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ENERGY-SAVING RECTIFICATION TECHNOLOGY WITH CONTROLLED MASS EXCHANGE CYCLES BETWEEN LIQUID AND VAPOUR

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ABSTRACT

An energy-saving technology of cyclic rectification with a continuous supply of heating steam and liquid to a mass-exchange column apparatus equipped with flake plates is proposed. The innovative method allows to prolong the time of contact of steam and liquid on plates up to 40-60 s and to reduce the time of overflow up to 1-1,7 s. In order to implement the technology, a rectification column design was proposed, equipped with flake plates with variable free cross-section.

Hydrodynamic modes of operation of perforated plates are determined to ensure their cyclic action: the lower critical velocity of steam in barbotage openings of meshes plates is 5,4 m/s, of scales plates is 6,5 m/s; the upper critical velocity at which fluid is carried away is 8 and 16 m/s, respectively. A prerequisite for spilling liquid through barbotage openings is an instantaneous reduction of the steam velocity in the openings to 1,5-1 m /s due to the instantaneous change of the free cross-section of the plate from 2,6 to 51,7 % and more.

Research of efficiency of the offered technology was carried out in industrial conditions in processes of distillation of alcohol-containing fractions and epuration of alcoholic fermentations distillates in the experimental column. This technical solution makes it possible to provide complete purification of ethyl alcohol from ethers, methyl acetate and isopropyl alcohol, to increase the degree of extraction of higher alcohols of sour oil and methanol by 38%, to increase the multiplicity of the concentration of head impurities by 25 %, the upper intermediate impurities - by 38%, the end impurities - by 37 %, as well as increasing the liquid throughput of the column by 34 %. Lack of drain and receiving devices on the plates makes it possible to increase the contact area between steam and liquid by 15-20 %. The coaxial arrangement of the flakes on *the plate web eliminates the possibility of formation of stagnant zones and allows to intensify the mass transfer between steam and liquid.*

Under these conditions, the specific heating steam consumption is reduced by 40% compared to typical installations: in the rectificational column decreases from 25 to 15 kg/dl of anhydrous alcohol introduced on the acceptance plate, and in the epyurating column - from 15 to 8,2 kg/dl of anhydrous alcohol.

Keywords: *mass exchange, plates, rectification, cycles, steam column apparatus, alcohol impurities.*

Problem statement. The use of cyclic rectification technology in the columns of bragorectificational plants is a relatively new approach for the intensification of the mass transfer process in alcohol production. Implementation of controlled cycles of liquid retention on the plates allows to prolong the time of its contact with steam, to create conditions in order to achieve a phase state close to equilibrium and to bring the efficiency of each real plate closer to the theoretical one (Maleta, Shevchenko, Bedryk & Kiss, 2105). In the above conditions, the driving force of the mass transfer process increases due to the increase in the concentration gradient of volatile components, improves the diffuse characteristics of contact devices, increases the efficiency of their operation and reduces the specific flow rate of heating steam (Bulii, Kuts & Shyian, 2019). The above-mentioned advantages of cyclic rectification seem to be very attractive for implementation in production.

Analysis of recent research and publications. Research of physical and chemical conditions of separation of multicomponent systems in the process of cyclic rectification, development of rational methods of calculation and design of cyclic rectification columns on the basis of laws of thermodynamics, works on computer modeling of cycles with the purpose of intensification of mass exchange have begun in 60-70-ies of the XX-th century. The scientific works of Whirter and Cannon, Sommerfield, Chien, Robinson and Angel, Schrodt, Helperin, Beiron, Frezer, Thomson, Schioni, Anton Kiss, Matsubar and others are devoted to solving this problem (Kiss, 2015; Kiss, Flores, Landaeta & Zondervan, 2012; Nielsen, Huusom & Abildskov, 2017). A lot of scientific works of outstanding scientists are devoted to energy saving, increase of efficiency of mass exchange on cyclic plates and nowadays. The studies conducted by V.P. Krivosheev, A.V. Anufriev, M.I. Farakhov, S.B. Azizov, H.R. Flodman, M. Matsubara, M. Petrus, etc. are noteworthy (Flodman & Timm, 2012; Lita, Bildea & Kiss, 2012, Krivosheev & Anufriev, 2015). At department of processes and devices of national university of food technologies under the direction of professor Taran V.M. theoretical bases of cyclic rectification are developed, designs of column apparatuses are offered, which action allowed to provide separate movement of phases in a rectification column, to intensify process of mass exchange by retention of liquid on plates (Maleta, Taran & Maleta, 2010).

