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DESIGN OF A RECTIFICATION PLANT FOR THE COMPLEX SEPARATION OF PROPANE-BUTANE MIXTURES

Iurii M. Symonenko
iu.symonenko@gmail.com
ORCID: 0000-0002-7827-0591

Bohdan H. Hrudka
bogdangennadievich@gmail.com
ORCID: 0000-0003-1200-5442

Eduard V. Matvieiev
kriogen.magistr@gmail.com
ORCID: 0000-0003-0749-689X

Odesa National University of Technology,
112, Kanatna Str., Odesa,
65039, Ukraine

This paper is devoted to the issues of designing and experimental research of a pilot-industrial rectification plant intended for the complex separation of propane-butane mixtures obtained during the preparation of natural gas and its liquefaction. The topic is considered relevant due to the urgent need to use environmentally friendly natural refrigerants, such as propane (R290) and isobutane (R600a), which are becoming increasingly popular in refrigeration technology. The authors emphasize the importance of deep processing of hydrocarbon raw materials. Thus, multi-stage processing allows to significantly increase the added value of the final product - propane and high-purity butane. A theoretical justification for the choice of the technological scheme is provided in the paper. During preliminary calculations, it was established that due to the significant difference in the thermophysical properties of binary systems (propane-isobutane and propane-propylene), the use of one rectification column is ineffective. The designed plant includes two columns with different geometric parameters, filled with an irregular mesh packing, which ensures effective heat and mass transfer. Particular attention is paid to an innovative approach to the heat and cold supply system organizing. The developed system integrates two vapor-compression refrigeration units directly into the technological cycle of the columns. The evaporators of the refrigeration machines, built into the column condensers, allow to create a flow of reflux necessary for rectification, and the condensers of the machines supply heat to the column cubes. This approach allows to maintain stable pressure in the devices and flexibly regulate temperature levels. Experimental plant testing during the processing of "dirty" propane confirmed the design expectations: 99.9% purity of the target product was achieved. The technical possibility of further propane purifying to 99.96% by means of repeated distillation in the second column was shown. The plant has high flexibility, which enables waste-free processing of the initial feedstock with the release of butane, isobutane and propylene as independent commercial products by changing the operating temperature modes. The obtained results demonstrate the high efficiency of the proposed technical solutions for production facilities.

***Keywords**: rectification, propane-butane mixture, natural gas, natural refrigerants, packed column, heating and cooling supply.*

Introduction

The modern development of global refrigeration and air conditioning technology is at the stage of global transformation, driven by the strict requirements of international environmental agreements. Thus, according to the provisions of such documents as the Montreal Protocol, the Kigali Amendment and European regulations on fluorinated greenhouse gases (F-gases) [1], it is necessary to almost completely phase out not only ozone-depleting substances, but also hydrofluorocarbons (HFCs), which have a high global warming potential (GWP). In these conditions, a return to natural refrigerants is of particular relevance, among which propane (R290) and isobutane (R600a) occupy leading positions. They have zero ozone depletion potential (ODP=0) and negligible global warming potential (GWP<3), and also demonstrate excellent thermodynamic and thermophysical properties [2], which often exceed the indicators of synthetic analogues. The source for the industrial production of such refrigerants is a wide fraction of light hydrocarbons, in particular the C_{2+} fraction [3], which is released during the preparation of natural gas (NG) for main transportation or immediately before its liquefaction (LNG production). The composition of the initial propane-butane mixtures, in addition to the main components, usually includes a wide range of other associated hydrocarbons: ethane (R170), ethylene (R1150), propylene (R1270) and normal butane (R600) [4]. Each of these compounds is a high-value raw material for the petrochemical industry or a promising component for low-temperature working fluids. The current state of development of the gas processing industry requires a transition from simple combustion or the use of propane-butane mixtures as low-quality fuel (LPG) [5]. Economic analysis of deep processing of hydrocarbons clearly indicates that sequential multi-stage separation allows to dramatically increase the added value of the final product. Passing 4–5 stages of separation and purification of raw materials increases the market value of the obtained high-purity components by 8–10 times compared to the initial gas [6].

