

Advantages of cyclic rectification for ethanol production

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Abstract

Keywords:

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Introduction. The efficiency of the ethyl alcohol rectification process depends on the degree of purification of the final product from organic impurities and the heating vapour flow rate in the distillation unit.

Materials and Methods. The object of the study was a cyclic column for concentration of impurities. The concentration of volatile impurities of alcohol was determined by chromatographic method, their extraction and concentration ratio – by calculation method. Water flow rate was controlled by means of Rate-Master® flow meters, vapor velocity in the free section of the column – by vortex flowmeter, in the holes of the plates – by calculation method.

Results and discussion. Studies of the efficiency of the technology of cyclic rectification of ethyl alcohol, providing for the implementation of controlled cycles of liquid delay on the column plates and its overflow without interruption of heating vapour supply have been carried out. The design of the rectification column for its realization is developed. Hydrodynamic modes of operation of sieve and flake plates in cyclic mode are established: vapour velocity in the free section of the column and plate openings for mass exchange between liquid and vapour and liquid preflow. The research results obtained in production conditions proved the advantages and feasibility of using the innovative technology: in the process of separation of alcohol-containing fractions in full measure head impurities of alcohol are allocated, the degree of extraction of the higher alcohols of sivush oil increases by 38%, methanol – by 15,6%, the multiplicity of concentration of head impurities increases by 25%, higher alcohols – by 40%, methanol – by 34%, acrolein – by 36%, isopropyl alcohol – by 42%. At the same time vapour consumption in the impurity concentration column is reduced by 40% in comparison with typical units operating in the stationary mode and does not exceed 12 kg/dal of absolute alcohol (a.a.) introduced with feed.

Conclusion. Increasing the contact time of vapor and liquid on each plate up to 30–40 s allows increasing the degree of purification of rectified alcohol from volatile impurities and reducing energy costs by at least 40%.

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Introduction

The energy dependency of industrialized nations requires for alternative sources of energy, one of which is ethyl alcohol (bioethanol). To ensure the competitiveness of bioethanol over carbohydrate energy sources, the development and implementation of innovative resource- and energy-saving technologies, and the enhancement of operational and technological characteristics of the rectification plants are a prerequisite. A promising direction to solve this problem is to use a non-stationary (cyclic) mode of operation of the plants, based on the alternating change of the periods of the steaming periods and liquid overflow (Botshekan et al., 2022; Nagy et al., 2015).

Investigation of physico-chemical conditions of separation of multicomponent systems in the mode of controlled rectification cycles, development on the basis of the laws of thermodynamics of rational methods of calculation and construction of rectification columns of cyclic action, work in computer modeling of delay cycles and liquid overflow began in the 60's and 70's of the XX-th century (Andersen et al., 2018; Bastian et al., 2012; Kiss, 2014; McWhirter and Cannon, 1961). The authors managed to achieve an increase in steam load by 48% with constant pressure drop without making structural changes in the column, double the capacity of the installation with cap plates compared to the typical at the same degree of separation, to develop a hydrodynamic model of rectification with alternate phase motion, the theory of intermittent cyclic distillation – a new method of periodic control in which the motion of the liquid depended on the ripple of the steam flow, and to propose a new type of plate was a traditional mesh plate with a special inclined section to slow down the liquid overflow (Nielsen et al., 2017; Rasmussen et al., 2020; Toftegard et al., 2016).

Due to rising energy prices, interest in the study of cyclic distillation has increased in Ukraine in recent years. At the National University of Food Technology, special valve contact devices have been proposed to provide controlled cycles of delay and liquid overflow (patent UA60566. Mass exchange contact device). Pilot tests of the devices proved the possibility of reducing the specific consumption of heating **vapor** in columned installations of cyclic action by 30% compared to typical ones. The disadvantages of their operation include the dependence of the operation of the overflow devices on the vapor pressure, the limitation of the rectification column in height, the need to install intermediate plates, the appearance of the impulse to delay the opening and closing of the floating valves (Maleta et al., 2011).

Despite the positive results of experimental studies, justified by the methods of mathematical modeling of the benefits of cyclic rectification, the above methods and models are not widely used. Absence of mass-exchange in the vapour period, fluctuations of vapour pressure in the collector, the bottom section and the deflegmator of the rectification column adversely affect the quality of the finished product and the work of other columns. And the complexity of the proposed constructive solutions to ensure a cyclic mode reduce the reliability of the rectification equipment.

