Dynamics of a change in the local density of liquid flow rate and efficiency of mixture separation at periodic irrigation of the structured packing

A N Pavlenko, V E Zhukov, N I Pecherkin, A D Nazarov, E Yu Slesareva, X Li, H Sui, H Li, and X Gao
Kutateladze Institute of Thermophysics SB RAS, Novosibirsk, Russia
E-mail: pavl@itp.nsc.ru; zhukov@itp.nsc.ru

Abstract. The use of modern structured packing in the distillation columns allows much more even distribution of the liquid film over the packing surface, but it does not completely solve the problem of uniform distribution of flow parameters over the entire height of the packing. Negative stratification of vapor along the packing height caused by different densities of vapor mixture components and higher temperature in the lower part of the column leads to formation of large-scale maldistributions of temperature and mixture composition over the column cross-section even under the conditions of uniform irrigation of packing with liquid. This work presents the experimental results obtained at periodic packing irrigation with total blocking of the spray nozzle. The experiments were carried out in the distillation column with the diameter of 0.6 m on 10 layers of the Sulzer 500X packing with the total height of 2.2 m. The mixture of R-21 and R-114 was used as the working liquid. To irrigate the packing, the liquid distributor with 123 independently controlled solenoid valves overlapping the holes with the diameter of 3.6 mm, specially designed by the authors, was used. Response of the column to the action of liquid distributor was observed in real time according to indications of 3 groups of thermometers mounted in 3 different cross-sections of the column. The flow meter located at a fixed point of the column cross-section allowed registration of dynamics of a change in the local flow rate density of liquid flowing from the packing.

1. Introduction
Distillation columns are widely used in oil refining, chemical, and food industries to separate the mixtures of various liquids. The use of modern structured packings, including the porous and mesh ones [1-7], allows much more even distribution of the liquid film over the packing surface, but it does not completely solve the problem of uniform distribution of flows over the packing. Negative stratification of vapor along the packing height due to different densities of vapor mixture components and higher temperature in the lower part of the column leads to formation of large-scale maldistribution of temperature and mixture composition over the column cross-section, even at uniform packing irrigation with liquid [1]. Experimental data on minimization of large-scale maldistribution by means of static and dynamic changes in the structure of the drip point pattern were presented in [8-9]. In this paper, the results of experiments obtained with periodic packing irrigation with complete blocking of the spraying nozzle are presented.
2. Set-up and method description
The experiments were carried out on a large-scale research setup “Large Freon Column”, whose detailed description is given in [1,10]. The structured Sulzer 500X packing with the height of 2.2 m, consisting of 10 layers, was installed in a distillation column with the diameter of 0.6 m. The mixture of freons R-21 and R-114 was used as a working liquid; characteristics of this liquid were selected to simulate separation of cryogenic mixtures. A liquid distributor specially developed by the authors was used to irrigate the packing. The distributor had 123 independently controlled solenoid valves, which blocked the holes with the diameter of 3.6 mm. The valves were controlled both manually and in automatic valve switching mode, according to any predetermined algorithm. The time shift of switching was 1 s. The response of the column to the action of the liquid distributor was observed in real time by indications of thermometers mounted in 3 different column cross-sections (distance $h$ from low edge of packing) along the packing height. In each section, 16 thermometers were installed. Their readings were displayed on the computer monitor in the form of topograms. Thermometers were installed in the lower (2 layers from the packing bottom), middle and upper (2 layers from the packing top) cross-sections of the packing. Information displayed on the monitor made it possible to evaluate both the structure of large-scale temperature maldistribution and value of this maldistribution. The value of maldistribution was characterized by standard deviation of temperature, calculated by the readings of 16 thermometers in each cross-section. Under the lower packing layer, a flow meter was mounted to measure the flow rate density of liquid flowing out of the packing. By means of a two-coordinate device, the flow meter was positioned at the predetermined points of the column cross-section. The schematic diagram of the flow meter is shown in figure 1. Cylindrical collector $l$ with the diameter of 28 mm is connected with permanently flooded escape channel 2. Pulsed thermal generator of vapor bubbles 3 and registering optical coupler 4 are located in the channel. Pulsed heat generation on a spiral forms a vapor phase, which is carried away by a liquid flow in the channel and recorded by an optical coupler. The flow velocity in the channel and, consequently, the flow rate of liquid were determined using these data. Before the experiments, individual calibration of the flow meter was carried out.

![Figure 1](image.png)  
**Figure 1.** Schematic diagram of liquid flow meter. $l$ – receiving collector; $2$ – escape channel; $3$ – vapor phase generator; $4$ – registering optical coupler.

During the experiment, the irrigating valves were periodically blocked. The times of valve opening and closing varied between 5 and 30 seconds. The density of liquid flow rate was measured at a fixed point of the column cross-section (coordinates: $x = 120$ mm, $y = 200$ mm) directly under the packing at reduced vapor velocity $K_v = 0.033 - 0.035$ m/s.

