ISSN 2074-9414. Technology and technology of food production. 2012. No. 4
A. N. Voinov, O. P. Zhukova, V. A. Pankov, N. Ah.

ETHYL ALCOHOL RECTIFICATION IN COLUMNS WITH SPIRAL PRISMATIC NOZZLE

Experimental data on the strengthening of raw ethyl alcohol in columns with a spiral-prismatic nozzle are presented, as well as the results of the investigation of contact steps with the conductance of the thermal nonadiabatic rectification.

Separation, packed column, contact stage, efficiency

Introduction. in recent years, there is a tendency to create periodically operating rectification installations of low and medium power with sufficiently high efficiency [1-4]. The design for ethanol production is based on columns with spiral prismatic nozzle [5], which are known to have high efficiency and allow to reduce the installation height by an order of magnitude in comparison with industrial analogues. Industrial technological scheme of rectification of ethanol, which includes five or more columns, is formed in line with the installation of one periodic working column, which allows to obtain from the mash by successive selection of the head and tail fractions of raw ethyl alcohol, followed by its strengthening and purification to the parameters of rectified alcohol.

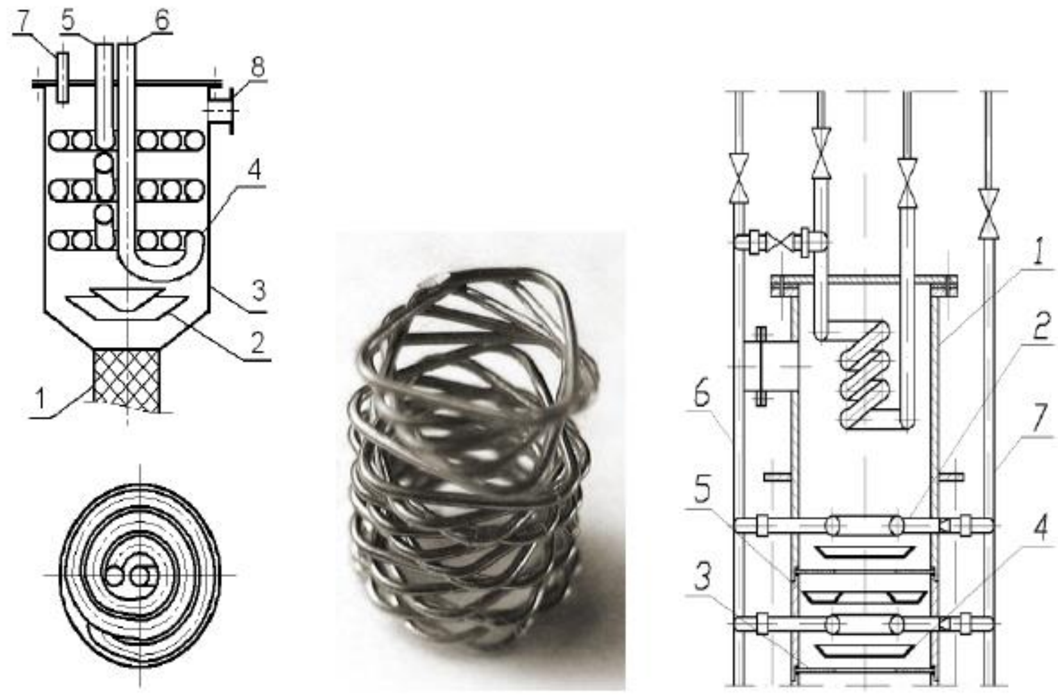
As shown by the experience of operation of such packing columns, obtaining the required quality of alcohol at periodically operating plants is complicated by large losses of the main product. The composition of alcoholic mixtures includes numerous impurities of low concentration, different in chemical nature [6], which have a determining effect on the quality of food rectified alcohol and complicate its purification.

In connection with the lack of information on the work of the above-mentioned rectification plants, the article presents experimental data accumulated in the development and operation of packed columns with a spiral-prismatic packing during the rectification of ethyl alcohol, and also shows a new design of the strengthening part of the rectification column , realizing the effects of thermal non-adiabatic rectification [7, 8], which made it possible to significantly improve the purification of ethanol, to reduce product losses, separation time and energy costs.