In order to improve the efficiency of mass-exchange by eliminating the above disadvantages, a method of controlled rectification, which provides for periodic (cyclic) motion of the liquid after its delay at the contact stages with continuous supply of heating vapour in the bottom section of the column was proposed. For the cyclic motion of the liquid on the plates of the column, the vapour flow is cut off at each plate by means of valves located on the bypass conduits (Krivosheev and Anufriev, 2015). According to the authors, this technical solution allows to ensure the periodic motion (weeping) of the liquid on the plates during the period of opening of the respective valves and the direction of vapour through the

respective bypass conduits and to exclude its mixing with the portions of the liquid located on the lower adjacent plates.

However, the method was not practically implemented due to the fact that, at the moment of opening of the valves, the vapour flow occurred both through the bypass conduits and through the slits of the upper plates, thus complicating the liquid overflow on the lower plates.

To solve the current problem, the staff of the Department of Biotechnology of Fermentation Products and Winemaking of the National University of Food Technology and LLC “TISER” developed an innovative technology of cyclic rectification, which provided for the delay of liquid on the plates and its cyclic overflow without interrupting the supply of heating vapour, as well as the design of rectification column for its implementation (Buliy et al., 2016; Ukrainets et al., 2018). The efficiency of technical solutions was determined in the process of ethyl alcohol extraction from alcohol-containing fractions enriched with organic impurities. For the purpose of the study, the experimental impurity concentration column was equipped with movable valves connected to actuators whose action was according to the controller program, and flake plates with valve openings.

The aim of the present research was to improve the technology of cyclic rectification, to develop the construction of a distillation column to ensure the cyclic motion of the liquid with continuous vapour supply, to study the effectiveness of innovative technology in the process of rectification of alcohol-containing fractions, to determine the optimal technological modes of operation of the impurity concentration column, specific vapour consumption, degree of extraction and multiplicity of concentration of volatile impurities of alcohol.

To achieve this aim and conduct production tests, the primary task was to determine the hydrodynamic mode of operation of the perforated plates (mesh and scale-shaped) to ensure their cyclic action:

1. Establishment of the working range of vapour speed in the free section of the impurity concentration column and in the slots (openings) of the plates — the lower boundary, beyond which the weeping of the plate stops from the upper plate to the lower plate, and the upper boundary, at which liquid entrainment occurs to the upper plates.
2. Determination of the vapor velocity in the slots (openings) of the plates, at which there is an intense weeping of the liquid from the upper plates to the lower ones.

Materials and Methods

Research objects

1. Experimental cyclic column for concentrating alcohol impurities (ICC).

The impurity concentration column is made of stainless steel AISI 304, equipped with flake plates. Technical characteristics: diameter – 426 mm; number of plates – 30; distance between the plates – 300 mm; the cross-sectional area of the holes – 19.42 mm²; thickness of the plate blade – 2 mm; free section of the plate: 5.5% – during the stay of liquid on the plates – 5.5%; during the overflow of liquid – 51.7% (Figure 1).

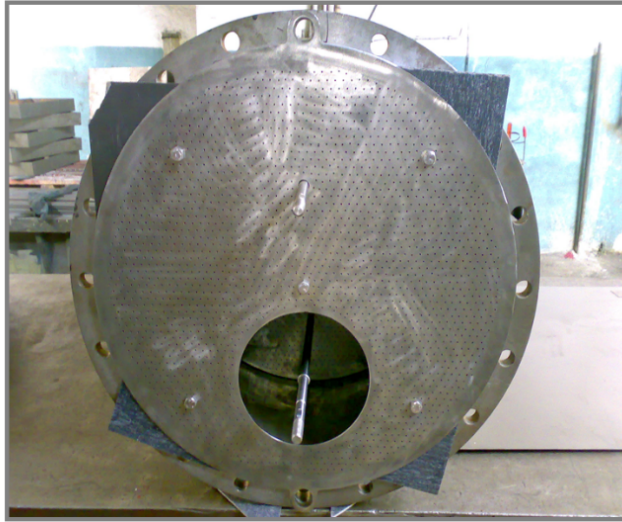


Figure 1. Cyclic plate with variable live section

A fragment of the impurity concentration column with movable rods and valves is shown in Figure 2 (patent UA139228. Column mass transfer apparatus of cyclic action).

