

## Overview of methods to control the liquid distribution in distillation columns with structured packing: Improving separation efficiency

A.N. Pavlenko<sup>a</sup>, N.I. Pecherkin<sup>a,\*</sup>, V.E. Zhukov<sup>a</sup>, G. Meski<sup>b</sup>, P. Houghton<sup>b</sup>

<sup>a</sup> Kutateladze Institute of Thermophysics SB RAS, Lavrentiev ave. 1, Novosibirsk, 630090, Russia

<sup>b</sup> Air Products, Inc., 7201 Hamilton Boulevard, Allentown, PA, 18195-1501, USA



### ARTICLE INFO

#### Keywords:

Distillation column  
Separation efficiency  
Pressure drop  
Structured packing  
Spreading of liquid  
Initial packing irrigation  
Turn angle of packing layers

### ABSTRACT

The paper presents experimental results on the effect of characteristics of liquid spreading over the surface of a structured packing, distribution of local flow parameters over the cross-section and height of the distillation column under various conditions of initial packing irrigation. It is shown that the corrugation angle of the structured packing sheets, microtexture parameters, presence of perforation and size of holes, as well as liquid flow rate determine the patterns of liquid spreading and wetting of the packing surface. The test results for a new method of dynamic control of the liquid distributor with independent valves for each drip point are presented with the aim of influencing large-scale flow maldistribution in the column and increasing the separation efficiency. The data obtained in these experiments showed that a targeted change in the position of active irrigation points based on the actual distribution of local parameters in the separation column can lead to a noticeable increase in the efficiency of mixture separation up to 20% compared to a regular distributor with a fixed position of drip points. The effect of variation of the turn angles of the structured packing layers (20°, 45°, 70°, and 90°) on the efficiency of mixture separation was studied. Using a passive method to decrease the nonuniformity of distribution of local flow parameters by increasing the rotation angle of the structured packing layers in the column also leads to an increase in the separation efficiency. For the rotation angles of the packing layers (70–90 degrees, the HETP decreased in comparison with the rotation angle of 20° by (10–12) %.

### Credit author statement

Aleksandr Pavlenko: Supervision, Conceptualization, Investigation; Nikolay Pecherkin: Investigation, Data curation, Writing - original draft, Writing - review & editing; Vladimir Zhukov: Investigation, Data curation, Methodology; George Meski: Conceptualization, Resources; Patrick Houghton: Conceptualization, Data curation.

### 1. Introduction

Distillation is a widely used technology for mixture separation in various fields. The energy intensity of the rectification process is associated with high heat of vaporization of the components of separated mixtures. The large size of the distillation columns is determined by low energy efficiency. Despite the high energy consumption, the trend is that the size of distillation columns and their production are continuously growing, Olubić et al. [1]. Modern distillation columns can be up to 16 m in diameter and up to 90 m in height. The high level of energy

consumption in industrial distillation columns, which are the most energy-intensive devices in chemical technology, raises also the problem of climate change and global warming. Increasing the efficiency of separation processes even by several percent, and even more so by tens of percent, reduces significantly the environmental load. The authors of the cited article [1] give an overview of new developed equipment used in separation technology, as well as existing problems and challenges.

One of the effective ways to reduce energy consumption in the process of mixture separation is the use of columns with dividing walls (DWC), Kaibel [2], Lorenz et al. [3]. Dividing Wall Columns are intensively developing systems for separating multicomponent mixtures. Columns with dividing walls got a new impulse for intensive development due to principal advantage in comparison with traditional distillation systems – organizing the process of preliminary and subsequent separation of multicomponent mixtures in one casing [4–6].

Gas plants are a vital component of any refinery. These columns have always been operated using well-established distillation techniques. However, the concept of DWC can generate a leaner and efficient

\* Corresponding author.

E-mail address: [pecherkin@itp.nsc.ru](mailto:pecherkin@itp.nsc.ru) (N.I. Pecherkin).

system. DWCs lower both equipment cost and energy consumption of most configurations. These novel distillation columns can even provide better products as compared to the conventional columns in existing and future gas plant configurations, Kalita et al. [7].

Most of the publications on DWC topics are devoted to the development of technological schemes for the separation of various multi-component mixtures, control and management of processes [8–11]. Significantly fewer papers are devoted to the study of mass transfer, hydrodynamics and separation efficiency in non-cylindrical columns [12]. New experimental results on the efficiency of mixture separation for square and semi-cylindrical structured packing were obtained by Pavlenko et al. [13]. The efficiency of mixture separation on non-circular packing as a rule decreases in comparison with conventional round columns. The presence of angular zones in semi-cylindrical and square columns leads to greater nonuniformity of the liquid flow and concentration of volatile component over the column cross-section. In the paper [13], it was shown that for structured packing with a corrugation angle of 60°, the parameters characterizing the efficiency of separation and the hydraulic resistance in round and square columns are almost identical. The data obtained show that the main advantages of dividing wall columns (the energy and capital saving) can be achieved without losing the performance and capacity, characteristic of the columns with a circular cross-section. The importance of reducing energy consumption in the processes of mixtures separation is also due to the need to solve the global problem of reducing carbon dioxide emissions and achieving sustainable industrial development [1].

Along with the search for the ways of reducing energy consumption and capital costs of production of equipment for the distillation process, there is a search for the methods of quick evaluation of efficiency and increase in visibility by presenting data in the form of diagrams for total energy demand and total operating costs for various process options [14].

Taking into account the high importance of the intensification of mass transfer in the processes of distillation and absorption, the authors of [15] identify several main strategies for solving this extremely important problem. The most important of these are an increase in the mass transfer coefficients between the vapor and liquid phases at the interface, as well as the increasing of the active area of the contact surface. The authors analyzed various physical methods of intensifying mass transfer: microwave radiation, exposure to electric and magnetic fields, ultrasonic impact, high-gravity forces, etc. The authors consider some of them perspective (microwave field) and conduct experimental testing of different aspects on the laboratory facilities [16].

The most realistic and feasible at present is the use of new materials to create contact devices with a developed three-dimensional structure in order to increase the effective wetted area of the interface surface. Such materials include solid foams, porous ceramics, including metal-based materials. Structured packings made of ceramic or solid foam have a large porosity and specific area, the pressure drop on them is small. In Refs. [17], the authors studied the process of liquid spreading on SiC foam packing. It is shown that the renewal of the liquid and the spreading coefficients in the axial and radial directions are higher than those of the currently used KATAPAK packing. In Ref. [18], the results of tests of a structured CFP (Carbon Foam packing) with various specific area and corrugation angles are presented. The pressure drop on this packing is lower than on the ceramic packing SSCP (SiC foam packing). Solid foam packing shows higher separation efficiency at low column loads, probably due to more complete wetting of the surface. In Ref. [19], the results of a study of the hydraulic characteristics and separation efficiency of a novel SiC foam structured packing are presented. In laboratory conditions, the packing showed higher separation efficiency than Metal Foam and wire mesh packings. An important outcome obtained in this work is the test results of this packing in a pilot installation for air separation on O<sub>2</sub>/N<sub>2</sub> (oxygen/nitrogen) and O<sub>2</sub>/Ar (oxygen/argon) systems. The results show the perspective of using structured packings of Carbon foam and SiC ceramic foam in reactive

distillation and cryogenic distillation columns.

