Improvement of alcohol distillation plant operation

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Abstract

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Introduction. The criteria for improving the operation of distillation plant include the degree of purification of rectified alcohol from impurities, the consumption of vapor, hot process water for hydroselection, and cooling water.

Materials and methods. The object of the study was the distillation plant equipped with columns of cyclic action. The concentration of volatile impurities was determined by the chromatographic method, degree of extraction and the multiplicity of concentration was determined by the calculation method, the flow rates of water, distillation residue and alcohol-containing vapor by using flow meters.

Results and discussion. Extending the residence time of the liquid on the plates of the impurity concentration column to 40 s ensures the complete removal of esters, methyl acetate and isopropyl alcohol, to increase the degree of extraction of higher alcohols of fusel oil and methanol by 38 %, to increase the multiplicity of concentration of the head impurities by 25 %, the upper intermediate and final impurities by 38 % and to reduce the consumption of heating vapor by 40 %. Reduction of the ethanol concentration in the epyurate to 25 % vol. allows to fully remove esters, reduce the concentration of aldehydes and fusel oil in the epyurate by 43% and isopropyl alcohol by 62%. Through the recirculation of the distillation residues of the rectification column, impurity concentration column and the extractive rectification column, the specific consumption of water and the energy required for her heating are reduced by 80%. Feeding alcohol-containing fractions in the form of vapor to the impurity concentration column in the amount of 2% allows reducing water consumption for cooling by 2.1%, and heating vapor by 1.4%. In case of an increase in the amount of non-condensed alcohol vapors from 2 to 5% water consumption is reduced by 5.1%, and of the heating vapor by 1.9%.

Conclusion. The research results can be used to improve the operation of distillation plant by reducing the consumption of heating vapor, process water and water for cooling heat-exchange equipment.

Introduction

For the further development of the alcohol industry the priority direction is the optimization of the operation of distillation plants (DP) by the development and implementation of resource- and energy-saving technologies (Shiyan et al., 2009). When using traditional methods of purification of alcohol from impurities in typical DP, some of them usually remain present in small quantities in commercial alcohol. For their more complete removal modern installations are retrofitted with an impurity concentration column, final purification, and extractive rectification columns, hydroselection of volatile impurities is carried out (Jacimovic et al., 2014; Tsygankov, 2010). The use of rational technological and design solutions allows to increase the degree of extraction and the multiplicity of concentration of the impurities, thereby improving the quality indicators of rectified alcohol, reducing the specific consumption of water and vapor, as well as increasing the yield of the finished product (Botshekan et al., 2022).

A relatively new and promising approach to solving the problem of optimizing the operation of the DP is the use of cyclic rectification technology (Andersen et al., 2018; Bedryk et al., 2023). Under the above conditions increase the driving force of the process of mass-exchange between liquid and vapor (Lita et al., 2014), increases the efficiency of plates operation and the specific consumption of heating vapor decreases (Chenyang et al., 2024; Kiss, 2014). Research in this direction began in the 60-70s of the twentieth century (Bastian et al., 2018; Nielsen et al., 2017), but due to the complexity of technical solutions installations did not find wide practical use (Rasmussen et al., 2020; Toftegard et al., 2016).

In the National University of Food Technologies (Ukraine) it has developed a design of column devices, the action of which allows for to extension of the contact time of vapor and liquid until the moment of reaching a state of phases close to equilibrium, and brings the efficiency of each physical plate closer to the efficiency of theoretical plate (patent UA 141245. Method of liquid pouring through plates of a column mass-exchange apparatus; patent UA 139228. Column mass-exchange apparatus of cyclic action). Almost on real plates such an equilibrium is rarely achieved. One of the good reasons for this is the short period of time that the contacting phases stay on the plate. To implement the method, the distillation column was equipped with plates with a variable free section, the movable segments of which were connected to mechatronic subsystems (Bulii et al., 2021). In the cyclic mode increases the driving force of the mass-exchange process and the efficiency of contact devices significantly (Flodman et al., 2012; Zhang et al., 2017).

One of the ways to optimize the work of the DP is carrying out of hydroselection of impurities. To increase the rectification coefficients of the head impurities of alcohol and the maximum removal of isopropanol hydroselection is carried out in the ether column (Linek et al., 2005). To do this the concentration of ethyl alcohol in the epyurate is reduced from 45–55 to 20–30 % vol. by supplying hot softened water or condensate of vapor to the upper plate of the column. Water consumption is 0.55-0.90 kg/kg of anhydrous alcohol (a.a.) introduced into the column. For extraction of head and upper intermediate impurities (fusel alcohol) in the impurity concentration column the concentration of distillation residue is maintained within 4–4.5 % vol., for the extraction of lower intermediate impurities (higher alcohols of fusel oil) in the extractive rectification column should not exceed 1.5–3.0 % vol. (Shiyan et al., 2009).

The disadvantages of the known methods are the increased consumption of process water and vapor for heating it (Nagy et al., 2015). To eliminate these shortcomings it is proposed an innovative technology (Bulii et al., 2021), the peculiarity of which is the recirculation of distillation residues of rectification column, impurity concentration column and extractive rectification column for hydroselection of impurities (patent UA 116733. Method of obtaining rectified alcohol).

The operation of heat exchange equipment is an important component of the functioning of a distillation plant (Jacimovic et al., 2014; Pinto et al., 2011). From practical experience, it is known that the thermal load on the condensers of the ether and rectification columns, impurity concentration column and final purification column is insignificant: 0.5-2% of the vapor condenses in them (Tsygankov, 2010). Known methods provide for the supply of alcohol-containing intermediates and by-products in columns from condensers in the form condensate of vapor with a temperature of 25-30 °C (Matsuda et al., 2011; Mischenko et al., 2020). For the effective isolation and concentration of impurities of alcohol a prerequisite is additional heat costs for heating them to a temperature of 75-80 °C, as well as an increase in the consumption of heating vapor by 28.7 % (Shiyan et al., 2009).

