Resource- and energy-saving methods of joint processing of by-products and intermediates in alcohol production

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Abstract

Introduction. The aim of the work was to study and substantiate the effectiveness of energy-saving methods for the joint processing of alcohol-containing fractions in a cyclic action column, to increase the degree of alcohol purification from volatile impurities.

Materials and methods. The studys was carried out in a typical impurity concentration column and a experimental cyclic action column. The liquid flow rate was monitored using constant differential pressure flowmeters, the concentration of ethyl alcohol and volatile impurities was determined by areometric and chromatographic methods, the degree of impurity emission and the multiplicity of their concentrating were determined by the calculation method.

Results and discussion. The use of the methods proposed by the authors makes it possible to carry out joint processing of by-products and intermediate products of alcohol production (head and fusel fractions) in a cyclic action rectification column equipped with scaly plates with a variable free cross-section, to obtain high-quality rectified alcohol, to increase its yield by 3.8-4.0% from one tonne of notional starch or by 10.8% compared to the known method and to reduce specific vapor consumption by 40% (from 20 to 12 kg/dal of anhydrous alcohol introduced to the feed plate). Extending the contact time of steam and liquid on the column plates to 40 sec allows for complete emission of esters, increasing the degree of aldehyde recovery by 25% and the higher alcohols of fusel oil and methanol by 40%. The proposed technical solutions and selected technological modes make it possible to increase the efficiency of separation of the alcohol-containing mixture in the decanter, increase the multiplicity of concentrating of aldehydes and esters by 26%, higher fusel oil alcohols by 40%, methanol by 37%, reduce the loss of ethyl alcohol with the impurity concentrate, the amount of alcohol-containing waste, the metal consumption of technological equipment and the cost of rectified alcohol.

Conclusion. The proposed methods allow the maximum purification of ethyl alcohol from head and intermediate impurities in a cyclic action rectification column, to obtain high-quality rectified alcohol, to reduce energy consumption and loss of alcohol with waste.

Introduction

The rise of energy prices is the reason for the development and implementation of energy-saving ways to increase the yield of rectified ethyl alcohol by processing alcohol-containing by-products. Intermediates products include vapor condensates from the condenser of the beer and rectification columns, from the carbon dioxide separator condenser and alcohol traps, fusel alcohol, fusel rinse water and unpasteurized alcohol.

It is known that the yield of rectified ethanol in typical distillation equipment is 93–95% of the amount of alcohol introduced with the beer. Part of the alcohol (0.8–1.2%) is lost with waste (stillage, luther water and non-condensable gases). With the head fraction and fusel alcohol, 3–5% of ethyl alcohol is emitted from the unit, with fusel oil 0.3-0.45% of the alcohol. The anhydrous part of the head fraction contains 92–97% ethyl alcohol and 3-8% volatile impurities. The inclusion of a impurity concentration column (ICC) in the technological scheme allows to increase the yield of rectified ethyl alcohol from 94–96 to 98–98.5% and to emitted volatile impurities from the unit in concentrated form (Shiyan et al., 2009). The composition of fusel alcohol contains 25–30% water, 45–60% ethanol, 5–20% higher alcohols $C_3...C_5$ (mainly propanol and isobutanol), 0.3-0.8% esters, small amounts of volatile nitrogenous substances, aldehydes and acids. The fusel alcohol is taken from the 18th, 20th, 22nd, and 24th plates of the rectification column in the amount of 0.8–2.5%, and the fusel fraction from the 5th, 7th, 9th, and 11th plates of the column in the amount of 3–5% of the amount of alcohol introduced to the feed plate. Its ethanol content is 5–40%. For ethanol extraction, fusel alcohol, together with the fusel fraction, is fed to a fusel column (Mendoza-Pedroza et al., 2021).

The inclusion of additional columns in the scheme of the distillation equipment requires increased energy consumption, increased metal consumption of equipment, which leads to a decrease in the cost of rectified alcohol (Biasi et al., 2020). Thus, the use of ICC requires an increase in the consumption of heating vapor to 20 kg/dal in terms of anhydrous alcohol (a.a) introduced into the column, and hot softened water for hydroselection in the amount of 11.7–20.5 kg/kg a.a, depending on the type of raw material.

The efficiency of the ICC is determined by the degree of distillation residue from volatile impurities formed at all technological stages of production. Theoretical developments prove that the degree of separation of organic impurities and ethyl alcohol depends on the difference in their evaporation coefficients. For the head and intermediate impurities, this difference reaches its maximum value at low alcohol concentrations in the solutions, and for the final impurities – at high ethanol concentrations. Effective emitted of head impurities (esters and aldehydes) occurs under conditions of deep hydroselection at a concentration of ethyl alcohol at the bottom of the column of 6–8% vol. To emitted intermediate impurities (higher alcohols of fusel oil), the concentration of ethyl alcohol must be reduced to 4–5% vol. To increase the coefficient of rectification of final impurities of low boiling point impurities (methyl alcohol) it is advisable to perform moderate hydroselection to ensure the concentration of ethanol on the plates within 60% mol. along the entire height of the column.

From practical experience, it is known that for the joint processing of head and fusel fraction in a typical ICC, to increase the number of contact devices to 51–57, and to increase the consumption of heating steam by 28.7% (from 2.56 to 3.59 kg/kg a.a) (Kiss et al., 2014). Despite this, the known processing methods do not ensure effective extraction of the head and upper intermediate impurities, which negatively affects the quality of rectified alcohol.

The efficiency of purification of ethyl alcohol, which is part of by-products and semi-products products, depends not only on the degree of extraction and multiplicity of concentrating volatile impurities, but also on the organization of their selection from the places

of their maximum accumulation. In order to increase the efficiency of this process, the deflemmator of ICC is connect with a decantator (Kiss, 2015).