The low efficiency of mixture separation is often due to the complex and uneven nature of liquid distribution at the countercurrent vapor flow both in the individual channels of the structured packing, and along the cross-section and height of the column as a whole [20–22]. This problem is most important for large diameter columns [23]. In one of the last reviews of Spiegel [22], there is a brief look at the history of this issue. For the structured packing the problem of maldistribution and performance dates back to the mid-60s. Experiments of Pavlenko et al. [21] with initial uneven liquid distribution also showed significant deterioration in separation efficiency. In this work, to achieve initial maldistribution of irrigation, the access of liquid to the holes in the bottom of distributor, through which it flows onto a structured packing, was blocked by lowering the metal plates of a given shape. Thus, the large-scale zones, under which the structured packing was not irrigated, were organized in the liquid distributor. It was established in the work that namely the presence of large-scale non-irrigated zones above the packing is the cause of significant deterioration in the efficiency of mixture separation. At the same time, the pressure drop is much less dependent on uniformity of the packing irrigation. In practice, to reduce the negative impact of maldistribution of vapor and liquid flows on the separation efficiency, the liquid redistributors are installed in the high columns [24,25].

Pavlenko et al. in Refs. [26–30] proposed a new approach to the formation of initial irrigation of a structured packing in a distillation column using a controlled liquid distributor. This method allows creating an arbitrary pattern of drip points in the liquid distributor during operation of the distillation column, at which the more effective mixture separation is observed.

The authors of this paper studied systematically various aspects of maldistribution and separation efficiency relationship in the large diameter column [31–34]. Measurements of distribution of mixture composition and local liquid flow rates over the column cross-section were carried out under real conditions of mixture separation. It is shown that large-scale maldistribution appears within the packing despite uniform initial distribution. After numerous tests, in search of the cause, it was found that the vapor phase was unstable due to the inverse density gradient (negative stratification), see Pavlenko et al., [34].

In recent years (10–15 years), there has been stable understanding of the extreme importance of studying the distribution of vapor and liquid flows inside distillation columns on the packings of various geometries. In this regard, promising new modern research methods based on the use of computer tomography to obtain an instant spatial picture of liquid distribution over the structured packing are being developed [35–41]. At this stage of development of these methods, the research is limited to the columns of relatively small diameters and hydrodynamic experiments. The special value of these data will be achieved if the results on distribution of liquid film over the surface of corrugated sheets of the structured packing with different characteristics are obtained under the conditions of mixture separation in the presence of a countercurrent vapor flow.

The density of irrigation points in the distributors of distillation columns is determined by the nature of liquid spreading over the surface of the structured packings and their characteristics. Experiments with irrigation of the packing with single jets have shown that the flow of liquid along the sheets is more intensive than in the transverse direction, Hoek et al. [42]. To equalize liquid distribution across the cross-section, each layer of the packing in the column is turned relative to the previous one. Therefore, the angle of rotation of the packing layers can be used to change the distribution of liquid in the rectification columns.

A simplified method of estimating the required density of drip points for a column with the structured packing is presented by Cai [43]. The method is based on determining the wetted surface area in the volume of the packing when a single jet of liquid flows onto the upper surface of the layer. The liquid from a single drip point will be separated or spread onto

four neighboring sheets. For each sheet, liquid will flow not only within two corrugated channels but also downwards into the channels below. When using this approach, the minimum drip point density is proportional to the specific surface of the packing. This method does not fully take into account the influence of complex microstructure of the corrugated sheets, perforation, as well as liquid properties on the wetted surface area.

It is shown in Pavlenko et al. [44] that at local irrigation of single elements of a structured packing liquid flows mainly along the lower plane and in the valley of irrigated channels. The wetted surface area of the irrigated packing sheet depends on the liquid flow rate. Targeted studies of this process were carried out in Ref. [45–49]. It is shown in detail that distribution of a liquid nitrogen film over the surface of structured packing sheets widely used in cryogenic distillation columns for air separation depends on the corrugation angle, characteristics of the surface microtexture, perforation, and flow parameters. The effect of contact points on redistribution of liquid along the width of the packing plug with different directions of microtexture is shown. It is revealed that with a small degree of irrigation, all liquid flows only along the valley of irrigated channel, not falling into the underlying channels. With a greater degree of irrigation, there is an intensive flow of liquid into the lower channels, which ensures wetting and liquid flow on a significant part of the corrugated plate surface in the non-irrigated zone. Similar conclusions were also made in Ref. [41] based on processing of data on liquid spreading in the structured packing obtained by the X-ray computer tomography.

In recent works of Pavlenko et al. [50,51] liquid spreading over the surface of a structured packing was studied at irrigation by a single jet. Data on the influence of liquid flow rate in the jet and position of the irrigating jet relative to the packing (the central part of the plug, the edge of the plug) on liquid distribution under one packing layer were obtained.

An overview of the studies on distribution of local flow parameters during mixture separation, influence of various types of initial irregularity of irrigation and their influence on separation efficiency, as well as the use of passive and active methods for controlling these processes, carried out at IT SB RAS, is presented in this paper.

## 2. Experimental equipment and research methods

The description of experimental large scale distillation column was given in Ref. [31]. The binary mixture of freons R114 and R21 was used as the test fluid. Absolute pressure in the column was 3 bar. The physical properties of this mixture are suitable for simulating the processes that occur during cryogenic air distillation. To study the effect of the regime parameters on heat and mass transfer and hydraulic characteristics of the separation process, it is possible to set independently the vapor and liquid flow rates, as well as the ratio between them. Different structured packings were tested both at the same molar flow rate of liquid and vapor  $L/V = 1$  (total reflux), and at  $L/V > 1$ . The total height of structured packings installed in the column can reach 4 m. Taking into account the available inserts in the main column, the packing size can be 0.6 and 0.9 m for the round columns, 0.54 m × 0.54 m for the square column, and 0.8 m for the semi-cylindrical column [13,21,32]. To irrigate the packing, there are liquid distributors with different structure and density of drip points, including a controlled liquid distributor.

Concentration of the volatile component (R114) in the liquid and vapor phases is measured by a gas chromatograph. The sensitivity of the device for measuring the mole concentration of the volatile component is 0.001%. The measurement uncertainty of the mole concentration in the range of 0.3% (bottom of the column) is 2%. The measurement uncertainty of the mole concentration in the range of 50% (top of the column) is 0.5%.

Hydraulic losses of the structured packing, distribution of the local liquid flow rate and mixture composition were also measured. The uncertainty of measuring the pressure and pressure drop is 1%. Methods

for measuring these parameters are given in Refs. [31,32, and 34]].

