

Improving the efficiency of mass-exchange between liquid and steam in rectification columns of cyclic action

Yurii Bulii, Anatolii Kuts, Ivan Yuryk, Andrii Forsiuk

National University of Food Technologies. Kyiv, Ukraine

Abstract

Introduction. The purpose of the work was to determine the optimal time of residence of the liquid on the plates, the grade of extraction and concentration ratio of volatile impurities of alcohol and the specific consumption of heating steam in rectification columns of cyclic action.

Materials and Methods. The studies were carried out in a rectification column, equipped with flaky plates with a variable free cross-section. Concentration of alcohol volatile impurities was determined by chromatographic method, the grade of their extraction and concentration ratio – by calculation method, other indicators – by commonly known methods.

Results and Discussion. The maximum extraction of volatile impurities was being achieved in a rectification column, equipped with flaky plates containing turnaround sections connected to drive mechanisms, the action of which is occurred according to a given algorithm. The optimal parameters of operating the column were: vapor velocity in the orifices of the flakes during the period of liquid retention on the plates 12-14 m/s; during liquid pouring 1-1.5 m/s; time of residence of the liquid on the plates 40 s, pouring time 1.7 s; pressure in the lower part of the column 12 kPa; the concentration of ethyl alcohol in the still liquid 3-4% vol. In order to provide the cycles, the free sectional area of the plates must change instantaneously from 5.5 to 51.7%. This technical solution allows to provide complete disposal of ethers, methyl acetate and isopropyl alcohol, to increase the grade of extraction of higher alcohols of sивush oil and methanol by 38%, the concentration ratio of aldehydes by 25%, higher alcohols by 38%, methanol by 37%, and to reduce specific consumption of heating steam by 40% compared to a typical column operating in stationary mode.

Conclusion. The innovative technology of cyclic rectification allows to increase the grade of extraction and the concentration ratio of volatile impurities of alcohol by 25-38% and reduce energy consumption by 40% compared with the known ones.

Keywords:

Alcohol
Rectification
Plates
Column
Impurities

Підвищення ефективності масообміну між рідиною і парою в ректифікаційних колонах циклічної дії

Юрій Булій, Анатолій Куц, Іван Юрик, Андрій Форсюк

Національний університет харчових технологій. Київ, Україна

Анотація

Вступ. Метою роботи було визначити оптимальний час перебування рідни на тарілках, ступінь вилучення і кратність концентрування летких домішок спирту і питому витрату гріючої пари в ректифікаційній колоні циклічної дії.

Матеріали та методи. Дослідження проводили в розгінній колоні, оснащій лускоподібними тарілками із змінним вільним перерізом. Концентрацію летких домішок спирту визначали хроматографічним методом, ступінь їх вилучення і кратність концентрування – розрахунковим методом, інші показники – загальновідомими методами.

Результати та обговорення. Максимальне видалення летких домішок досягалось в ректифікаційній колоні, оснащій лускоподібними тарілками, що містять поворотні секції, з'єднані з приводними механізмами, дія яких відбувається за заданим алгоритмом. Оптимальними параметрами роботи колони були наступні: швидкість пари в отворах лусок в період затримки рідини на тарілках 12-14 м/с, в період переливу рідини 1-1,5 м/с, час перебування рідини на тарілках 40 с, час переливу 1,7 с; тиск в нижній частині колони 12 кПа, концентрація етилового спирту в кубовій рідині 3-4 % об. Для забезпечення циклів площа вільного перерізу тарілок повинна миттєво змінюватись від 5,5 до 51,7 %. Технічне рішення дозволяє забезпечити повне видалення естерів, метилацетату та ізопропілового спирту, збільшити ступінь вилучення вищих спиртів сивушного масла і метанолу на 38 %, кратність концентрування альдегідів – на 25 %, вищих спиртів – на 38 %, метанолу – на 37 %, а питомі витрати гріючої пари зменшити на 40 % в порівнянні з типовою колоною.

Висновки. Інноваційна технологія циклічної ректифікації дозволяє підвищити ступінь вилучення і кратність концентрування летких домішок спирту на 25-38 % і зменшити енерговитрати на 40 % в порівнянні з відомими.

Ключові слова: спирт, ректифікація, тарілки, колона, домішки.

Introduction

Technical progress in the alcohol industry is inextricably linked to the development and implementation of highly efficient column apparatuses and energy-saving ways of mass transfer between the liquid and steam on their plates [1-3]. One of the ways of solving the mass transfer process problem is the use of cyclic mode of phase motion, which is based on alternate change of two periods: the steam passing up the column period and the period of liquid pouring on its plates [4,5]. Implementation of controlled cycles of liquid retention on the plates allows to prolong the time of its contact with steam, to create conditions in order to achieve a phase state close to equilibrium and to bring the efficiency of each real plate closer to the theoretical one [6,7]. This significantly reduces the specific consumption of heating steam, decreases the volume of alcohol-containing waste and minimizes the cost of equipment [8-10].

There are well-known ways of increasing the residence time of liquid on the plates by organizing the flow of separate steam–liquid jets with their mutual collision or additional installation of baffles and reflectors, directing the steam through the appropriate bypass pipelines, etc. [11-14]. Despite the obtained positive results in reducing energy costs, the known methods and apparatuses of cyclic operation have not found wide practical application due to the lack of mass transfer in the steam period, the steam pressure dependence of pouring devices' operation, the fluctuations of the steam pressure in the collector, the inability to stabilize the hydrodynamic mode of plates, the mixing of liquid on adjacent plates during its pouring, the low apparatuses' steam and liquid throughput capacity, and the complexity of constructive solutions [15-21].