Despite the obtained positive results in reducing energy costs, the known methods and apparatuses of cyclic operation have not found wide practical application due to the lack of mass transfer in the steam period, the steam pressure dependence of pouring devices' operation, the fluctuations of the steam pressure in the collector, the inability to stabilize the hydrodynamic mode of plates, the mixing of liquid on adjacent plates during its pouring, the low apparatuses' steam and liquid throughput capacity, and the complexity of constructive solutions.

The authors proposed an innovative rectification technology (patent UA 141245. Method of pouring the liquid on the plates of mass-exchange column apparatus), which excludes earlier mentioned disadvantages and provides periodic liquid pouring from one plate to another at continuous supply of liquid and heating steam into the column. To implement the technology, the design of a rectification column equipped with plates with variable free cross-section was developed (patent UA 139228. Column mass-exchange apparatus of cyclic action). These include scales, meshes, tubular, wavy, flake and other plates. Advantages of perforated plates - simplicity of a design, low cost of manufacturing and installation, rather small hydraulic resistance. The main drawback is the short interval of change of speeds of steam and a liquid within which their steady and effective work, insufficient time of contact of phases, and also mixing of a liquid on adjacent plates is supported.

The innovative technology provides the implementation of controlled cycles of liquid retention on perforated plates and its periodic overflow through all the openings from the upper plates to the lower ones due to the instantaneous change of steam speed. While the liquid is on the plates, there is a mass transfer between the liquid and the steam, which goes through the contact device holes. During this cycle period, the movable valve closes the overflow port and the fluid is held on the plate. When the overflow opening is opened, the steam velocity in the barbotage openings becomes lower than the critical speed at which it is held on the plate and the liquid is transferred to the lower plates. The versatility of the operation of the barbored-perforated plates is in the alternation of the steam velocity in the contact device holes by changing the free cross-section of the plates in a given range of values according to a given algorithm (Buliy, Shiyan & Kuts, A.,2016).

Purpose of the article: the definition of the hydrodynamic mode of operation of barbotage perforated plates, of the efficiency of the technology of cyclic rectification in the mass-exchange columns equipped with barbotage perforated plates with variable free cross-sectionin and determining the consumption of heating steam in the rectificational and epyuratin columns.

Materials and methods. Research methods - analytical, chemical, physico-chemical with the use of instruments and research methods used in the production of rectified ethyl alcohol. Fluid consumption was controlled with the help of flowmeter RM, air velocity in the free section of the column - anemometer MS-13, in the holes of the plates - by calculation method. The concentration of volatile impurities of alcohol was determined on a gas chromatograph with a column HP FFAP 50 m × 0.32 m. Analysis of research samples was performed according to the State Standard of Ukraine 4222:2003 "Vodka, ethyl alcohol and water-alcohol solutions. Gas chromatographic method for determination of microcomponents content" (Plutowska & Wardenski, 2008).

A summary of the main findings of the study. Efficiency of operation of barbotage perforated plates in the conditions of cyclic rectification depends on the adopted hydrodynamic regimes that determine the boundaries of stable operation of columns mass exchange apparatuses. The necessary condition for their determination is the establishment of the maximum allowable steam velocity (upper and lower critical velocity) in the free section of the column and in barbotage holes.