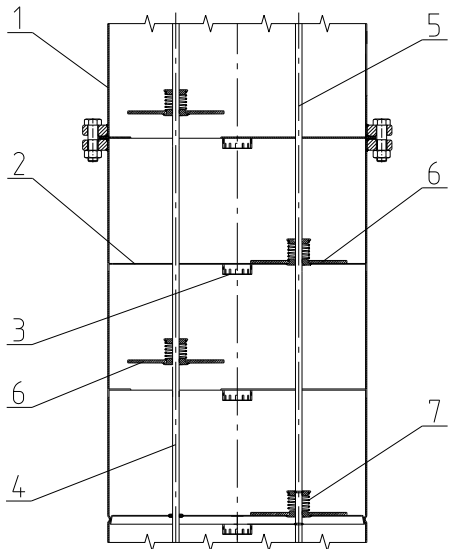


Figure 2. Fragment of a prototype LC with movable valves:

- 1 – body; 2 – plates; 3 – contact elements; 4,5 – rods;
- 6 – valves; 7 – springs.

The column contained a body 1, plates 2 with contact elements 3, movable rods 4 and 5, on which valves 6 and springs 7 were fixed. The rods moved up and down periodically under the action of drive mechanisms (double-acting pneumatic cylinders of DNT type of

FESTO Company). In this case, valves 6 alternately closed and opened holes for liquid overflow. Pneumatic cylinders were controlled according to the program of controller M340 of Schneider Electric Company.

The operation of the column provided for conducting adjustable in time cycles of liquid residence on the plates and its synchronous overflow from plate to plate over the entire height of the column in two successive stages, repeating periodically in time, alternately, according to the specified algorithm without interrupting the liquid and steam supply in the column (patent UA89874. Method of liquid overflow on plates of column apparatus in the process of mass-exchange between vapour and liquid). The time interval of liquid retention was determined experimentally depending on the degree of extraction of volatile alcohol impurities and the multiplicity of their concentration and was 30-40 s.

The technical solution allowed for one-stage (full) and two-stage (fractional) overflow of liquid from the upper plate on the lower plate. The single-stage method ensured that all liquid overflowed. According to the two-stage method (patent UA 141245. Method of overflow of liquid on plates of mass-exchange column apparatus) at the first stage a part of liquid (30-70% of its volume) was overflowed from the upper plate to the lower one, and after the set time of liquid delay its rests were overflowed.

The experimental impurity concentration column was incorporated into the circuit of an operating distillation unit.

2. Installation for extraction of ethyl alcohol from alcohol-containing fractions

The technological scheme of inclusion of the experimental impurity concentration column in the scheme of the distillation unit is shown in Figure 3.

The scheme provided for the introduction on the feed plate of the impurity concentration column of the head fraction of ethyl alcohol, distillate cuts from the condensers of the distillation column and CO₂ separator condenser, fusel alcohol and rinse water for fusel oil. The installation included impurity concentration column 6, the plates of which are connected to the pneumatic cylinders bilateral action 7, and their upper and lower parts with vacuum breakers 4, evaporator 5, condenser 9, alcohol catcher 10, softened water container 1 for hydroselction, intermediate collections of distillation residue 17 and alcohol-containing fractions 20, flowmeters 3, 12, 13, 14 and 15, centrifugal pumps 2, 18, 19 and decanter 11.

The consumption of vapour, water for hydroselction, fusel ad ester-aldehyde concentrate and distillation residue was determined using flowmeters.

Research methods

To evaluate the obtained research results, analytical, chemical, physicochemical, and computational methods were used with the use of devices and research methods used in the production of rectified ethyl alcohol.

Liquid consumption. The consumption of alcohol-containing fractions, water for hydroselction, distillation residue and rectified alcohol was monitored using RM flowmeters (Polulyah and Topolov, 2002).

The principle of their operation is based on the perception of the dynamic head of the controlled medium, which depends on the flow rate, by a sensing element (float) placed in the flow. As a result of the flow, the sensing element moves along the height of the flowmeter, and the amount of movement serves as a measure of flow. The readings were taken on the scale of the flowmeter, graduated by water in dm³/h.

Vapour velocity. The vapour velocity in the free section of the column was determined using a vortex flowmeter (VFM), which has an ejection rod located on the path of the steam flow from which the vortices are ejected. The frequency of rejection varies linearly with the speed of the steam. Placing a piezoelectric sensor below the flow and calculating the peak frequency of its signal allows you to fix the speed of steam and its flow rate (Dattarajan, 2019).

