USE OF SPIRAL INSERTS IN THERMAL-DIFFUSION COLUMNS

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The influence of spiral inserts on the efficiency of the fractionation of petroleum products in thermodiffusion columns is investigated.

Beginning with the 1950s the thermal-diffusion method of fractionation has been widely used both in analytical research to separate narrow fractions of petroleum products in order to determine their chemical structure [1-10] and to study the possibility of its application in technological processes of obtaining petroleum oils with valuable new properties [11-14]. The apparatus used for this purpose has been described in a number of publications [15-18].

Various structural modifications of thermal-diffusion columns have been suggested to increase their efficiency (a column with horizontal partitions [19], a flat inclined column [15, 20], a column containing a packing [21]) in which the common idea of decreasing the velocity of convective flows is employed. In this case, although one is able to increase the degree of separation in the nonsampling mode of operation, in the sampling mode the productivity of such columns is lower than that of ordinary columns with an open gap.

In 1962 one other method was suggested for intensifying the fractionation process in a thermal-diffusion column [22], consisting in the inner cylinder of the column being wound with a wire spiral fully spanning the working gap. According to the data in [23], which used this method, the introduction of spirals not only decreased the time it takes to reach the steady state in the nonsampling mode of column operation but also considerably increased the degree of fractionation of a cetane — decalin mixture in comparison with a column with an open gap. We note that further tests on the fractionation of the kerosene fraction of camomile oil were carried out by these authors only in a column with a spiral insert and were not compared with tests in a column with an open gap. Such a comparison is also absent in [8, 9]. The sole paper in which a column containing a spiral was studied only in the sampling mode is the one of Yeh and Ward [24]. In it the fractionation of n-heptane — benzene and toluene — isobutyl alcohol was studied as a function of the winding angle of the wire spiral at different column productivities. It was established that at low sampling rates there is some optimum

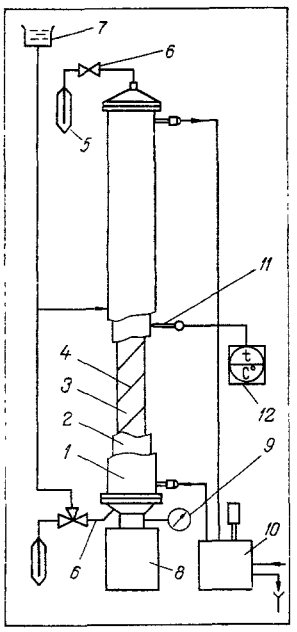

Fig. 1. Diagram of experimental installation: 1) jacket of condenser; 2) outer cylinder; 3) inner cylinder; 4) spiral; 5) fraction collector; 6) valve; 7) supply vessel; 8) vapor generator of two-phase closed thermosiphon; 9) manometer; 10) thermostat; 11) thermocouple; 12) thermometer.

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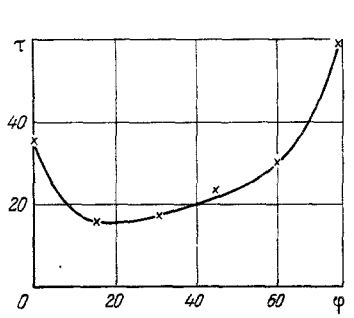

Fig. 2

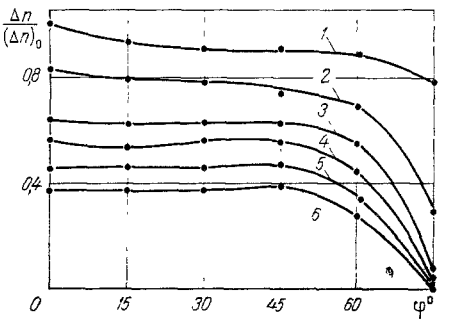

Fig. 3

Fig. 2. Dependence of the time τ it takes to reach the equilibrium state on the winding angle of the spiral.