Objects and methods of research

Schemes of nodes of the investigated structures of rectification columns are shown in Fig. 1, the scheme of the built-in reflux condenser is shown in Fig. 2. Food sugar was used as feedstock in the preparation of ethanol. Anaerobic fermentation was carried out in a yeast-containing vat with a volume of 1 m3 for six days with an ethanol output of 0.5 kg / kg. Breast rectification was carried out in a periodic manner with the selection of the head fraction and the production of raw alcohol, including: ethanol, 89-94% vol .; aldehydes in terms of vinegar in anhydrous alcohol is not more than 35 mg / l; Esters in terms of acetic-ethyl in anhydrous alcohol not more than 38 mg / l; fusel oils in terms of a mixture of isoamyl and isobutyl alcohols (3: 1) in anhydrous alcohol, not more than 658 mg / l; Methanol in terms of anhydrous alcohol is not more than 0.004%.

The resulting raw alcohol was diluted with technical water to a concentration of 50% by volume. Then, repeated rectification was performed with the selection of the main fraction. The technological parameters of the process are presented in Table. 1. The ethanol sampling was stopped when the pressure in the lower part of the column deviated by 10% from the steady state. The composition of ethyl alcohol was determined using a YCD plus chromatography-mass spectrometer (column-30 m, 0.25 mm-D-HP-58). The main design and technological characteristics of the rectification columns studied are presented in Table. 1. Fig.



а б в

1. Schemes of rectification plants: **a** - packed column with built-in reflux condenser from Archimedes spirals:
1 - packed column; 2 - the distributor of reflux; 3 - case of the reflux condenser; 4 - a reflux condenser; 5, 6 - input and output of cooling water;

b - spiral-prismatic nozzle;

c - column with stages of thermal rectification:
1 - shell of the column; 2 - reflux condensers; 3, 4, 5 - elements of the contact stage; 6, 7 - collectors for supply and discharge of water.

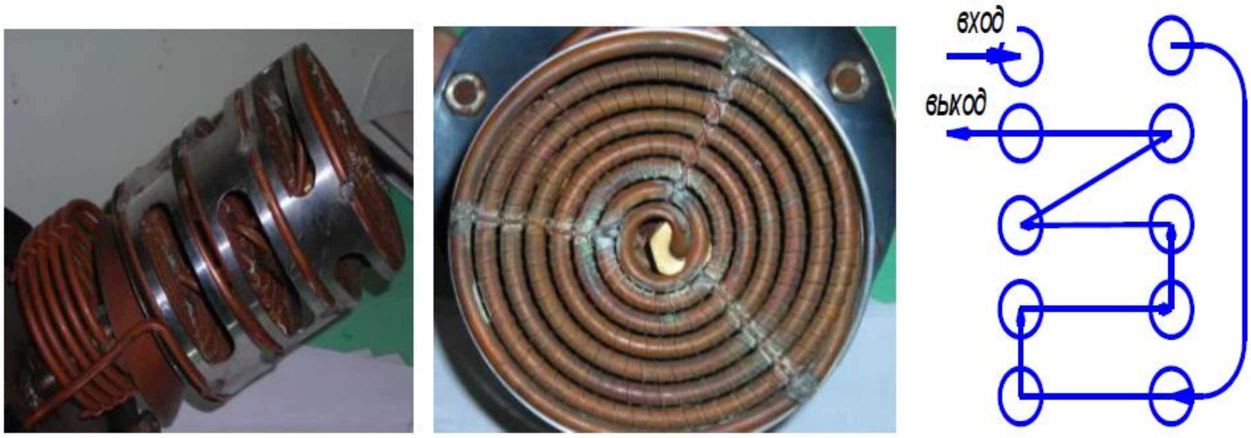


Fig. 2. The design of the built-in reflux condenser of the rectification packed column: **a** - appearance; **b** is a view of the reflux condenser from the end; **c** - diagram of the cooling water connection to the reflux condenser

The heat transfer surface of the reflux condenser, in the form of Archimedes' spirals [9], from copper tubes with a diameter of 8x1 mm, in spirals 4 pcs., the distance between them is 50 mm, The size of the gap between the turns of the helix is 1.2-1.5 mm. To remove the condensate film from the surface of the tubes, a copper wire 0.1 mm thick was wound on them in 35 mm steps. The ratio of internal diameter of the column at the location of the reflux condenser to the diameter of the main part of the packed column was 1.5. A helical-prismatic nozzle measuring 4x3x0.18 mm, made of nichrome wire with a bulk density of 1300 kg / m3. The bottom part of the column with a capacity of 0.5 m3 is equipped with a jacket with built-in electric heaters. Consumption of water in the reflux condenser was 0.04-0.5 m3 / h at its initial temperature of 7-13 ° C. Before entering the reflux condenser, water was passed through a filter made from the grid, in order to eliminate solid inclusions.
Increasing the initial temperature of the cooling water leads to an increase in its consumption. The elements of the column considered in the work in the periodic mode and blowing the nozzle with steam before. We have been operating for more than ten years.
The combined column, on the contact steps of which in the strengthening part was