To the optimize the operation of heat exchange equipment by reducing consumption vapor and water for cooling, a promising direction is the organization of supplying the columns with alcohol-containing vapor that has not condensed in dephlegmators (Sheikus, 2019; Simon et al., 2009). It has developed an innovative energy-saving technology, which provides for the supply of the impurity concentration column with alcohol-containing fractions in a vaporous state (patent UA 126533. Method for obtaining rectified alcohol). The technical solution allows for to reduction of the specific consumption of heating vapor, cooling water and the cost of installation due to the absence of condensers in the technological scheme.

The aim of research was to study the effectiveness of innovative ways to improve the operation of distillation plant: carrying out the processing of alcohol-containing fractions and epuration of beer distillate in cyclic mode, the recirculation of distillation residue of rectification and ether columns, impurity concentration column and extractive rectification column, as well as of the method which provides for the supply to the impurity concentration column of power in a vaporous state.

Research objectives:

1. To determine the degree of extraction, multiplicity of concentrating of volatile alcohol impurities and consumption of heating vapor in the impurity concentration column and ether column of cyclic action;
2. To determine the specific and total consumption of process water and energy costs for its heating for hydroselection in the ether column, impurity concentration and extractive rectification columns when the recirculation of their distillation residues;
3. To determine the water consumption for cooling of dephlegmators of distillation, ether, rectification and impurity concentration columns and vapor consumption for heating them in case of supplying the impurity concentration column with alcohol-containing vapors.

Materials and methods

Research objects

1. Experimental ether column (EC) and impurity concentration column (ICC) of cyclic action

To conduct research in the production conditions of the «Storonibabsky distillery» (Ukraine) an experimental EC with a diameter of 1200 mm and ICC with a diameter of 950 mm where installed of cyclic action (Figure 1). The EC contained 35 scaly plates, the distance between which was 300 mm. The ICC contained 30 scaly plates. In terms of design features the ICC did not differ from the EC.

The rotary segments of the plates were connected to standard double-acting pneumatic cylinders, the action of which took place in accordance with the controller's program. The movable segments opened and closed the overflow holes of the plates according to a given algorithm in such a way that the liquid synchronously overflowed through the formed overflow hole and all the holes of the plates (patent UA 124733. Column mass-exchange apparatus of cyclic action; patent UA 123918. Mass-exchange contact plate).

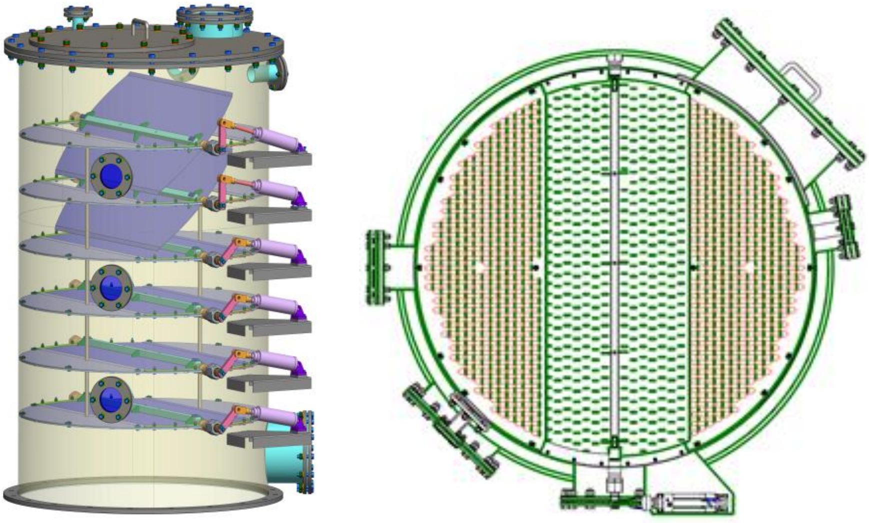

Figure 1. Fragment of an experimental column of cyclic action

2. Distillation plant (DP) with recirculation of distillation residues of rectification column, impurity concentration column (ICC) and extractive rectification column

Studies of the effectiveness of innovative technology with the recycling of distillation residue for hydroselection of alcohol impurities were carried out in the production conditions of the «Chudniv Distillery» (Ukraine). The technological scheme of resource- and energy-saving DP is presented in Figure 2.

The installation included distillation, ether and rectification column, an impurity concentration, extractive rectification, final purification columns and appropriate heat exchange equipment. To heat the columns heating vapor (HV) was supplied to their bottoms parts. Vapor condensate (C) was removed from the evaporators.

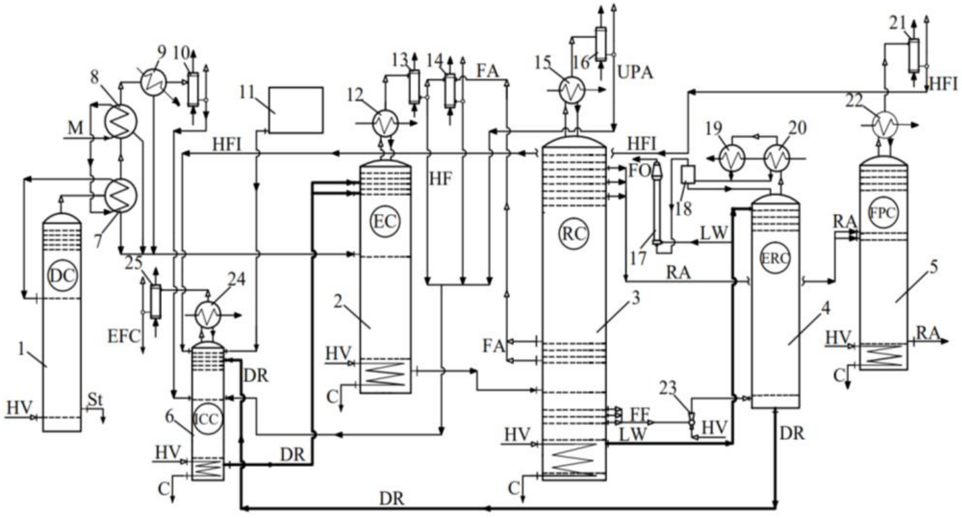

Figure 2. Resource- and energy-saving of distillation plant (DP) with distillation residues recirculation