Fig. 3. Dependence of relative variation in the degree of fractionation on the winding angle of the spiral at different sampling rates: 1) $\sigma = 0$; 2) 1; 3) 3; 4) 5; 5) 8; 6) 12 ml/h.

winding angle of the spiral at which the degree of fractionation is maximal, with this maximum decreasing and shifting toward smaller angles as the sampling rate increases, and disappearing when it reaches a certain value, as a result of which a column with a spiral gives a lower degree of fractionation than a column with an open gap in this case. These dependences were quantitatively different for each of the mixtures studied. We note that, on the basis of the approximate theory of Yeh and Ward [24], the presence of the maxima indicated above is determined by the physical characteristics of the mixture, the geometry of the column, and the temperature conditions of its operation.

Considering that in the creation of a thermal-diffusion apparatus, both for analytical and for technological purposes, the obtainment of narrow fractions of petroleum products in the maximum amounts is one of its important technicoeconomic indices, we conducted a special investigation with the aim of studying the influence of the angle of the spiral winding on the efficiency of the fractionation of petroleum products in a thermal-diffusion column.

The tests were carried out in parallel on three columns which had the same indices of the working gaps: height 630 mm, depth 560 ± 20 μm, mean perimeter 155.2 mm. The construction of the column is partially described in [18]. The inner cylinder is made of Kh18N9T stainless steel with a wall thickness of 4 mm while the outer cylinder with a wall thickness of 8 mm is made of 45 steel with a nickel coating (Fig. 1). Each column is heated from an individual steam generator 8 by saturated steam whose temperature T_S was determined from the pressure, measured by the manometer 9. The electric power required by the steam generators was regulated by autotransformers. The advantages of such a means of heating are expounded in [25]. The outer cylinders were cooled by water from three thermostats 10 whose delivery rates were the same (within limits of ±8%) and equaled 210 liters/h. With this flow rate and with the condenser construction used the heat-exchange coefficient with the water side was $\alpha_W = 2000$ W/m^2 · deg, as was determined earlier [18], while the heat-exchange coefficient with the side of the heating steam was $\alpha_S \approx 11{,}000$ W/m^2 · deg. On the basis of the data presented above, as well as the results of the measurement of the water temperature T_W by thermocouples 11 in the midsection of the condensers, we found the heat flux per unit column length

$$q_l = 9\pi (T_s - T_w) \text{ W/m},$$

the temperature at the inner surface of the outer cylinder

$$T_2 = T_w + \frac{q_l}{\pi}\left(\frac{1}{2\lambda_{oc}} \ln \frac{d_{oc}}{d_2} + \frac{1}{\alpha_w d_{oc}}\right),$$

and the temperature difference

$$\Delta T = \frac{q_l (d_2 - d_1)}{2\pi\lambda_{liq} d_1}$$

between the outer surface of the inner cylinder (T_1) and the inner surface of the outer cylinder, where λ_{liq} and λ_{oc} are the coefficients of thermal conductivity of the mixture being fractionated and of the wall of the outer

TABLE 1. Difference $\Delta n \cdot 10^2$ in Indices of Refraction between Lower and Upper Fractions Obtained in a Thermal-Diffusion Column with a Spiral Insert as a Function of the Delivery Rate and the Winding Angle of the Spiral in the Fractionation of IS-45 Industrial Oil

Sampling rate, ml/h	φ					
	0°	15°	30°	45°	60°	75°
0	5,43	5,08	4,95	4,97	4,81	3,66
1	4,47	4,34	4,29	4,06	3,77	1,61
2	—	3,78	3,80	3,81	3,77	—
3	3,49	3,34	3,40	3,46	2,98	0,38
4	3,33	3,10	3,21	3,25	2,72	—
5	3,05	2,91	3,08	2,95	2,48	0,20
8	2,45	2,52	2,48	2,57	1,89	0,10
12	2,04	2,05	2,04	2,19	1,52	—
25	0,95	1,28	1,35	1,48	0,75	0,0

cylinder; d_{oc}, d_2, and d_1 are the outside and inside diameters of the outer cylinder and the outside diameter of the inner cylinder, respectively.

