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Industrial experience in using cyclic distillation columns for food grade alcohol purification

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ABSTRACT

This study provides novel details about the industrial use of cyclic distillation in the production of food-grade alcohol, which confirmed the theoretical predictions of increasing separation efficiency. Increasing the profitability of the production of ethanol food grade is primarily associated with an increase in product quality and reduction of energy costs per unit of production. One of the ways to solve these problems is to improve the ethanol purification technology by using cyclic distillation, which allows reduction of energy costs and higher productivity by removing impurities at a higher concentration (leading also to waste reduction). The purification of ethanol from impurities (head and intermediate type) is carried out in hydro-selection columns. Critically, the volatility of most components depends on the actual ethanol concentration on the stage. This study investigated the distribution of ethanol on the trays depending on the water feed stage to the column and showed the optimal distribution of hydro-selective water in an industrial column to allow the highest possible separation efficiency of components during cyclic distillation. A cyclic distillation column with 15 Maleta trays was superior in separation capacity and performance as compared to a traditional column with 50 bubble cap trays.

1. Introduction

Mass transfer processes are widely used in many industries, and the number of production processes (which includes rectification processes) is growing every year. During the past decades, many advanced distillation technologies were implemented in the chemical process industry in order to reduce the capital and operating costs. Such intensified fluid separation technologies include dividing-wall column, heat-integrated distillation column, reactive distillation, and cyclic distillation (Kiss, 2013). Among them, cyclic distillation stands out as a new contender due to a different way of contacting the liquid and vapor phases (Maleta et al., 2011). Cyclic distillation uses separate phase movement (SPM) that can be achieved with specific internals and a periodic operation mode (Kiss and Bildea, 2015). One operating cycle consists of two parts: a vapor flow period (when the thrust of rising vapor prevents liquid down flow) followed by a liquid flow period (when the liquid flows down the column, dropping by gravity, first to a lock chamber and then moving to the tray below). This leads to several key advantages, such as increased column throughput, significantly reduced energy requirements, and much better separation performance.

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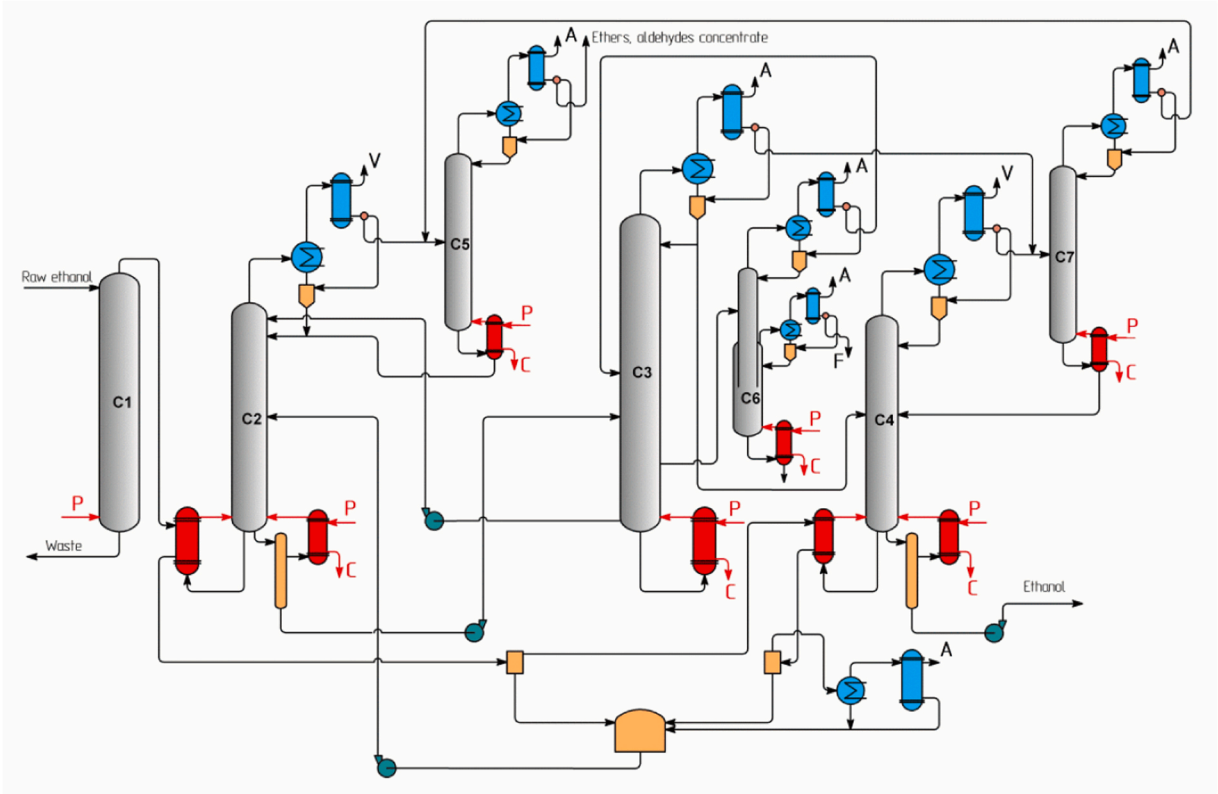