Concentration of ethyl alcohol in water-alcohol solutions. The concentration of ethyl alcohol in the liquid distillation residue of the aldehyde concentrate was determined by areometric method). The test solution was poured into a 250 cm³ glass cylinder, the temperature was measured with a thermometer with a division price of 0.1 °C, and then the ASP-1 alcoholmeter was immersed. The actual concentration of ethyl alcohol at a temperature of 20 °C was determined from the readings of the alcoholmeter and using special tables to make appropriate corrections for temperature.

Concentration of volatile alcohol impurities. Concentration of aldehydes was determined by reaction with fuchsin-sulfur reagent I; fusel oil – by the method based on the reaction of higher alcohols with salicylic aldehyde solution in the presence of sulfuric acid; free acids – by the amount of sodium hydroxide solution used for titration; complex esters – by titrometric method after their saponification with sodium hydroxide solution; volume fraction of methyl alcohol – by method based on the reaction of methanol oxidation with potassium permanganate and sulfuric acid to form formaldehyde, which forms a color with fuchsin-sulfur reagent II (Arslan, 2021).

The concentration of head volatile impurities (aldehydes, higher alcohols of fusel oil, acrolein, isopropyl alcohol) in alcohol-containing fractions, distillation residue of impurity concentration column, concentrate of impurities and rectified ethyl alcohol was determined on a gas chromatograph with an HP FFAP 50 m × 0.32 m column (Dewulf, 2002; Plutowska et al., 2008; Steven et al., 2002). The analysis of the experimental samples was carried out three times. The average values were chosen as determinative.

Grade of extraction and concentration ratio of volatile alcohol impurities.

The degree of extraction (α) and multiplicity of concentration (β) of key organic impurities of alcohol were calculated by the formulas:

$$\alpha = \frac{X_l}{X_{cub}}; \beta = \frac{X_{seac}}{X_l};$$

where X_l , X_{seac} , X_{cub} – accordingly, the concentration of volatile alcohol impurities on the feed plate, sivush and ester-aldehyde concentrate and cube liquid, mg/dm³ in terms of a.a. (Linek, 2005; Shiyan et al., 2009).

Studied modes

In the first stage, the hydrodynamic regimes of failure plates were investigated to ensure the cyclic mode of operation of the experimental impurity concentration column. The efficiency of the perforated plates in a cyclic mode depends on the adopted hydrodynamic modes, which determine the boundaries of stable operation of the column (Kiss et al., 2009; Kiss and Bildea, 2011; Kiss, 2012). There are no general methods for calculating the boundaries of hydrodynamic modes for bubble plates. Therefore, when designing the plant-

like devices, the vapor velocity corresponding to the lower and upper bounds of the plate was determined by calculation, and then the working speed vapor velocity was determined in the free section of the column and in the slits of the plates. The lower limit corresponded to the vapor velocity at which the weeping of the liquid from the plate stopped. The upper limit corresponded to the vapor velocity at which the liquid entrainment onto the upper plate was observed. In this case, the contact surface of the phases sharply decreased. Such plates are called versatile or variable cross-sectional plates. Many studies have been devoted to determining the conditions under which bubbling occurs on the plates (the fluid is retained on the plate) and the overflow of the liquid through the overflow openings and its weeping through the slits of the contact devices (Lita, 2014; Pătruț et al., 2014; Premkumar and Rangaiah, 2009). In order to eliminate the possibility of the liquid being transferred to the top plate, the vapor velocity in the free section of the column equipped with mesh plates should not exceed 0.7 m/s. The vapour velocity in the holes at which the liquid is retained on the plate is 4-5.5 m/s. When using scale-shaped plates with an optimal live cross-section of 10%, the vapor velocity in the free column cross-section may reach 1.2 m/s or more, and in the openings of the scales must exceed the first critical velocity of 6.5-7.5 m/s. The liquid weeping occurs at a vapor velocity in the openings of 1.5-1 m/s (Dejanovic et al., 2010; Flodman and Timm, 2012).

At the second stage, the effectiveness of the innovative technology in the process of distillation and separation of alcohol-containing fractions was investigated: determined the optimal technological mode of operation of the impurity concentration column, the specific vapour consumption, the degree of extraction and multiplicity of concentration of key organic impurities of alcohol.