The authors proposed an innovative rectification technology, which excludes earlier mentioned disadvantages and provides periodic liquid pouring from one plate to another at continuous supply of liquid and heating steam into the column [22,23]. To implement the technology, the design of a rectification column equipped with plates with variable free cross-section was developed [24]. For stable operation of plates in the column hydrodynamic regimes were maintained, providing effective mass transfer between liquid and steam without entrainment of liquid on upper plates during the fluid retention period and its intensive pouring through pouring and barbotage holes after the end of the retention time.

The aim of the work was to study the efficiency of mass-exchange between liquid and steam in column apparatuses of cyclic action: to determine the grade of extraction and the concentration ratio of volatile impurities of alcohol during its extraction from alcohol-containing fractions and to identify the specific rate of heating steam in the studied rectification column.

Research objectives:

1) to determine the grade of extraction and the concentration ratio of alcohol impurity concentrations under conditions of typical and cyclic rectification (in columns equipped with moving valves and turning plate sections);

2) to determine the optimal technological parameters of the studied column and the residence time of the liquid on its plates, by which the maximum extraction of volatile impurities is provided without reducing the liquid throughput of the column;

3) to determine the specific rate of heating steam in a rectification column of cyclic action.

Materials and methods

Research objects

1. Rectification columns of cyclic action with moving valves (RC).

The RC is made of stainless steel AISI 304, equipped with flaky plates of arched type. Technical characteristics: diameter – 426 mm; number of plates – 30; distance between the plates – 300 mm; the cross-sectional area of flakes' holes – 19,42 mm²; thickness of the plate fabric – 2 mm; free cross-section of the plate: 5,5% – during the residence of the liquid on the plates – 5,5%; during the liquid pouring – 51,7%.

A fragment of the RC with movable rods, valves and hydraulic shutters is shown in Figure 1a (patent UA 116565. Rectification column with controlled cycles). The operation of the column provided the conducting of the adjustable in time cycles of liquid residence on the plates and its synchronous pouring from one plate to another 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 (patent UA 89874. Method of liquid pouring on plates of column apparatus in the process of mass transfer between steam and liquid). The interval of liquid retention was being determined experimentally depending on the grade of extraction of volatile alcohol impurities and their concentration ratio.

The experimental RC was included in the scheme of the bragorectificational plant (BRP). The column contained corps *1*, plates *2* with contact elements *3*, movable rods *4* and *5*, on which valves *6* and springs *7* were mounted. The rods moved up and down under the action of drive mechanisms (double-acting pneumatic cylinders of DNT type manufactured by FESTO). At that, valves *6* closed and opened the holes of pouring pipes *8* alternately. The operation of pneumatic cylinders was managed in accordance with the M340 controller program of 'Schneider Electric' company. Pipes *8* were inserted into glasses *9* and together with them served as water traps, which prevented steam breakthrough through all the holes during liquid pouring.

Figure 1b shows an experimental RC with movable rods and valves without hydraulic shutters (patent UA 139228. Column mass-exchange apparatus of cyclic action). The technical solution allowed one-stage (full) and two-stage methods of liquid pouring on plates (Figure 2). The one-step method involved pouring all the liquid from one plate to another (Figure 2a). According to the two-stage method (patent UA 141245. Method of pouring the liquid on the plates of mass-exchange column apparatus) part of the liquid had been pouring from the upper plate to the lower one (30-70% of its volume), and after a specified delay time, its remnants were poured (Figure 2b).

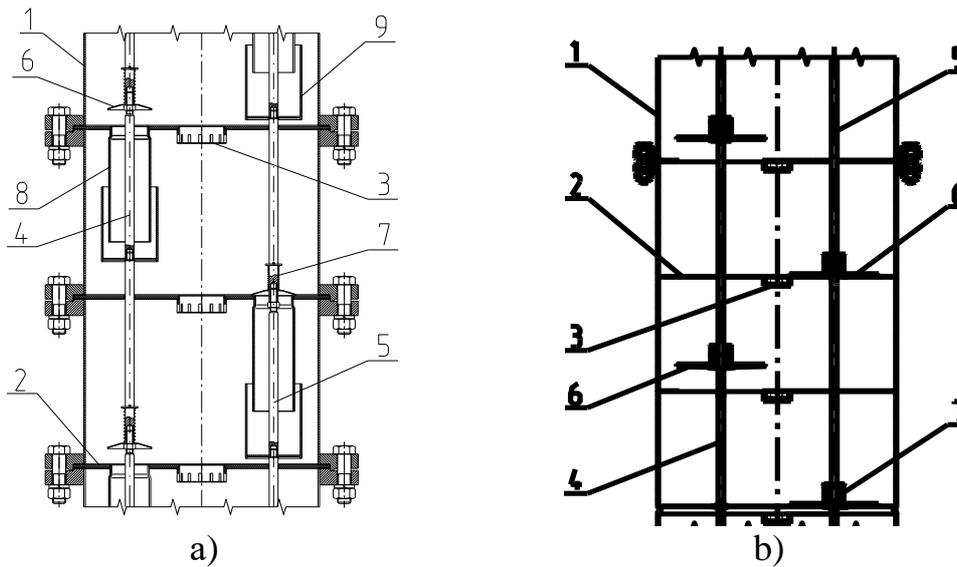


Figure 1. Fragments of the studied RC with movable valves: with hydraulic shutters (a) and without hydraulic shutters (b): 1 – body; 2 – plates; 3 – contact devices; 4, 5 – rods; 6 – valves; 7 – springs; 8 – pouring pipes; 9 – glasses.