The experiments were carried out with the fixed number of drip points in the liquid distributor. The initial drip point pattern corresponded to uniform packing irrigation. When column operation was stabilized in the quasi-stationary state, the program of liquid distributor valve control was switched on. All valves were closed for time $t_2$, and then opened for time $t_1$. Later, the valves were closed and opened periodically with period $t = t_1 + t_2$. The state of liquid distributor valves and their arrangement are shown in figure 2.
3. Results and discussions

The experiments were performed at \( P = 3 \) bar under the reflux conditions at reduced vapor velocity \( K_v = 0.03 - 0.035 \) m/s. Under the condition of uniform packing irrigation (Uniform pattern, figure 2a), large-scale maldistribution of mixture concentration and, consequently, temperature was formed throughout the packing cross-section along the entire height of the column. As a rule, the presence of diametrically located poles of high and low temperature is characteristic of the structure non-uniformity revealed in the experiments. The topograms of temperature distribution in three different packing cross-sections along the column height are shown in figure 3 for Uniform drip point pattern at reduced vapor velocity \( K_v = 0.03 \) m/s. For each topogram, the value of standard deviation \( StDev \) is shown by indications of all thermometers mounted in the given packing cross-section.

As it follows from the analysis of presented topograms, large-scale maldistribution spreads over the entire height of the packing and has the areas of increased and decreased temperature in approximately the same parts of the column cross-section. The region of decreased temperature (dark area in topograms) corresponds to the region with higher concentration of volatile component and, as a rule, higher density of liquid flow rate in the given cross-sectional projection.

Evolution of temperature distribution in the packing cross-section at the level of the 5th layer under the conditions of periodic irrigation is shown in figure 4 on the time scale (\( \tau \)) of one period. As it can
be seen from the above-mentioned topograms, the large-scale maldistribution does not collapse at a short-term stop of packing irrigation, but only the degree of maldistribution, characterized by the value of standard deviation of temperature in the cross section, changes.

Figure 4. Dynamics of a change in temperature distribution in the column cross-section at the level of the 5th packing layer at periodic irrigation. \( t_1 = 30 \text{ s}; t_2 = 10 \text{ s. a) } - \tau = 1 \text{ s, StDev} = 0.81 \text{ K; b) } - 6, 1.03; c) - 12, 1.12; d) - 17, 1.14; e) - 23, 1.14; f) - 28, 1.10; g) - 33, 0.93; h) - 39, 0.81; i) - 44, 0.69. \)

The diagram of the density of local liquid flow from the packing at the point of column cross-section with coordinates \( x = 120 \text{ mm, } y = 200 \text{ mm} \) is presented in figure 5. These coordinates correspond to the region of reduced temperature in the packing cross-section. As it can be seen from the diagram, the local density of liquid flow rate under the packing changes periodically in accordance with the period of packing irrigation. In this case, the nature of a change in the flow rate density is
close to the sinusoidal one, while the law of packing irrigation by liquid is close to the step-wise one. The time for switching-on of all the valves is 3 s, the shutdown is almost instantaneous (about 100 ms).

Figure 5. Diagram of a change in the flow rate density above the packing at the fixed point of cross-section at periodic irrigation. $t_1 = 10$ s; $t_2 = 10$ s; $2 - 20; 10; 3 - 30; 10; 4 - 20; 20; 5$ – uniform irrigation.

Under different regimes of periodic irrigation, different maldistributions of the local density of the liquid flow rate were observed at the packing outlet. As it can be seen from the diagram, the greatest maldistribution of the flow was observed in cases 1 and 4. In these cases, the ratio of times of valve closing and opening $t_2/t_1$ equals 1. Dependence of the efficiency of mixture separation at periodic packing irrigation is shown in figure 6.

Figure 6. Height of transfer unit ($HTU$) vs. relative period $t_{rel} = t_2/t_1$. The error of HTU measurement is less than 10 mm. $K_v = 0.033 - 0.035$ m/s. Packing 500X, $d = 0.6$ m, $h = 2.2$ m, $t_2 = 1 - 5$ s; $2 - 10; 3 - 20$.

As it can be seen from the above diagram, complete periodic stop in irrigation of the Sulzer 500X packing leads to deterioration of separation efficiency. If the time of irrigation stop is commensurate with the time of liquid film flow over the packing (about 10 s), then the efficiency of separation
depends almost linearly on the value of relative period \( t_{\text{rel}} = t_2/t_1 \). In the case of a longer time of irrigation stop \((t_2 = 20 \, \text{s})\), the efficiency of separation deteriorates significantly.

### 4. Conclusions

According to the studies performed, large-scale maldistribution does not collapse at short-term periodic stop of packing irrigation, but only the degree of this maldistribution changes. In the studied range of irrigation times \( t_1 \) and flow blocking \( t_2 \), the higher the ratio \( t_2/t_1 \), the greater maldistribution of the liquid flow rate density at the packing outlet. The conducted experiments showed that complete periodic stop of irrigation of the Sulzer 500X packing with the diameter of 0.6 m in the studied range of times of valve opening and closing does not lead to the improvement of separation efficiency of the column. The higher the irrigation period corresponding to the closed state of the valves, the greater the change in the density of liquid flow under the packing and the lower the efficiency of mixture separation of the column.

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