Alcohol-containing fractions of 135 dm³/h were calculated on the feed plate of the impurity concentration column in terms of absolute alcohol (a.a.). Of these, the head fraction of ethyl alcohol is 8.5%, the distillate cuts from the condenser of the distillation column – 9.4%, the CO₂ separator condenser – 3.0%, fusel alcohol – 1.5%, rinse water for fusel oil – 1.5% of the a .a. of mash. The delay time of the liquid on the plates was 30 s, the overflow time – 2 s. The pressure in the bottom part of the DC was 17.5 kPa, in the upper part – 2–5 kPa. The temperature in the bottom of the column was equal to 102–103 °C, in the upper part – 90-91 °C, water for cooling at the condenser inlet – 15 °C, at the outlet after the deflegmator – 65 °C.

Water for hydroselction in the amount of 2400 dm³/h was supplied from water container 1 through a flowmeter 3 to the top plate of the column by pump 2. The concentration of ethyl alcohol on the plates of the concentration part was 7% vol., and in the bottom part – 5–6% vol. The alcohol-containing fractions were warmed by the heat of luther water (residue water) in the container 20, from which the feed plate was fed by the pump 18. Released from the head and part of the intermediate volatile impurities distillation residue was pumped into the upper zone of the concentration part of the ether column by centrifugal pump 18. Vapour containing volatile impurities from the upper part of column 6 was sequentially fed into the deflegmator 8 and the condenser 9. In the dephlegmator mainly water, higher alcohols, partially esters and ethanol condensed. There was the condensation of low-boiling aldehydes and methyl alcohol in condenser, which vapours did not condense in the deflegmator. Reflux from the dephlegmator 8 and most of the condensate of the head and final impurities from the condenser 9 was sent to the decanter 11. In the decanter, the heterogeneous mixture was separated, forming the upper layer – FEAC containing esters, higher alcohols and part of aldehydes, and the lower layer – water released from the head, part of the intermediate and final impurities. The FEAC was removed from the installation through the flowmeter 13 and the aqueous-alcoholic liquid from the lower part of the

decanter, which in the form of phlegma flowed on the top plate of the impurity concentration column for irrigation. To reduce the concentration of methyl alcohol in the distillation residue part of the phlegma was removed from the installation in the form of methanol concentrate (MC) through flowmeter 11. From the condenser 9 aldehyde concentrate (AC) was selected through flowmeter 14.

A mnemonic diagram of the impurity concentration column inclusion in operation with controlled cycles of mass-exchange and liquid overflow is shown in Figure 4.

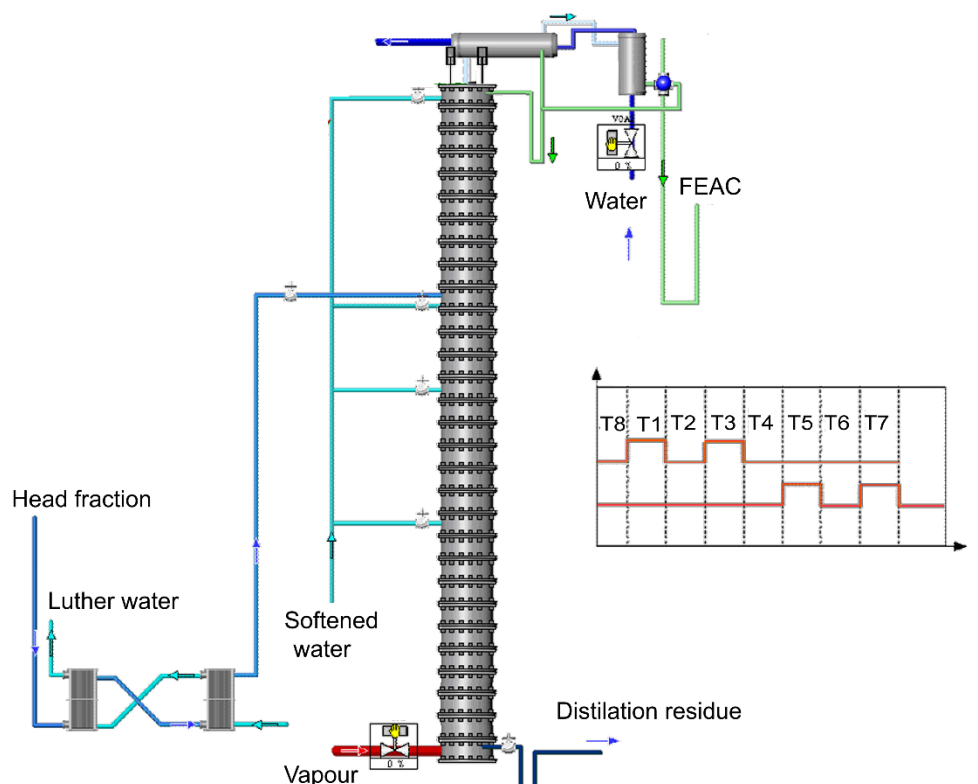


Figure 4. Mnemonic diagram of the operation of an column concentrating impurities in the mode of controlled cycles of liquid retention and overflow