In the decanter the condensate of vapour (phlegm) from the dephlegmator is separated into upper and lower layers. The upper layer concentrates water insoluble impurities, which are removed from the unit. The purified aqueous-alcoholic liquid from the bottom of the decanter is fed on the top plate of the column. In case of poor separation of the impurities can enter the column acetic, formic, crotonic aldehydes, some fusel oil alcohols (n-propyl and isopropyl alcohols), acrolein – impurities that significantly impair the quality of rectified ethyl alcohol. The most of these impurities enter the liquid at the bottom of the column and further into the beer. Increasing their concentration in the beer leads to increased consumption of heating steam in the beer column. The known processing methods do not provide effective demulsification of higher alcohols of fusel fractions due to the temperature in the decanter being much higher than 25–35 °C. Under such conditions, higher alcohols, which are concentrated in the upper layer of the decanter, retain more water and ethanol, resulting in a decrease in the yield of rectified ethanol (Mendoza-Pedroza et al., 2015).

The use of cyclic rectification technology is a relatively new approach to solving the problem of energy saving and increasing the degree of alcohol impurities. A significant number of scientific works by famous scientists are devoted to research in this area, but the known processing methods have not found wide practical application (Andersen et al., 2018; Kiss, 2014; Bastian et al., 2012; Nielsen et al., 2017; Toftegard et al., 2016; Rasmussen et al., 2020).

To solve the actual problem, the authors proposed resource- and energy-saving methods for the extraction of ethyl alcohol from alcohol-containing by-products and semi-products of alcohol production and its effective purification from impurities in the mode of controlled rectification cycles (Maleta et al., 2015, Buliy et al., 2019). To implement them, was developed a design of a ICC (patent UA 124733. Column mass-exchange apparatus of cyclic action) and an equipment and technological scheme for its inclusion in operation.

The adopted technical solutions made it possible to eliminate the disadvantages inherent in the known method, to process fractions enriched with head, intermediate and terminal impurities in one column, to improve the quality of rectified ethyl alcohol by achieving a state of contacting phases (vapor and liquid) close to equilibrium on its plates, to maximize the removal of volatile impurities, and to reduce the specific consumption of heating steam for the processing process (Bulii et. al., 2021).

The aim of the work was to research and justificate of the efficiency of energy-saving methods of joint processing of by-products and intermediates of alcohol production in the ICC of cyclic action: selection of optimal technological modes of operation, at which the degree of extraction and multiplicity of concentrating of head, intermediate and terminal impurities of alcohol will be maximized; determination of the specific consumption of heating steam in the experimental column.

Research tasks:
- To establish the optimal technological parameters of ICC operation for efficient joint processing of fractions enriched with head, intermediate and terminal impurities;
- To determine the degree of extraction and the multiplicity of concentrating of organic volatile impurities of alcohol in a typical column operating in a stationary mode and a cyclic action experimental column;
- To investigate and select the optimal technological modes for effective phlegm stratification in the decanter, increase of multiplicity concentrating impurities, degree of purification of phlegm and distillation residur in the experimental column;
- Determine the specific consumption of heating steam in the experimental column of cyclic action.

Materials and methods

Research objects

1. Experimental column of cyclic action for impurity concentrating (ICC). The experimental column was made of AISI 304 stainless steel and equipped with scale plates (Figure 1).

Figure 1. General view of a impurity concentration column

Technical characteristics of the column: diameter – 950 mm; number of plates –30; distance between plates – 300 mm; free cross-section of the plate during the period of liquid staying on its surface – 2.5%, during the period of liquid overflow – 51.5%.

The absence of overflow and receiving devices made it possible to increase the surface of phase contact on each plate by 15%, and the coaxial arrangement of the scales to increased the efficiency of mass- exchange and eliminated the possibility of liquid droplets being carried to the upper plates. All the plates contained movable sections connected to pneumatic cylinders and modern computer-integrated means (patent UA 136561. Mass-exchange contact plate).

The operation of a cyclic mass-exchange plate is shown schematically in Figure 2.
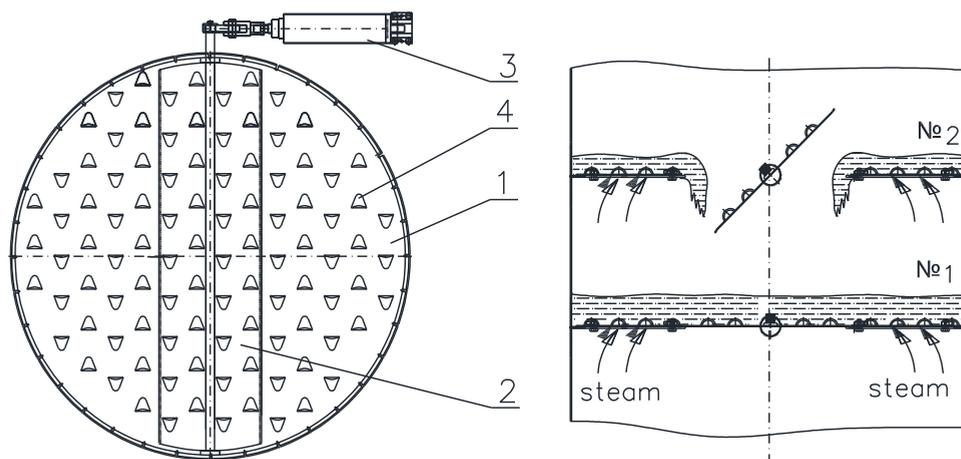
Figure 2. Cyclic mass–exchange plate with a variable free cross-section
1 – plate web; 2 – movable segment; 3 – drive mechanism; 4 – scales

The column was operated by continuously supplying heating steam to its lower part and liquid to the upper plate and periodically overflowing liquid from plate to plate from top to bottom after a set time of its delay on each plate. The ratio of the cross-sectional area of the overflow hole to the cross-sectional area of the plate at the time of overflow of (0.5:1) ensured an instantaneous decrease in the velocity of vapor in the holes of the scales to 1.5–1 m/s, as a result of which the liquid spilled through all the holes. Thus, the cycles of mass-exchange and overflow occurred alternately according to a given algorithm of pneumatic cylinders (patent UA 123917. Method of mass-exchange between liquid and vapor in a column apparatus).