The lower limit corresponds to the steam velocity at which the "sink" of the liquid from the upper plates to the lower ones stops, the upper limit corresponds to the steam velocity at which the liquid from the lower plates begins to drift to the upper ones, which leads to a reduction of the phase contact surface. The research was carried out on an experimental rectification column equipped with replaceable contact devices - meshes and scales in the water-air system. Characteristics of the column: diameter - 300 mm; number of plates - 5; distance between plates - 300 mm; diameter of barbotage holes – 2,4 mm; area of section of holes of arched type scales – 19,42 mm^2; thickness of a cloth of a plate - 2 mm; free section of a plate – 2,6 %; height of a layer of a liquid on plates - 35 mm. For the meshes plates the air flow rate was changed in the range of 1-15 dm^3/s, which corresponds to the change of speed in the barbotage openings of 1,5-10 m/s, the irrigation density fluctuated in the range of 4 to 11 m^2/(m^2·h). For scales plates the irrigation density varied from 5 to 15 m^3/(m^2·h).

It is known that in order to increase the efficiency of plate of various designs it is advisable to delay the liquid on their canvas by organizing the flow of separate vapor-liquid jets with mutual collision of jets or additional installation of partitions and reflectors. Liquid brake devices are made in the form of holes, nozzles, etc. The area of free cross-section of holes with a counter flow of steam is 2-3 % of the total area of the plate. To increase the duration of the contact between steam and liquid and to intensify the mass transfer, vertical dividers are installed in some areas of the plate. Contact devices with partial compensation of direct flow were first studied by A.N. Prokhorov. The studies conducted in this direction by V.P. Krivosheev, A.V. Anufriev, M.I. Farakhov, S.B. Azizov, A.A. Kiss, H.R., Flodman, M. Matsubara, M. Petrus and others deserve attention (Buliy, Shiyan, Kuts & Melnic, 2019; Van Gerven & Stankiewicz, 2009).

In spite of positive results, proved by methods of mathematical modeling, the known methods and models have not found wide practical application because of complexity of the chosen constructive decisions, absence of mass exchange in the steam period, fluctuations of pressure of steam in a collector and low throughput of column mass exchange apparatuses on steam and a liquid.

In order to solve the set tasks the authors proposed the technology of cyclic rectification (patent UA 136560. Method of mass-exchange between liquid and steam in a column apparatus), which allows to exclude the above mentioned disadvantages, and the construction of a column mass exchange apparatus equipped with perforated plates of cyclic action (patent UA 136561. Mass-exchange contact plate). The method provides for periodic overflow of liquid from the plate to the plate due to the forced operation of overflow devices containing moving elements associated with the drive mechanisms. To implement the technology, the column apparatus equipped with mesh or flake plates with coaxial arrangement of scales. Such technical solution allows to exclude unidirectionality of steam and liquid flows over the whole area of the plate, and in case of separation of mixtures containing suspended particles (for example, in the distillation column of the distillation unit), to exclude the possibility of formation of stagnant zones, sticking of suspended particles on the surface of the plates and thus to increase their service life without stopping for preventive work.

For realization of the offered method and carrying out of researches of a plate of an experimental column have been equipped by the rotary segments equipped with microprocessor pneumatics of firm FESTO (connected to standard pneumocylinders of bilateral action of type DNT 63-50-PPV-A) which moved in turns according to the program of controller M340 of firm "Schneider Electric". Movable segments opened and closed the overflow openings of the plates in such a way that the liquid overflow occurred periodically from top to bottom in the height of the column (Fig. 1).

The column includes plates 1 and rotary segments of plates 2 associated with the drive mechanisms 3. On the plates 1 and segments 2 are placed flakes 4 so that the direction of the flakes arranged in one row is opposite to the direction of the scales located in adjacents rows. The mass exchange contact plate works as follows. Liquid enters on the sheet 1 of plate No. 1 through the overflow hole, which is formed after the opening of the rotary segment 2. During this period, the rotary segment of the web 1 of the plate No. 1 is closed for a specified period of time. From the bottom upwards, heating steam flows continuously through the flakes' slots and comes into contact with the liquid on the plate web. Mass exchange between liquid and steam takes place in barbotage mode. After the set time of liquid delay, the moving segment 2 of plate No. 1 is opened, and the liquid is poured to the bottom of the plate through the hole, which was formed, and spilled through all the slots of the flakes (Krivosheev & Anufriev, 2015).