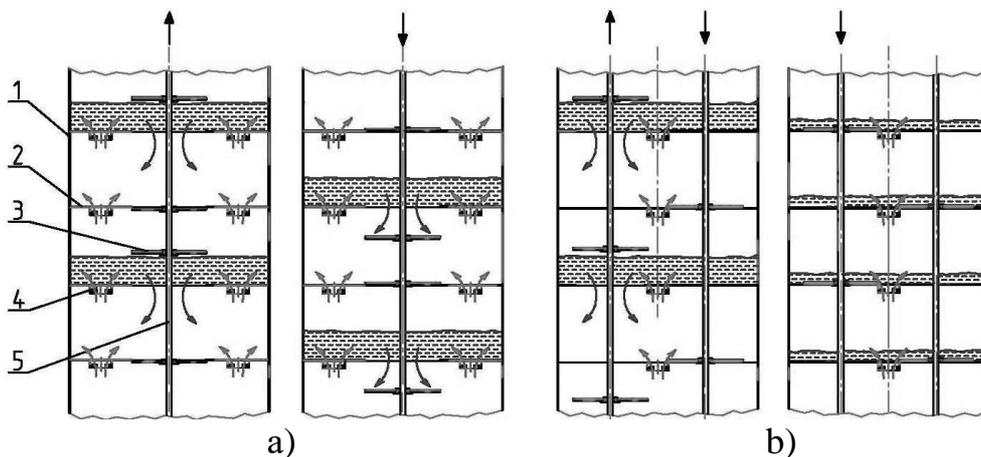


Figure 2. One-stage and two-stage methods of liquid pouring on the plates of the cyclic action RC:

1 – body; 2 – plate; 3 – valve; 4 – contact element; 5 – moving rod.

2. The plant for ethyl alcohol extraction from alcohol-containing fractions.

The scheme of the implementation of the studied RC into the BRP one is shown in Figure 3.

The plant included the experimental column 6, the upper and lower parts of which are connected to the vacuum breakers 4, evaporator 5, deflegmator 7, condenser 8, alcohol-collecting vessel (trap) 9, softened water collector 1, intermediate collectors of cube aqueous-alcoholic liquid 15 and alcoholic fractions 18, flow-meters 3, 11, 12 and 13, centrifugal pumps 2, 16, 17 and decantator 10.

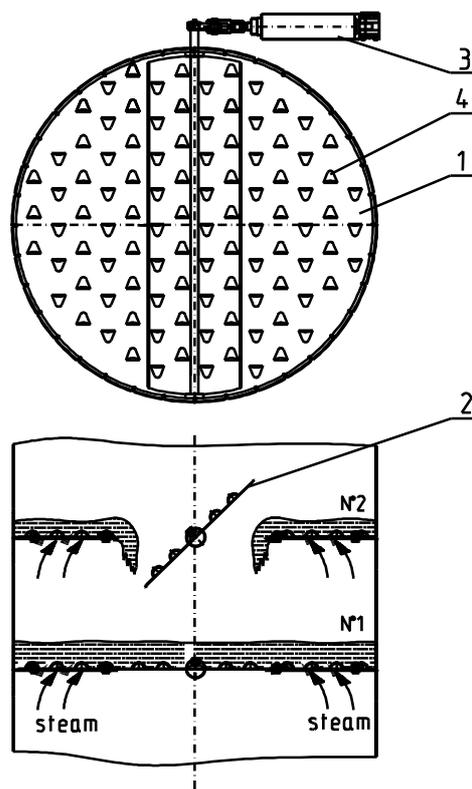


Figure 4. Flaky plate of cyclic action with coaxial placement of flakes and variable free cross-section
1 – plate fabric; 2 – moving section; 3 – drive mechanism (pneumatic cylinder); 4 – flakes.

The moving sections opened and closed the pouring holes of the plates so that the liquid pouring occurred periodically. The coaxial placement of the flakes made it possible to eliminate the ‘one-way’ steam and liquid flow and the chance of forming stagnant zones. Pneumatic cylinders and technological parameters operation control (i. e. temperature, pressure) was carried out with the help of automatic sensors, the signal from which was transmitted to the microprocessor controller.

The head fraction of ethyl alcohol, steam condensate from the condensers of the distillation column and carbon dioxide separator, as well as sivush alcohol and sivush rinse water were served on the feeder plate of the column in total amount of 688.3 dm³/h (250 dm³/h in terms of anhydrous alcohol (a.a.)). The aldehyde-methanol concentrate from the condenser and the sivush ester-aldehyde concentrate from the upper part of the decanter were sorted to the impurity concentrate collector.

Research methods

1. Liquid consumption. The consumption of alcohol-containing fractions, water for hydroselection, the cube liquid and rectified alcohol was monitored using PM flow-meters [25].