After the rotation of the movable segment 2, the liquid overflowed onto the web 1 of the plate 1 from the plate 2 above it through the hole that had formed. During this period, the movable segment 2 of the web 1 of the plate № 1 was closed. The heating steam entered through the slits of the scales 4 and came into contact with the liquid on the plate 1. During the period of mass-exchange, the steam velocity in the slits of the scales 4 was maintained within 12–14 m/s, so the liquid was kept on the plate. After the set time of the liquid staying on the plate № 1, due to the action of the pneumatic cylinder 3, its movable segment 2 was turned, and the liquid instantly overflowed to the plate below through the hole formed.

2. The unit for ethyl alcohol extraction from alcohol-containing fractions. The processing of alcohol-containing fractions enriched with head, intermediate and terminal impurities was carried out in three ways. The hardware and technological scheme of the unit for the implementation of method I is shown in Figure 3 (patent UA 137550. Method for the joint distillation of alcohol-containing fractions enriched with head, intermediate and terminal impurities).

Figure 3. Installation for joint processing of head and fusel fractions by method I:
1 – water container; 2,10 – centrifugal pumps; 3,9,11,14 – flowmeters; 4 – impurity concentration column (ICC); 5 – hydraulic shutter; 6 – dephlegmator; 7 – condenser; 8 – decanter; 12 – cooler; 13 – extractor; 15 – impurity concentrate container; 16 – alcohol-containing fractions container.

Notation conventions:
CW – cooling water; HS – heating steam; DR – distillation residue; WH – water for hydroselection; FRW - fusel rinse water; FC – feed of column; FEAC – fusel and ester-aldehyde concentrate; AMC – aldehyde and methanol concentrate; LW – luther water; BC – beer column; EC – ether column; CBC – condenser of beer column; CDSC – carbon dioxide separator condenser; IC – impurity concentrate.

The equipment and technological scheme of the unit for processing by-products and intermediates of alcohol production according to method II is shown in Figure 4 (patent UA 137553. Method of joint processing of head and fusel fractions).
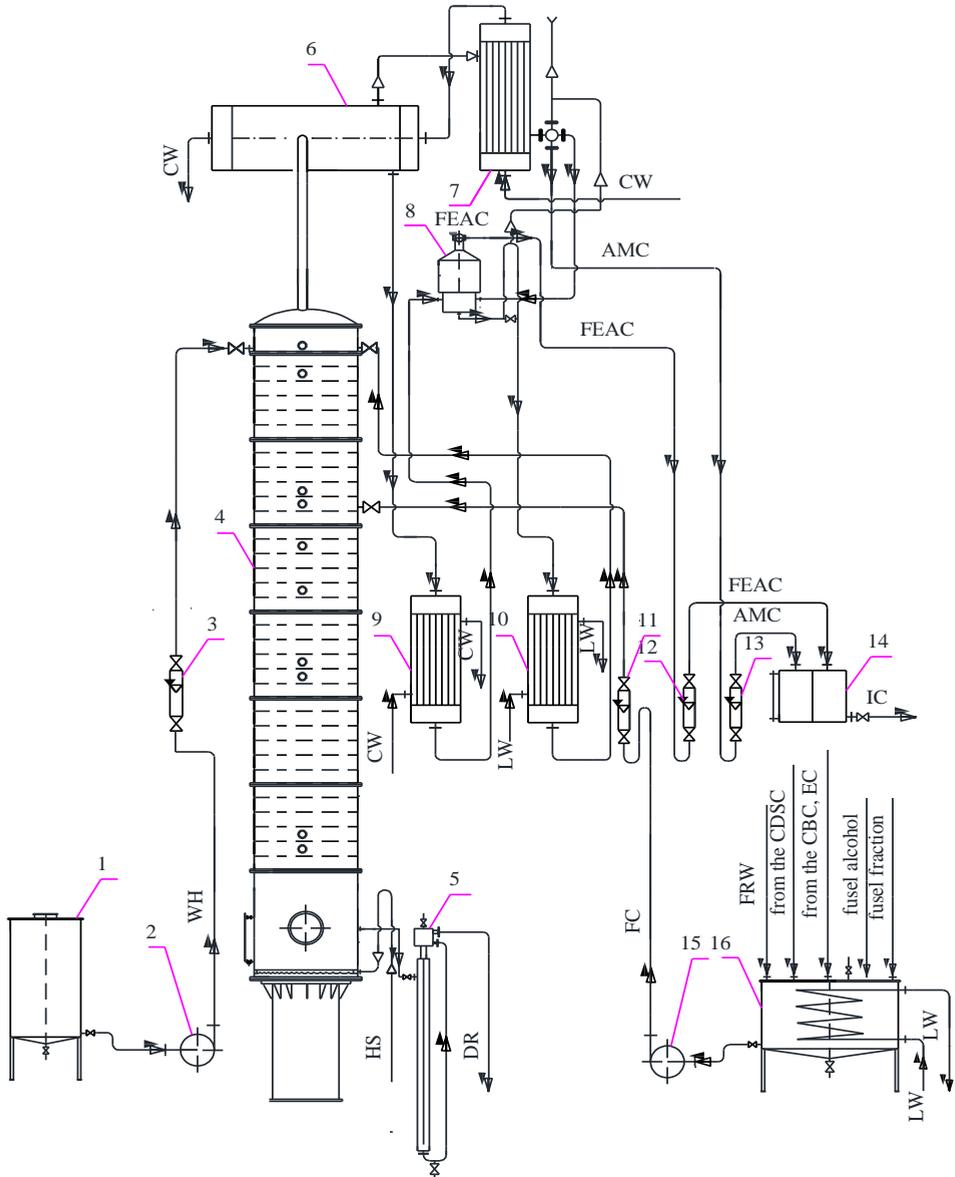
Figure 4. Unit for joint processing of head and fusel fractions by method II:
1 – water container; 2,15 – centrifugal pumps; 3,11,12,13 – flowmeters;
4 – impurity concentration column (ICC); 5 – hydraulic shutter; 6 – dephlegmator;
7 – condenser; 8 – decanter; 9 – cooler; 10 – heater; 14 – impurity concentrate
container; 16 – alcohol-containing fractions container.