Data on separation efficiency are presented as dependences of the height of transfer unit (HTU) and height equivalent to the theoretical plate (HETP) on the vapor load ( $F$ -factor). The values of HTU and HETP are calculated by the measured mixture composition in the vapor and liquid phases at the inlet and outlet of the column and vapor-liquid equilibrium data. Uncertainty in calculating HETP and HTU values – 2.7%. The data on the efficiency of the mixture separation and the pressure drop on the structured packing are presented in the form of corresponding diagrams on the flow parameters, in particular on the  $F$ -factor, which is determined by the superficial vapor velocity. The uncertainty of definition  $F$ -factor is 4%.

## 3. Distribution of local flow parameters on the structured packing

Liquid distribution over a structured packing at mixture separation is determined by many factors. They include, first of all, the characteristics of distributors such as the number of drip points and their arrangement with respect to the individual elements of the regular packing. The liquid load and geometric parameters of the structured packings determine the patterns of liquid film flow in the unit channels of the packing. The number of packing layers and angle of their rotation determine liquid distribution both over the cross-section and height of the column.

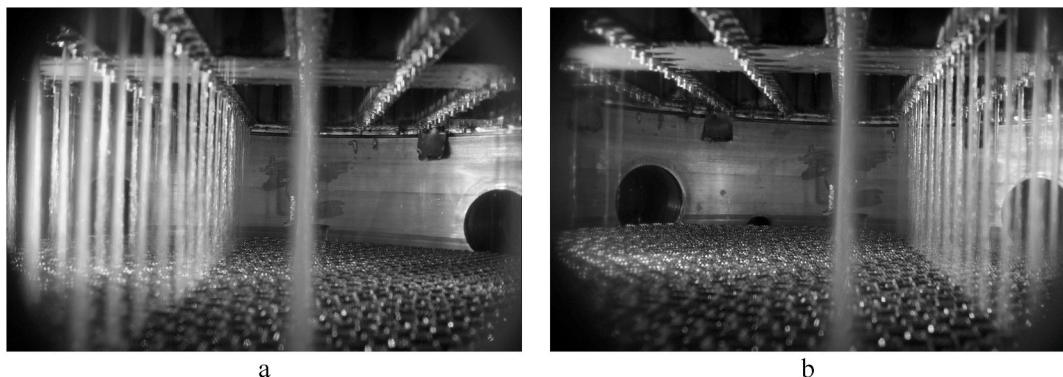
### 3.1. The effect of initial liquid distribution on separation efficiency

The importance of setting the initial uniform irrigation of regular packings was noted almost simultaneously with the beginning of a wide industrial use of structured packings in the processes of absorption and distillation [52,53]. Liquid distributors of distillation columns are designed to ensure uniform initial distribution of liquid. Their main characteristics are the density and pattern of irrigation points. The influence of the number of irrigation points on distribution of liquid over the packing cross-section and column wall, as well as on the separation efficiency, was studied in Ref. [32,33]. As the height of the packing bed increases, the proportion of liquid flow falling down the column wall increases substantially.

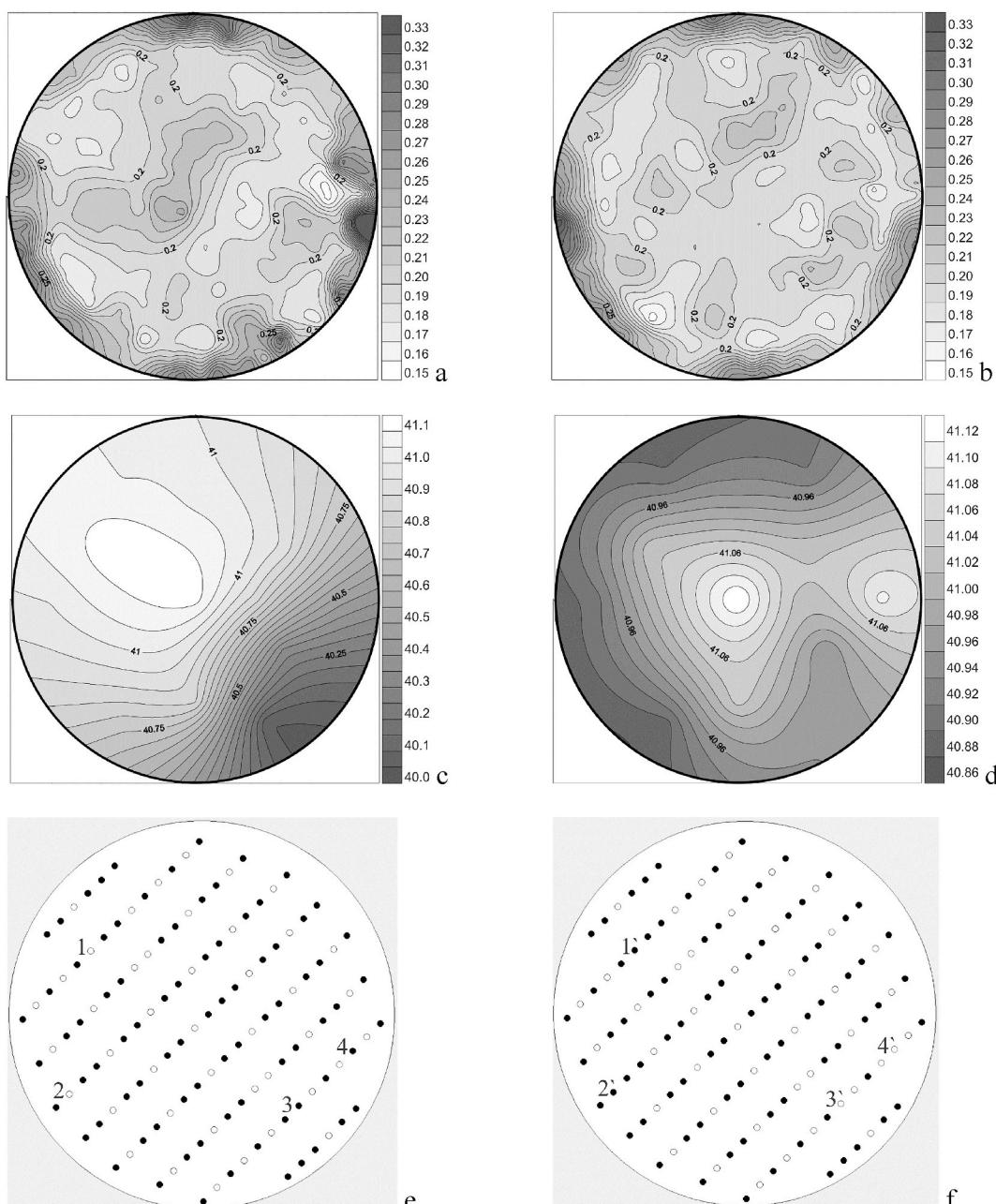
A decrease in the density of irrigation points from 449 1/m<sup>2</sup> to 230 1/m<sup>2</sup> at their uniform distribution over the cross-section does not influence the separation efficiency [33]. The creation of large-scale zones with a reduced liquid flow rate at the column inlet has a more significant effect on these parameters. A decrease in separation efficiency at such uneven irrigation can be up to 50% [21,33]. The influence of maldistribution on separation efficiency depends on the magnitude of irregularity. When the non-irrigated area of the column cross-section increases from 10% to 20%, the separation efficiency decreases by (30–50) %. Irregular irrigation of the packing contributes to the appearance and development of temperature nonuniformity in the column, which extends to the entire height of the packing bed without changing its location. The location of zones with a high liquid temperature (and, consequently, low concentration) corresponds to the location of zones with poor packing irrigation. The development of nonuniformity of the temperature and concentration fields in the column occurs during stabilization of column operation due to increasing gradient of vapor phase density over the packing height [34].