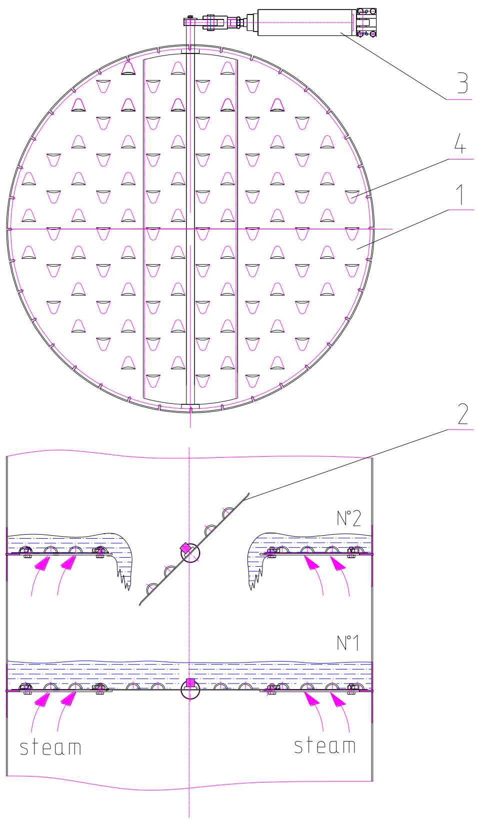

Figure 1. Flake plate of cyclic action:

1 - plate; 2 - rotary segment; 3 - drive mechanism (FESTO pneumatic cylinder); 4 – flakes

At the first stage of researches hydrodynamic modes of stable work of mesh and scaly plates in a mode of controllable cycles of a delay and overflow of a liquid have been established, maximum admissible values of speed of air in barbotage apertures and in a free section of a column at which the liquid is kept on plates and at which its carrying away (ejection) on the top plates begins are defined. It is established that the lower critical air velocity in the barbotage openings of meshes plates *(V_{hole})* was 5,4 m/s; the linear air velocity in the free section of the column *(V_{lin})* – 0,25 m/s. For scales plates: *(V_{hole})* is equal to 6,5 m/s; in barbotage mode of plate *(V_{lin})* was 0,5-0,9 m/s, in transition – 0,9-1,3 m/s and in jet – 1,3-2,0 m/s.

At the second stage of the studies, the air velocity in the barbotage holes *(V_{drift})* was determined, at which the entrainment of liquid onto the upper plates began. In the columned apparatus with meshes plates *(V_{drift})* was 8 m/s; while *(V_{lin})* was equal to 0,7 m/s; the relative value of fluid entrainment (e) did not exceed 0,01 kg per 1 kg of air. In a column with scales plates *(V_{lin})* was equal to 1,3-1,5 m/s; in mode of barbotage not more than 0,1 kg/kg, in jet mode – 0,2 kg/kg. Intense fluid overflow was observed at steam speeds below critical. For perforated plates the steam speed should not exceed 1,5-1 m/s. The obtained experimental data can be used in the development of mass transfer apparatus with perforated plates operating in a cyclic mode of rectification.

Research of efficiency of the offered technology was carried out in production conditions of Stonibabsky place of business of the State Enterprise Ukrspirt in processes of distillation of alcohol-containing fractions and epuration of distillate in a hydroselection mode. For research, an experimental cyclic column with perforated plates was mounted (Fig. 2).

Figure 2. Experimental column with controlled mass exchange cycles between liquid and vapour

The column had a diameter of 950 mm and was equipped with flake plates in the amount of 30 pieces. The distance between the plates was 300 mm. Rotating segments of the plates were connected to mechatronic subsystems, which were controlled by modern computer-integrated means.