realized nonadiabatic thermal rectification process (see Figure 1c), consisted of two parts. To the bottom part of the column with a diameter of 130 mm and a length of 1.4 m was filled with a spiral-prismatic nozzle. Upper part of the column with a diameter of 200 mm and a height of 0.8 m was made of 10 contact steps, each of which were placed three plates with a diameter 160-210 mm, made of copper sheet 0.25 mm thick (see Figure 1b, positions 3, 4, 5), as well as refluxers made of copper-tube with a diameter of 8 mm with a heat exchange surface of 0.01 m2. A column that realizes the non-adiabatic The thermal rectification (see Table 1) had [7] diameter 200 mm, 25 contact steps 70 mm high, made of 0.25 mm thick plates (see Fig. 1c, positions 3, 4, 5) were installed on it.

Table 1. Parameters of the rectification columns studied with the reinforcement of raw ethyl alcohol

Parameters	Column diameter, mm					
	51	80	130	195	130/200	200
	Packing				Combined	Column thermal rectification
heating power, kw	2,0	5,0	13,0	30,0	15,0	15
height of spp layer, m	1,7	1,9	1,25	1,7	1,4	–
number of stages, pieces	–	–	–	–	10	25
condenser surface, square meters	0,045	0,09	0,28	0,6	0,3	0,45
spp weight, kg	7,0	11	23	71	26	–
distillate consumption, l per hour	1,4	3,5	9,0	26	15	–
abv	96,0	96,6	96,2	96,4	96,6	96,7
water out temp. from the reflux condenser,	71	76	74	74	76,2	–
Reflux ratio: 1. in the selection of ethanol 2. when selecting a head fraction	4,0 50	4,0 50	4,0 46	3,0 35	2,5 6,0	3,0 –
Selection time (volume of raw alcohol 100 liters), min: head fraction ethyl alcohol	250 –	250 1714	250 667	250 230	8 400	– –
Number of head fraction (volume of raw alcohol 100 liters), l	–	3	4,5–5	4,0	0,4	–
Specific energy inputs, kWh / kg	–	1,6	2,0	2,4	1,0	1,0

Results and its discussion

As established, in a dephlegmator made of spirals of Archimedes, (see Figure 2), at low costs vapor and liquid, the drip flow regime is observed with the formation of phlegm and its disruption in the form of droplets from the surface of the turns. At the rate of reflux 12-27 l / h and the gas velocity in the gap between the turns of the spiral 0.9-1.5 m / s, the flow regime corresponding to the operation of the failed disc is observed. At speed vapors of the mixture above 2 m / s, a hang-up regime is established with subsequent flooding of the column. Hydraulic resistance of the reflux condenser without Irrigation is not more than 350 Pa. At the values of the Reynolds test of cooling water in copper tubes of a reflux condenser equal to 2500-3000, the value of the heat transfer coefficient was not more than 500 W / (m2 K). And the highest values were obtained by connecting the reflux- tor to the cooling system according to the scheme presented by in Fig. 2c. The cooling water temperature at the outlet from the reflux condenser was 71-76 ° C (see Table 1). FROM increase in the cooling water temperature by output from the reflux condenser column efficiency Murphrey and the stability of its work are increasing. Reducing the power of heaters in the column cube leads to a reduction in the flow of cooling water in dephlegmator, which requires its design use of copper pipes of small diameter for the purpose of providing turbulent regime. In this connection, the use of columns with a diameter of less than 80 mm The built-in reflux condenser is inefficient, since due to the low temperature head pressure in the column the required heating of the reflux is ensured. Measurement of the concentration of ethanol in vapors and in liquids, passing through the reflux condenser, it was possible to calculate its Murphrey efficiency, which did not exceed 0.1, while the composition of rectificated alcohol before and after the reflux condenser was practically no different. AT this connection can be concluded that the main function of the reflux condenser in the columns under consideration is reduced to providing a reflux temperature close to the boiling point of the mixture located in the upper layers of the nozzle. Therefore, of the reflux condenser by performing certain gaps between coils of Archimedes' spirals [2], and also between the body and the coils is not justified, this only complicates the manufacture of the reflux condenser. In the packed columns tested, when ethanol was strengthened, the height of the nozzle layer 100 mm was equivalent to one theoretical step.