Fig. 1 – Flowsheet of an ethanol production plant: C1 – beer column, C2 – hydro-selection column, C3 – rectification column, C4 – column for end-cleaning, C5 – column for concentrating impurities, C6 – fusel column, C7 – methanol column, P – steam, C – condensate, A – atmosphere, V – vacuum, F – fusel alcohol.

A review of ways to increase the efficiency of mass transfer equipment in recent years has shown that the use of cyclic distillation with separate phase movement is successful at industrial scale and it is gaining momentum (Bildea et al., 2016). In addition, several papers and reviews on cyclic distillation are available in literature, covering its history, working principles, modeling and simulation (Bildea et al., 2016), design and control (Patrut et al., 2014; Andersen et al., 2018), perfect displacement model and working lines (Maleta et al., 2011), simultaneous vs consecutive cycling operation mode (Toftegård et al., 2016), driving force based design (Nielsen et al., 2017), mathematical modeling (Krivosheev and Anufriev, 2018), impact of operating parameters on the performance (Buetehorn et al., 2015), new tray designs (Maleta and Maleta, 2012), pilot-scale industrial studies (Maleta et al., 2015), industrial equipment and applications (Kiss and Maleta, 2018), and revamping of conventional columns to cyclic distillation (Kiss and Bildea, 2015).

The selection and design of fluid separation processes is very important in all chemical processes (Blahušiak et al., 2018). In this respect, to ensure high and stable distillation quality, the typical scheme of ethanol fermentation-distillation plants includes 6–7 columns that are required for the separation and purification of food grade alcohol, as shown in Fig. 1 (Maleta et al., 2019). There are many flowsheets for the production of ethanol food grade. Some of them are aimed at saving energy, some are aimed at obtaining a high degree of purification, and others (like this one) try to combine both (Madson, 2003). To prevent a drop in productivity, additional small columns are installed on the main columns, which allow the removal of impurities at a higher concentration. The main columns include: C1 – beer column (removes all volatile impurities from beer), C2 – hydro-selection column (removes head impurities such as ethers, esters and aldehydes), C3 – rectification column (raises the concentration of ethanol to 96.3%vol and removes fuselage), C4 – column for end-cleaning (removes end impurities, such as methanol). Additional columns used include a column for concentrating impurities (C5), a fusel column (C6), and a methanol column (C7). To obtain high quality ethanol without additional columns, the amount of waste can be up to 20%. Using the additional columns, the amount of waste drops to only 3%. In this process, energy savings are obtained by using the heat of condensation of the vapors of the beer column (C1) to heat the hydro-selection column (C2) and final purification column (C4), which operate under vacuum. Additional energy savings could be obtained by using heat pump assisted operation (Kiss and Infante Ferreira, 2016).

Overall, the main goals in the purification of food-grade alcohol are to obtain efficiently a product with a minimum content of impurities and to maximize the yield of products. Experimental studies in the field of rectification have shown that the process of separation of the binary mixture into fractions by the traditional method does not fully unleash the potential of such installations (Maleta et al., 2015).

This original research paper – invited for the special issue of *Chemical Engineering Research and Design*, dedicated to the 12th International Conference on Distillation & Absorption – focuses on the industrial experience in using cyclic distillation for the purification of food-grade alcohol (Bedryk et al., 2022).

2. Problem statement

The alcohol industry aims to improve the purification of ethanol product. Its purification with the production of high-quality food alcohol involves the sequential distillation of the

water-alcohol mixture in the fermentation-distillation unit, which ensures the separation of impurities (more than 70 species) from the zones of their maximum concentration. Their concentration and removal as a by-product occur in the hydro-selection columns. In this respect, it is necessary to increase the water supply for hydro-selection in these columns, which reduces the concentrations. This, in turn, disrupts the distribution of intermediate impurities in the distillation column, complicating their separation.

To solve this problem, this original research focuses on determining the optimal conditions for the removal of impurities due to the uniform distribution of ethanol over the entire height of the distillation column, for a fixed value of the concentration of the bottom fluid. The maximum effect of the exit of the main amount of impurities from the column is achieved at a low concentration of ethanol on the trays. This is facilitated by hydro-selection (extractive distillation with water). However, the distribution of water in the column depends on where the water is fed into the column. The aim is to determine (theoretically and experimentally) the optimal conditions for the removal of impurities in the column in view of the uniform distribution of ethanol concentration over the column.