### 3.2. Dynamic control of initial distribution of liquid in the separation column

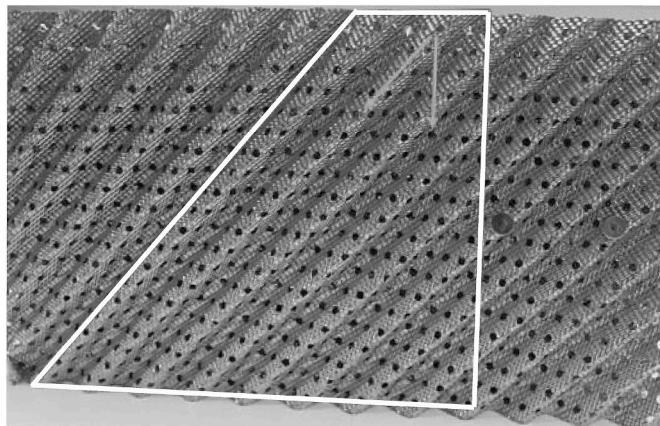
To study the dynamic effect of liquid distribution and correct initial irrigation of the structured packing, a controlled liquid distributor was developed for the experimental distillation column [26,27]. The controlled liquid distributor is a cylindrical vessel with a flat bottom, where 126 removable nozzles with different diameters of holes are evenly mounted. An electromagnet is installed above each nozzle, and



**Fig. 1.** Jet flow from the liquid distributor to the structured packing at periodic switching of valves in the controlled liquid distributor [26,27].



**Fig. 2.** Distribution of liquid flow rate (a, b) and temperature (c, d) at the packing outlet and location of active drip points in the distributor (e, f) at initial packing irrigation (left pictures) and after correction (right pictures);  $F_v = 1.1 \text{ Pa}^{1/2}$  [54].



**Fig. 3.** Surface area wetted by a single drip point, Cai [43].

the spherical end of this magnet armature is a shutoff valve for the nozzle. When de-energized, all valves are closed. Developed software allows independent control of the state of each valve during column operation, thus setting the pattern of active points of structured packing irrigation. An example of periodic irrigation of left (a) and right (b) halves of the column is shown in Fig. 1 [26,27].

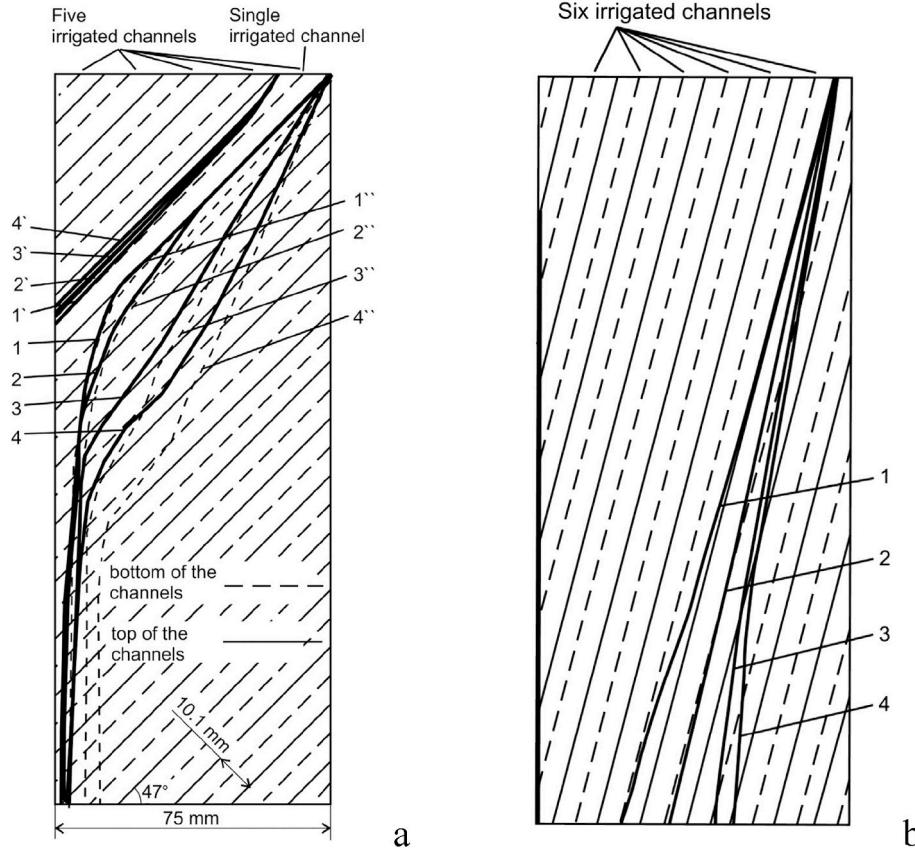
Possibilities of control of initial liquid distribution using this distributor in order to increase the separation efficiency are shown in Fig. 2 [30,54,55], where  $F_v = \sqrt{\rho_v} U_v$ ,  $\rho_v$  is the density of vapor;  $U_v$  is superficial vapor velocity. Data on local liquid flow distribution on Fig. 2 are presented in ml/s; data on temperature - in degrees Celsius ( $^{\circ}\text{C}$ ). The corresponding scales are located to the right of the topograms. The location of active drip points is corrected based on the distribution of local liquid flow rate (Fig. 2a) and temperature distribution of the liquid

phase under the packing (Fig. 2c). During the correction, some closed holes (open circles 1 and 2, Fig. 2e) in the controlled liquid distributor were opened, and some of the previously opened holes (dark circles 3 and 4) were closed (Fig. 2f). As a result of correction of the initial packing irrigation, liquid distribution under the packing became more uniform (Fig. 2b). Changing the position of only two active drip points (1' and 2' instead of 3 and 4, Fig. 2 e, f) changed significantly the distribution of liquid flow rate (Fig. 2b), temperature and mixture composition under the packing (Fig. 2d). The standard deviation of the liquid flow rate decreased from 14.5% to 11.7%, temperature difference in the packing layer decreased from 1.1 K to 0.3 K, and the height of transfer unit HTU decreased approximately by 20%. A decrease in the standard deviation of the local liquid flow rate indicates that its distribution over the column cross section becomes more uniform. Reducing maldistribution of the liquid leads to a decrease in the uneven distribution of the concentration of the volatile component, as indicated by the equalization of the temperature of the liquid phase over the cross section (temperature difference in the packing layer decreased from 1.1 K to 0.3 K). As a result of a decrease in the spread of local values of the L/V ratio and the value of local concentration head, an increase in the efficiency of separation of the mixture is observed, which is expressed in a decrease in the value of HTU to 20%.