For clarity, let's consider the classic technological chain: natural gas → ethane → ethylene → polyethylene → finished plastic products. The market value of pure olefins (ethylene and propylene) is approximately three times higher than the cost of a mixture of propane and butane. Processing them into polymers (polyethylene, polypropylene) increases the cost by 7–8 times, and the output of finished products – by 12 times. Obtaining high-purity propane fractions and isobutane-propane separated products (over 99.5–99.9%) opens direct access to the high-tech refrigerant market. However, the main scientific and technical problem on the way to obtaining individual hydrocarbons of propane purity is the complexity of their separation. Since propane-butane fractions are multicomponent non-azeotropic mixtures with close boiling points of individual components (for example, propane and propylene), their clear separation by classical methods of single distillation is impossible. It is necessary to use rectification mass transfer devices. Traditional high-power rectification plants used in oil refineries have huge dimensions, significant metal content and colossal energy consumption. However, for small-tonnage production, local gas distribution stations or experimental and industrial facilities, flexible, compact and highly efficient units are required. The use of packed columns (in particular, with irregular mesh packings) allows to significantly intensify heat and mass transfer processes and reduce the dimensions of the equipment [7].

A separate critical challenge in the design of such small-sized units is the organization of thermodynamically efficient heat supply and removal. Rectification requires constant heating of the column cube to evaporate the mixture and simultaneous cooling of the dephlegmator (condenser) to create a reverse flow of liquid (reflux). The creation of autonomous and energy-efficient heat and cold supply systems that integrate refrigeration cycles directly into the rectification process circuit is an advanced research direction that allows to utilize condensation heat and minimize external energy costs.

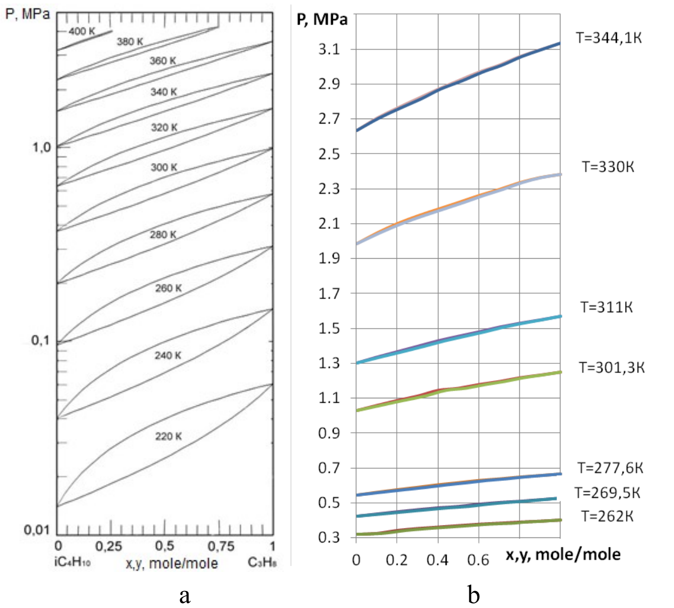
The paper is devoted to solving the specified scientific and technical tasks, namely the design, optimization and experimental study of a pilot-industrial rectification plant for the complex separation of the propane-butane fraction of natural gas into components. The main goal is to develop a highly efficient technological scheme based on two packed columns and an integrated vapor-compression refrigeration machine capable of providing stable production of promising environmentally safe working fluids for refrigeration equipment with a high degree of purity.

Design of a rectification plant

Preliminary calculations of packed columns [6] showed that for the separation of the propane-butane fraction with the removal of propane, due to the large difference in the thermophysical properties of the binary propane-isobutane and propane-propylene mixtures (Fig. 1), it is impossible to use a single column. At least two columns with different diameter-height ratios for the separation zone are required.