3. Results and discussion

The focus of this work is a hydro-selection column (another name for the impurity concentration column), which is associated with a wide range of demonstrations of its capabilities of the proposed method and equipment. Note that the hydro-selection column performs two functions: 1) Reduces waste from alcohol production (by 3–10% of the plant's productivity) due to a higher concentration of the removed impurities and thus less alcohol wasted in the impurities stream. 2) Improves physico-chemical and organoleptic qualities of the product by processing / removing a larger volume of waste from the main columns.

Different impurities are removed under different conditions in the hydro-selection column. By changing the feed location and the amount of water for hydro-selection, one can influence the composition of the removed impurities. The hydro-selection column operates in cyclic distillation mode. Such columns are already used in industry and provide the greatest efficiency of separation of components. Based on a mathematical model for cyclic distillation processes Maleta Cyclic Distillation has created a program to calculate the distribution of alcohol concentration on the plates of the column. This model was used to simulate this dependence (Maleta et al., 2011), but other models could be also used as described in the literature (Patrut et al., 2014; Nielsen et al., 2017).

The simulation of the column was performed on the example of a plant capacity of 30,000 L/day on 15 plates. The feed supply was on the 8th stage. The input simulation parameters are shown in Table 1.

Several options for the water supply for hydro-selection are considered:

- Water and reflux are fed to the first plate of the top section of the column (Fig. 2a);
- Water is fed to the 1st plate from the top, while reflux is fed to one of the lower plates (Fig. 2b);
- Water is fed to the feed tray, while the reflux is fed to the first tray at the top (Fig. 2c).

Table 1 – Parameters used in the simulation of the cyclic distillation column.

Description	Unit	Value
Consumption of steam	kg / h	200
Feed flowrate	L / h	200
Water flowrate (for hydro-selection)	L / h	820
Power supply	% vol.	90,00
Bottom product concentration	% vol.	15,00

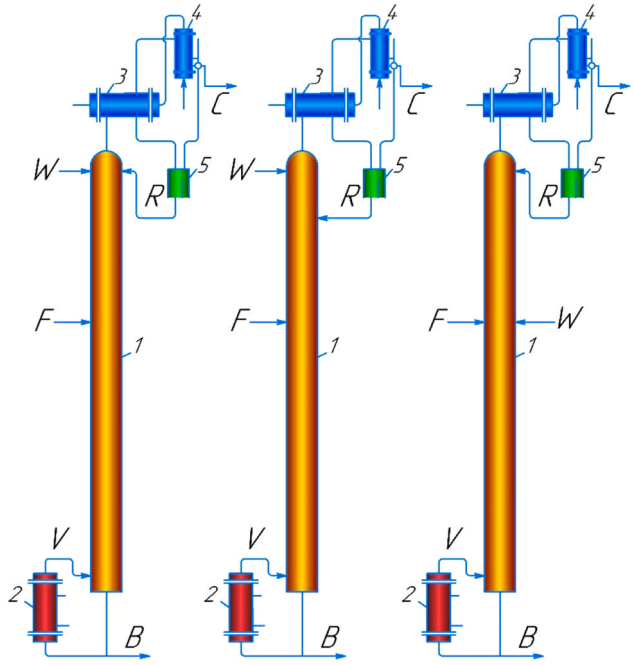

Fig. 2 – Scheme of flows in the hydro-selection column: a) water and reflux are both fed on the first plate from the top; b) water is fed on the first tray from the top, while reflux is fed on the lower trays; c) water is fed on the same stage as the feed (diluting the feed), while reflux is fed on the first stage at the top. Notation: 1 - column; 2 - boiler; 3 - reflux condenser; 4 – heat exchanger; 5 – reflux drum; C - main fraction concentrate; F - feed supply; R - reflux; B - bottom product; V - steam; W - water.

The scheme of flow distribution in the column is shown in Fig. 2, in which there is a single feed of water to two possilbe places (top of the column or the feed stage), with two options for supplying the reflux to the very top of the column and somewhete below the top. The results of the calculations of the hydro-selection column are illustrated in Fig. 3, which shows that the location of the water feed significantly affects the distribution of the concentration of alcohol along the height of the column and accordingly, the conditions for the removal of impurities.