1- distillation column (DC), 2 – ether column (EC), 3 – rectification column (RC), 4 – extractive rectification column (ERC), 5 – final purification column (FPC), 6 – impurity concentration column (ICC); 7, 8 – beer heaters; 9 – water section of the beer heater; 10, 13, 16, 19, 21, 25 – condensers; 12, 15, 20, 22, 24 – dephlegmators; 11 – pressure collector of hot softened water; 14 – fusel alcohol condenser; 17 – fusel oil extractor; 18 – decanter; 23 – vapor ejector.

Symbols: M – beer; St – stillage; HV – heating vapor; HF– head fraction of ethyl alcohol; LW – luther water; FO – fusel oil; FF – fusel fraction; RA – rectified alcohol; FA – fusel alcohol; EFC – ester-fusel concentrate; UPA – unpasteurized alcohol; C – condensate; HFI – head and final impurities; DR – distillation residue.

The beer (M) was sequentially fed through the second **8** and first **7** sections of the beer heater, in which it was heated with water-alcoholic vapor of the distillation column **1** to a temperature close to the boiling point and then directed to the upper plate of the distillation column, in which alcohol and related organic impurities were extracted. The beer distillate from the beer heaters **7** and **8** and the water section of the heater **9** was supplied to the feeding plate of the EC **2** and the vapor condensate from the condenser **10** to the feeding plate of the ICC **6**. From the bottom part of the column **1** stillage was removed. Concentration and extraction of the head and part of the intermediate impurities of alcohol were carried out in the EC **2**. For this purpose, distillation residue from the ICC **6** was supplied to the upper zone of its concentration part.

Concentrated impurities in the form of the head fraction of ethyl alcohol (HF) were taken from the condenser **13**. Fusel alcohol (FA) was taken from the vapor phase of 18-23rd plates of the rectification column **3** were fed into the interpipe space of the condenser **14** and after condensation of the vapor was removed from the unit together with the HF. The fusel fraction (FF) was taken from the vapor phase of the **5, 7, 9**, and **11**th plates of the rectification column **3**. In the upper pasteurization part of this column the concentration of the head impurities was carried out, which were not fully removed in EC **2.** These impurities in the form of unpasteurized alcohol (UPA) were discharged through condenser **16** together with HF from condenser **13**, fractions from condenser **10** and **14** to the feeding plate of the ICC **6.**

Rectified alcohol (RA) was taken from the liquid phase of the upper plates of column **3**, which then went to the feeding plate of the final purification column **5**. Its upper part was connected to the dephlegmator **22** and the condenser **21**. In column **5** alcohol was purified from the head and final impurities (HFI) were taken from condenser **21** and then directed to the upper plate of column **6**. Commercial rectified alcohol was taken from the bottom part of the column **5**. Vapors of the FF were mixed with heating vapor in ejector **23** and fed into the bottom part of the extractive rectification column **4**. Hot luther water (LW) from the bottom part of the rectification column **3** was supplied to its upper plate. In column **4** fusel oil (FO) alcohols were extracted and concentrated. The condensate of FO vapors from the dephlegmator **20** and condenser **19** was directed to the decanter **18**, in which the mixture was delaminated: the liquid from the bottom part of the decanter **18** was returned in the form of reflux to the upper plate of column **4** and the FO was sent to the extractor **17**. LW was supplied to the bottom part of the extractor to flush the oil. Concentrated of FO in the form of a marketable product was taken from the upper part of the extractor **17**.

Distillation residue (DR) of column **4** was supplied to 3-5th plate, counting from above, of the ICC **6** for hydroselection of intermediate impurities, including fusel oil components, and their concentration. In the process of separation of alcohol-containing fractions in column **6**, reflux was returned from the dephlegmator **24** to her upper plate, and ester-fusel concentrate (EFC) was taken from condenser **25**. To carry out hydroselection in the EC **2** in the upper zone of its concentration part the distillation residue purified from head and part of intermediate impurities from the ICC **6** was fed to the upper zone of its concentration part.

3. The distillation plant (DP) with feeding of the impurity concentration column with alcohol-containing fractions in the form of vapour

In the production conditions of the «Chudniv Distillery» the reconstruction of the existing indirect action DP equipped with the ICC was carried out. The technological scheme of the installation after its reconstruction is presented in Figure 3.

The operation of the unit was as follows. The matured alcohol beer (M) entered the beer heater **3**, in which it was heated with water-alcoholic vapor from distillation column (DC) **2** to a temperature of 70-85 °C, then into separator **1** to get rid of carbon dioxide and other non-condensed gases, after which it entered the DC **2** feeding plate. In separator **1** together with non-condensed gases a certain amount of alcohol was carried out. To extract it vapor from the upper part of the separator **1** was directed to the concentration part of the ICC **11** through a flow meter FIT4 and an analog damper VGA4. Heating vapor (HV) was continuously supplied to the bottom parts of DC **2**, EC **5**, rectification column (RC) **7** and ICC **11.** From the bottom part of the DC **2** stillage freed from alcohol and volatile impurities was removed. Water was supplied to the pipes of the dephlegmators **4, 6, 8, 9, 13**, condenser **17** and alcohol trap **18** for cooling.