The equipment and technological scheme of the unit for processing by-products and intermediates of alcohol production according to method III is shown in Figure 5 (patent UA 137555. Method of ethyl alcohol extraction from head and fusel fractions).
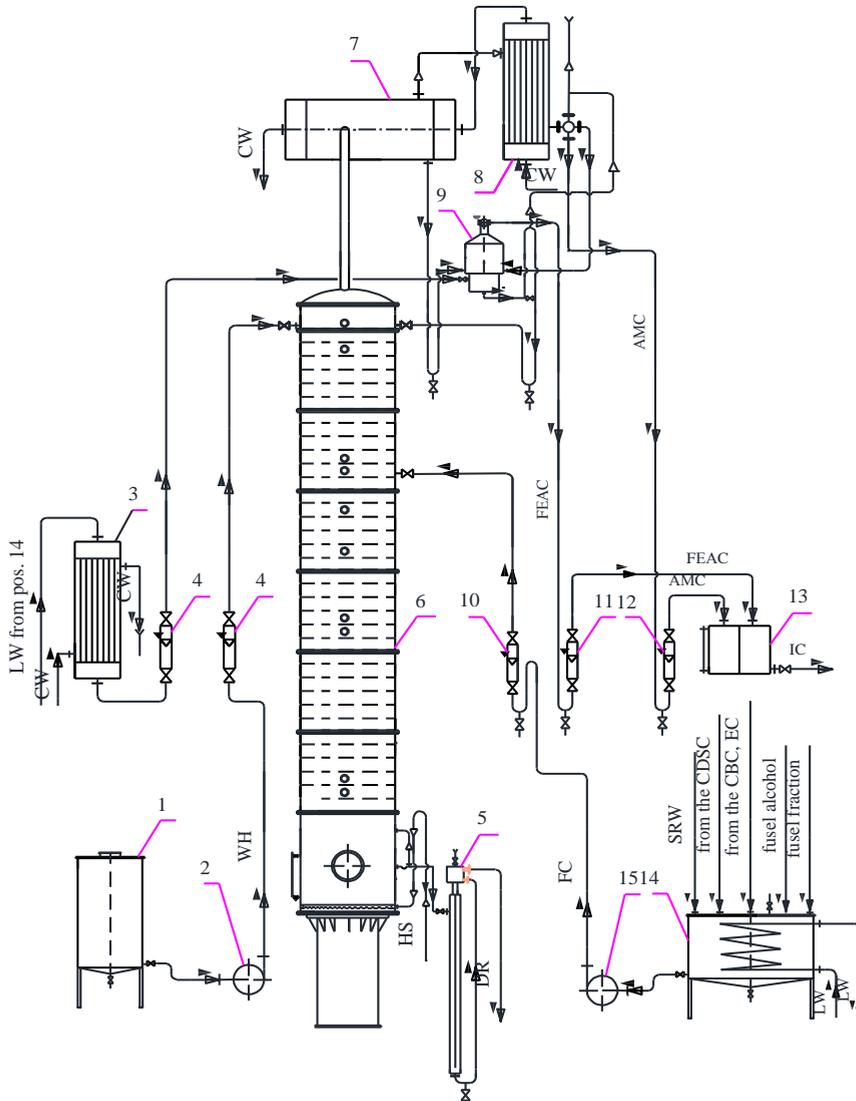
Figure 5. Unit for joint processing of head and fusel fractions by method III
1 – water container; 2,15 – centrifugal pumps; 3 – cooler; 4,10,11,12 – flowmeters;
5 – hydraulic shutter; 6 – impurity concentration column; 7 – dephlegmator;
8 – condenser; 9 – decanter; 13 – impurity concentrate container;
14 – alcohol-containing fraction container.

Research methods

Analytical, chemical, physico-chemical and computational methods were used to evaluate the results obtained, using instruments and research methods used in the production of rectified ethyl alcohol.

Liquid consumption. The consumption of alcohol-containing fractions, water for hydroselection, distillation residue from the impurity concentration column and rectified alcohol was monitored using flowmeters (Polulyah et al., 2012).

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 dm^3/h.

Concentration of ethyl alcohol in water-alcohol solutions. The concentration of ethyl alcohol in the aqueous-alcoholic liquids was determined by the areometric method (Yanchevskiy et al., 2002). The test solution was poured into a $250\ sm^3$ glass cylinder, the temperature was measured with a thermometer with a division price of 0.1 ºC, 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. The concentration of volatile impurities in alcohol by-products and intermediates, the distillation residue of the impurity concentration column, the impurity concentrate and the 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.

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

$$\alpha = \frac{X_{fc}}{X_{dr}}, \ \beta = \frac{X_{feac}}{X_{fc}}$$

where X_{fc}, X_{feac}, X_{dr} – the concentration of volatile impurities of alcohol on the feed plate of colunn, in the fusel ester-aldehyde concentrate and the distillation residue of the impurity concentration column (ICC), mg/dm^3 in terms of a.a. (Shiyan et al., 2009).