Concentration barriers are possible on the plates of the column. In this context, this is a local increase in the concentration of ethanol, which reduces the volatility of impurities and reduces the efficiency of ethanol purification. Obviously, this situation should be avoided. Industrial experiments confirmed the presence of such an effect in the column and also confirmed the correctness of our assumptions about the tools to eliminate these problems. The concentration barrier is a new terminology that we have proposed to describe the process in the hydro-selection column (Bedryk et al., 2022). This can be defined as an

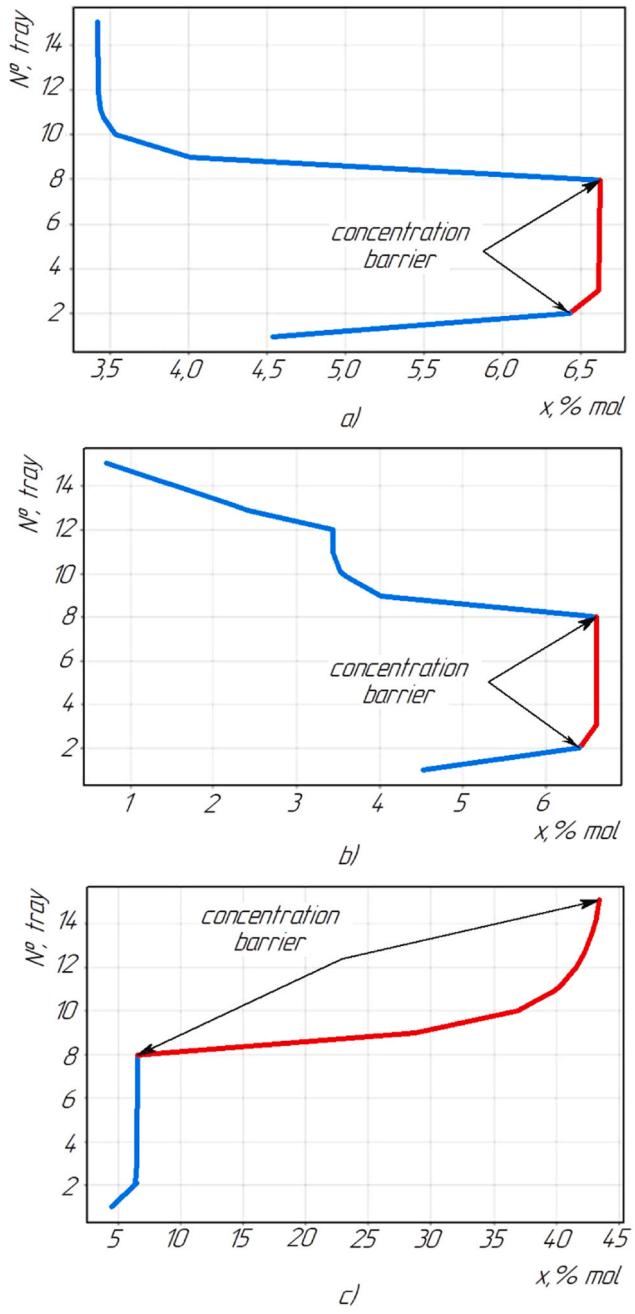

Fig. 3 – Column profiles showing the distribution of alcohol concentration on the trays of the hydro-selection column: a) water and reflux are fed on the first plate from the top; b) water is fed on the first plate from the top, and reflux is fed on the lower plates; c) water is fed on the feed stage, and reflux on the first plate at the top.

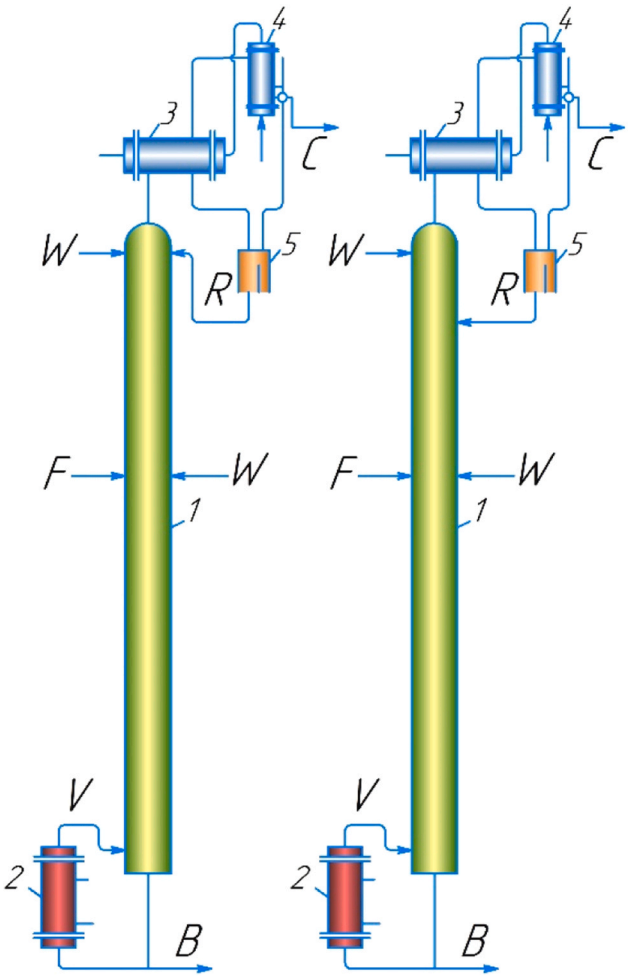

Fig. 4 – Flow distribution scheme in the hydro-selection column of the cyclic mode: a) Mode I – water is fed on 15th tray and also in on 8th stage to dilute the feed, while the reflux is fed on 15th tray (at the top); b) Mode II – similar to mode I but the reflux is fed on 12th tray (i.e. 3 stages below the top tray). Notation: 1 - column; 2 - reboiler; 3 - reflux condenser; 4 – heat exchanger; 5 – reflux drum; C - main fraction concentrate; F - feed supply; R - reflux; B - bottom product; V - steam; W - water.