Condensate of water-alcoholic vapor from the heater **3** and the water section of the dephlegmator **4** was supplied to the upper part of the EC **5**. Freed from the head (esters and aldehydes), parts of the upper intermediate (higher alcohols of fusel oil) and final (methyl alcohol) impurities of alcohol beer distillate — epyurate (E) was supplied as a power supply to the RC **7**. Low-boiling vapors of the head fraction (HF) uncondensed in the dephlegmator **6** were directed through the flow meter FIT3 and analog damper VGA3 to the concentration part of the ICC **11.** In the RC **7** ethyl alcohol was purified from intermediate and final impurities and its concentration. Rectified (pasteurized) alcohol (RA) was taken from the 5th, 7th and 10th plates, counting the RC **7** from above.

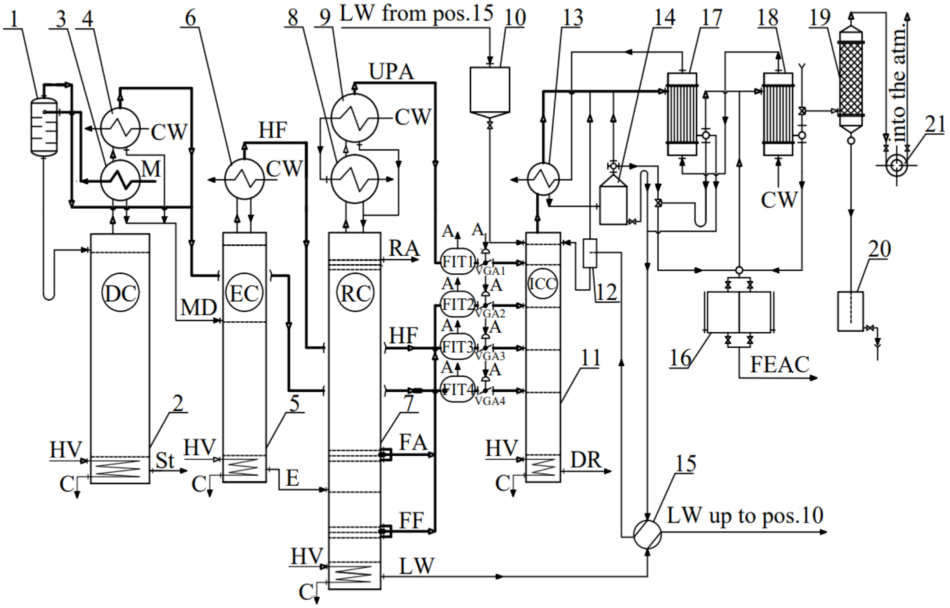

Figure 3. Resource- and energy-saving of distillation plant (DP) with feeding of the impurity concentration column alcohol-containing vapor

1 – carbon dioxide separator, 2 – distillation column (DC), 3 – beer heater, 4, 6, 8, 9, 13 – dephlegmators; 5 – ether column (EC); 7 – rectification column (RC); 10 – pressure collector of luther water, 11 – impurity concentration column (ICC), 12 – reflux separator, 14 – decanter; 15 – heat exchanger; 16 – collection of FEAC; 17 – upper stage condenser; 18 – alcohol trap; 19-barometric condenser; 20-barometric box; 21 – vacuum pump.

Symbols: M – beer; MD – beer distillate; St – stillage; CW – cooling water; E – epyurate; HF– head fraction of ethyl alcohol; HV – heating vapor; LW – luther water; FEAC – fusel-ester-aldehyde concentrate; FA – fusel alcohol; FF – fusel fraction; RA – rectified alcohol; DR – distillation residue; C – condensate; FIT – flow meter; VGA – analogue trap; A – analogue signal.

Intermediate impurities were removed from this column in the form of two products: fusel fraction (FF), which was taken from the vapor space of the 5, 7, 9 and 11th plates, counting from the bottom of the column, and fusel alcohol (FA), which was taken from the vapor space 17-20th and 25th plates, counting from the bottom. To extract ethyl alcohol FF and FA vapors were directed to the concentration part of the ICC **11** through the flow meter FIT2 and the analog damper VGA2. Low-boiling water-alcoholic vapors enriched with main and final impurities that did not condense in dephlegmators **8** and **9** through the flow meter FIT1 and analog damper VGA1 was directed to the upper zone of the concentration part of the ICC **11.**

All flow meters and dampers were installed on the corresponding vapor pipelines. The dampers were controlled by an analog signal (A), which came from the controller. For the supply of non-condensed vapor from the separator **1**, dephlegmators **4, 6, 9** and FF and FA pairs to the ICC **11,** the pressure difference at the points of their selection and introduction into the column was 4.9–9.8 kPa (patent UA 126533. Method of obtaining rectified alcohol).