Studied modes

Joint processing of by-products and intermediates was carried out in stationary and cyclic modes of work of the impurity concentration column (in the existing typical and experimental column), whiches alternately operated as part of an indirect-acting distillation unit (Bulii et al., 2021; Mischenko et al., 2020). The movement of volatile alcohol impurities in the columns of the distillation plant is shown schematically in Figure 6.

The plate of feed of the impurity concentration column was supplied with the head fraction of ethyl alcohol, fusel fraction and fusel alcohol from the rectification column, fusel rinse water, fractions from the condenser of the beer column and the condenser of the carbon dioxide separator. The total amount of alcohol containing fractions was 700 dm^3/h (250 dm3/h in terms of a.a). The concentration of ethyl alcohol in them was 35.7% vol. Hot luther

water in the amount of 3500–4100 m³/h was continuously supplied to the upper plate of column for the hydroselection of alcohol impurities. The temperature of the luther water was 90–92 °C. In steady-state mode, the liquid was poured from plate to plate from top to bottom continuously, while in cyclic mode, it was poured periodically after a set delay time on each plate.
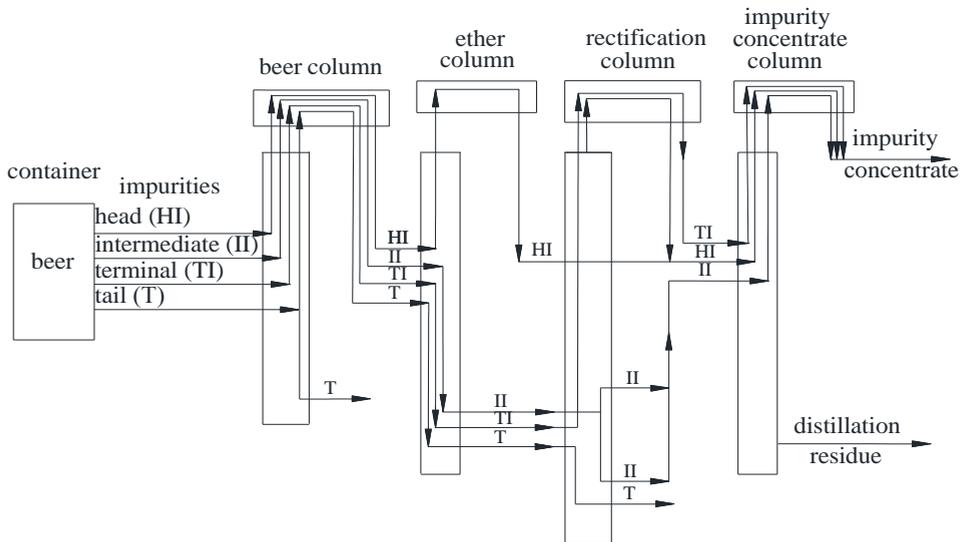
Figure 6. The movement of volatile alcohol impurities in the columns of the distillation plant of indirect action
HI – head impurities; II – intermediate impurities; TI – terminal impurities;
T– tail impuritys

The fluid retention time was 40 s, and the time for its overflow from the upper plate to the lower plate was 1.7 s. The height of the liquid layer on the plates was 35–40 mm. The pressure in the lower part of the column was maintained within 120 kPa, and in the upper part 0.3 kPa. The temperature at the bottom of the column was 101 °C, and above the top plate 94 °C. The concentration of ethyl alcohol of the distillation residue at the bottom of the column was regulated by the flow rate of luther water and maintained within 3.8% vol.

In the experimental impurity concentration column, the vapor velocity in the holes of the scales during the period of liquid retention on the plates was 12−14 m/s, and during the period of its overflow 1.5−1 m/s (Bulii et al., 2021; Krivchun et al., 1986). The change in the vapor velocity occurred by changing the free cross-section of the plate at the time of opening and closing the overflow hole. The liquid throughput of the experimental column was 5000 dm³/h. The phlegm from the deflagmator was sent to the decanter, where the liquid was stratified at an optimum temperature of 20–40 °C (Bulii et al., 2022). The water-alcohol liquid freed from most of the impurities from the lower part of the decanter in the form of phlegm was sent to the upper plate of the column for its irrigation. From the upper part of the decanter, SEAC was taken in the amount of 0.4–0.6% of the amount of rectified alcohol. AMC was taken from the condenser acceleration column in a ratio of (1:2) to FEAC in the amount of 0.2–0.3%

Stages of research

At the first stage, we studied the efficiency of joint processing of alcohol-containing by-products and intermediates using a well-known method with the separation of all concentrated alcohol impurities in the form of one product – SEAC in a experimental column operating in a stationary mode (Patent UA 69511. Rectification unit for the extraction of ethyl alcohol from fractions enriched with organic impurities).

During the rectification process, the alcohol-containing mixture was divided into two streams: the upper one, which contained head and intermediate alcohol impurities, and the lower one, which contained the water-alcohol mixture freed from them. The vapors coming out of the upper part of the column were fed first to the deflagrator and then to the condenser. Most of the vapors condensed in the deflagrator (high-boiling components – water, higher alcohols, esters and ethanol), while low-boiling components – aldehydes, esters, a small amount of higher alcohols and methyl alcohol condensed in the condenser.

The phlegm from the deflagrator was fed to the top plate of the column for its irrigation, and the condensate from the condenser flowed by gravity to the decanter. In the decanter, the heterogeneous mixture was stratified to form an upper layer – fusel and ester-aldehyde concentrate (FEAC), which included esters, aldehydes, esters, fusel oil alcohols, and a lower layer – an aqueous-alcoholic liquid free of head, part of intermediate and terminal impurities. The FEAC was removed from the unit into a separate container, and the water-alcohol liquid was fed to the upper plate of the column for its irrigation. The yield of FEAC was 0.4–0.6% of the amount of rectified alcohol. The aqueous-alcoholic liquid from the bottom of the impurity concentration column, was pump into the beer.

