylindrical cross-section with structured packing. The effect of vapor and liquid flow rates, packing height on separation efficiency, pressure drop, distribution of local liquid flow rates, temperature, and mixture composition as under the structured packing so in different positions inside the column was studied experimentally. An increase in the ratio of the molar flow rates of the liquid and vapor phases \( L/V \) results in a decrease in the height equivalent to the theoretical plate HETP. Increasing of the height of the structured packing leads to deterioration in the efficiency of the mixture separation. To analyze the effect of the shape of the column cross-section on the separation efficiency, the similar experiments were carried out in a round column. The main regularities in the influence of the structured packing height, ratio of liquid and vapor flow rates, and other operating parameters obtained for the round column are also valid for the column with a semi-cylindrical cross-section.

1. Introduction

Columns with dividing walls (DWC) are the intensively developing systems for separation of multicomponent mixtures. They have a huge potential for saving energy and reducing capital costs, much greater than the traditional distillation systems of separation. The beginning of industrial construction of divided columns dates back to the mid-1980s, now their number exceeds hundreds of installations [1]. Most of publications on this topic deal with the development and analysis of thermodynamic schemes of the entire technological cycle for separation of multicomponent mixtures, including the issues of thermal integration [2-4]. Much less works are devoted to the issues of development of design systems and calculation methods for mass transfer and hydrodynamics in non-cylindrical packings [5-6].

Construction of divided columns assumes separation of the internal space of the column at least into two parts with mounting the separation walls, and production and installation of the structured packing, whose cross-sectional shape corresponds to the divided columns. Depending on the number of separation walls, they can be the segments, squares, triangles, etc. [7]. There are other arrangements of the heat integrated DWC [8]. The counter-current flow of vapor and liquid in the structured packing of non-circular shape has a number of features due to presence of the corner zones. This paper deals with experimental study of binary mixture separation in a semi-cylindrical column.
2. Experimental setup
The experiments were carried out in the experimental large-scale model of a distillation column with a diameter of 0.9 m [9]. The insert with a semi-cylindrical shape of cross-section 0.4 × 0.8 m in size was installed inside the main column. The unused part of the cross-section of the main column overlapped at the top and bottom with hermetically sealed flanges. The semi-cylindrical insert consists of several sections for carrying out experiments on a structured packing of different heights.

In this study, the structured packing with specific surface area of 500 m²/m³ and corrugation angle of 45° was used. The height of the structured packing varied from 1.2 to 3.3 m. The experiments were carried out at two L/V values of 1 and 1.7. The mixture of refrigerants R114 and R21 at the pressure of 0.3 MPa in the column was used as the working liquid. The effect of vapor and liquid flow rates, packing height on separation efficiency, pressure drop, distribution of local liquid flow rate, temperature and mixture composition was studied experimentally. Similar experiments were carried out in a round column with a diameter of 0.6 m. Compositions of vapor and liquid phases at the inlet and outlet of the column were measured by gas chromatography. Data on separation efficiency are presented as dependences of the height of transfer unit HTU and height equivalent to the theoretical plate HETP on parameters characterizing the vapor load of the column (Fv, Rev) or the liquid load (Ref).

3. Distribution of the local parameters on the column cross-section
The results and technique of measuring the distribution of mixture composition and temperature inside the round column in different cross-sections along the height are given by [10]. To measure the distribution of the temperature in several cross-sections of the column, the miniature sensors (16 in each cross-section) are mounted on the top of the structured packing layer. Then, the measured temperature of the liquid phase is recalculated into local concentration assuming that the mixture is in equilibrium state. Figure 1 shows the distributions of local temperatures and flow rates under the packing at different distances h from the lower edge of the packing. The distribution of the local liquid flow rate under the packing is characterized by an increased liquid flow rate with several local maxima in the central part of the cross-section and reduced flow rate at the corners of the column (figure 1c). In the vicinity of the column walls, there are also the areas of reduced liquid flow rate. The change in the local flow rate, temperature of liquid and composition of the mixture under the packing reflects the changes in distribution of liquid and vapor flows within the structured packing.