The tests were carried out using IS-45 industrial oil as the working mixture at $T_s = 158.1°C$, $T_w = 92°C$, $T_2 = 96°C$, and $T_1 = 147°C$. The degree of fractionation was determined by measuring the indices of refraction of the individual fractions on an IRF-22 refractometer. The steady state was assumed to be reached when the samples taken off over 4 h at intervals of 1 h did not differ in index of refraction within the error limits of the refractometer. Before the start of a test the oil was filtered and then vacuum evaporated in a 10-liter vessel, from which it was passed into the reservoir 7 by a siphon (see Fig. 1). The columns were filled through the lower fitting 6.

The first series of tests was set up in the absence of a spiral winding in the working gap (an open gap) in the nonsampling and sampling modes. In the latter case, the columns were fed in their midsections and samples of equal sizes were taken from the upper and lower samplers 6. The results of these tests are presented in the first column of Table 1, in which the angle 0° corresponds to operation with an open gap, and they display the natural law of a decrease in Δn with an increase in the sampling rate. After this series of experiments was conducted the columns were disassembled and spirals of nickel wire 0.55 mm in diameter were wound on the inner cylinders. The spirals were wound with respect to gauges set for angles $\varphi = 15$, 30, 45, 60, and 75°, reading from the vertical. The ends of the spirals were rigidly fixed with screws in notches made in the centering bands of the inner cylinder. The inner cylinder was inserted into the outer cylinder after the latter, together with the condenser, was heated to a temperature of about 150°C by pumping through it oil from a special thermostat, which increased the diameter by 6-7 μm. Such an assembly method eliminated the possibility of displacing the spiral turns and assured the reproducibility of the tests. The results obtained are presented in Table 1, while the dependence of the time it takes to reach the equilibrium state on the winding angle of the spirals is shown in Fig. 2. It is seen from this figure that in the range of values of $0 < \varphi < 60°$ the time of establishment of equilibrium is less than that in a column with an open gap, and its lowest value is attained at $\varphi = 15°$. This result agrees with the data of [23], in which the time it takes to reach equilibrium for a cetane — decalin mixture for a winding angle of 56° was reduced by about 1.5 times in comparison with that for a column with an open gap. But, in contrast to that report, the degree of fractionation Δn (the difference in the indices of refraction between the lower and upper fractions) decreases with an increase in the angle φ, as seen from the first row of Table 1.

The influence of the sampling rates on the fractionation efficiency as a function of the winding angles of the spirals is shown in Fig. 3, in which the ratio of the degree of fractionation to that in the nonsampling mode of operation of a column with an open gap $(\Delta n)_0$ is laid out along the ordinate. As seen from the figure, in the entire range of variation of the sampling rates the fractionation efficiency decreases upon the introduction of a spiral winding into the gap. The lower three curves are characterized by the presence of only weak maxima in the region of winding angles of 30-45°; i.e., in our tests we detected no extremal values of φ at which the fractionation efficiency grows sharply with a decrease in the sampling rate, as occurred in the tests of Yeh and Ward [24].

Thus, both in the nonsampling mode and in the sampling mode the introduction of a spiral winding into the working gap decreases the fractionation efficiency. The fact that a pronounced increase in the degree of fractionation was noted in [23] upon the introduction of a spiral with a winding angle of 56° into the gap can only be explained by the fact that the column used in [23] did not satisfy the strict demands on the geometry of

the gap and did not provide a high degree of isothermy of the working surfaces of both cylinders. As shown in [25], when these demands are not met parasitic convection currents develop in the column which considerably reduce the fractionation efficiency. The electric heater used in [23] promotes the creation of a temperature asymmetry over the perimeter of the column and degrades its operating conditions owing to the nonuniformity of the heat flux, causing nonuniformity of the pressing of the fiber-glass fabric against the surface of the cylinder and nonuniformity of the arrangement of the turns of the electric spiral.

In this case the introduction of spiral inserts into the working gap should have a favorable effect on correcting the defects of the geometry of the gap and increasing the degree of fractionation. In other words, spiral inserts are justified in columns of poor construction quality whose heating and cooling are organized without observance of the necessary conditions for maintaining uniform temperatures at the working surfaces of the column [18].