2. Concentration of ethyl alcohol in water-alcohol solutions. The concentration of ethyl alcohol in the cube liquid of the RC was determined by areometric method [26].

3. Concentration of volatile alcohol impurities. The concentration of volatile impurities in the head fraction, in the condensate steam from the condensers of the distillation column and carbon dioxide separator, in sivush alcohol, in sivush rinse water and in the feed of the column were implemented on a gas chromatograph with an HP FFAP 50 m × 0.32 m column [27,28]. Three-time repetition samples were taken for chromatographic analysis. The mean values were chosen as the determining ones.

4. The grade of extraction and concentration ratio of volatile alcohol impurities. The grade of extraction (α) and concentration ratio (β) of key organic alcohol impurities were calculated by the formulas:

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

where X_l , X_{seac} , X_{cub} – the concentration of volatile alcohol impurities on the feed plate, sivush ester-aldehyde concentrate and cube aqueous-alcoholic liquid, mg/dm³ in terms of a.a. [1].

Studied modes

It is known, that for flaky plates the lower critical speed of steam in holes, at which liquid spilling stops, is 6,5-7,5 m/s, linear speed in free cross-section of the column in barbotage mode is 0,5-0,9 m/s, in transitional 0,9-1,3 m/s and in jet 1,3-2,0 m/s. Upper critical speed of steam is 15-16 m/s [29]. Intensive liquid pouring through the holes of the plates occurs at steam velocities of 1.5-1 m/s [30].

Considering the above, the velocity of steam in the holes of flakes during the liquid residence on the plates of the studied RC was maintained within 12-14 m/s.

The extraction of ethyl alcohol from alcohol-containing fractions was carried out under the circumstances of moderate and deep hydroselection. Therefore, the upper plate of the column was provided with steam condensate, the temperature of which was 90-92 °C. The condensate consumption was being increased from 2000 to 4500 m³/h. Yet the concentration of ethyl alcohol in the cube liquid of the column varied from 2.8 to 8% vol. Depending on the quantity of liquid, the residence time on the plates was being varied from 20 to 60 s, the pouring time — from 7 to 1.7 s. The height of the liquid layer on the plates was 35–40 mm. Depending on the quantity of alcohol-containing fractions and water for hydroselection the pressure at the bottom of the column was being varied between 12 and 18 kPa. For an effective separation of the heterogeneous mixture, the decanter temperature of the RC was being maintained around 30-35 °C [1]. The aldehyde-methanol concentrate and the sivush ester-aldehyde concentrate were being changed from 12 to 1 dm³/h, while controlling the quality parameters of the RC cube aqueous-alcoholic liquid and rectified ethyl alcohol.

Stages of research

At the first stage, the efficiency of the mass transfer process in the typical [31] and cyclic [32] rectification in the existing and experimental RC with hydraulic gates was investigated (Figure 1a). The head fraction of ethyl alcohol, steam condensate from the condensers of the distillation column and carbon dioxide separator, as well as sivush alcohol were served on the feeder plate of the column in total amount of 96 dm³/h in terms of anhydrous a.a. Heating steam was continuously provided to the lower part of the column and hot softened water – to the upper plate in order to hydroselect the impurities, which ranged the concentration of ethanol in the cube liquid from 4-5% vol. The residence time of the liquid on the plates was 23 s and the pouring time through the hydraulic shutters was 7 s.

At the second stage the efficiency of mass exchange between liquid and steam in an experimental RC of cyclic action without hydraulic shutters was investigated (Figure 1b). The technical solution suggested by the authors provided time-controlled cycles of liquid residence on the plates and its pouring from the upper plates to the lower ones, thanks to instantaneous change of steam velocity in the holes from 12-14 to 1,5-1 m/s by changing free cross-section of the plates from 5,5 to 51,7 %. While the valves were being lifted at the moment the pouring holes were opened, the steam velocity in the holes became lower than critical and the liquid was pouring simultaneously through all the holes to the underlying plates.

At the third stage of the research the optimal parameters of mass exchange process of the experimental RC operation (Figure 3), equipped with flaky plates with turnaround sections, presented in Figure 4. The research included liquid sampling at the feeder plate (FL) as well as samples of the head fraction (HF), sivush alcohol (SA), sivush rinse water (SRW), fractions from the distillation column condenser (DCC) and carbon dioxide separator condenser (CDSC). To determine the efficiency of processing alcohol-containing fractions in a given hydrodynamic mode, the concentration of volatile impurities of alcohol in the cube liquid (CL), impurities concentrate (IC), epyurate (E), and rectified ethyl alcohol (REA) were studied. The results of the chromatographic analysis of the studied samples are shown in Tables 1 and 2.

Results and discussion

Studies on the efficiency of mass exchange between liquid and steam in RC, equipped with moving valves and plates with hydraulic shutters.

Studies have shown that in the experimental column the esters were completely removed. The grade of extraction of higher alcohols of sivush oil and methanol in the cyclic mode increased by 25%, the concentration ratio of head impurities – by 21%, higher alcohols and methanol – by 30% in comparison with the column operating in the stationary mode. That said, it reduced the specific heating steam consumption by 38% and 1.2 kg/dal of a.a. introduced into the column. This is explained by the fact that when the phase contact time had been prolonged from 13 to 23 s, the difference in concentration of volatile impurities in steam and liquid decreased, thus increasing the grade of phase equilibrium [33].