Figure 1. Distribution of local temperature (a, b) and liquid flow rate (c) in semi-cylindrical column, $F_v = 1.6 \text{ Pa}^{1/2}$: a – $h = 0.8$ m; b – $h = 0.2$ m; c – under packing.
The data on the temperature distribution inside the column show that the temperature difference in one section (figure 1a) is close to the temperature difference between the top and bottom of the column. The change in the concentration of the volatile component over the column cross-section is also comparable to the difference in concentration between the top and bottom of the column. Maldistribution of the mixture composition over the cross-section leads to the fact that the driving force of the mass transfer process on the interface varies along the cross-section and along the height of the column, which affects the total efficiency of mixture separation.

4. Effect of the $L/V$ ratio on separation efficiency

Most experimental studies of separation efficiency on the tested structured packings and trays are carried out under the conditions of total reflux. The molar liquid and vapor flow rates are the same, their ratio is $L/V = 1$. In real distillation columns, the value of $L/V$ can be either greater or less than unity. It is believed that the efficiency of structured packing for $L/V$ not equal to 1 is the same as for total reflux [11]. Experiments on the investigation of the effect of the reflux ratio on separation efficiency and pressure drop were carried out in a round column 0.9 m in diameter on 10 layers of the Koch1Y structured packing [12] and 19 layers of a Mellapak packing with a specific surface of 350 $m^2/m^3$ [13]. In these studies, it was shown that with an increase in the reflux ratio HETP decreases.

Figure 2 shows the results of the experimental study of the efficiency of separation in a semi-cylindrical column. A comparison with the predictive models [14, 15] is given. The SRP model [14] gives close, but understated estimates of HETP and HTU in comparison with the experiments for $L/V = 1$. For $L/V = 1.7$, this model predicts the values of HETP and HTU overestimated by about 25% as compared to the experiment. The Delft model [15] gives significantly overestimated values of HETP and HTU for all values of the reflux ratio. None of the models predict deterioration in separation efficiency after the loading point. In addition, the calculations for the presented models show a different influence of $L/V$ on the values of HETP and HTU. The Delft model predicts a significant increase in HETP when reflux ratio is changed from 1 to 1.7, while the results of the experiments show an inverse relationship. The variation of HTU with increasing $L/V$ is the same in both experiments and models (figure 2).

Data on the efficiency of separation in the form of HTU and HETP are usually used to compare the results obtained under different conditions. The main disadvantage of these quantities is that they are beyond traditional generalization. We presented our data on the efficiency separation in semi-cylindrical column at different $L/V$ values in the form of the dependence of the overall mass transfer coefficient $K_{OG}$ on the Reynolds number of the liquid film flowing along the sheets of the structured packing $Re_f$ (figure 3). The maximum in dependence of the mass transfer coefficient and its

![Figure 2](image-url)
subsequent decrease correspond to the beginning of mass transfer flooding. For \( L/V = 1.7 \), the maximal mass transfer coefficient is achieved at \( Re_f = 80 - 90 \), and for \( L/V = 1 \), it is achieved at \( Re_f = 60 - 70 \). The same figure shows the data on mass transfer coefficients for six layers of the Koch1Y packing at \( L/V = 1 \) in a round column with the diameter of 0.9 m with \( a = 420 \, \text{m}^2/\text{m}^3 \), \( L/V = 1 \) by [9]. Data for the packings with different specific surfaces and shapes of cross-section, but the same height, are in satisfactory agreement.

Data on the pressure drop obtained in experiments with different \( L/V \) ratios are shown in figure 4 as a function of the total pressure loss factor versus the Reynolds number of the vapor phase. The hydraulic crisis for \( L/V = 1.7 \) starts earlier than for \( L/V = 1 \).

### 5. Effect of the height of packing

This paper presents investigation results on the effect of the structured packing height on separation efficiency in a semi-cylindrical column. Data for \( L/V = 1 \) were obtained on six and eleven layers (figure 5). Separation efficiency increases with a decrease in the packing height. On the packing with a smaller number of layers, the mass transfer crisis occurs at the higher column load. We have obtained similar results on the effect of Koch1Y and Mellapak 350Y packings height on separation efficiency and pressure drop for the round column of the large diameter [9, 16]. Perhaps, the deterioration of the separation efficiency with increasing the height of the structured packing occurs as a result of maldistribution of the vapor and liquid flows within the column.

---

**Figure 3.** Overall mass transfer coefficient vs. film Reynolds number. 1, 2 – semi-cylindrical packing, \( L/V = 1 \) and 1.7, respectively; 3 – Koch1Y packing.