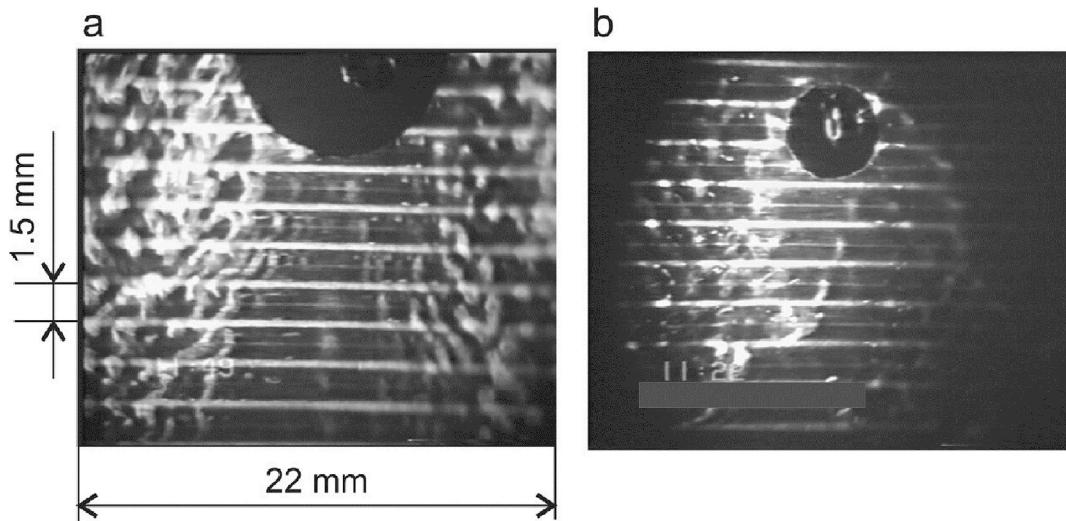
### 3.3. Liquid spreading over the surface of structured packing sheets

As it was noted above, Cai [43] has determined the surface of the packing, which can be wetted with liquid from a single drip point, based on the simplest model. It consists of two parts, a rectangular cuboid and a triangular prism, Fig. 3. The right boundary of the wetting region in the form of a vertical line can be obtained under the assumption of an intensive flow of liquid over edges of ribs and through the holes.

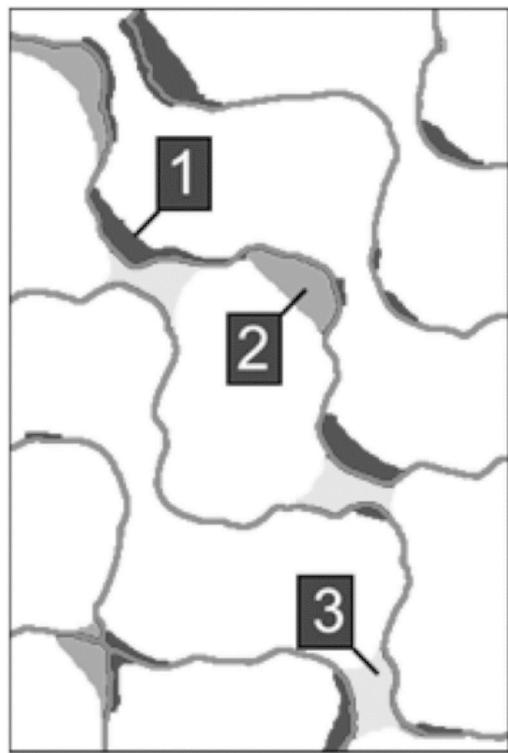
Data on spreading of liquid nitrogen over the surface of single sheets



**Fig. 4.** The boundaries of liquid spreading zones on the plates with different angles of rib inclination: a –  $45^{\circ}$ , b –  $75^{\circ}$  [49].



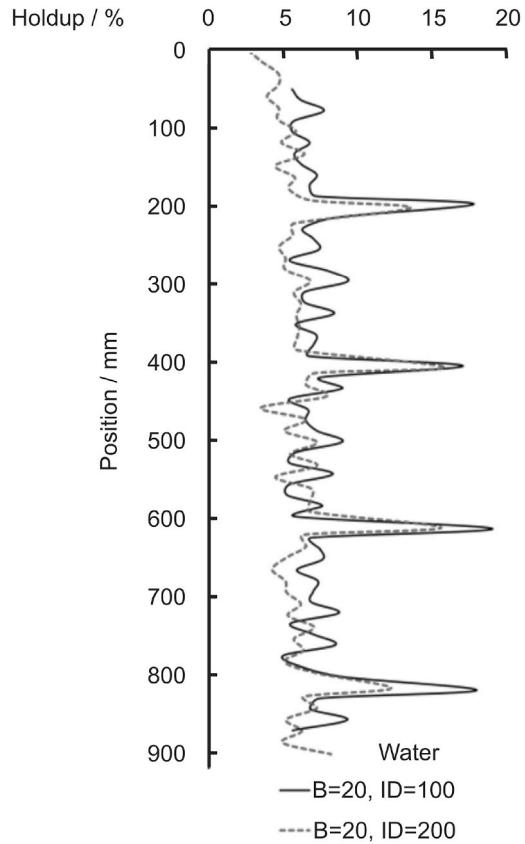
**Fig. 5.** The flow of liquid nitrogen on perforated sheets with microtexture,  $Re = 215$ . Diameter of holes: a – 10 mm, b – 4 mm; [49].



**Fig. 6.** Examples of flow of liquid films (1), rivulets (2) and bridges (3) [41].

of the structured packing obtained by Pavlenko et al. show a much more complex character [44–49]. The wetted area for one irrigated channel is located between the upper (1'-4') and lower (1–4) borders (Fig. 4a).