The disadvantages were the low liquid capacity of the column (750 dm³/h), its mixing on adjacent plates during pouring and a 15% reduction in the working area of the plate due to the presence of the hydraulic shutters.

Studies on the efficiency of mass exchange between liquid and steam in RC, equipped with moving valves and plates without hydraulic shutters.

Design changes allowed to increase the liquid throughput by 34% (750 to 1000 dm³/h) without reducing the liquid retention time by lessening the pouring time from 7 to 2 s. Due to the absence of hydraulic shutters, the contact area of the phases on each plate has increased by 15%, which has improved the performance of the plates and the efficiency of the mass exchange: the grade of extraction of higher alcohols of sivush oil and methanol was increased by 29 %, the concentration ratio of aldehydes was increased by 23 %, higher alcohols – by 33 % and methanol by 34 % compared to a column operating in stationary mode.

One-stage (full) and two-stage pouring methods.

The one-stage (full) pouring method did not provide an even distribution of liquid on the plates due to a lack of liquid on the paired plates while it being held on the unpaired plates and vice versa (Figure 2a). This technical decision made it impossible to maintain a stable hydrodynamic regime along the height of the column [34].

In order to optimize the operation of the RC and to increase the efficiency of mass exchange, the pouring of liquid from plate to plate was carried out in two stages (Figure 2b). The method allowed to operate all the plates simultaneously, to ensure that the liquid level on the plates is the same throughout the height of the column and to stabilize the hydrodynamic mode of their operation. At that, the RC liquid throughput has increased by 20% (from 1000 to 1200 dm³/h), the grade of extraction of higher alcohols of sivush oil and methanol — by 38%, the concentration ratio of head impurities has increased by 25%, higher alcohols – by 38%, methanol – by 37% compared to a column operating in stationary mode.

The disadvantage of the one- and two-stage methods of liquid pouring is the impossibility of autonomous regulation of liquid residence time on each individual plate, because moving elements of pouring devices of paired and unpaired plates were set in motion by one drive mechanism.

Studies on the efficiency of mass exchange between liquid and steam in RC, equipped with plates with rotary sections.

To eliminate the disadvantages mentioned above, the authors have proposed a method of processing alcohol-containing fractions in a column equipped with plates with rotary sections (patent UA 136560. Method of mass-exchange between liquid and steam in a column apparatus). The results of chromatographic analysis of alcohol-containing fractions entering the column and the distribution of impurities in its cube liquid, concentrate, epyurate and rectified alcohol are presented in Tables 1 and 2.

The criterion for the RC optimization was the concentration of acetaldehyde, higher alcohols of sivush oil (including isopropyl alcohol) and methanol in the cube liquid and in the rectified ethyl alcohol. The determinants of mass exchange efficiency between liquid and steam were the grade of extraction and concentration ratio of volatile alcohol impurities in the studied RC.

Table 1

The results of the chromatographic analysis of alcohol-containing fractions

Impurity name	Concentration, mg/dm ³					
	HF	CDC	CDSC	SA	SRW	FL
Ethanol, % об.	92,5	48,8	60	89	17,5	30,5
Aldehydes	1135,2	37,2	126,2	4,9	7,0	318,7
acetaldehyde	926,1	37,2	90,9	4,9	7,0	242,3
methylacetate	209,1	traces	35,3	traces	traces	76,4
Esters	2394,9	186,4	39,7	20,2	68,3	40,5
ethylacetate	2223,6	165,8	traces	2,1	traces	trace
isobutylacetate	23,0	13,0	7,9	10,1	traces	11,1
isoamylacetate	90,6	7,6	31,8	8,0	68,3	29,4
ethylbutyrate	57,7	traces	traces	traces	traces	traces
Methanol, %	0,49	0,025	0,1445	0,013	0,0032	0,18
Sivush oil	3113,1	18820	12583	48824	197726	105883
isopropanol	4,9	4,9	1,7	1,1	traces	1,2
n-propanol	1186,4	1403	699,6	14741	36681	20002
isobutanol	1640	606,1	4082	27557	36826	20297
n-butanol	2,7	6,4	16,5	35	705,2	362
isoamylol	279,1	1863,5	7783	6485,3	123514	65221

Table 2

Concentration of impurities in the cube liquid, impurities concentrate, epyurate and rectified ethyl alcohol

Impurity name	Concentration, mg/dm ³			
	CL	IC	E	REA
Ethanol, % об.	3,7	67	30,1	96,5
Aldehydes	2,8	2302,2	0,3	0,18
acetaldehyde	2,8	1396,7	0,3	0,18
methylacetate	traces	905,5	traces	—
Esters	traces	446615	traces	—
isobutylacetate	traces	3234,8	traces	—
isoamylacetate	traces	494,4	traces	—
ethylbutyrate	traces	442886	traces	—
Methanol, %	0,004	2,69	0,0023	0,0003
Sivush oil	721,7	726464	1179,8	0,88
isopropanol	traces	22,4	0,4	0,88
n-propanol	677,5	220,6	121,4	—
isobutanol	4,9	357247	326,0	—
n-butanol	2,7	1003,8	2,0	—
isoamilol	13,8	367970	728,5	—

According to the results of the study, optimal technological parameters of RC operation were:

- the liquid retention time on the plates is 40 s;
- the time of liquid pouring from the upper plate to the lower one is 1.7 s;
- the pressure in the cubic part of the column is 11.5-12 kPa;
- pressure in the upper part of the column is up to 0.03 kPa;
- the temperature in the cubic part is 100.5-101.5 °C;
- the temperature in the steam phase above the upper plate is 93.5-94 °C;
- temperature in the steam phase on the plate of feed is 93.2-94 °C;
- the water temperature for hydroselection is 95-98 °C;
- the temperature of the mixture in the decanter is (30–35) °C;
- water consumption for hydroselection is 4050-4500 dm³/h;
- the temperature in the inner-tube space of the condenser is 45-50 °C;
- water temperature for cooling after the dephlegmator is 85-87 °C;
- concentration of ethyl alcohol in the cubic liquid is 3-4 % vol.;
- withdrawal of aldehyde-methanol concentrate (AMC) from the RC is 7-9 dm³/h;
- concentration of ethyl alcohol in the AMC is 70.5% vol.;
- withdrawal of the sivush and ester-aldehyde concentrate (SEAC) from the decanter is 2-3 dm³/h.

The calculated values (α) and (β) at RC operation in the selected hydrodynamic mode and the specified optimal technical parameters are shown in Table 3.

Table 3

Calculated values of the grade of extraction (α) and concentration ratio (β) of volatile alcohol impurities

Name of impurities	Typical rectification		Cyclic rectification	
	α	β	α	β
Aldehydes	85,4	5,3	113,8	7,2
acetaldehyde	63,7	4,3	86,5	5,8
methylacetate	∞	8,8	∞	11,9
Esters	79,7	8163,7	∞	11027
isobutylacetate	57,8	214,7	∞	291,4
isoamylacetate	∞	12,3	∞	16,8
Methanol	27,6	9,3	45	14,9
Sivush oil	89,8	4,1	146,7	6,9
isopropanol	87	10,9	∞	18,7
n-propanol	17,9	0,005	29,5	0,01
isobutanol	2414,4	10,5	4142,2	17,6
n-butanol	82,9	1,6	134,1	2,8
isoamylol	3953,2	3,3	4726,2	5,6

Result Analysis

Analysis of the obtained results showed that by increasing the contact time of steam and liquid on the RC plates to 40 s the grade of extraction and concentration ratio of volatile alcohol impurities increased by 25-38%. At the same time, complex esters, methylacetate and isopropyl alcohol are completely extracted – those are the impurities that significantly degrade the quality of rectified alcohol in small amounts. This can be explained by the fact that there was more of a complete steam saturation with its volatile components on the plates of the column and liquid with volatile steam components, the mixing of liquid on adjacent plates during its pouring was excluded, so that the grade of phase equilibrium achievement was increased [35,36]. The prolonging in the residence time of the liquid on the plates longer than 40 s proved to be impractical due to an increase in the specific heating steam consumption without a significant increase in the grade of impurity extraction.

Specific consumption of heating steam in experimental RC decreased by 40% (from 20 to 12 kg/dal of a.a. injected to the feed plate) compared to the column operating in the stationary mode. This is explained by the fact that the free cross-sectional area of the plates in the column of cyclic action was 50-75% smaller than that of the column operating in the stationary mode, and was 2.5-5.5% [37].

After the experimental distillation column for concentrating impurities was put into operation, the yield of rectified ethyl alcohol increased by 3.8% due to its extraction from the head fraction and other alcohol-containing waste without deteriorating its qualitative indicators. The use of the RC liquid purified from volatile impurities for carrying out hydroselection in the epurating column made it possible to reduce the consumption of hot softened water by 2000 dm³/h (patent UA 119277. Method of producing rectified alcohol) [38].

Conclusion

1. To increase the efficiency of mass exchange between liquid and steam in rectification columns the expediency of using a cyclic rectification technology that provides periodic pouring of liquid from plate to plate at continuous supply of alcohol-containing fractions and steam in the column is proved.

2. To implement the technology, the plates have to be equipped with moving sections connected to driving mechanisms (e.g., pneumatic cylinders), which are controlled according to the program of the controller in consonance with a predetermined algorithm.

3. Equipping the columns with flaky plates allows to increase their capacity by 34% due to intensification of liquid pouring by doing so simultaneously through the pouring and barbotage holes.

4. At the moment of liquid pouring, steam velocity in barbotage holes should be 1,5-1 m/s. At this speed the pouring occurs within 1.7 s.

5. To ensure stable operation of the plates during the period of liquid retention and in order to intensify its pouring, their free cross-section area should instantly change from 5.5 to 51.7%.

6. In working environment the optimal technological parameters of column operation of cyclic action were established. It is experimentally proven, that prolonging the contact time of steam and liquid up to 40 s allows to increase the grade of extraction and concentration ratio of volatile impurities of alcohol by 25-38% compared to a column operating in stationary mode. In doing so, the complete extraction of esters, methyl acetate and isopropyl alcohol is provided.

7. The coaxial placement of the flakes on the plate fabric allows to eliminate the possibility of formation of stagnant zones and intensify the mass transfer between steam and liquid.

8. The use of innovative technology makes it possible to reduce the specific consumption of heating steam during processing of alcohol-containing fractions by 40% compared to the known ones.