To ensure this mode the ICC **11** operated under vacuum, which was maintained by means of a vacuum pump **21** and a barometric condenser **19** connected to a barometric box **20**. To carry out hydroselection the upper plate of the column **11** from the pressure collector **10** continuously received LW with a temperature of 90–95 ºC in the amount that provided the concentration of alcohol in the distillation residue within 4–8 % vol. In the ICC **11** alcohol-containing vapors of by-products and intermediate products of rectification were divided into two streams: the upper one, enriched with the head (esters, aldehydes) and intermediate (higher alcohols of FF and FA, including C_3, C_4, C_5), alcohol impurities, and the lower one — distillation residue. The vapor, coming out of the upper part of the column, was condensed in the dephlegmator **13** and its condensate flowed by gravity into the decanter **14**. In the decanter the heterogeneous mixture was delaminated into fusel-ester-aldehyde concentrate (FEAC), which was taken from the upper part of the decanter, and the lower layer enriched with water, which was directed to the upper plate of the column **11** in the form of reflux for irrigation. The reflux was preheated with LW heat in heat exchanger **15** to a temperature of 90-95 ºC, which corresponded to the temperature at the upper plate of column **11**, and passed through separator **12** to separate gases. FEAC was taken in the amount of 0.4-0.5% of the amount of alcohol introduced into DC **2**, and the distillation residue, freed from the head, intermediate and part of the final impurities, was continuously removed from the lower part of the ICC **11** and then fed to the upper plate of DC **2.** The water pressure in the working collector was 245 kPa and provided water supply to the dephlegmators and its required speed in the pipes. For calculations, the water temperature at the inlet to the dephlegmators was 20 °C, at the outlet – 70 °C.

Research methods

To evaluate the results of the research, analytical, chemical, physicochemical and calculation methods were used using devices and methods used in the production of rectified ethyl alcohol.

Liquid consumption. The flow rates of alcohol-containing fractions, water, distillation residue, rectified alcohol and concentrate of impurities were monitored using constant differential pressure flow meters RM (Polulyah et al., 2012). The principle of their operation is based on the perception of the dynamic pressure of the controlled medium, which depends on the flow, by a sensing element (float) placed in the flow. As a result of the flow action, the sensing element moved along the height of the flow meter, and the amount of displacement served as a measure of flow. The readings were taken on the scale of a flow meter graduated by water in l/h.

Vapor cost. The flow rate of alcohol-containing steam was determined using TVA flow meters from Spirax Sarco. Their principle of operation is based on the measurement of mechanical stress, which causes an instantaneous flow rate in the measuring cone in one direction. This voltage is converted into mass-compensated flow rate and transmitted through 4-20 mA outputs with a single-loop power supply. The TVA flow meter design provides high ranges of parameter changes and high measurement accuracy (Dattarajan et al., 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 (Polygalina, 1999). The test solution was poured into a 250 ml 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. 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). 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:

$$\alpha = \frac{X_f}{X_{cl}}, \quad \beta = \frac{X_{seac}}{X_f},$$

where X_f, X_{seac}, X_{cl} – accordingly, the concentration of volatile alcohol impurities on the feed plate, fusel and ester-aldehyde concentrate and distillation residue, mg/l in terms of a.a. (Linek, 2005; Shiyan et al., 2009).

Research stages

At **the first stage** the efficiency of the technology of epyuration of the beer distillate and separation of alcohol-containing fractions in the specified hydrodynamic modes in the EC and the ICC of stationary and cyclic action was investigated. To improve the process a mathematical model, a control program and a human-machine interface (SCADA) were developed. Beer distillate was fed to the *EC* feeding plate. For maximum release of the epyurate from the head and upper intermediate impurities, their hydroselection was carried out by supplying the upper plate of the column of distillation residue from the bottom part of the ICC, freed from impurities, in the amount of 240 dal/h. Under such conditions the concentration of ethyl alcohol in the epyurate decreased to 25 % vol. In the process of its epyuration the head impurities of alcohol were extracted and concentrated, which were taken from the condenser in the form of the head fraction in the amount of 5% of the amount of rectified alcohol. The epyurate freed from these impurities was fed to the 16th plate of the rectification column. The pressure in the bottom part of the EC was maintained within 11.3–11.8 kPa; the temperature in the bottom part was 100.5–101 °C and above the upper plate 93.5–94 °C.

The feed plate of the experimental ICC was fed with the head fraction of ethyl alcohol from the EC condenser and alcohol-containing fractions from the condensers of the distillation column, the carbon dioxide separator, fusel alcohol, fusel rinse water and distillates from the alcohol traps. The total number of feed fractions was 688.3 l/h (250 l/h in terms of a.a.). For hydroselection of impurities hot softened water in the amount of 4050-

4500 l/h was continuously supplied to the upper plate, which provided a concentration of ethyl alcohol in a distillation residue of 3.8 % vol. The distillation residue from the ICC purified from impurities was supplied to the upper zone of the concentration part of the EC for hydroselection of the main and part of the intermediate impurities, including isopropyl alcohol. The liquid retention time on the column plates in cyclic mode was 40 s, the time of its overflow from plate to plate was 1.7 s. Aldehyde-methanol concentrate was taken from the ICC condenser in the amount of 7–9 l/h. From the upper zone of the decanter after separation of the water-alcoholic mixture ester-fusel concentrate was taken in the amount of 2–3 l/h. Both concentrates were mixed in a concentrate of impurities.

At the **second stage** the efficiency of a typical DP indirect action with the recirculation of distillation residue was investigated. For hydroselection of impurities a distillation residue of the rectification column (luther water) was fed to the upper plate of the extractive rectification column; for 3–5th plate, counting from above, the ICC supplied the distillation residue of the extractive rectification column purified from the upper intermediate impurities and the distillation residue of the ICC freed from the head impurities and isopropanol was sent to the upper zone of the concentration part of the EC. The use of the luther water did not affect the quality of the RA, since the distillation residue of the extractive rectification column with a low content of ethyl alcohol (1.5–3.0 % vol.) freed from higher fusel oil alcohols was fed into the concentration zone of the ICC, in which intermediate impurities, including fusel oil components, were extracted and concentrated. In the process of research, the specific and total consumption of process water and energy consumption for its heating for hydroselection were determined.

