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# Studies of Freon mixture separation using a large-scale model of distillation column

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Abstract. The operation efficiency of distillation columns with structured packing is maximal if the distribution of countercurrent vapor and liquid film flows over the column cross-section on the mass transfer surface is most uniform. Various types of structured packing are widely used in distillation columns. Formation of the temperature field maldistribution in the column cross-section is observed in large-scale distillation columns. The sizes of large-scale maldistributions on zones are commensurate with the column diameter. The aim of this work is to obtain experimental data on the separation efficiency in large-scale distillation columns and dynamics of formation of large-scale maldistributions of local parameters of vapor and liquid in a countercurrent flow over a structured packing during mixture separation. Separation of the R114/R21 freon mixture was carried out on a structured Mellapack 350.Y packing with a diameter of 0.9 m and height of 2100 mm. The experiments were carried out under conditions of complete reflux in the range of reduced vapor velocity of 0.017 <  $K_v$  < 0.035 m/s. Experimental data were obtained on the efficiency of mixture separation, pressure drop over the structured packing and parameters determining the formation dynamics of the large-scale temperature field maldistribution in the column cross-section. The presented experimental data will be used for the construction and verification of a new model of mass transfer and efficiency of mixture separation in large-scale distillation packed columns.

#### 1. Introduction

Rectification is a process of separation of binary, multicomponent or continuous mixtures into almost pure components or their mixtures (fractions) that differ in boiling points (for binary and multicomponent mixtures) or boiling ranges (for continuous mixtures) [1-4]. Distillation columns are widely used in industry to perform the rectification process, represented by mass transfer occurring in both directions between two phases of mixture, one of which is liquid and another is vapor. This is a multiple contact interaction of nonequilibrium phases along the height (length) of the distillation apparatus under the conditions of their countercurrent flow. The driving force of heat and mass transfer between vapor and liquid in the apparatus is the temperature and concentration differences in the countercurrent phases. The efficiency of distillation column depends substantially on the degree of uniformity of distribution of vapor and liquid film countercurrent flows over the column cross-section on the packing surface and column wall. To ensure the most uniform distribution of the film of the falling liquid phase over the contact devices and reduce energy consumption for pumping the vapor phase, various structured packings have been and are being developed, differing from each other in geometry and methods for applying a specific surface structure (packing sheet corrugation, including

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those varying along the height of separate packing layers; various forms of microtexture; holes with different areal densities, diameters and shapes; various shapes of wipers and clips restricting the liquid flow on the column wall, etc.) [5 - 14]. However, in large-scale distillation columns, formation of temperature field non-uniformity is observed in the column cross-section, and the sizes of the characteristic zones of this non-uniformity are comparable with the column diameter [15, 16]. These studies are carried out in the framework of research aimed at developing new methods for modeling the processes of mixture separation in large-scale industrial apparatuses [17,18]. This work is part of a series of studies aimed at obtaining the experimental data on separation efficiency of large-scale columns and formation dynamics of large-scale maldistributions of local parameters of vapor and liquid countercurrent flows over a structured packing during mixture separation.

#### 2. Setup and method description

The experiments were carried out on a model separation column designed to study the integral and local characteristics of mixture separation by distillation on a structured packing. To simulate the process of liquefied air separation at ambient temperature, the mixture of R114/R21 freons was selected. The studies were carried out using this mixture. The main elements of setup are schematically presented in Figure 1.



The mixture was separated on the structured Mellapack 350.Y packing (1) with a diameter of 0.9 m and height of 2100 mm (10 packing layers). The mixture poured into the column bottom (bottom space) was supplied by a circulation pump to the evaporator (2), and then it was fed to the column bottom in the form of vapor. After the perforated sheet (3), which served as a vapor phase distributor, vapor passed through the structured packing and turned completely into the liquid phase in the condenser (4). Liquid from the condenser was supplied using a circulation pump to the liquid distributor (5), which was a cylindrical vessel of 500-mm height with uniformly distributed vapor channels. At the bottom of liquid distributor, there were the holes for installing nozzles to irrigate the packing. A schematic arrangement of irrigation nozzles in the liquid distributor is shown in *Figure 2*. A detailed description of the setup is given in [15, 16,19].





Figure 2. Arrangement of irrigation nozzles in liquid distributor.

The efficiency of mixture separation was determined by measuring the concentration of vapor and liquid phases at the inlet and outlet of the distillation column. The concentration was determined by gas chromatography. The measurements were carried out in a continuous cycle of transporting the mixture samples through the sampling lines. The time of one sampling was 90 s. The accuracy of measuring the molar concentration of the mixture in the region of small values (column bottom, 0.1-1%) was about 0.01%; in the region of high values of concentration (column top, 30-50%), it was 0.2%.