9. It is advisable to use the results of the research to design column mass transfer apparatuses of cyclic action.

References

1. Shyian P.L., Sosnytskyi V.V., Oliinichuk S.T. (2009), *Innovatsiini tekhnolohii spyrtovoi promyslovosti. Teoriia i praktyka: Monohrafiia*, Vydavnychiy dim Askania, Kyiv.
2. Kyziun H.O., Mishchenko O.S., Mikhnenko Ye.O. i inshi (2006), Enerhozberezhennia na brahorektyfikatsiinykh ustanovkakh, *Koleha, NUKhT*, 6-8, pp. 52-54.
3. Martseniuk O.S. Malezhyk I.F., Zotkina L.V. (2019), Tarilchasti aparaty ta yikh udoskonalennia, *Zhurnal Naukovi pratsi NUKhT*, 25(2), pp. 121-133.
4. Maleta V.N., Kiss A.A., Taran V.M., Maleta B.V. (2011), Understanding process intensification in cyclic distillation systems. *Chemical Engineering and Processing: Process Intensification*, 50(7), pp. 655-664.
5. Kiss AA, Flores Landaeta SJ, Edwin Zondervan CJ. (2012), Cyclic distillation – towards energy efficient binary distillation. *Chemical Engineering and Chemistry*, 30, pp. 697-701.
6. Maleta B.V., Taran V.N., Maleta V.N. (2010), Spivstavlennia tsyklichnoho ta statsionarnoho protsesu rektyfikatsii, *Zhurnal Naukovi pratsi NUKhT*, 33, S.S. 95-97.
7. Buliy Y., Shiyani P., Kuts A., Melnic I. (2019), Technology of cyclic rectification of alcohol production, *Journal of Food Science and Technology*, 13, pp. 104-111.
8. Kiss AA, Olujic Z. (2014), A review on process intensification in internally heat-integrated distillation columns, *Chem Eng Process*, 86, pp. 125-144.
9. Kiss A. (2015), Pilot-scale studies of process intensification by cyclic distillation // *AIChE Journal*, 61, pp. 2581-2591.

10. Maleta B., Taran V., Maleta V. (2010), Energy-saving technology of mass transfer in tray columns with separate phase movement, *The 6 th European Meeting on Chemical Industry and Environment EMChIE (Mechelen, Belgium), Conference Proceeding*, 2, pp. 1149-1155.
11. Krivosheev V., Anufriev A. (2015), Fundamentals and efficiency of cyclic modes of rectification process. Basic Research, *Scientific Journal of Basic Research*, 11(2), pp. 267-271.
12. Kiss AA, Bildea CS. (2011), A control perspective on process intensification in dividing-wall columns. *Chemical Engineering and Processing: Process Intensification*. 50(3), pp. 281-292.
13. Pătruț C, Bildea CS, Liță I, Kiss AA. (2014), Cyclic distillation – design, control and applications. *Separation and Purification Technology*, 125, pp.326–336.
14. Kiss A. (2015), Pilot-scale studies of process intensification by cyclic distillation, *AIChE Journal*, 61, pp. 2581-2591.
15. Lita I., Bildea C. S., Kiss A. A. (2012), Modeling, design and control of cyclic distillation systems, *Procedia Engineering*, 42, pp. 1311-1322.
16. Flodman H. R., Timm D.C. (2012), Batch distillation employing cyclic rectification and stripping operations, *ISA Transactions*, 51, pp. 454-460.
17. Kiss A.A. (2014), Distillation technology—still young and full of breakthrough opportunities, *J Chem Technol Biotechnol*, 89, pp. 479-498.
18. Toftegard B., Clausen C.H., Jorgensen S.B., Abildskov J. (2016), New Realization of Periodic Cycled Separation, *Ind. Eng. Chem. Res*, 55(6), p. 1720.
19. Rasmus Fjordbak Nielsen, Jakob Kjøbsted Huusom, Jens Abildskov, Driving Force Based (2017), Design of Cyclic Distillation, *Industrial & Engineering Chemistry Research*, 56(38), pp. 10833-10844.
20. Bastian B. Andersen, Rasmus F. Nielsen, Isuru A. Udugama, Emmanouil Papadakis, Krist V. Gernaey, Jakob K. Huusom, Seyed Soheil Mansouri, Jens Abildskov (2018), Integrated Process Design and Control of Cyclic Distillation Columns, *IFAC-PapersOnLine*, 51(18), pp. 542-547.
21. Jess Bjørn Rasmussen, Seyed Soheil Mansouri, Xiangping Zhang, Jens Abildskov, Jakob Kjøbsted Huusom (2020), A mass and energy balance stage model for cyclic distillation, *AIChE Journal*, 66(8), pp. 1002.
22. Buliy Y., Shiyani P., Kuts A. (2016), Distillation of alcoholic distillate in controlled cycles mode, *Food Science for Well-being (CEFood 2016): 8th Central European Congress on Food 2016*, Kyiv, NUFT, p. 230.
23. Ukrainets A., Shiyani P., Buliy Y., Kuts A. (2018), Increasing the efficiency of the distillation unit, *Scientific Works of National University of Food Technologies*, Kyiv, NUFT, 24(6), pp. 160-166.
24. Buliy Yu.V., Kuts A.M., Shiyani P.L. (2019), Pidvyshchennia ekspluatatsiinykh kharakterystyk masoobminnykh kolonnykh aparativ tsyklichnoi dii, *Zhurnal Naukovi pratsi NUKhT*, 25(5), pp. 48-54.
25. Yarovenko V.L., Ustinnikov B.A., Bogdanov YU.P., Gromov S.I. (1981), *Spravochnik po proizvodstvu spirta*, Legkaya i pishchevaya promyshlennost', Moskva.