At the second stage, were studied the efficiency of processing alcohol-containing fractions using Method I (Figure 3).

The fractions enriched with head (esters and aldehydes), intermediate (higher alcohols of fusel oil), and terminal (methyl alcohol) impurities were fed to the alcohol-containing fraction tank 16, where they were heated by the heat of hot luther water to a temperature of 78–80 °C. Then they were fed to the feed plate of column 4 by a centrifugal pump 10. In the process of extractive rectification, the alcohol-containing mixture was separated into two streams: the upper one, which contained the head, intermediate and terminal alcohol impurities, and the lower one, which contained the water-alcohol liquid freed from them. The vapors from the upper part of the column were fed first to the dephlegmator 6 and then to the condenser 7. In its lantern, the mixture was stratified: water-insoluble aldehydes, esters, and higher alcohols were concentrated in the upper layer, and water-soluble aldehydes and methanol were concentrated in the lower layer, which were taken from the condenser to the container 15 in the form of aldehyde and methanol concentrate (AMC). The vapor condensate from the dephlegmator 6 with a temperature of 65 °C and the upper layer of liquid from the condenser lantern 7 flowed by gravity into the middle part of the decanter 8. To reduce the concentration of ethyl alcohol in the mixture from 65–68 to 30–40% vol., the calculated amount of hot luther water was supplied from the container 1 to the lower part of the decanter 8 by the centrifugal pump 2 through the flowmeter 3. The mixture was separated into an upper and a lower layer in the decanter. From the upper part of the decanter, FEAC was taken through the flowmeter 11, which was cooled in the cooler 12 to a temperature of 20–35 °C and sent to the lower part of the extractor 13. In the extractor, the FEAC was washed with luther water, which had a temperature of 20–35 °C and pH 5.0–5.5, to extract ethyl alcohol. The FEAC freed from alcohol was sent to container 15, and the fusel rinse water containing 12–15% vol. of ethyl alcohol was returned to container 16. The distillation residue from the

bottom of the impurity concentration column, free from volatile impurities, was fed to the upper zone of the concentration part of the ether column for hydroselection of impurities.

At the third stage, we studied the efficiency of processing alcohol-containing fractions using Method II (Figure 4). The method involved cooling the phlegm, which was taken from the dephlegmator 6, to a temperature of 20–35 °C in a cooler 9, separating the mixture in the decanter 8 into two layers – the upper (FEAC) and the lower (water-alcohol liquid purified from impurities), heating this liquid in the heat exchanger 10 with the heat of hot luther water, which had a temperature of 102–103 °C, to a temperature of 90–92 °C and supplying the hot liquid in the form of phlegm to the upper plate of the impurity concentration column for its irrigation.

At the fourth stage of the studies, joint processing of the alcohol-containing fractions were processed according to method III (Figure 5). To do this, hot luther water was first sent to the alcohol-containing fraction container 14 to heat the power supply, then it was cooled in the refrigerator 3 to a temperature of 10–15 °C and then fed to the lower part of the decanter 9. After mixing the cooled luther water with phlegm coming from the dephlegmator 7 in the decanter, the temperature of the mixture decreased from 65–68 to 20–35 °C, the concentration of ethyl alcohol in it decreased to 30–40% vol, and the pH was reduced to 5.0–5.5.

To determine optimal technological parameters of work of the column of cyclic action, in which the degree of extraction and multiplicity of concentrating of head, intermediate and terminal impurities of alcohol were the highest, a comparative analysis of efficiency of known and proposed by the authors methods of processing alcohol-containing fractions. In the course of research, samples were taken of fractions coming to the plate of feeding of the column (FC), the distillate residue (DR), impurity concentrate (IC), formed after mixing in the container of FEAC and AMC, rectified ethyl alcohol (REA) and carried out their analysis by chromatographic method. The results of the analysis of the test samples are presented in Table 1.

The calculated values of the degree of extraction *(α)* and the multiplicity of concentration *(β)* of volatile alcohol impurities under typical and cyclic rectification conditions are given in Table 2.

Results and discussion

Processing of by-products and intermediates by a known method in an impurity concentration column working in a stationary mode.

The use of a well-known method of processing alcohol-containing fractions made it possible to increase the yield of rectified ethyl alcohol by 3.5–3.7% from one ton of conditional starch and to remove volatile organic impurities from the unit in the form of a single product, which simplified the method of selection, storage and transportation of waste, but did not exclude the disadvantages listed below.

1. Table 1 shows that in the process of joint processing of the head and fusel fractions by the known method, the physical and chemical parameters of rectified ethyl alcohol did not meet the standard for the high-quality alcohol. The mass concentration of fusel oil, in terms of a mixture of isoamyl and isobutyl alcohols (1:1) in anhydrous alcohol, exceeded the standard by 10% (Pang et al., 2017).

Concentration of volatile impurities in the feed, distillation residue, impurity concentrate and rectified ethyl alcohol

Impurity name	Concentration, mg/dm³ in terms of a.a.						
		A well-known method			Methods I, II, III		
	FC	DR	IC	REA	DR	IC	REA
Ethanol,% vol.	30.5	3.8	75	96.3	3.8	67	96.3
Aldehydes	318.7	4.7	1689.1	1.2	2.8	2302.2	0.18
- acetaldehyde	242.3	4.7	1041.9	1.2	2.8	1396.7	0.18
- methylacetate	76.4	traces	672.3	–	traces	905.5	–
Esters	40.5	2.5	330630	–	traces	446615	–
- ethylacetate	traces	2.5	traces	–	traces	traces	–
- isobutylacetate	11.1	traces	2383.2	–	traces	3234.8	–
- isoamylacetate	29.4	traces	361.5	–	traces	494.4	–
- ethylbutyrate	traces	traces	327885	–	traces	442886	–
Methanol,%	0.18	0.007	1.7	0.005	0.004	2.7	0.0006
Fusel oil	105883	1180.4	434120	2.2	721.7	726464	0.88
- isopropanol	1.2	0.015	13.1	2.2	traces	22.4	0.88
- n-propanol	20002	1117.4	100.4	–	677.5	220.6	–
- isobutanol	20297	8.4	213118	–	4.9	357247	–
- n-butanol	362	4.4	579.2	–	2.7	1003.8	–
- isoamylol	65221	16.5	215230	–	13.8	367970	–

Notation conventions:
FC – feed of column; DR – distillation residue; IC – impurity concentrate;
REA – rectified ethyl alcohol.