At the **third stage** the efficiency of the operation of the DP in the mode of supplying the ICC with alcohol-containing fractions was investigated, which entered the feed plate in the form of vapor that did not condense in the dephlegmators of the main columns. According to the known method of rectification 98% of the amount of vapor coming out of the upper part of each column was condensed in the dephlegmators of the main columns, of which vapor condensate in the amount of 2% was directed to the ICC feeding plate.

The effectiveness of the innovative technology was investigated in two ways. According to Method I uncondensed in the carbon dioxide separator and dephlegmators of the main columns vapor was supplied to the ICC in the amount of 2%. According to Method II the water consumption for cooling the dephlegmators was reduced in such a way that 95% of the vapor from the amount of vapor coming out of each column condensed in the dephlegmators, and uncondensed vapor in the amount of 5% was supplied to the ICC feeding plate. In the course of the research, the quality indicators of rectified ethyl alcohol were monitored for compliance with the requirements of the standard.

Results and discussion

At the **first stage** the efficiency of the process of epyuration of the beer distillate in the EC of cyclic action was determined using hydroselection and without hydroselection and the process of extraction of ethyl alcohol from alcohol-containing fractions in the ICC of stationary and cyclic action (patent UA 136560. Method of mass-exchange between liquid and vapor in a column apparatus). Physicochemical parameters of beer distillate (BD), head fraction (HF), epyurate obtained without hydroselection (E1) and using hydroselection (E2) are given in Table 1.

Table 1

Composition of experimental samples of beer distillate (BD), head fraction (HF), and epyurate (E)

Impurities	Concentration of impurities, mg/l, in terms of a.a.			
	BD	HF	E1	E2
Ethanol, % vol.	50	92	40	25
Aldehydes	31.5	5252.0	4.5	1.8
methylacetate	traces	630.1	traces	traces
acetaldehyde	27.6	4621.9	2.7	1.8
Esters	83.1	14302.6	1.6	traces
ethylacetate	80.0	13905.6	traces	traces
isoamylacetate	3.1	325.2	1.6	traces
ethyl butyrate	traces	50.49	traces	traces
Methanol, %	0.044	2.4	0.027	0.010
Fusel oil	14116.7	4526.3	7786.3	4492.1
isopropanol	3.6	21.9	2.1	0.8
n-propanol	2565.8	558.2	1137.6	408.3
isobutanol	4014.2	3696.0	1627.1	566.5
n-butanol	39.4	8.2	38.8	38.4
isoamylol	7491.2	50.8	4966.9	3478.1
n-pentanol	4.5	191.1	13.8	traces

Table 1 demonstrates that in E2 epyurate the concentration of aldehydes decreased by 60%, acetaldehyde by 33.4%, methanol by 63%, fusel oil by 42.3%, and isopropyl alcohol by 62% compared to E1 epyurate. Furthermore, esters and n-pentanol were completely eliminated. This reduction in concentration of impurities can be attributed to the decrease of ethanol in the water-alcohol solutions to 30% vol. and below leading to rectification coefficients of the alcohol impurities become greater than one (Shiyan et al., 2009). At the same time the intermediate and final impurities of alcohol take on the character of the head ones. The calculated values of the degree of extraction of key alcohol impurities (α) in the EC of cyclic action using hydroselection and without hydroselection are presented in the form of a diagram (Figure 4).

As can be seen from the diagram, in the hydroselection mode the degree of extraction of all impurities increased by 1.7–2.8 times. The head impurities of alcohol were best removed.

The results of chromatographic analysis of alcohol-containing fractions (F), which were supplied to the ICC, are given in Table 2.

They included HF, shoulder straps made from condensers of the distillation column (CDC) and carbon dioxide separator (CDSC), fusel alcohol (FA) and fusel rinse water (FRW). In the process of processing alcohol-containing fractions samples of distillation residue (DR), impurity concentrate (IC) and rectified ethyl alcohol (RA) were taken

The results of chromatographic analysis of experimental samples and the calculated values of the degree of extraction of volatile impurities of alcohol in the stationary (α1) and cyclic (α2) modes of operation of the ICC are given in Table 3.

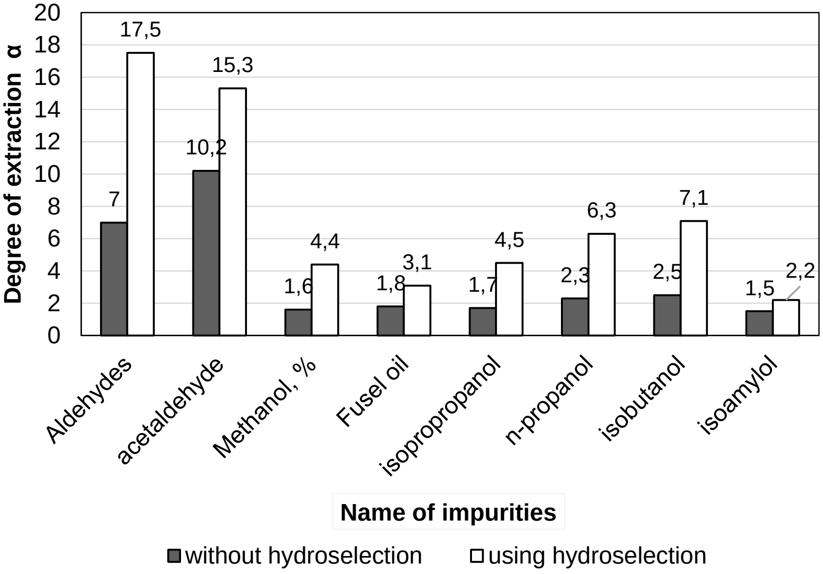

Figure. 4. Degree of extraction of volatile impurities of alcohol (α) in ether column (EC) of cyclic action with and without hydroselection

.