**Figure 4.** Effect of the ratio of vapor and liquid flow rates on the total pressure loss factor in semi-cylindrical column.

**Figure 5.** Effect of packing height on separation efficiency in semi-cylindrical column, \( L/V = 1 \): 1 – 1.2 m; 2 – 2.3 m; 3 – round column, 2.3 m.

**Figure 6.** Effect of packing height on separation efficiency in semi-cylindrical column, \( L/V = 1.7 \): 1 – 1.2 m; 2 – 3.3 m.
This figure also shows that in the range of $F$-factor from 1.6 to 2.4 Pa$^{-1/2}$, the separation efficiency in a semi-cylindrical column decreased significantly as compared to a round column with a diameter of 0.6 m. At low vapor load and in the packing flooding regimes, the separation efficiency in the semi-cylindrical and round columns is approximately the same.

The effect of the packing height on separation efficiency in a semi-cylindrical column at $L/V = 1.7$ is shown in figure 6. In the plateau area, before the mass-transfer crisis, HETP on six packing layers is approximately 30% lower than on sixteen layers. After the beginning of mass transfer flooding, the difference decreases significantly. The relative pressure drop before the hydraulic crisis beginning does not depend on the packing height, and after crisis beginning, the pressure drop at the higher packing becomes lower.

6. Conclusion
The effect of the packing height and ratio of the vapor and liquid flow rates on the efficiency of mixture separation and pressure drop for a semi-cylindrical column is similar to that for a round column. With increasing packing height, separation efficiency in the semi-cylindrical column deteriorates. The HTU and HETP values reduce significantly, when the reflux ratio increases.

Acknowledgments
This work was carried out within joint project of the Kutateladze Institute Thermophysics SB RAS and Air Products Inc., USA and the financial support of the FASO of Russia for the BSI SAS Program for 2013-2020 (project No. III.18.2.3) in the part of experimental studies on the distribution of the local mixture composition and temperature in the column.

References
[1] Yildirim Ö, Kiss A A and Kenig E Y 2011 Separation and Purification Technology 80 403
[2] Staak D, Grützner T, Schwegler B and Roederer D 2014 Chem. Eng. and Processing 75 48
[3] Wang S J and Wong D S H 2007 Chem. Eng. Sci. 62 1010
[4] Dejanović I, Halvorsen I J, Skogestad S, Jansen H and Olujić Ž 2014 Chem. Eng. and Processing 84 71
[5] Olujić Ž, Jödecke M, Shilkin A, Schuch G and Kaibel B 2009 Chem. Eng. and Processing 48 1089
[6] Chen W, Huang K, Chen H, Xia C, Wu G and Wang K 2014 Chem. Eng. and Processing 75 90
[7] Wilson J, Sunder S and Houghton P. Structured packing. US Patent: US 2013/0233016 A1, Sep. 12, 2013
[8] Kiss A A and Olujić Ž 2014 Chem. Eng. and Processing 86 125
[9] Pavlenko A N, Pecharkin N I, Chekhovich V Yu, Zhukov V E, Sunder S, Houghton P, Serov A F and Nazarov A D 2006 Theor. Found. of Chem. Eng. 40 329
[10] Pavlenko A N, Zhukov V E, Pecharkin N I, Chekhovich V Y, Sunder S and Houghton P 2010 Theor. Found. of Chem. Eng. 44 869
[11] Kister H Z 1992 Distillation design (McGrawHill, Inc.)
[12] Pavlenko A, Pecherin N, Chekhovich V, Zhukov V, Sunder S, Houghton P, Serov A and Nazarov A 2005 Journal of Engineering Thermophysics 13 1
[13] Pavlenko A, Zhukov V, Pecherin N, Chekhovich V, Vолодин О, Shilkin A and Grossmann C 2014 AIChE J. 60 690
[14] Bravo J L, Rocha J A and Fair J R 1985 Hydrocarbon Processing 64 91
[15] Olujić Ž, Kamerbeek A B and de Graauw J 1999 Chem. Eng. and Processing 38 683
[16] Pavlenko A, Zhukov V, Pecherin N, Nazarov A, Li X, Liu M, Sui H and Li H 2017 Effect of the structured packing height on efficiency of freons mixture separation in a large-scale model of distillation column MATEC Web of Conf. 115 08003