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

Impurity name	A well-known method		Methods I, II, III	
	α	β	α	B
Aldehydes	67.8	5.3	113.8	7.2
- acetaldehyde	51.6	4.3	86.5	5.8
- methylacetate	max	8.8	max	11.9
Esters	16.2	8163.7	max	11027
- isobutylacetate	max	214.7	max	291.4
- isoamylacetate	max	12.3	max	16.8
Methanol	25.7	9.4	45.0	14.9
Fusel oil	89.7	4.1	146.7	6.9
- isopropanol	80.0	10.9	max	18.7
- n-propanol	17.9	0.005	29.5	0.01
- isobutanol	2416.3	10.5	4142.2	17.6
- n-butanol	82.3	1.6	134.1	2.8
- isoamylol	3952.8	3.3	4726.2	5.6

2. An increase in the concentration of esters and higher alcohol of fusel oil in the distillat rtdidue from the bottom of the column, and then in the beer, led to an increased in the consumption of heating steam for their extraction in the beer and ether columns. This is due to the fact that at high distillation temperatures of alcohol beer (95–105 ºC), simultaneously with the release of volatile impurities, the interaction of alcohols, acids, aldehydes, aminoacid breakdown products, sulfur compounds and other beer compounds occurred and a number of substances (aldehydes, esters, acetals of organic acids, etc.) were formed that deteriorate the quality of alcohol and reduce its yield. To ensure the efficient extraction of alcohol and related organic impurities from the beer, the beer column was operated with an excess of 5-10% steam at a steam excess coefficient of $\beta = 1.05-1.1$ (Bulii et al., 2022).

The continuous flow of distillation residue into the beer, which contained residues of intermediate and terminal impurities, led to their accumulation in the beer, an increase in the content of undesirable newly formed compounds in the beer distillate, the removal of which requires increased water consumption for their hydroselection in the ether column, and, accordingly, an increase in the consumption of heating stram (Simon et al., 2009).

3. In order to increase the degree of volatile impurities extraction and achieve the standard quality indicators of rectified ethyl alcohol a necessary condition was to increase the specific consumption of heating steam in the impureties conctntration column to 20 kg/dal a.a. introduced to the feed plate. This is due to the fact that with the continuous overflow of liquid along the height of the column, the time of its stay on the plates and contact with steam was insufficient to create conditions close to the equilibrium state of the phases, under which the maximum extraction of impurities occurs. In addition, the method did not provide for cooling the phlegm to the temperature (25–30 °C) optimal for demulsification of higher fusel oil alcohols (Patil et al., 2002). At an elevated phlegm temperature (65–68 ºC), the quality of the FEAC deteriorated: higher fusel oil alcohols, which were concentrated in the upper layer of the decanter, retained more water and ethanol, resulting in a decrease in the yield of rectified alcohol. Due to the high concentration of ethyl alcohol in the phlegm (65–70% vol.), enriched with head and intermediate impurities, the phlegm did not effectively separate in the decanter. As a result, water-soluble undesirable impurities (methyl alcohol, crotonic aldehyde, acrolein, isopropanol, etc.) from the bottom of the decanter together with the liquid first got to the upper plate of the column, and then to the at the bottom part of it and beer. Method for the txtraction of ethyl alcohol from fusel fractions. It is known that for effective stratification of the water-alcohol mixture in the decanter, the actual content of ethyl alcohol in the liquid should not exceed 34% (40.8% vol.) (Osypenko O. et al., 2013).

Processing of by-products and intermediates in a experimental impurity concentration column in a cyclic mode by methods I, II, III.

The results of the study of the efficiency of processing alcohol-containing fractions enriched with head, intermediate, and terminal alcohol impurities by method I (Figure 3) showed that when mixing phlegm at a temperature of 65 °C and hot hydroselection water at a temperature of 92 °C, the process of mixture stratification in the decanter accelerated, but the quality of the FEAC deteriorated. Higher alcohols of fusel fractions, which were part of the FEAC, retained more water and ethyl alcohol at an increased demulsification temperature. Washing of the FEAC with luther water and extraction of ethyl alcohol in the extractor of the distillation column made it possible to reduce its losses with the impurity concentrate by 8% and increase the concentration of higher fusel oil alcohols by 40% compared to the known method.

Cooling of the phlegm supplied from the dephlegmator to the decanter to a temperature of 20-35 °C according to Method II (Figure 4) allowed for effective separation of the mixture in the decanter, thereby increasing the degree of concentration of head, intermediate, and terminal alcohol impurities by 26, 37, and 40%, respectively. Heating of the water-alcohol liqui free of impurities, which was fed from the bottom of the decanter to the upper plate of the column, with the heat of luther water to a temperature of 90–92 °C made it possible to reduce the specific consumption of heating steam for the processing process.

Dilution of the phlegm from the dephlegmator in the decanter with luther water at a temperature of 10–15 °C to an alcohol concentration of 30–40% vol. in the mixture, lowering the pH of the mixture to 5–5.5, and reducing its temperature from 65–68 to 20–35 °C according to Method III (Figure 5) made it possible to increase the multiplicity of concentration of head, intermediate, and terminal alcohol impurities by 26, 37, and 40% respectively.