During the periodic movement of the rotating segments alternately opened and closed the overflow holes of the plates according to the given algorithm in such a way that the overflow of liquid occurred cyclically along the height of the column from top to bottom with the continuous movement of steam from bottom to top. When the overflow opening was opened by the movable segment, the free area of the plate instantly changed from 2,6 to 51,7 %. As a result, the steam velocity in the barbotage openings became less than 1,5-1,0 m/s, and the liquid simultaneously overflowed through the overflow opening and all flakes. The technical solution made it possible to intensify the overflow process, reduce the overflow time to 1-1,7 s and increase the throughput capacity of the column apparatus.

The head fraction of ethyl alcohol and the alcohol-containing fractions from the condensers of the distillation column, carbon dioxide separator,sivush alcohol, sivush rinse water and streams from alcohol traps were fed into the column. The total supply volume was 688,3 dm3/h or 250 dm^3/h in terms of anhydrous alcohol (a.a). The upper plate of the column continuously received hot softened water in the amount of 4050-4500 dm^3/h for hydroselection of impurities. The specified water consumption provided concentration of ethyl alcohol in a cube liquid within the limits of 3,5-3,8 % vol. Under such conditions, the rectification coefficient of all impurities of alcohol (including higher alcohols of sivush oil) exceeded one, and impurities were effectively released. The cube liquid released in the process of distillation from volatile impurities was supplied to the upper zone of the concentration part of the epyurating column for double hydroselection of head impurities and effective extraction together with the head fraction of isopropyl alcohol. Such a solution allowed to reduce the consumption of hot softened water for hydroselection of impurities to 2000 dm^3/h. The retention time of the liquid on the plates was 40-60 s, the time of its overflow was 1-1,7 s. The pressure in the cub part of the column was maintained within 1,15-1,20 meters of the water column, the temperature was 100,5-101 °C, the temperature above the upper plate was 93,5-94 °C. Aldehyde-methanol concentrate was taken from the upper stage condenser in the amount of 7-9 dm^3/h. Esters and higher alcohols of sivush oil were taken out of the unit from the upper decanting zone in the form of ester-sivushnogo concentrate in the amount of 2-3 dm^3/h after the separation of the water-alcohol mixture. Aldehyde-methanol and sivush ester-aldehyde concentrates were mixed in the admixture concentrate collector.

In the course of the research, samples of feed *(F),* cube fluid *(CL),* impuritys concentrate *(IC),* epyurate *(E),* head fraction *(HF)* and rectified ethyl alcohol *(RA)* were taken. The results of chromatographic analysis of the research samples and calculated values of the extraction degree (α) and multiplicity of concentration (β) volatile alcohol impurities at the operation of the perforated plates in the selected hydrodynamic mode, and the rectificational column at the above technological parameters are given in Table 1.

The results of the chromatographic analysis of the test samples and the calculated values of the degree of extraction (α) and the multiplicity of concentration (β) of volatile impurities of alcohol

Table 1

A group of impurities	Concentration, mg/dm^3 in terms of a.a.						α	β
	F	CL	IC	E	HF	RA		
Ethanol, % об.	30,5	3,7	67	30,1	92,5	96,5	8,2	2,2
Aldehydes	318,6	2,75	2302	0,29	1135	0,18	115,9	7,2
Esters	40,5	—	448615	—	2395	—	∞	11077
Methanol, %	0,18	0,0003	2,69	0,0023	0,49	0,0003	600	14,9
Sivush oil	105882	726,9	726463	1179,8	3113	0,88	145,7	6,9

The analysis of the received results has shown that at prolongation of time of stay of a liquid on flakes plates up to 40 s esters and isopropyl alcohol are completely removed. It is known that by the content of isopropanol the quality and power of distillation units are estimated. The degree of extraction of higher alcohols of sivush oil and methanol increased by 38%, the multiplicity of concentration of head impurities increased by 25 %, of upper intermediate impurities - by 40 %, of end impurities - by 37 % in comparison with a typical installation operating in stationary mode. It is established that in the cyclic mode of rectification the specific consumption of heating steam in the process of distillation of alcohol-containing fractions decreased from 25 to 15 kg/dl a.a., introduced on the feeding plate, and in the process of epyuration of distillate - from 15 to 8,2 kg/dl a.a. in comparison with column apparatuses operating in stationary mode. After inclusion of an experimental rectificational column in the scheme of distillation unit the output of distilled ethyl alcohol increased by 3,8 %. According to all indicators, the ethyl distilled alcohol obtained met the requirements for the variety “Lux”.