Lines (1"-4") indicate the lower boundaries of the wetting zone for five irrigated channels. Lines (1, 1', 1'') correspond to the minimum Reynolds number of the film,  $Re = 65$ , and lines (4, 4', 4'') correspond to the maximum  $Re$  number,  $Re = 215$ . At the low Reynolds numbers, the liquid flows along the channels, and at high flow rates, the liquid overflows through the crests of the ribs. Analysis of results obtained on the sheets with different microtextures, corrugation angles, and diameters of holes shows a significant effect of these parameters on the surface wetting [39,48]. The wetted area on the surface of the packing with an angle of ribs inclination of  $75^\circ$  occupies almost the entire surface

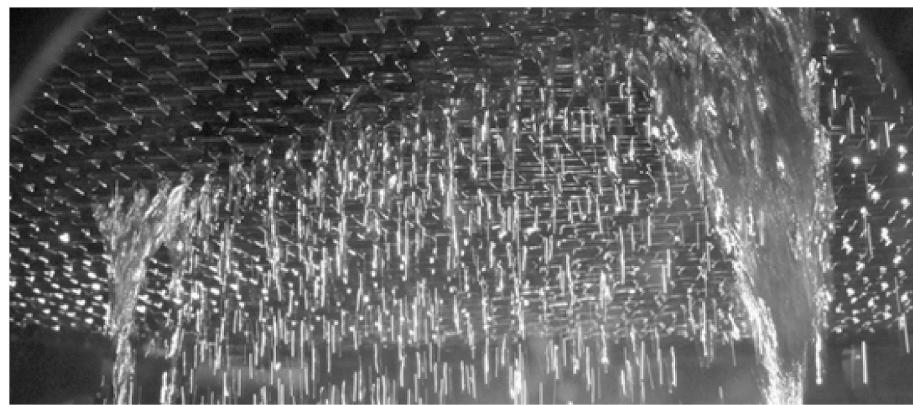


**Fig. 7.** Holdup profiles along the packing bed Mellapak 500Y at  $F = 0$  and irrigation rate  $B = 20 \text{ m}^3/(\text{m}^2 \text{ h})$  [41].

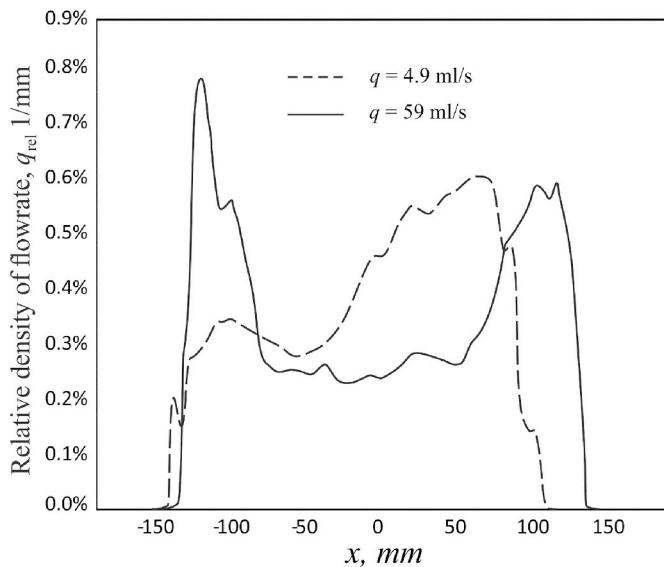
of the plate (Fig. 4b). Lines (1, 2, 3, 4) correspond to Reynolds numbers  $Re = 57, 132, 177$  and  $317$  in this figure.

Examples of fluid flow around the holes on the packing sheets with microtexture are shown in Fig. 5 [44,49]. The part of liquid flowing through the holes to the non-irrigated side depends on the flow rate of liquid and size of the holes. The size of holes also affects significantly the structure of the wavy surface of the liquid film.

The paper of Wehrli et al. [41] presents data on the liquid flow on the surface of sheets of the structured packing obtained by X-ray computer



**Fig. 8.** Liquid flow in the Sulzer 500X packing. The flow rate through the drip point is 59 ml/s [51].



**Fig. 9.** Distribution of the liquid flow rate under the packing for different irrigation rates [51].

tomography. On Fig. 6 it can be seen that the flow of liquid on the surface of the structured packing takes various forms, including flow of the liquid film at the corners of the channel, which coincides with the above observations by Pavlenko et al.

The wavy shape of the liquid holdup profiles observed in Fig. 7 is due to packing sheets perforation, characterized by rows of holes arranged uniformly at defined axial positions, Wehrli et al. [41]. This figure also shows the periodic zones of 15–20 mm in size, where the liquid holdup is 3–4 times higher than the average holdup in the column. Authors do not provide a satisfactory explanation of this effect. The figure shows that the position of the holdup maxima coincides with the boundary between the individual layers in the packing bed and this is most likely caused by accumulation of liquid at the junction of layers when it flows from the overlying layer to the underlying one. An increased liquid amount at the boundary between the layers can lead to an increase in the pressure drop component at the boundary not only due to a change in the direction of the vapor phase flow, but also due to an increase in liquid holdup in this zone.

The authors of the present study have measured liquid distribution under one layer of the structured packing at its irrigation by a single jet [50,51]. An example of a liquid flow on the packing with corrugation angle of 60° is shown in Fig. 8. At a large flow rate of liquid, powerful flows are observed along the edges of the region bounded by the inclined channels of adjacent irrigated sheets of the packing plug.

**Table 1**

The range of variation of the layer turn angle [57].

The angle of layers rotation	20°	45°	70°	90°
The total turn angle of layers in the packing bed, $\pi$ rad	1.11	2.5	3.9	5.0
The total turn angle of layers on the unit packing height, $\pi$ rad/m	0.49	1.1	1.71	2.2

The measured values of the local flow rate of liquid under the packing obtained in these experiments are shown in Fig. 9. It is seen from the diagram that liquid spreads at a distance of  $\pm 130$  mm from the location of irrigation point within the sector formed by the inclined channels of neighboring corrugated sheets. The results obtained in these experiments show a strong influence of fluid flow rate on its distribution. At higher flow rates a portion of liquid flowing down in the lateral channels of the irrigation sector increases significantly. Currently, the effect of velocity of the countercurrent vapor flow on the liquid flow over the surface of structured packing is being studied.