For calculation degree of extraction (α) and multiplicity of concentration (β) of key organic impurities of alcohol during the research period, samples of alcohol-containing fractions entering the column were taken, distillation residue, reflux, fusel and ester-aldehyde concentrate and samples of liquid from the 2nd, 6th, 11th, 17th and 26th plates of the column were taken and their chromatographic analysis was performed. The studies were carried out in triplicate. The average values were chosen as the determining ones.

Stages of research

At the first stage of research it was established that the vapour velocity in the free section of the column equipped with mesh plates should not exceed 0.7 m/s. At such steam velocity the possibility of liquid entrainment to the upper plate is excluded. The vapour velocity in the holes, at which the liquid is retained on the plate, is 4–5.5 m/s; the optimum velocity for mass-exchange is 7.5–8.0 m/s. When using flake plates with an optimum live section of 10%, the vapour velocity in the free section of the column can reach 1,2 m/s and more. In the holes of scales the vapour velocity should exceed the critical velocity of 6.5–7.5 m/s, the optimal velocity is 12–14 m/s (Buliy et al., 2019). Intensive overflow of liquid occurs at vapour velocity in holes 1.5–1 m/s (patent UA 123917. Method of mass-exchange between liquid and vapor in a column apparatus).

At the second stage, it was investigated that to obtain high-quality alcohol, the extraction of FEAC should be 0.23–0.27% of the amount of ethyl alcohol in the fermentation. Of these, AC should be selected in the ratio of 1:2–1:2.5 to FEAC. And MC should be sampled if the concentration of methanol in distillation residue exceeds 0.001% vol.

It's been practically proven that the organization of differentiated selection of alcohol impurities from places of their maximum accumulation, autonomous regulation of their ratio, depending on the qualitative composition of feed, allowed to increase the efficiency of rectification (patent 69511. Rectification unit for alcohol extraction from fractions enriched with organic impurities). Direction of phlegma of dephlegmator and condensate from the condenser to the decanter to separate the heterogeneous mixture and the additional selection of water-alcohol mixture from the lower part of the decanter helped to reduce the content of the end liquid, parts of the head, intermediate and atypical types of impurities, isopropyl alcohols, crotonaldehyde, acrolein) and esters (methyl acetate, ethyl acetate), due to which the quality of rectified alcohol improved.

The results of the chromatographic analysis of the test samples are given in Table 1. The analysis of the experimental data Table 1 has shown that in the process of rectification of alcohol-containing intermediates and rectification products in the mode of controlled rectification cycles together with the head ones, the intermediate and final impurities of alcohol are effectively removed. Under the given conditions, aldehydes (acetaldehyde), esters (methyl acetate, ethyl acetate, isobutyl acetate, isoamyl acetate), atypical impurities (acrolein, crotonaldehyde), n-pentanol and isopropyl alcohol are completely removed. It is known from practical experience that isopropanol is one of the volatile impurities that is most difficult to separate from ethanol. Therefore, the content of it in the final product evaluates the efficiency of the rectification plant. The concentration of isopropanol in commercial alcohol should not exceed 1.5 mg/dm³ (Xiao-Na Pang et al., 2017).

Among the esters, ethyl acetate had the highest concentration, and isobutyl acetate has the lowest. Of the higher fusel alcohols, isobutanol was most effectively concentrated, n-propanol had the least recovery rate and concentration. During rectification, the concentration of ethyl alcohol on the plates of the column did not exceed 13% vol.

The calculated values of (α) and (β) for aldehydes, esters, higher fusel alcohols, methyl alcohol and atypical alcohol (acrolein) impurities under typical and cyclic distillation are given in Table 2.