The analysis of Table 1 showed that in the process of joint processing of alcohol-containing fractions in a cyclic mode, provided that any of the methods proposed by the authors is used, the concentration of aldehydes (acetaldehyde) in the finished product decreased by 85%, esters by 35%, methyl alcohol by 87%, and fusel oil by 60% compared to the known processing method. This is due to the fact that by prolonging the contact time of vapor and liquid on the plates to 40 s, the degree of extraction of volatile alcohol impurities increased, and by creating and maintaining optimal conditions for liquid separation in the decanter, the multiplicity of concentration of impurities increased significantly.

A comparative analysis of the calculated values *(α)* and *(β)* given in Table 2 confirmed that in the experimental column of cyclic action, provided that deep hydroselection of alcohol impurities was carried out, esters were completely removed. In the selected hydraulic mode of operation of the plates, the degree of extraction of aldehydes, higher alcohols of fusel oil and methyl alcohol increased by 40%. At the same time, the multiplicity of concentration of aldehydes and esters increased by 26%, higher alcohols of fusel oil by 40%, and methyl alcohol by 37% compared to the known method. As a result, the physicochemical parameters of rectified ethyl alcohol, which are shown in Table 1, have significantly improved.

Thus, the high efficiency of the innovative methods of processing by-products and intermediates of alcohol production proposed by the authors was proved in production conditions. The use of resource- and energy-saving methods I, II, and III made it possible to eliminate the disadvantages inherent in the known method, to obtain rectified ethyl alcohol with a high degree of purity and to increase its yield by 3.8–4.0% from one ton of conditional starch. At the same time, the loss of alcohol with impurity concentrate decreased by 8%, and the specific consumption of heating vapor in the processing process by 40% (from 20 to 12 kg/dal a.a. introduced to the feed plate) compared to the known method.

Conclusion

1. The known methods of processing by-products and intermediate products of alcohol production do not allow for the joint processing of head and fusel fractions in the impurity concentration column which works in stationary mode. The inclusion of an additional fusel column requires higher energy costs and increased metal consumption in the distillation plant.
2. The studies have confirmed the high efficiency and expediency of using a joint method of processing fractions enriched with head, intermediate and terminal alcohol impurities

in the selected technological mode in the impurity concentration column of a cyclic action equipped with scaly plates with a variable free cross section.

3. The use of innovative processing methods makes it possible to increase the yield of rectified ethyl alcohol from 3.5–3.7 to 3.8–4.0% from one ton of conditional starch, reduce the loss of alcohol with impurity concentrate by 8% and reduce the specific consumption of heating steam by 40% (from 20 to 12 kg/dal of a.a introduced to the feed plate) compared to the known method.

4. Extending the contact time of vapor and liquid on the column plates to 40 s allows for complete removal of esters, increase of the degree of aldehydes, higher alcohols of fusel oil and methanol recovery by 40% and increase of the multiplicity of concentration of head impurities by 26%, higher alcohols by 40% and methanol by 37%.

5. To increase the degree of concentration of volatile impurities of alcohol, it is advisable to mix the phlegm after the deflagrator and part of the water for hydroselection in a decanter, assuming that the concentration of ethyl alcohol in the mixture should be 30–40% vol.; for effective separation of liquid the temperature should be 20–35 °C and pH 5–5.5.

6. Maintaining the above-mentioned technological modes in the impurity concentration column of cyclic action and decanter allowed to reduce the concentration of aldehydes in the finished product by 85%, esters by 35%, methyl alcohol by 87%, fusel oil by 60% and to obtain rectified ethyl alcohol with a high degree of purity using innovative methods proposed by the authors.

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Ресурсо- та енергозберігаючі способи сумісної переробки побічних продуктів і напівпродуктів у спиртовому виробництві

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Вступ. Метою роботи було дослідження і обгрунтування ефективності енергозберігаючих способів сумісної переробки спиртовмісних фракцій в колоні циклічної дії, підвищення ступеню очистки спирту від летких домішок.

Матеріали та методи. Дослідження проводили в типовій розгінній колоні і колоні циклічної дії. Витрати рідини контролювали за допомогою витратомірів постійного перепаду тиску, концентрацію етилового спирту і летких домішок визначали ареометричним і хроматографічним методами, ступінь вилучення домішок і кратність їх концентрування – розрахунковим методом.

Результати та обговорення. Використання запропонованих авторами способів дозволяє здійснювати сумісну переробку побічних продуктів і напівпродуктів спиртового виробництва (головної та сивушних фракцій) в розгінній колоні циклічної дії, оснащеної лускоподібними тарілками із змінним вільним перерізом, отримати високоякісний ректифікований спирт, збільшити його вихід на 3,8-4,0 % із однієї тони умовного крохмалю або на 10,8 % в порівнянні з відомим способом і зменшити питому витрату пари на 40 % (від 20 до 12 кг/дал безводного спирту, введеного на тарілку живлення). Подовження часу контакту пари і рідини на тарілках колони до 40 с дозволяє в повній мірі видаляти естери, збільшити ступінь вилучення альдегідів на 25 %, а вищих спиртів сивушного масла і метанолу на 40 %. Запропоновані технічні рішення і обрані технологічні режими дають можливість підвищити ефективність розділення спиртовмісної суміші в декантаторі, збільшити кратність концентрування альдегідів та естерів на 26 %, вищих спиртів сивушного масла на 40 %, метанолу на 37 %, зменшити втрати етилового спирту з концентратом домішок, кількість спиртовмісних відходів, металоємність технологічного обладнання і собівартість ректифікованого спирту.

Висновки. Запропоновані способи дозволяють максимально очищати етиловий спирт від головних і проміжних домішок в колоні циклічної дії, отримати високоякісний спирт, зменшити енерговитрати і втрати спирту з відходами.

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