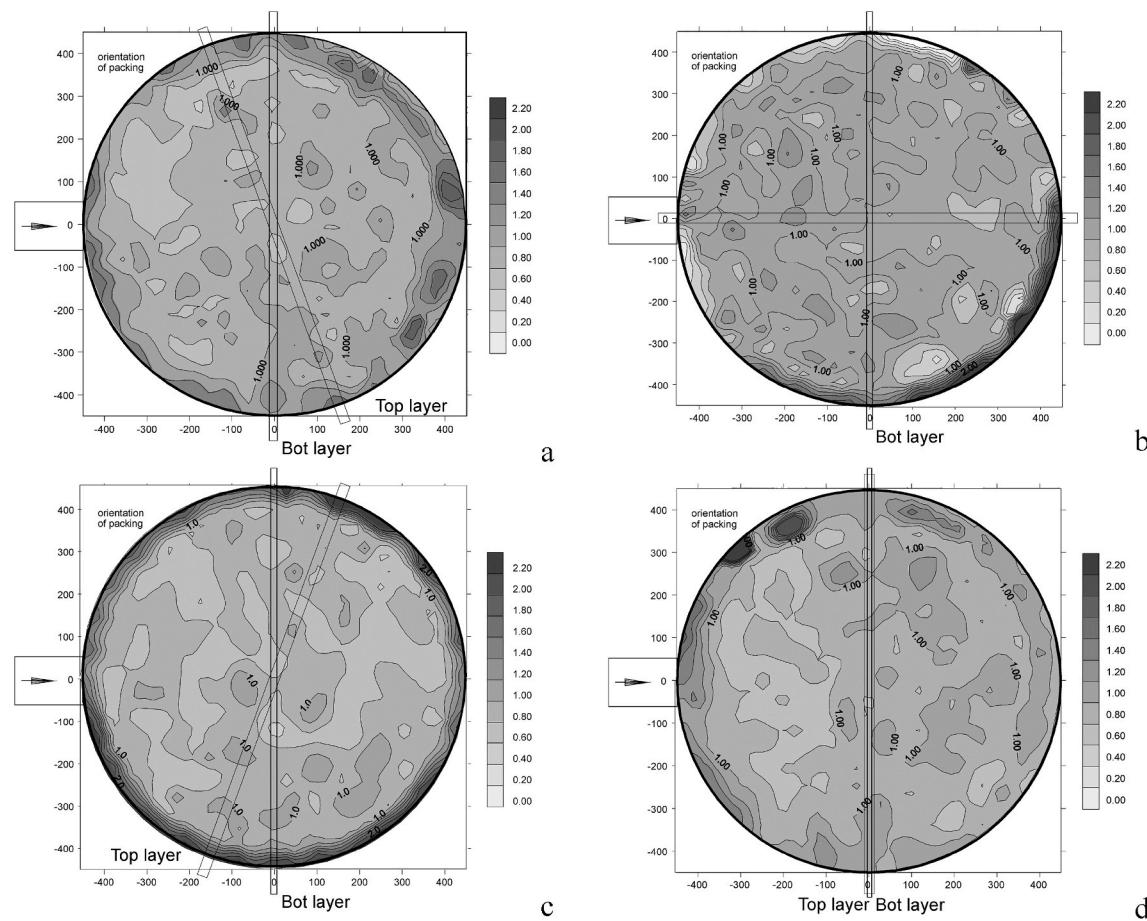
#### 4. The effect of turn angle of packing layers on distribution of local parameters

One of the passive methods of influencing the distribution of liquid over the column cross-section is a change in the turn angle of layers of the structured packing. In the experiments of Pavlenko et al. [33] with initial maldistribution, it was shown that the position of zones with increased or reduced liquid flow rate at a small turn angle of layers (20°) is kept at a large height of the packing in the column, what is accompanied by a decrease in separation efficiency in comparison with turn angle of 70°.

Further experiments to investigate the effect of turn angle of packing layers on distribution of liquid in the column were carried out at uniform packing irrigation and total reflux [56,57]. The drip point density of liquid distributor was 450 1/m<sup>2</sup>, approximately. Eleven packing layers each of 0.2 m height with a specific area of 500 m<sup>2</sup>/m<sup>3</sup> and corrugation angle of 45° were installed in the column with a diameter of 0.9 m. The values of the turn angles of layers are given in Table 1. The full turn angle of the packing layers characterizes the intensity of liquid redistribution over the cross-section and height of the column.

The distribution of fluid flow at the outlet of the packing bed is highly uneven. At the packing periphery the zones with an increased liquid flow rate are always observed, Fig. 10. The distribution of relative liquid flow rate is the most uniform for the layer turn angles of 70 and 90°.

The distribution of mixture composition for different turn angles of the packing layers corresponds generally to the distribution of the local flow rate, Fig. 11. Maximal maldistribution of mixture composition under the packing bed is observed for small turn angles (Fig. 11 a, b) and



**Fig. 10.** Distribution of local liquid flow rates under packing bed,  $F_v = 1.4 \text{ Pa}^{1/2}$ . Turn angle of layers: a – 20°; b – 45°; c – 70°; d – 90° [57].

minimal ones of 70° (Fig. 11 c).

##### 5. The effect of turn angle of packing layers on separation efficiency

The results of experiments on the study of the effect of arrangement of the structured packing layers on separation efficiency are shown in Fig. 12. For all experiments a significant decrease in separation efficiency is observed with a decrease in the  $F$ -factor less than 1.2–1.3. For example, for the layers rotation angle of 70°, the HETP increases from 180 to 220 mm at decreasing of the vapor load, i.e. separation efficiency is reduced by about 20%. At the low vapor load of the column, the Reynolds number of the liquid film on the surface of the structured packing becomes very small. At the same time, along with a decrease in the mass transfer coefficients in the liquid film, the proportion of the non-wetted surface of the packing may increase with a decrease in irrigation density [13]. A decrease in local mass transfer coefficients in the liquid phase, as well as a decrease in the effective area of the mass transfer surface, cause a significant increase in HETP with a decrease in the column load. Under these conditions, the role of more uniform distribution of liquid in the distillation column for increasing the separation efficiency increases significantly.

At low vapor load ( $F_v \approx 1 \text{ Pa}^{1/2}$ ), an increase in the rotation angle of the layers from 20° to 90° result in an increase in separation efficiency by about 10%. At higher vapor load ( $F_v \approx 1.3 \text{ Pa}^{1/2}$ ), a more significant increase in separation efficiency is observed, HETP decreases by 15–18%. The HETP data (Fig. 13) show the increasing of separation efficiency with increasing turn angle of layers from 20° to 90° approximately by 10%.

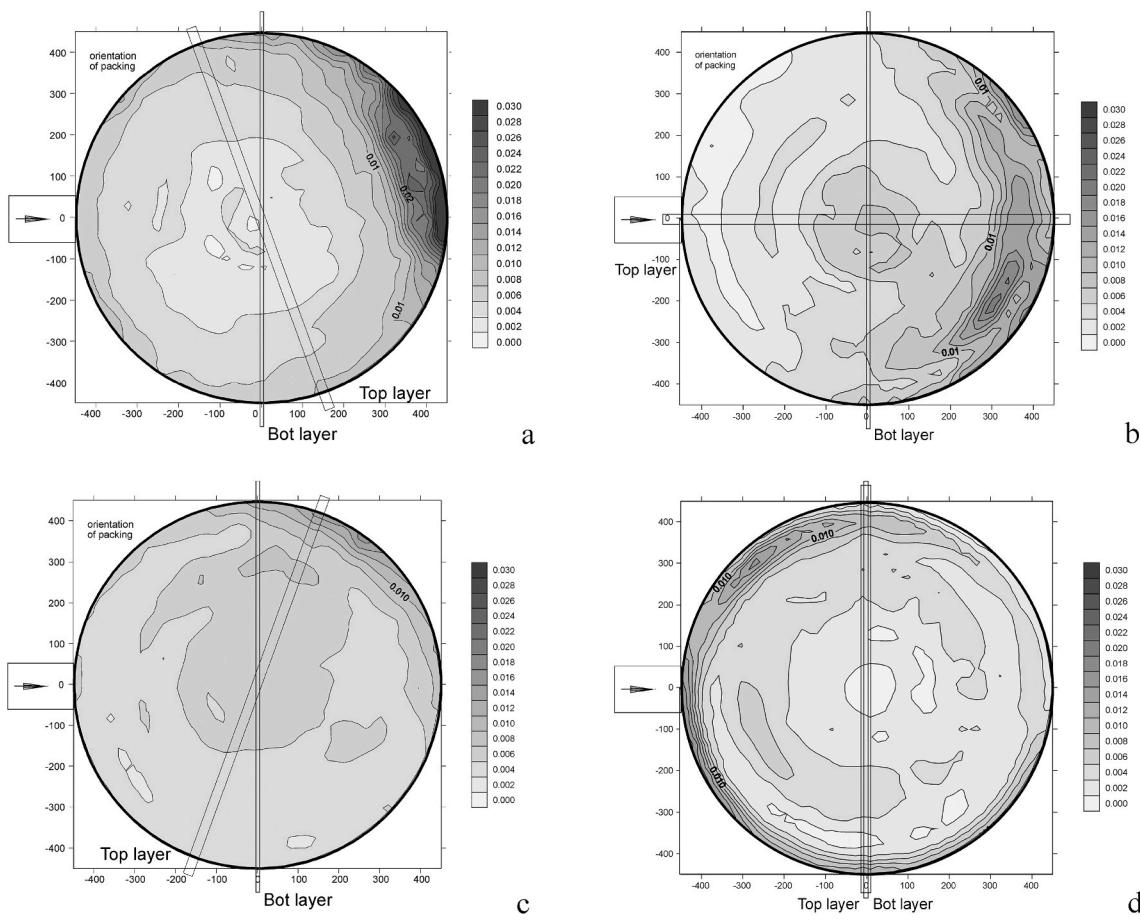
As shown in Table 1, at large angles of rotation of the layers, the