Table 1

Distribution of volatile organic impurities of alcohol along height of the impurity concentration column

Name of the impurity	Concentration, mg/dm ³ (in terms of a. a.)								
	Distillate cuts	Distillate residue	The plate number					Reflux	EEAC
			2	6	11	17	26		
Acetaldehyde	605.1	<i>traces</i>	<i>traces</i>	2.6	3.3	5.3	16.7	12304.2	13591.2
Methyl acetate	32.3	<i>traces</i>	<i>traces</i>	<i>traces</i>	<i>traces</i>	<i>traces</i>	<i>traces</i>	756.1	770.8
Ethyl acetate	509.4	<i>traces</i>	<i>traces</i>	4.7	8.3	18.6	<i>traces</i>	14448.9	15344.4
Isobutyl acetate	14.6	<i>traces</i>	<i>traces</i>	<i>traces</i>	<i>traces</i>	9.1	47.5	122.9	137.2
Isoamyl acetate	81.4	<i>traces</i>	<i>traces</i>	<i>traces</i>	<i>traces</i>	<i>traces</i>	<i>traces</i>	1573.2	1652.9
Isopropanol	2.0	<i>traces</i>	<i>traces</i>	<i>traces</i>	1.0	4.4	12.5	47.7	42.2
n-propanol	10063.8	1080.3	1419.3	2331.1	4894.4	8035.3	6254.5	49045.5	53570.4
Isobutanol	6851.2	6.5	7.6	47.9	347.5	1749.3	6718.3	218161	204757
n-butanol	36.1	<i>traces</i>	5.4	8.6	23.4	24.1	28.7	422.4	379.9
Isoamylol	10354.8	7.1	8.0	156.7	731.9	1672.4	8531.7	211033	203311
n-pentanol	<i>traces</i>	<i>traces</i>	<i>traces</i>	<i>traces</i>	<i>traces</i>	<i>traces</i>	<i>traces</i>	12.3	17.4
Acrolein	25.9	<i>traces</i>	<i>traces</i>	<i>traces</i>	<i>traces</i>	<i>traces</i>	<i>traces</i>	446.9	447.6
Crotonaldehyde	<i>traces</i>	<i>traces</i>	<i>traces</i>	<i>traces</i>	<i>traces</i>	<i>traces</i>	<i>traces</i>	121.8	136.7
<i>Aldehydes</i>	605.1	<i>traces</i>	<i>traces</i>	2.6	3.3	5.3	16.7	12304.2	13591.2
<i>Esters</i>	637.7	<i>traces</i>	<i>traces</i>	4.7	8.3	27.7	47.5	16901.1	17905.2
<i>Fusel oil</i>	27307.9	1093.9	1440.2	2544.4	5998.2	11485	21545	478712	462078
<i>Methanol, % vol.</i>	0.026	0.001	0.001	0.002	0.004	0.003	0.005	2.9	2.1
<i>Ethanol, % vol.</i>	82.0	5.4	10.0	13.0	13.0	12.4	7.0	65.0	68.0
<i>Atypical</i>	25.9	<i>traces</i>	<i>traces</i>	<i>traces</i>	<i>traces</i>	<i>traces</i>	<i>traces</i>	446.9	447.6

Table 2

Degree of extraction (α) and concentration multiplicity (β) of impurities of alcohol during typical and cyclic rectification

Name of the impurity	Conventional distillation		Cyclic distillation	
	α	β	α	β
Aldehydes	86.4	16.9	max	22.5
Esters	79.7	21.1	max	28.1
Fusel oil	21.1	10.0	25.0	16.9
Methanol	16.2	67.4	26.0	102.3
Acrolein	64.7	11.2	max	17.4

Concentration multiplicity (β) of key volatile alcohol impurities (aldehydes and higher alcohols of sivush oil) are presented in the diagram (Fig. 5).