Table 1 shows the main geometric characteristics of the packed columns adopted for manufacture. Both columns are filled with a mesh saddle-shaped packing 10×10×0.2 mm. The overall dimensions of the columns were limited by the funds allocated for their manufacture.

A simplified diagram of the rectification plant (a) and a design of the control panel (b) are shown on Fig. 2. The first column is designed to separate the heavy fraction from propane – isobutane, butane and heavier hydrocarbons that are part



a b

Fig. 1. Equilibrium isobars of the propane-isobutane (a) [8] and propylene-propane (b) systems [9]

of the fraction in micro concentrations. The gaseous fraction (propane purified from butanes) enters the second column for the separation of propylene, ethylene, ethane and lighter components of the propane-butane mixture.

To ensure the operation of the column, heat is constantly supplied to the cube, and an equivalent amount of heat is removed from the upper part (column condenser), for example, using an external refrigerant. Between the condenser and the cube, there is a working part of the packed column, where heat and mass exchange takes place, filled with an irregular packing. The diameter and height of the working part are calculated values, determined by the type of separated mixture, balance equations and the type of mass exchange surface.

In the column condenser, a liquid that flows down the packing is formed. The gas fraction contains practically no high-boiling impurities. The liquid entering the cube contains almost no propane. It evaporates when heat is applied. Similar processes occur in the second column.

Heat Q_{X1} and Q_{X2} removal from the column dephlegmators is carried out at temperatures 5–7 degrees lower than the condensation temperature of the main low-boiling component at operating pressure (Fig. 3).

At the first stage, when obtaining propane, cascade refrigeration cycles on ammonia, propane or propylene in the lower cascade can be used as a source of cold [10]. When purifying from propylene – on propylene, ethane or ethylene. A promising method of cooling at this stage may be machine-free devices for transferring cold from a lower temperature level (for example, from liquid nitrogen) through an intermediary substance (thermosiphon [11]).

However, the search for equipment with specified characteristics showed that it is possible to provide the necessary cooling parameters (Table 2) by using only a serial refrigeration unit.

Table 1. Geometric characteristics of rectification columns (mm) of the pilot plan

Device name	Dimensions of the packing part		Dimensions of heat exchangers		
			Height		Diameter
	Height, *h*	Diameter, *d*	$L_Д$	L_K	$D_D=D_K$
Column K1	4000	200	1300	1800	400
Column K2	6000	250	1300	1800	400

D – column condenser (dephlegmator); K – column cube

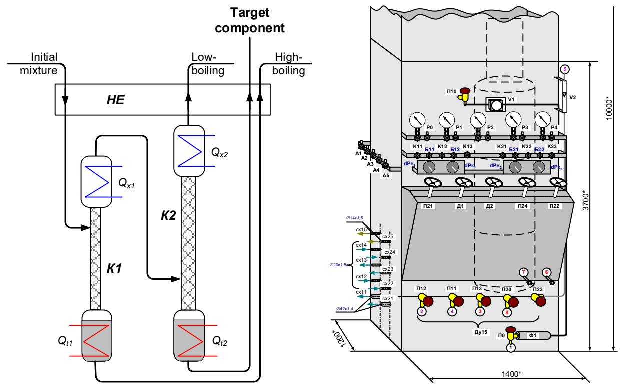


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Fig. 2. Simplified scheme (a) of obtaining propane from a mixture of C_{2+} hydrocarbons and the control panel of the plant (b), located in the lower part of the cold unit:
K1, K2 – packed rectification columns; HE – recuperative heat exchanger