To study the dynamics of formation of large-scale temperature field maldistribution during mixture separation, the miniature temperature sensors were installed at three levels of the packing cross-section. Sensors in three layers were installed at distances of 420, 1050 and 1680 mm from the lower packing edge, respectively. In each layer, 16 sensors were evenly distributed. Silicon diodes, whose temperature sensitivity is higher as compared to platinum and copper thermometers and thermocouples, were used as the sensors. They have high long-term stability of characteristics and good linearity. Each thermometer was powered by an individual precision REF200 generator with a current of 100  $\mu$ A. Under the conditions of a gaseous medium, the heating of thermometer itself was about 0.06 K. At that, the intrinsic heat release was  $6 \cdot 10^{-5}$  W. The voltage drop across the thermometer was measured with a 24-bit LTR114 ADC. The sampling time of all 48 thermometers was 5 s. Data was transferred to a PC and displayed in real time in the form of temperature fields for three packing cross-sections. To calibrate the thermometers, the ThermoHaake DC30 thermostat with temperature maintenance stability of 0.02°C and temperature accuracy of 0.1°C was used.

#### 3. Results and discussions

The experiments were carried out at a pressure of 0.3 MPa, which corresponded to temperatures in the upper and lower parts of the column of 34 - 40.5°C, respectively. A series of experiments was carried out under the complete reflux conditions at a constant vapor velocity through the column. Values of the column average velocity (U) were in the range of 0.15–0.3 m/s, which corresponded to the reduced

vapor velocity of 0.017 <  $K_v$  < 0.035 m/s, where  $K_v = U_v \sqrt{\frac{\rho_{vap}}{\rho_{liq} - \rho_{vap}}}$ . The time required for the

column to reach the stationary regime of mixture separation was, as a rule, 1.5–3 hours. During this time, all elements of the column were heated to the operating temperature, the flows of liquid and

vapor were stabilized, and the external heat release and heat removal were balanced. The diagram of changes in local temperatures in the middle cross-section of the packing is shown in *Figure 3*.



**Figure 3.** Diagram of a change in the local temperatures in the middle (h = 1.05 m) cross-section of the packing during stabilization of column operation.  $K_v = 0.03$  m/s. Readings of thermometerss: 1 - s14.

As it can be seen in the diagram, at the first stage of the transition process, the temperature distribution in the middle cross-section of the packing is quite uniform. Then, while approaching the stationary regime of column operation, the formation of significant temperature maldistribution over the packing cross-section is observed. The regions of high and low temperature are large and they are located diametrically in the packing cross-section. The temperature range of the mixture along the packing height is 35 - 40.5°C. At a pressure of 0.3 MPa, the equilibrium molar concentration of the liquid phase of mixture varies in the range of 0.28–0.005, and the vapor phase concentration varies in the range of 0.35–0.01. Thus, the conditions for mass transfer processes within one packing cross-section are very ambiguous, and therefore, the efficiency of mixture separation in large-scale columns has the potential to increase when eliminating such significant unevenness in the distribution of flow parameters.

Data on the efficiency of mixture separation and pressure drop on the structured Mellapack 350.Y packing are shown in Figure 4.



**Figure 4.** Efficiency of mixture separation and pressure drop on the structured Mellapack 350.Y packing. Packing height is 2.1 m. 1 – separation efficiency, HTU; 2 – pressure drop on the packing.

As it can be seen in the diagram, with increasing vapor flow through the column, the efficiency of mixture separation is significantly improved. This improvement is valid for a range of vapor velocities far from the regimes of droplet entrainment.

To perform the activity aimed at increasing the efficiency of large-scale distillation columns, it is necessary to know the mechanism of formation of large-scale non-uniformity of local parameters over the column cross-section. One of the possible reasons for the formation of these non-uniformities may be convective down flows with a scale of the column diameter. These flows can be either a consequence of hydrodynamic stability loss under conditions of negative stratification of the vapor density, or a consequence of a vapor flow conjugated with the liquid flow along the column wall. The improvement in the efficiency of mixture separation with increasing vapor velocity through the column is possibly associated with the complete dominance of the countercurrent vapor flow through the packing over the convective flows. Another factor determining an increase in the efficiency of mixture separation is an increase in the proportion of the wetted surface with a corresponding increase in the flow rate of liquid through the structured packing.

#### 4. Conclusions

Experimental data on the dynamics of formation of large-scale non-uniformity of the temperature field in the cross-section of a distillation column with Mellapack 350.Y packing of the 0.9-m diameter and 2.1-m height were obtained.

Experimental data on the separation efficiency and pressure drop on a Mellapack 350.Y packing with a height of 2.1 m were obtained. The improvement in the efficiency of mixture separation with an increase in the vapor velocity through the column is due to the factors of an increase in the proportion of the wetted packing surface and the complete dominance of the countercurrent vapor flow through the packing over the convective flows.

The obtained experimental data will be used to verify the model of mass transfer in large-scale distillation packed columns developed in the framework of the RFBR 19–58–18004 project.

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