During the operation of the packed column, three hydrodynamic regimes were identified: film, suspending, flooding. Stable operation of the plant is observed in the range of the speed factor change:

F ω ρ (0.36 0.5) n, Pa0.5,

where is the average flow rate of steam across the section columns, m / c; n is the vapor density of the mixture, kg / m3. The mixture, obtained with the strengthening of raw alcohol, has the following composition: ethanol 96-96.4% vol .; mass concentration of aldehydes in terms of acetic aldehyde in anhydrous alcohol, no more than 5.5 mg / l; mass concentration of esters in terms of on acetic-ethyl in anhydrous alcohol, no more than 4 mg / l; mass concentration of fusel oil in recalculation on a mixture of isoamyl and isobutyl alcohols (3: 1) in anhydrous alcohol no more than 2 mg / l; the volume concentration of methyl alcohol in terms of anhydrous alcohol is not more than 0.002%. Losses ethanol with a head fraction with a reflux ratio 35-50 amounted to 3-5%, and the specific costs for strengthening raw spirit - 1.6-2.4 kWh / kg. Large losses of ethanol with a head fraction are primarily due to supercooling of reflux on the heat transfer surface of the reflux condenser. Dependence of the hydraulic resistance of the packed column on the separation factor for rectification of the mixture under consideration is shown in Fig. 3. The resistance of the investigated columns was 1000-4000 Pa, and the density of irrigation - up to 3000 kg / (m2 × h). With increasing steam velocity along the column cross-section, the holding capacity of the packing decreases (Figure 4).In order to intensify the process of separation and reduction of product losses, a combined distillation column, a top part of which consisted of stages of nonadiabatic thermal rectification performed in the form of plates (see Figure 1c). Reflux condensers 2, placed at each contact stage, allowed [7, 8] to ensure the presence of a reflux and its partial evaporation on plates with the formation of a secondary vapor with high concentration of volatile components. In the condensate formed under the lower surface of the plates, due to the effect of fractional rectification, concentration of the high-boiling component, which leads to a significant intensification of the process division. Murphrey efficiency of contact stages, at the concentration of ethanol in mixture of 20-96% vol. was 0.6-1.2, whereas in the absence of dephlegmators 2 (see Fig. 1c), in the steps considered, their

efficiency does not exceed 0.1.
As the gas velocity between plates increases, the efficiency of the degree increases. With reflux ratio 6.2 and the amount of the collected head fraction 0.4% of the total mixture in the combined column, the composition of the mixture includes: ethanol 96.6%; the mass concentration of aldehydes in terms of vinegar in anhydrous alcohol not more than 2.0 mg / l; mass the concentration of esters in terms of aceticethyl in anhydrous alcohol is not more than 0.5 mg / l; mass concentration of fusel oil in terms of A mixture of isoamyl and isobutyl alcohols (3: 1) in anhydrous alcohol not more than 0.8 mg / l; volume concentration of methyl alcohol in terms of anhydrous alcohol - traces, which indicates a high separation capacity of the combined column. The current costs of the process due to the reduction
the time of selection was halved (see Table 1). The developed column is efficient with high efficiency at sufficiently low reflux number of 2.5-3.0. At the same time, the reduction in the retention capacity of the contact steps liquid by an order of magnitude, which ensures low losses of the processed product. It should be noted that factor of separation of the lower part of the combined The column increased and amounted to 0.8-0.9 Pa0.5.
The hydraulic resistance of the contact stage, which realizes the process of thermal rectification, was no more than 20 Pa. Important advantages of the developed column are low hydraulic resistance and achieving efficient separation of a multicomponent mixture by change the temperature of the coolant supplied in the dephlegmators.
Thus, the data accumulated in the design and study of heat-mass-exchange low-tonnage columns indicate the possibility of implementing an effective separation of multicomponent mixtures using a combined column with contact stages of thermal nonadiabatic rectification. At the same time, a new rectification method, protected by a patent, has been tested and its potential advantage is shown in comparison with the known ones. High ethanol purification efficiency achieved from impurities at low hydraulic resistance and height of the column.