increase in the concentration of ethanol in any part of the column, preventing the removal of impurities.

In Fig. 3a and b, the concentration barrier is located in the stripping part of the column. In Fig. 3c, the concentration barrier is located in the rectification part of the column. This distribution of hydro-selection water leads to different conditions for the removal of impurities (different concentrations of ethanol) for different parts of the column, which negatively affects the separation process since the local increase in the concentration of ethanol reduces the volatility of impurities and consequnelty reduces the efficiency of ethanol purification. However, equalization of ethanol concentrations along the height of the column is possible in the case of redistribution of water between the feed stage and the first plate above.

Two modes of operation of the column were simulated, as these modes allow controlling the concentration of ethanol along the entire height of the column. The input parameters of the calculation of the hydro-selection column do not change compared to the previous cases.

- Mode I: water feed on 15th tray and dilution of the feed on 8th stage, with reflux supply on 15th tray (at the top).
- Mode II: water feed on 15th tray and dilution of feed on 8th stage, with reflux supplied to 12th tray.

Fig. 4 illustrates the scheme of distribution of flows in the simulated cyclic distillation column. There are two feeds of water which are supplied in the same two places at once (top of the column and the feed stage), and again with two options for supplying reflux to the very top of the column and somewhere below the top. The simulations show how this affects the ethanol distributions across the column trays. It allows achieving the maximum separating capacity of the

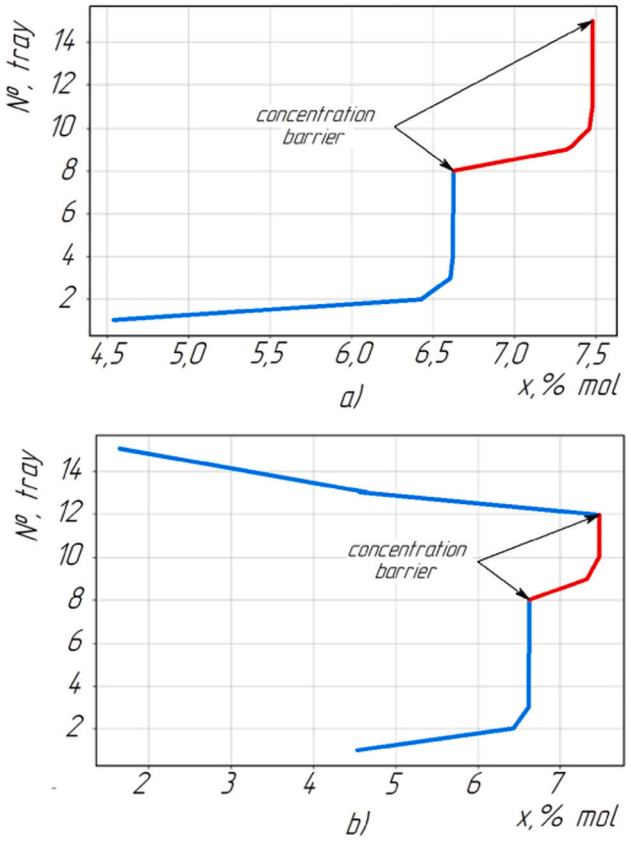

Fig. 5 – Distribution of ethanol concentration on the plates of the column (Ethanol concentration on the feed tray 30% vol.).