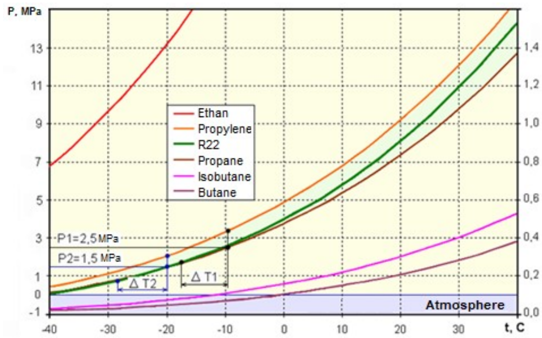

Fig. 3. Selection of refrigerant for the column cooling system [2]:
ΔT_1 and ΔT_2 – temperature difference between the condensation temperature of propylene in the upper part of the column K2 and the refrigerant

Table 2. Operating parameters of the refrigeration machine in different operating modes of the plant

No.	Technological process	Column	
		First	Second
1. Propane obtaining			
	1.1. Temperature level,°C	-30 ... -35	-35 ... -40
	1.2. Cooling capacity, kW	15	30...45
2. Propylene purification			
	2.1. Temperature level,°C	-38 ... -45	-38 ... -45
	2.2. Cooling capacity, kW	15	30...45
3. Isobutane obtaining			
	3.1. Temperature level,°C	-5	-10
	3.2. Cooling capacity, kW	10…15	25…30

The heat and cold supply system (HCSS) of rectification units is developed on the basis of a vapor compression refrigeration machine (VCRM) with a regenerative heat exchanger (RHE, Fig. 4, 5 [10]), built into the rectification plant.

The working substance is compressed in the compressor (process 1–2) and enters the condenser of the refrigeration machine, where it is liquefied (process 2–3). To increase the throttling effect, heat exchange between the direct and reverse flows in the RHE is used (process 3–4). After throttling (process 4–5), the working substance enters the VCRM evaporator located in the column condenser (process 5–6). The gas heated in the RHE (process 6–1) is supplied to the column compressor for compression. To cool the column condensers, an evaporator of the corresponding refrigeration machine is built into each of them. Due to this, part of the substance condenses and a flow of reflux that irrigates the packings is created. To evaporate the reflux in the cube zone, refrigeration machine condensers built into the column cubes are used. The heat of the working substance condensation is supplied to the product.

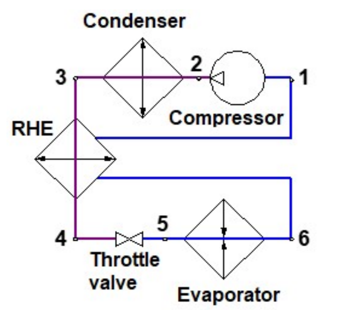

Fig. 4. Traditional refrigeration system diagram

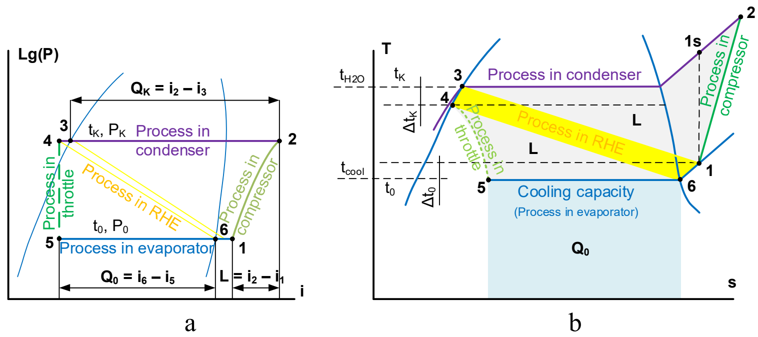

Fig. 5. Refrigeration cycle in lgP-i (a) and T-s (b) diagrams

The cooling capacity $Q_0 = i_6 - i_5$ corresponds to the given heat flow required to cool the column condenser Q_X. The heat flow from the VCRM condenser $Q_K = i_2 - i_3$ is supplied to the column cube.

From the energy balance of the refrigeration cycle it follows that

$$i_2 - i_3 = (i_6 - i_5) + (i_2 - i_1)$$

or

$$Q_K = Q_0 + L\,, \qquad)$$

where L is a compressor operation.