Table 2

Composition of alcohol-containing fractions

Impurities	**Concentration of impurities, mg/l, in terms of a.a.**					
	HF	**CDC**	**CDSC**	**FA**	**FRW**	**F**
Ethanol, % vol.	92.5	48.8	60	89	17.5	30.5
Aldehydes	1135.2	37.2	126.2	4.9	7.0	318.7
methylacetate	926.1	37.2	90.9	4.9	7.0	242.3
acetaldehyde	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	traces
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
Methanol, %	0.49	0.025	0.1445	0.013	0.0032	0.18
Fusel 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 3

Compositions of experimental samples and calculated values of the degree of extraction (α) of alcohol impurities

Impurities	Concentration of impurities, mg/l, in terms of a.a.						Degree of extraction	
	DR1*	IC1*	DR2*	IC2*	RA1*	RA2*	α1	α2
Ethanol,% vol.	3.8	67	3.8	67	96.3	96.3	8.0	8.0
Aldehydes	3.7	1689.1	2.8	2302	0.20	0.18	85.4	113.8
Esters	0.5	334961	traces	446615	traces	traces	79.7	∞
Methanol, %	0.006	1.67	0.004	2.69	0.007	0.0003	28.5	45.0
Fusel oil	1163.5	434120	721.7	726463	1.2	0.88	91.0	146.7

* 1 – operation of the ICC in stationary mode

* 2 – operation of the ICC in cyclic mode

The analysis of the results showed that esters were fully removed in the cyclic mode and the degree of extraction of higher alcohols of fusel oil and methanol increased by 38 %. This is explained by the fact that when the time of stay of the liquid on the plates of the column was extended to 40 s the time of its contact with vapor increased and the efficiency of mass-exchange increased due to an increase in the gradient of concentrations of volatile components, and there was a more complete saturation of vapor with volatile components of the liquid, and liquids with heavy-volatile components of vapor. As a result of optimizing the operation of the ICC, the quality of rectified alcohol improved (Kiss et al., 2014; Mischenko et al., 2020).

In the cyclic mode the multiplicity of concentration (β) of the head impurities increased by 25%, the upper intermediate impurities – by 40% and the final ones – by 37% (Figure 5).

This can be explained by the fact that there was more of a complete vapor saturation with it volatile components of liquid on the plates of the column, the mixing of the liquid on adjacent plates, so that the grade of phase equilibrium achievement was increased (Chen et al., 2010). 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 vapor consumption without a significant increase in the grade of impurity extraction. The determining impurity, by the content of which the degree of purification of rectified alcohol was assessed, was isopropyl alcohol. It is known that its concentration in the epyurate and the finished product should not exceed 1.5 mg/l (Pang et al., 2017).

It was found that the specific consumption of heating vapor in installations of cyclic action decreased by 40 % compared to installations operating in stationary mode: in the process of processing of alcohol-containing fractions from 25 to 15 kg/dal of a.a. introduced to the feed plate, and in the process of epuration of the beer distillate — from 15 to 8.2 kg/dal of a.a. This is explained by the fact that due to the increase in the contact time of vapor and liquid on the plates of columns of cyclic action the degree of phase equilibrium achievement increased. The consequence of this was a reduction in vapor consumption (Chenyang et al., 2024).

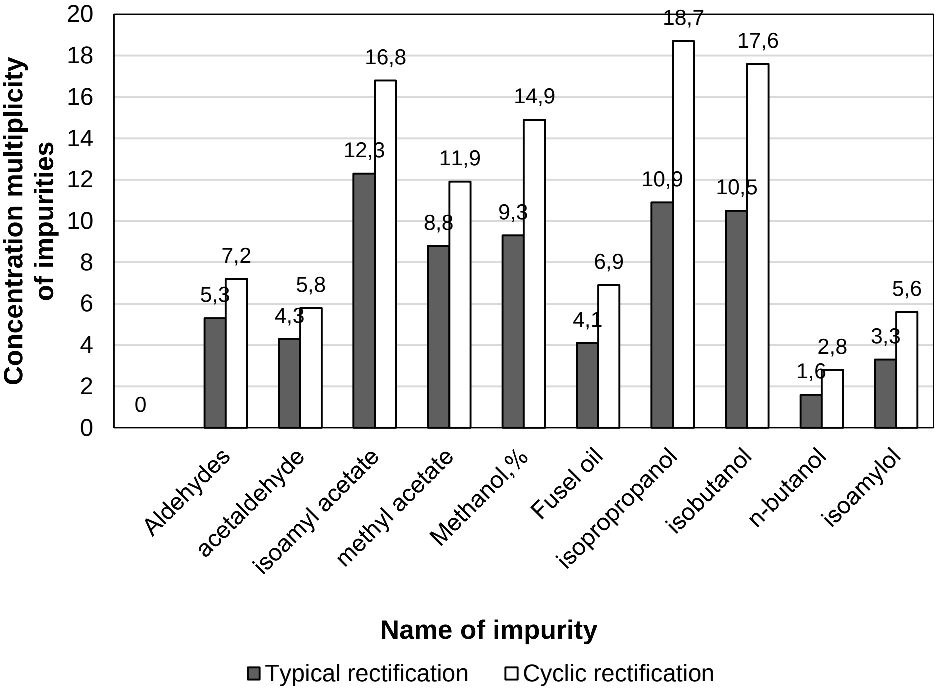

Figure 5. The multiplicity of concentration (β) of volatile alcohol impurities in impurity concentration column (ICC) under typical and cyclic rectification conditions

After the inclusion of the experimental ICC in the DP scheme the yield of rectified ethyl alcohol increased by 3.8 %, the total consumption of hot softened process water for hydroselection decreased by 80% and the quality indicators of rectified ethyl alcohol corresponded to the normative ones. The increase in the yield of alcohol was due to its extraction from the head fraction. Reduction of water consumption was due to the use of distillation residue of the ICC for hydroselection in the EC (Bulii et al., 2021).