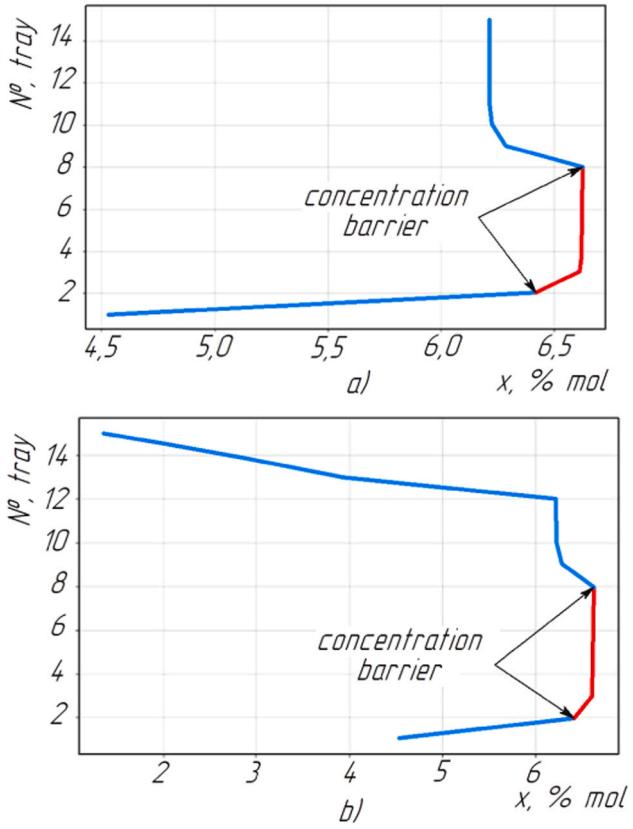

Fig. 6 – Distribution of ethanol concentration on the plates of the column (Ethanol concentration on the feed tray 35% vol.).

column by creating conditions for the same volatility (same concentration of ethanol on trays) of impurities along the entire height of the column, using hydro-selective water as a tool for this.

The raw material used is a mixture of streams from all columns of the distillation system containing the largest amount of impurities (e.g. head fraction, extracted impurities from various columns). The feed stream is pre-heated by the bottom product stream.

The distribution of ethanol concentration in mode I of the hydro-selection column is shown in Fig. 5a, Fig. 6a, and Fig. 7a. The supply of water on the 15th stage and the dilution of the feed on 8th stage with reflux fed to the 12th stage in mode II is shown in Fig. 5b, Fig. 6b, and Fig. 7b. Hydro selective water dilutes the feed to a concentration of ethanol on feed stage of 30%, 35% and 33% vol., respectively. Note that at a concentration of 30% the concentration barrier will be at the top of the column, while at a concentration of 35% ethanol on the feed stage the concentration barrier will be at the bottom of the column. However, at a concentration of 33% ethanol on the feed stage, the concentration barrier disappears (see Fig. 7). Fig. 5, Fig. 6, and Fig. 7 show that the location of the water supply for hydro-selection and its distribution between the feed points drastically affects the concentration of alcohol.

The hydro-selective column separates head impurities (such as acetaldehyde, ethyl acetate, acetone, and others) and fusel impurities (such as propanol, butanol, amylol, and others). Note that there are about 300 impurities that can be found in food ethanol. These impurities are divided into four large groups according to their volatility relative to ethanol (as described later in the experimental validation). To remove each group of impurities, special conditions are created in the distillation column.

Simulation results have been confirmed under industrial conditions by chromatographic analysis of the bottom and top products. The degree of purification from impurities was determined by the amount of impurities in the bottom product. The concentration of the bottom product may be different, depending on the type of impurities that need to be removed from the ethanol. Note that the column concentrating the impurities removes several types of impurities: head (volatile) impurities, intermediate impurities and end (heavy) impurities. Accordingly, the conditions for their separation are somewhat different. For head impurities, the concentration of ethanol at the bottom of the column should be 15–20% (Fig. 7a). For intermediate impurities, the concentration of ethanol at the bottom of the column should be 5–10% (Fig. 7b). For end impurities, the column configuration shown in Fig. 2c should be used. At the same time, the amount of water for hydro selection should be minimized to reduce heating costs for the subsequent processing of the bottom product. Overall, the most effective option was the one shown in Fig. 7b, since the ethanol concentration at the top of the column was minimal.

4. Experimental validation at industrial scale

Experimental studies focused on the impurity concentration column in industrial conditions were carried out at the plant for ethanol food grade production of the Limited Liability Company '*Chervonoslobidskiy Alcohol Factory*' (Ukraine). The column has a height of 5.5 m and a diameter of 560 mm, and consisted of 16 plates (Maleta trays). The feed stream is

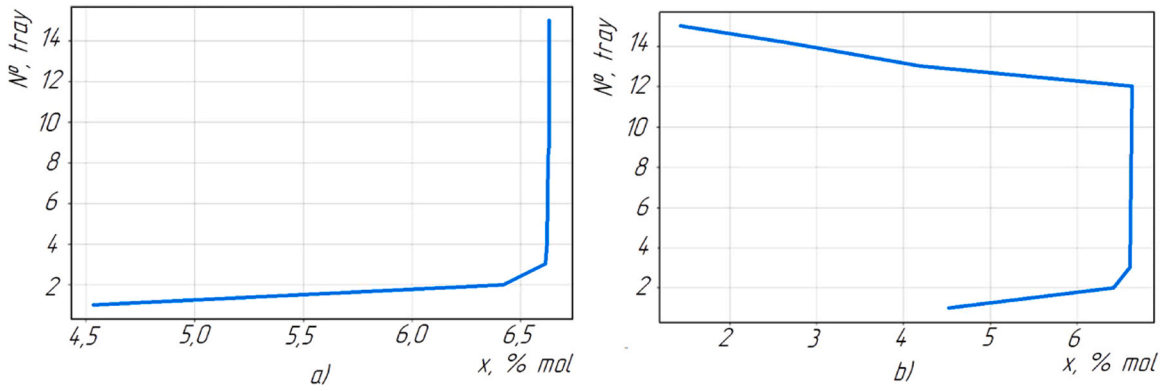

Fig. 7 – Distribution of ethanol concentration on the plates of the column (Ethanol concentration on the feed tray 33% vol.).