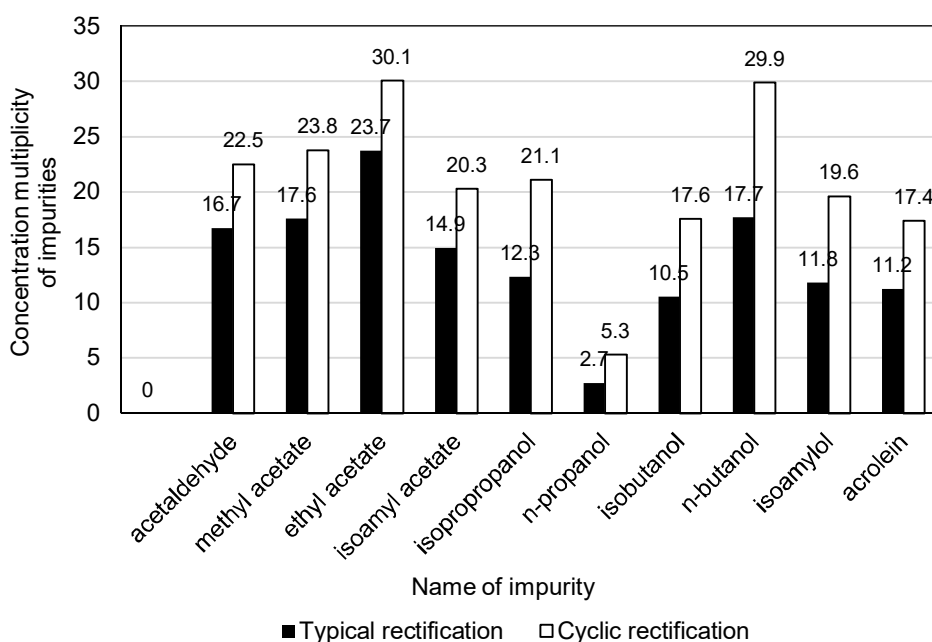


Figure 5. Multiplicity of concentration of (β) of key alcohol impurities (aldehydes and higher alcohols of fusel oil)

The data analysis in Table 2 and the diagrams show the advantages of cyclic rectification over typical rectification. It is experimentally proved that in the process of distillation of alcohol-containing fractions aldehydes, esters (head impurities) and acrolein are removed in full measure, and the degree of extraction (α) of higher alcohols of sivush oil (upper intermediate impurities) increased by 38 %, methanol – by 15.6 %. The multiplicity of concentration (β) of key impurities was increased by 25%, higher alcohols by 40%, methanol by 34%, acrolein by 36%, and isopropyl alcohol by 42%.

When included in the scheme of rectification plant of the impurity concentration column output of rectified ethyl alcohol increased by 3.5–3.7%, its indicators corresponded to the of regulatory documentation.

The costs of heating vapour for the process of rectification were determined from the heat balance by the cost of water for cooling and its temperature at the inlet of the condenser and the outlet of the dephlegmator of column. It was found that, in the conditions of cyclic distillation, heating vapour consumption was reduced by 30% compared to typical plants and did not exceed 12 kg/dal a.a. supplied with feed.

It is proved that for effective separation of the mixture in the mode of controlled cycles of delay and liquid overflow in the impurity concentration column, it is sufficient to install the 30 contact devices mentioned above.

From the experience of operating of the impurity concentration column, it is known that distillation residue is usually returned for a re-cycle of rectification, namely, on the top plate of the ether column (Shiyan et al., 2009). This is due to the fact that the rectification of impurities under the conditions of typical distillation does not always ensure the complete release of the residue from the key organic impurities, which are further concentrated in the process of rectification and impair the quality of the rectified ethyl alcohol. Therefore, rectification plants equipped with typical impurity concentration column require an increased consumption of heating vapour to produce a high quality final product.

The use of innovative cyclic rectification technology allows to obtain the distillation residue, as free from impurities of alcohol. When increasing the contact time of vapour and liquid on the column plates up to 30–40 s in the selected hydrodynamic mode of its operation, the total amount of impurities in the in the marketable product decreases by 94–96% of their quantity in the feedstock. It is expedient to use such distillation residue to conduct hydroselection of impurities in the ether column. The technical solution allows to reduce the consumption of hot softened water for hydroselection, the consumption of heating vapour and prevents the concentration of organic impurities concentration in the distillation unit (Buliy et al., 2019).

Conclusions

1. Hydrodynamic modes of operation of mesh and flake plates in cyclic mode are established: vapour velocity in the free section of the column equipped with mesh plates should not exceed 0.7 m/s, flake plates – 1.2 m/s; optimum vapour velocity in the holes of the mesh plate is 7.5–8.0 m/s, flake plate – 12–14 m/s; for intensive overflow of liquid the vapour velocity in the holes should not exceed 1.5–1.0 m/s.
2. The results of production studies proved the advantages of cyclic rectification for the production of high quality alcohol: in the process of separation of alcohol-containing fractions in full measure head impurities of alcohol are allocated, the degree of extraction of the higher alcohols of sivush oil increases by 38%, methanol – by 15.6%, the multiplicity of concentration of head impurities increases by 25%, higher alcohols – by 40%, methanol – by 34%, acrolein – by 36%, isopropyl alcohol – by 42%.
3. Vapour consumption in the impurity concentration column is reduced by 40% in comparison with typical units operating in the stationary mode and does not exceed 12 kg/dal a.a. introduced with feed.

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