Experimental rectificational column of cyclic action works on Storonibabsky plant of the State Enterprise Ukrspirt in a given hydrodynamic mode and set technological parameters to the present time.

The expected payback period of the rectificational column with controlled mass exchange cycles between liquid and vapour does not exceed 5 months.

Conclusion

1. The authors proposed the technology of cyclic rectification in column apparatuses equipped with barbotage perforated plates with variable free cross-section. Experimental data of the maximum permissible steam velocity in the free section of the column and in barbotage openings of meshe and flakes plates during the period of mass exchange and liquid overflow are obtained.

2. It is established that the increase of the time of liquid stay on the plates up to 40-60 s in the chosen hydrodynamic mode allows to increase the degree of extraction and the multiplicity of concentration of head, intermediate and end impurities by 25-40 %, and the instantaneous change of the steam speed in the barbotage holes below the critical one allows to reduce the liquid overflow time to 1-1,7 s and thus to increase the throughput of the column apparatus by 34 %.

3. Due to the increased phase contact time, the specific heating steam consumption in the processes of distillation of alcoholic fractions and epuration of

fermented distillate is reduced by at least 36 % in comparison with column units operating in stationary mode.

4. The expected payback period of the proposed energy-saving mass transfer column apparatuses of cyclic action does not exceed 5 months.

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ЕНЕРГОЗБЕІГАЮЧА ТЕХНОЛОГІЯ РЕКТИФІКАЦІЇ З КОНТРОЛЬОВАНИМИ ЦИКЛАМИ МАСООБМІНУ МІЖ РІДИНОЮ І ПАРОЮ

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Анотація. *Запропонована енергозберігаюча технологія циклічної ректифікації за безперервної подачі гріючої пари та рідини в масообмінний колонний апарат, оснащений провальними тарілками. Інноваційний спосіб дозволяє подовжити час контакту пари і рідини на кожній тарілці до 40-60 с та скоротити час її переливу до 1,5-1,7 с. Для реалізації технології запропонована конструкція ректифікаційної колони, оснащеної лускоподібними тарілками із змінним вільним перерізом.*

Визначені гідродинамічні режими роботи тарілок: нижня критична швидкість пари в отворах сітчастих тарілок становить 5,4 м/с, лускоподібних — 6,5 м/с; верхня критична швидкість — 8 та 16 м/с відповідно. Для здійснення почергових циклів та інтенсифікації переливу рідини необхідною умовою є миттєве зменшення швидкості пари в отворах або щілинах до 1,5-1 м/с завдяки миттєвій зміні вільного перерізу тарілки від 2,6 до 51,7 % і більше.

Дослідження проводились у виробничих умовах в процесах розгонки спиртовмісних фракцій і епюрації бражного дистиляту. Спосіб дозволяє

забезпечити повне видалення естерів, метилацетату та ізопропілового спирту, збільшити ступінь видалення вищих спиртів сивушного масла і метанолу на 38 %, підвищити кратність концентрування головних домішок на 25 %, верхніх проміжних домішок — на 38 %, кінцевих — на 37 %, а також підвищити пропускну здатність колони по рідині на 34 %. Відсутність зливних і приймальних пристроїв на полотнах тарілок дозволяє збільшити площу контакту пари і рідини на 15-20 %. Коаксіальне розташування лусок на полотні тарілки дає можливість виключити односпрямованість потоків пари і рідини та підвищити ефективність масообміну між рідиною і парою.

За таких умов питомі витрати гріючої пари зменшуються на 40 % в порівнянні з типовими установками: в розгінній колоні від 25 до 15 кг/дал безводного спирту, введеного на тарілку живлення, а в епюраційній — від 15 до 8,2 кг/дал безводного спирту.

Ключові слова: *масообмін, тарілки, ректифікація, цикли, колонний апарат пара, домішки спирту.*