added on the 8th plate. Water for hydro-selection can be supplied to the 8th and 16th plates. Reflux was added on the 16th and 12th plates. The supply of steam and the feed to the column were not changed during the experiments (although they were subjected to some fluctuations), and amounted to 250 kg/h and 200 L/h, respectively. The industrial operating conditions of the column are characterized by a large range of scatter in the composition of impurities entering the column. However, there was no possibility of taking feed stream samples, so the efficiency of the column was determined by comparing the composition of the concentrate at the top of the column and the composition of impurities at the bottom of the column, all measured by gas chromatography (GC).

There are several main factors affecting the concentration of impurities: 1) the concentration of ethanol in the liquid on the tray, 2) the driving force of the process in the form of the stripping factor (diffusion potential factor), and 3) the equilibrium conditions in a multi-component mixture at high concentrations of impurities. The degree of separation of impurities and ethanol depends on the difference in their relative volatility. The optimal range of ethanol concentration in solution is 6–8% vol for the main impurities (e.g. aldehydes, ethers, esters), and 4–5% vol for intermediate impurities (e.g. fuel fumes). Regarding the driving force, for a cyclic process, the Murphrey efficiency is proportional to the stripping factor ($\lambda = mG / L$).

In industrial research, the qualitative side of the theoretical modeling can be evaluated. The main idea of the experiments is to create conditions for uniform distribution of ethanol over the trays of the column, which will make it possible to eliminate the presence of a concentration barrier from alcohol, increase the volatility of impurities and obtain the maximum separation efficiency of the components. All impurities that must be separated from alcohol in the hydro-selection column should be conditionally divided into several groups: ethers, esters and aldehydes, intermediate impurities (fuel fumes), isopropanol and methanol. The separation efficiency can be assessed by their amount in the top and the bottom of the column. In addition, the key impurity in the alcohol purification is isopropanol, which forms an azeotrope with water (boiling point 80.2 °C) that is very close to ethanol (boiling point 78.3 °C).

Fig. 8 shows the operating modes tested in the industrial experiments, while Table 2 provides the measured concentration of impurities in the top and bottom products when using these operating modes. To create a concentration barrier in the rectification section of the column, the

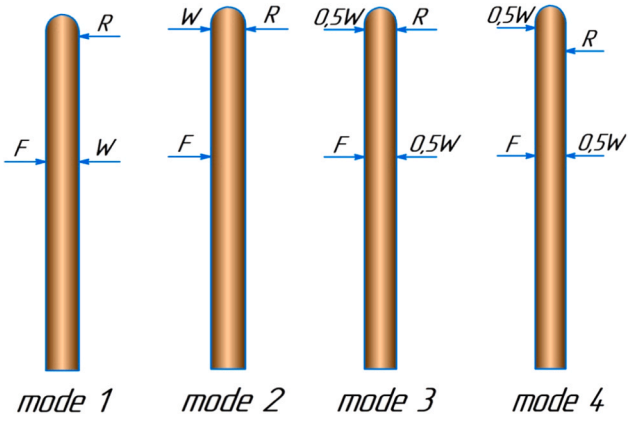

Fig. 8 – Operating modes tested at industrial scale operation of cyclic distillation.

operating mode was chosen with the supply of water to the feed stage (Mode 1 in Fig. 8). In industrial conditions, this mode of operation can be used with an increased concentration of methanol in the alcohol. Analysis of the experimental results showed that in the presence of a high concentration of ethanol in the distillate, the concentration of fusel oils in it decreases significantly; while isopropanol is practically not separated (it goes both to the top and bottom). Almost all the fuselage goes down the column, while most of the methanol goes up and it is removed in the top product. For ethers, esters and aldehydes, overcoming the concentration barrier is not a problem. They are almost all concentrated in the distillate product.

The concentration barrier in the stripping section of the column (Mode 2 in Fig. 8) occurs when the hydro-selective water is supplied to the 1st tray from the top of the column. The behavior of impurities changes this time. The amount of fusel oil in the distillate product already exceeds the concentration at the bottom of the column by several times. The same is true for isopropanol which gets more concentrated in the top product. On the contrary, for methanol its concentration at the bottom of the column is several times higher than at the top. In this series of experiments, the mutual influence of the impurity volatility and the stripping factor on the separation efficiency is observed. At the maximum water supply for hydro-selection (maximum volatility of head impurities), their presence is observed at the bottom of the column. This is due to a decrease in the stripping factor.