As for the tests of Yeh and Ward [24], in accordance with the approximate theory which they proposed, the optimum angle at which the greatest fractionation of a binary mixture is achieved in the range of variation of mass concentrations of from 0.3 to 0.7 is determined by the equation

$$\cos\varphi = \left(\frac{\sigma L}{2.52K}\right)^{1/4}, \tag{1}$$

where L is the height of the column;

$$K = \frac{g^2\rho^3\beta^2\delta^7(\Delta T)^2 B}{9!\eta^2 D}; \tag{2}$$

β, η, and D are the coefficients of thermal expansion, dynamic viscosity, and diffusion; ρ is the density of the mixture; ΔT is the temperature difference; δ and B are the sizes of the gap and of its perimeter, with Eq. (1) being valid when

$$0.319y_e^2\frac{K}{L} < \sigma < \frac{2.52K}{L}, \tag{3}$$

while $y_e/4$ is the degree of fractionation in the nonsampling mode.

As seen from (1) and (2), the right-hand side of inequality (3) may not be satisfied at some values of σ, K, and L. In this case the concept of an optimum winding angle of the spiral loses meaning. This can occur with small gaps and a high viscosity of the initial product, as seen from (2). Although these results pertain to a binary mixture, one can assume that they can also be extended qualitatively to multicomponent mixtures, which is what petroleum products are. It is quite possible that with an increase in ΔT, δ, and the average temperature in the gap one can, at certain sampling rates, achieve conditions under which the degree of fractionation is higher than in a column with an open gap.

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MASS TRANSFER IN A CONICAL CHANNEL IN THE PRESENCE OF PROCESSES OF EVAPORATION AND CONDENSATION AT THE CHANNEL WALLS

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The transfer of the wall material of a channel, caused by the presence of a temperature gradient along the channel axis, is discussed.

In the presence of a temperature gradient along the z axis of a channel the transfer of matter evaporating from the walls takes place from regions with high temperatures to regions with lower temperatures. If the temperature level is not very high, the vapor flow takes place in the free-molecule mode. The problem of the mass transfer in a cylindrical channel in the presence of a constant temperature gradient along the channel axis was solved in [1]. But the transfer of matter evaporating from the walls can lead to a change in the channel geometry, so that it is interesting to investigate mass transfer in channels with a more complicated geometry. The mass transfer in a conical channel is studied in the present paper. With slow variation of the radius of a real channel along the z axis the shape of the channel in the vicinity of any point can be approximated by a cone. Thus, knowing the solution for a conical channel, one can approximately calculate the mass transfer in a channel of more complicated shape.

We will take the coefficient of condensation as equal to unity and the velocity distribution of the evaporating molecules as Maxwellian at the wall temperature. In this case the geometrical quantities determining the vapor flow coincide with the corresponding quantities for a noncondensing gas with reflection of a diffuse character. Using the values calculated for them in [2-4], we can write the expression for the mass flux passing through the channel cross section at the point z. In doing this we will assume that a temperature T_0 and the vapor saturation pressure P_0 corresponding to it are maintained at the channel entrance (at $z=0$), while at the other end the channel opens into a vacuum:

$$G=\frac{1}{2}\sqrt{\frac{\pi m}{2k}}\frac{P_0}{\sqrt{T_0}}\left[\frac{z^2}{\cos^2\theta}+2r_0^2+2r_0r'z-\frac{z}{\cos^2\theta}\sqrt{z^2+4(r_0^2+r_0r'z)\cos^2\theta}\right]+\sqrt{\frac{\pi m}{2k}}\int_0^L\frac{P(z')}{\sqrt{T(z')}}\left[\frac{z'-z}{\cos^2\theta}+r_zr'\right.$$

$$\left.-\operatorname{sign}(z'-z)\frac{1}{\cos^2\theta}\,\frac{(z'-z)^2+2r_z^2\cos^2\theta+3r_zr'(z'-z)\cos^2\theta}{\sqrt{(z'-z)^2+4[r_z^2+r_zr'(z'-z)]\cos^2\theta}}\right]dz', \qquad (1)$$

where T(z) is the temperature of the channel walls in degrees Kelvin; P(z) is the vapor saturation pressure at the temperature T(z).

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