26. Polygalina G.V. (1999), *Tekhnohimicheskij kontrol' spirtovogo i likero-vodochnogo proizvodstva*, Kolos, Moskva.
27. Plutowska B., Wardenski W. (2008), Application of gas chromatography-olfactometry (GC-O) in analysis and quality assessment of alcoholic beverages – a review, *Food Chemistry*, 107, pp. 449-463.
28. Polyakov V.A. i dr. (2007), *Instrukciya po tekhnohimicheskomu i mikrobiologicheskomu kontrolyu spirtovogo proizvodstva*, DeLi print, Moskva.
29. Marija Stojkovic, Vincent Gerbaud, Nataliya Shcherbakova. (2018), Cyclic operation as optimal control reflux policy of binary mixture batch distillation, *Computers & Chemical Engineering*, 108, pp. 98-111.
30. Van Gerven T., Stankiewicz A. (2009), Structure, energy, synergy, time—the fundamentals of process intensification, *Ind Eng Chem Res*, 48, pp. 2465-2474.
31. Mishchenko O. S., Kyziun H. O., Mozharovska A. A., Oliinyk S. I. (2020), Enerhoefektyvna tekhnolohiia pererobky fraktsii holovnoi etylovoho spyrtu z otrymanniam spyrtu etylovoho rektyfikovanoho, *Naukovyi zhurnal Kharchova promyslovist*, NUKhT, 28, pp. 115-122.
32. Maleta B.V., Shevchenko A., Bedryk O., Kiss A.A. (2015), Pilot-scale studies of process intensification by cyclic distillation, *AIChE Journal*, 61, pp. 2581-2591.
33. Ayat Bozeya, Abeer Al-Bawab, Stig E. Friberg & Clarence A Miller. (2013), Spontaneous Emulsification and Phase Equilibria in the System Water, Ethanol, and Benzene, *Journal of Dispersion Science and Technology*, 34(10), pp. 1429-1436.
34. Chu G. W., Gao X., Luo Y., Zou H. K., Shao L., Chen J. F. (2013), Distillation studies in a two-stage counter-current rotating packed bed, *Separation & Purification Technology*, 102, pp. 62-66.
35. Chen H., Huang K., Wang S. (2010), A novel simplified configuration for an ideal heat-integrated distillation column (ideal HIDiC), *Separation & Purification Technology*, 73, pp. 230-242.
36. Shiyani P.L., Tsygankov P.S., Buliy Yu.V., Mozgovaya O.I. (1991), Fazovoe ravnovesie v sisteme etanol–voda pri davleniyakh nizhe atmosferного (soobshchenie II). Eksperimental'nye issledovaniya i termodinamicheskaya proverka rezul'tatov, *Zhurnal «Izvestiya vuzov. Pishchevaya tekhnologiya»*, 1(3), pp. 114-116.
37. Bausa J., Tsatsaronis G. (2001), Reducing the energy demand of continuous distillation processes by optimal controlled forced periodic operation, *Computers & Chemical Engineering*, 25, pp. 359-370.
38. Ukrainets, A. I., Shiyani P.L., Sosnytskyi V.V. (2006), Perspektyvni napriamky enerhozberezhennia v spyrtovomu vyrobnytstvi, *Zhurnal Kharchova i pererobna promyslovist*, 4, pp. 4-12.

Authors' Information

1. Yurii Volodimirovych Bulii (*responsible author*)

Candidate of Technical Sciences, Associate Professor, Department of Biotechnology of Fermentation Products and Winemaking

National University of Food Technologies

Address: 01601 Kyiv-33, Volodymyrska St., 68, building "Zh", k. 500

phone (044) 287-96-30, tel. mob. (098) 762-65-52 (*vyber*)

e-mail: yvbuliy@gmail.com

2. Anatolii Mikhailovich Kuts

Candidate of Technical Sciences, Associate Professor, Head of the Department of Biotechnology of Fermentation Products and Winemaking

National University of Food Technologies

Address: 01601 Kyiv-33, Volodymyrska St., 68, building "Zh", k. 508

phone (044) 287-96-30, tel. mob. (067) 463-13-03 (*vyber*)

e-mail: anatolykuts@ukr.net

3. Ivan Ivanovych Yuryk

D. in Physics and Mathematics, Professor, Head of the Department of Higher Mathematics named after Prof. V.I. Mozhar

National University of Food Technologies

Address: 01601 Kyiv-33, Volodymyrska St., 68, building "B", k. 208

phone (044) 284-30-04, tel. mob. (097) 916-30-29 (*vyber*)

e-mail: i.yu@ukr.net

4. Andrii Vasilyovich Forsiuk

Candidate of Technical Sciences, Associate Professor, Dean of the Faculty of Automation and Computer Systems

National University of Food Technologies

Address: 01601 Kyiv-33, Volodymyrska St., 68, building "A", k. 430

phone (044) 287-96-06, tel. mob. (067) 445-65-34 (*vyber*)

e-mail: forsyuk-andrii@ukr.net

10.06.2021 p.