To make the concentration barrier smooth, the hydro-selection water stream was split into two equal parts and

Table 2 – Concentration of impurities in the top and bottom products, using four operating modes (shown in Fig. 8).

Water used for hydro-selection (L/h)	Ethanol (% vol.)		Aldehydes (mg/L)		Ethers + Esters (mg/L)		Methanol (% vol.)		Isopropanol (mg/L)		Fusel oil (mg/L)	
	Bottom	Top	Bottom	Top	Bottom	Top*	Bottom	Top	Bottom	Top	Bottom	Top
Mode 1												
1200	6	94.5	17	232	24	373 K	0.066	0.15	8.56	15	8210	440
900	11.5	94	0	103	0	347 K	0.018	0.14	5.68	14.84	465	115.54
600	10	95	0	111	131	178 K	0.022	0.113	23.15	25.6	3224	38
Mode 2												
1200	6	82.5	26.5	383	9.5	373 K	0.22	0.054	0	17	1261	3023
900	9.5	82.5	0	247	2.7	388 K	0.15	0.025	4.12	42.5	121	1383
600	9.5	84.5	0	220	0	353 K	0.09	0.035	3.22	56.3	784	1427
Mode 3												
1200	8	84	0	410	0	353 K	0.42	0.015	3.08	19	197	2344
900	15	84	0	121	0	290 K	0.01	0.073	1.9	32	142	681
600	17	82	0	341	0	84 K	0.14	0.07	5.3	18.8	1146	1280
Mode 4												
1200	8	78.5	0	255	0	125 K	0.2	0.05	0	18.9	501	1226
900	13	79	0	72	0	320 K	0.01	0.06	0	44	68	1277
600	10	85.5	0	368	0	286 K	0.015	0.07	0	25	131	978

* **Note**: K stands for kilo (10^3), meaning that 320 K = 320,000 mg/L

carried to the top of the column (1st stage) and to the feed stage. The two modes in this configuration differed only in the feeding of the reflux stream. When reflux was also added to the top of the column on the 1st stage (Mode 3 in Fig. 8), ethers / esters and aldehydes were completely removed from the mixture for all changes in the amount of hydro-selective water. Concentrating the mixture on the top tray due to reflux contributed to the removal of methanol. Isopropanol, as the dominant impurity, responded to the concentration of ethanol on the top tray, and did not drop to zero at the bottom of the column. The fusel fraction increased its volatility and the concentration in the top product significantly exceeded the composition at the bottom of the column. The last option (with the reflux feed below the top water feed) turned out to be the most acceptable operating mode (Mode 4 in Fig. 8), with all the positive aspects of the third option. In this case, there is no isopropanol at the bottom of the column (and also no aldehydes and ethers / esters), which indicates the high efficiency of the cyclic distillation column in the purification of ethanol from impurities.

5. Conclusions

The introduction of new technology is an integral part of innovative development in distillation processes. The hydro-selection technology proposed in this work makes it possible to create optimal conditions for the removal of various head impurities and fusel impurities by equalizing the concentration of ethanol throughout the height of the column. The use of cyclic distillation technology makes it possible to reduce water consumption for hydro selection by increasing the efficiency of separation of components, as well as to increase the degree of purification of ethanol from accompanying impurities. Moreover, in terms of separation efficiency, the cyclic distillation with only 15 plates manages to outperform the traditional distillation column with 50 bubble cap trays, thus proving additional yield of alcohol (up to 3–4%), improving the physico-chemical and organoleptic characteristics of alcohol, and reducing the energy requirements per unit of ethanol product (by up to 30%).

Considering that the relative volatility of the bulk of the impurities increases with decreasing ethanol concentration, mode II is preferred for practical operation as the concentration on the plates of the upper and lower parts of the column is lower than on the middle stages. For the studied conditions, the optimal ethanol concentration on the feed tray is 33% vol. Other concentration ranges around the optimal point were used as an example. Range extensions on both sides only aggravate the separation conditions (increase the concentration barrier) so they are not considered. The optimal distribution of water supply for hydro-selection is in the proportion: feed tray - 42% and 15th plate - 58%. However, when changing any of the input parameters (such as feed flowrate, feed composition, steam flowrate), the optimal value must be recalculated accordingly.

The experimental validation at industrial scale showed that the operation using the optimal mode leads to complete removal of isopropanol, aldehydes and ethers / esters from the bottom product (while most of the fusels accumulate in the top product), which indicates the high efficiency of the cyclic distillation column in the purification of food grade alcohol from a large range of impurities.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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