At the **second stage** the effectiveness of innovative resource- and energy-saving technology, which provided for the recirculation of distillation residues of rectification column, the ICC and extractive rectification column was investigated. For hydroselection of impurities distillation residue from the ICC was supplied to the EC, and distillation residue of the rectification column was supplied to the column of extractive rectification, so there was no consumption of hot process softened water in the EC and the column of extractive rectification. The specific consumption of process water and heating vapor for its heating (in terms of 1 kg of a.a. introduced into the column) is given in Tables 4 and 5.

Table 4

Specific and total process water consumption for hydroselection of volatile alcohol impurities

Method	Specific flow rates of process water, l/kg of a.a.			Total flow rates process water	
	ICC	EC	Column of extractive rectification	l/kg of a.a.	%
Typical	4.7	0.7	18.1	23.5	100
Innovative	4.7	—	—	4.7	20

Table 5

Specific and total energy consumption for heating process water for hydroselection of volatile impurities of alcohol

Method	Specific energy consumption for heating process water, kJ/kg of a.a.			Total energy consumption	
	ICC	EC	Column of extractive rectification	кJ/kg of a.a.	%
Typical	590.8	88.0	2275.2	2951.0	100
Innovative	590.8	—	—	590.8	20

According to the tables it can be seen that the innovative method allows you to reduce the total consumption of process water and energy costs for its heating by 80%. This is explained by the fact that the supply of alcohol-containing fractions enriched with organic impurities from the EC condensers and rectification column to the ICC feeding plate, the distillation residue of the extractive rectification column above the feeding plate (3–5th plates, counting from above) and hot softened water for hydroselection of impurities to upper plate allows you to reduce the concentration of ethyl alcohol on the upper plates of the concentration part of the ICC and thereby reduce the consumption of process water in the ICC. The supply of distillation residue of the extractive rectification column to the ICC allows you to form a zone for effective concentration of intermediate impurities, including fusel oil components (Bausa et al., 2001).

At the **third stage** the effectiveness of the innovative technology was investigated, which provided for the supply of alcohol-containing fractions to the ICC feeding plate in the form of alcohol-containing vapor that did not condensed in the condenser of the carbon dioxide separator, dephlegmators of the distillation column, the EC (head fraction) and rectification column (unpasteurized alcohol), as well as fusel fraction vapors and fusel alcohol. Such a technical solution eliminated the need for additional consumption of heating vapor for preheating of these fractions to the boiling point for their evaporation in the ICC and made it possible to reduce the consumption of heating vapor and water for cooling the heat exchange equipment due to the absence of condensers in the technological scheme. The results of the research are given in Table 6.

The data presented in Table 6 indicates that using Method I resulted in a reduction of total water consumption for cooling by 0.013 m^3/dal of a.a. (2.1%).

Table 6

Water (W) and heating vapor (HV) consumption in distillation plant (DP) of indirect action equipped with the impurity concentration column (ICC)

Columns of distillation plant	W, m^3/dal of a.a.			HV, kg/dal of a.a.		
	known method	innovative		known method	innovative	
		method I	method II		method I	method II
Distillation	0.065	0.064	0.062	20	20	20
Ether	0.154	0.151	0.146	12	12	12
Rectification	0.282	0.276	0.268	22	22	22
ICC	0.128	0.125	0.121	10	9.1	8.8
DP	0.629	0.616	0.597	64	63.1	62.8

Additionally, the consumption of heating vapor in the ICC decreased by 0.9 kg/dal of a.a. (1.4%). With Method II, water consumption decreased by 0.032 m^3/dal of a.a. (5.1%), while heating vapor consumption dropped by 1.2 kg/dal of a.a. (1.9%). Importantly, the organoleptic and physicochemical parameters of the rectified ethyl alcohol remained within normative standards. Furthermore, when utilizing Method II the profit for a plant with a capacity of 3000 dal of alcohol per day increased by 33.3% compared to Method I. This is explained by the absence in the technological scheme of condensers and the reduced consumption of cooling water and steam to heat the alcohol fractions reaching the feed plate of the ICC.

Conclusions

1. Extension of the residence time of the liquid on the plates of the ICC up to 40 s in the selected hydrodynamic mode allows to increase the degree of extraction and the multiplicity of concentration of the head, intermediate and final impurities of alcohol by 25–40 %.
2. The consumption of heating vapor in the processes of epyuration of the beer distillate and processing of alcohol-containing fractions in cyclic mode is reducing by at least 40 %.
3. Recirculation of distillation residue of rectification column, ICC and extractive rectification column allows reducing the specific consumption of process water and vapor for its heating by 80% in the DP of indirect.
4. Feeding the ICC with alcohol-containing fractions in the form of vapor in the amount of 2% allows you to reduce the total water consumption for cooling by 0.013 m^3/dal of a.a. (2.1%) and consumption of heating vapor by 0.9 kg/dal of a.a. (1.4%).
5. In case of an increase in the amount of non-condensed vapor in the dephlegmators of the main columns from 2 to 5% and passing it to the ICC, the cooling water consumption is reduced by 0.032 m^3/dal of a.a. (5.1%), and of the heating vapor by 1.2 kg/dal of a.a. (1.9%).
6. The absence of condensers and alcohol traps of the main columns in the technological scheme allows you to reduce the metal intensity of the DP by 10%.

Taking into account the positive results obtained, it is advisable to continue research in this direction. Promising studies are those on the efficiency of the main columns in cyclic mode, as well as the operation of the ICC and other DP columns, provided that the volume of alcohol-containing vapors entering their feed trays increases to 10–25% or more.

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