The formulas show that more heat is removed in the refrigeration cycle than can be obtained in the cold in the VCRM evaporator. If this balance does not correspond to the set one required for the rectification process, excess heat may form in the column. As a result, the reflux will gradually evaporate, and the pressure in the rectification apparatus will increase. To achieve stability in the column, part of the heat of the refrigeration cycle should be removed to the external water condenser, which is part of any refrigeration unit.

As a result of the conducted research, a serial compressor-receiver unit on R-22 AKR-50 was adopted to ensure the rectification process [12].

As was shown above, the waste fractions obtained in the process of propane extraction contain other valuable components. The main ones for the heavy fraction are butane and isobutane, for the light fraction - propylene. For their processing, it is necessary to change the temperature level of cooling of the column condensers and supplying heat to the cubes (see Table 2).

Results of experimental studies

The results of propane purification with a purity of about 90% in a pilot plant are given in Table 3, and Table 4 shows the parameters of the columns and HCSS.

A sample certificate for a multicomponent mixture used to obtain propane is shown on Fig. 6, a. The plant's productivity for the initial mixture at the first stage corresponded to the design one – 30 nm^3/h, the purity of the product propane – 99.9%.

To obtain a product of 99.96% (Fig. 6, b), double distillation (further purification from propylene) is required in the second column.

Table 3. Results of "dirty" propane processing

Composition of the model mixture:	Product:
Methane – 0.31% Ethane – 0.49% Propane – 89.6% i-Butane – 4.2% n-Butane – 5.4%	Propane of 99.9% purity Impurities: ethane 351.18 ppm propylene 325.18 ppm isobutane 323 ppm
Propane productivity – 1200 kg per day (50 kg/h=24.8 nm^3/h)	

Table 4. Column operating parameters supported during the purification process

Parameter	Column K1	Column K2
Column pressure (head) kg/cm^2	2.62	2.57
Pressure of refrigerant R22 at the inlet to the VCRM evaporator (p.5, fig.4) kg/cm^2	1.88	0.55
Cooling temperature of K (°C)	256 (-17)	243 (-30)

Паспорт якості

Газ вуглеводневий скраплений паливний

(по ДСТУ 4047-2001)

№ з/п	Найменування показника	Норма показника	Фактичні результати аналізу газу
1	Масова доля компонентів, %		
	– сума метану, етану і етилену	не нормується	2,24
	– сума пропану і пропілену	не нормується	66,80
	– сума бутанів і бутиленів	не більше 60	30,93
2	Об'ємна доля рідкого залишку при 20 °С, %	не більше 1,6	0,4
3	Тиск насичених парів, надлишковий, МПа, при температурі +45 °С	не більше 1,6	1,31
4	Масова доля сірководню та меркаптанової сірки, %, в тому числі сірководню	не більше 0,013 не більше 0,003	0,0004 –
5	Вміст вільної води і лугів	Відсутність	відсут.
6	Інтенсивність запаху, бали	не менше 3	4
7	Густина, при 20 °С, кг/м³	не нормується	517

Газ вуглеводневий скраплений відповідає ДСТУ 4047-2001

Зав. хім. лабораторії

Дослідження проводив лаборант

Дата

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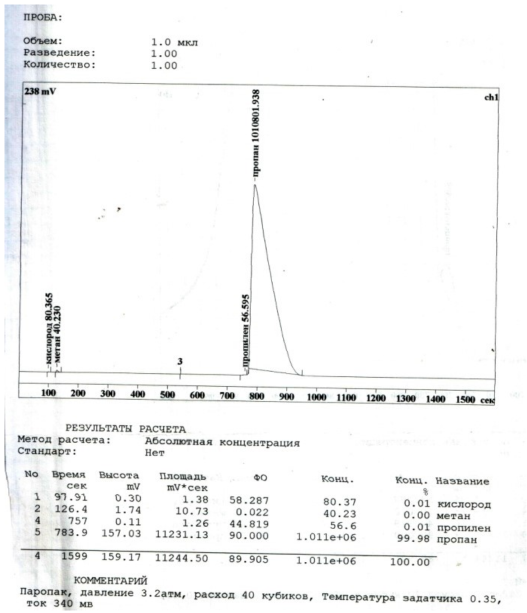

b

Fig. 6. The initial product (a) and chromatogram of propane obtained during processing in a pilot-scale rectification plant