lower layers in the column are rotated relative to the top layer by 4–5 full turns, and for small ones, respectively, only 1–2 full turns. At each rotation, the layers, being a self-distributor for the underlying layers, more uniformly distribute the liquid over the cross section of the column. With a more uniform distribution of the liquid, with other conditions remaining the same, there is an increase in the efficiency of separation of the mixture. However, due to other reasons for the development of the maldistribution of vapor and liquid flows in the column, namely, uneven distribution of liquid over the surface of the packing, edge effects, development of instability during countercurrent vapor and liquid flow and many others, it is impossible to achieve an ideal distribution of liquid even in very high columns. Topograms of the distribution of local parameters at the outlet of the column in our experiments always show the presence of irregularities, the magnitude of which correlates with the separation efficiency. The HETP data (Fig. 13) show the same trend of increasing separation efficiency with increasing angle of layers turn as the data on distribution of local fluid flow and mixture composition (Figs. 10 and 11). Hydraulic resistance of a structured packing is practically independent of the turn angle of packing layers [33,57].

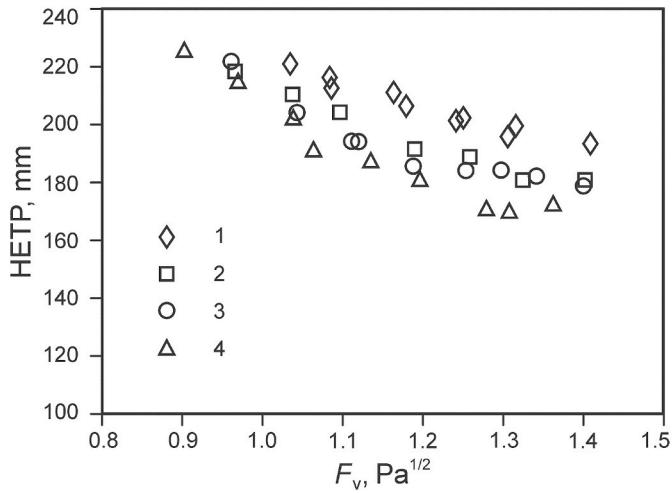
Results of experiments with different flow regimes, initial irrigation, and height of structured packing have shown that efficiency of mixture separation is closely related to the distribution of liquid in the column. It is shown that the most optimal turn angles of packing layers from the view point of mixture separation efficiency are in the range of (70–90)° because of more intensive mixing of liquid in the distillation column.

##### 6. Conclusion

The paper presents an overview of the latest studies on distribution of local flow parameters during separation of mixtures, liquid spreading



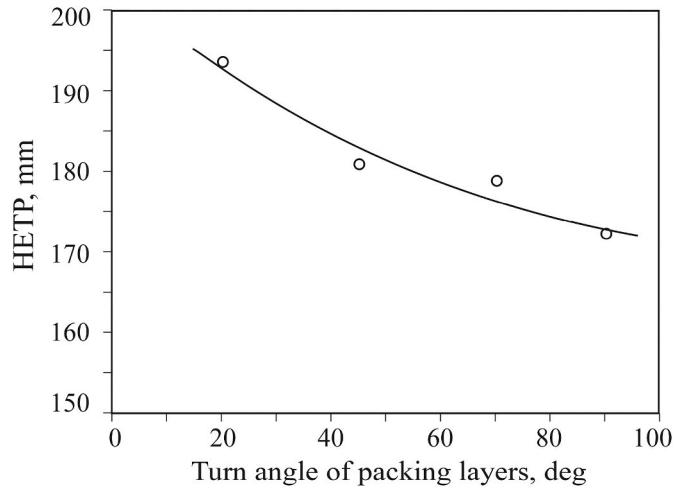
**Fig. 11.** Distribution of mixture composition in liquid phase under the packing bed,  $F_v = 1.4 \text{ Pa}^{1/2}$ . Turn angle of layers: a – 20°; b – 45°; c – 70°; d – 90° [57].



**Fig. 12.** Dependence of HETP on the vapor load. Turn angle of layers: 1–20°; 2–45°; 3–70°; 4–90° [57].

over the surface of the structured packing, the study of the influence of various types of initial irrigation nonuniformity and their influence on separation efficiency, as well as the experience of using the methods of passive and active control of these processes developed at IT SB RAS.

The results of experiments on the influence of flow parameters and characteristics of microtexture, perforation and contact points on the liquid film flow over the surface of structured packing are analyzed. It is shown that the amount of liquid held in the channels of the structured



**Fig. 13.** Effect of the turn angle of the structured packing layers on the efficiency of mixture separation,  $F_v = 1.4 \text{ Pa}^{1/2}$  [57].

packing depends significantly on the load of liquid.

The distribution of flow parameters over the cross-section and height of the distillation column can be controlled by the passive methods (arrangement of packing elements, initial packing irrigation, installing the vapor and liquid redistributors in the column). It is shown that the most optimal turn angles of packing layers from the view point of mixture separation efficiency are in the range of (70–90)°.

Using the controlled distributor of liquid for changing the number

and order of the active drip points during separation process can be an effective tool to destroy maldistribution in the column and increase separation efficiency.

## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

## Acknowledgments

The studies have been carried out within the joint project of Kutateladze Institute of Thermophysics SB RAS and Air Products Inc., USA, and under the state contract with IT SB RAS, Project No. III.18.2.3.

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