Conclusions

Propane-butane mixtures, as a rule, also include other hydrocarbons that can be used as natural working fluids of refrigeration machines. Since binary mixtures formed by the components of the mixture are non-azeotropic, the extraction of each individual component is possible by phase separation methods. For given conditions (low consumption), it is rational to use two columns filled with an irregular packing.

The system of supplying heat to the cube of the columns and removing heat from the condensers is organized on the basis of two vapor-compressor refrigeration machines built into the technological cycle.

Testing of the pilot plant during the supply of the initial propane-butane mixture with a given productivity showed that as a result of processing it is possible to obtain propane with a purity of 99.9%. The purity of propane can be increased by repeated distillation in the second column.

The developed plant allows for waste-free processing of raw materials to obtain butane, isobutane and propylene. To do this, it is needed to change the temperature level of cooling the column condensers and supplying heat to the cubes.

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Проєктування ректифікаційної установки для комплексного розділення пропан-бутанових сумішей

Ю. М. Симоненко, Б. Г. Грудка, Е. В. Матвєєв

Одеський національний технологічний університет,
65039, Україна, м. Одеса, вул. Канатна, 112

Дана робота присвячена питанням проєктування й експериментального дослідження дослідно-промислової ректифікаційної установки, призначеної для комплексного розділення пропан-бутанових сумішей, що одержуються при підготовці природного газу та його зрідження. Актуальність теми зумовлена гострою необхідністю використання екологічно безпечних натуральних холодоагентах, таких, як пропан (R290) та ізобутан (R600a), які стають все більш затребуваними в холодильній техніці. Автори наголошують на значущості глибокої переробки вуглеводневої сировини. Так, багатоступенева переробка дозволяє суттєво підвищити додану вартість кінцевої продукції – пропану й бутану високої чистоти. У статті надано теоретичне обґрунтування вибору технологічної схеми. У ході попередніх розрахунків встановлено, що через значну різницю в теплофізичних властивостях бінарних систем (пропан-ізобутан і пропан-пропілен) застосування однієї ректифікаційної колони неефективне. Проєктована установка включає дві колони з різними геометричними параметрами, заповнені нерегулярною сітчастою насадкою, що забезпечує ефективний тепломасообмін. Особливу увагу приділено інноваційному підходу до організації системи теплохолодопостачання. Розроблена система інтегрує два парокомпресійні холодильні агрегати безпосередньо в технологічний цикл колон. Випарники холодильних машин, вбудовані в конденсатори колон, дозволяють створювати потік флегми, необхідної для ректифікації, а конденсатори машин підводять тепло до кубів колон. Даний підхід дозволяє підтримувати стабільний тиск в апаратах та гнучко регулювати температурні рівні. Експериментальна перевірка установки під час переробки «брудного» пропану підтвердила проєктні очікування: досягнуто 99,9% чистоти цільового продукту. Показано технічну можливість доочищення пропану до 99,96% за допомогою повторної перегонки у другій колоні. Установка має високу гнучкість, що уможливлює безвідходну переробку вихідної сировини з виділенням бутану, ізобутану й пропілену як самостійних *товарних продуктів шляхом зміни робочих температурних режимів. Отримані результати демонструють високу ефективність запропонованих технічних рішень для виробничих потужностей.*

***Ключові слова**: ректифікація, пропан-бутанова суміш, природний газ, натуральні холодоагенти, насадкова колона, теплохолодопостачання.*

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