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RESEARCH ARTICLE

Contact crystallization of substances from solutions using evaporating refrigerants

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Objectives. *The aim of this study was to analyze the possibility of using contact crystallization with evaporating refrigerants for the isolation of substances from their aqueous solutions using salts [KNO_3, NaI, and $(NH_2)_2CO$] as extraction examples and sucrose. Isobutane was used as a refrigerant.*
Methods. *The analysis of the influence of the main technological parameters (i.e., solution's cooling temperature, initial concentration, and compressed refrigerant vapor pressure) on the separation process and identification of its regularities was performed using mathematical dependencies previously developed by N.I. Gelperin and G.A. Nosov for each stage of the contact crystallization process. The authors studied the influence of these parameters on the yield of crystalline and liquid phases, refrigerant consumption, and compressor power.*
Results. *The study showed that the use of evaporating refrigerants can significantly intensify the process of separating the mixture and spent refrigerant from the resulting crystalline suspension. This occurs owing to the evaporation of the liquid refrigerant that is in contact with the solution, which is accompanied by intense cooling. This process can be carried out at the temperature difference between the refrigerant and crystallizing mixture in the range of 0.5–1.0°C.*
Conclusions. *Contact crystallization with evaporating refrigerants can be successfully applied to separate various substances from aqueous solutions. An important advantage of this process is the relatively low refrigerant consumption because heat removal from the solution is carried out as a result of changes in the aggregate state of the refrigerant. The use of contact crystallization can also considerably simplify the equipment.*

Keywords: *crystallization, contact cooling, evaporating refrigerants, aqueous solutions*

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НАУЧНАЯ СТАТЬЯ

Контактная кристаллизация веществ из растворов с применением испаряющегося хладагента

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Цели. *Статья анализирует возможность применения контактной кристаллизации с использованием испаряющихся хладагентов для выделения веществ из их водных растворов на примере извлечения некоторых солей (KNO_3, NaI, $(NH_2)_2CO$) и сахарозы. В качестве хладагента использован изобутан.*

Методы. *Изучение влияния основных технологических параметров – температуры охлаждения раствора, его исходной концентрации и давления сжатых паров хладагента – на ход рассматриваемого процесса разделения, а также выявление закономерностей его протекания проводилось с помощью выведенных ранее Н.И. Гельпериным и Г.А. Носовым математических зависимостей для каждой стадии процесса контактной кристаллизации. Авторы исследовали влияние указанных параметров на выход кристаллической и жидкой фаз, расход хладагента и мощность компрессора.*

Результаты. *Установлено, что применение испаряющихся хладагентов позволяет существенно интенсифицировать процесс кристаллизации и облегчает отделение отработанного хладагента от образующейся кристаллической суспензии. Это обусловлено тем, что при контакте жидкого хладагента с раствором происходит его испарение, которое сопровождается интенсивным охлаждением раствора. Установлено, что такой процесс может осуществляться при разности температур хладагента и кристаллизующейся смеси порядка 0.5–1.0 °С.*

Выводы. *Контактная кристаллизация с использованием испаряющихся хладагентов может быть успешно применена для выделения различных веществ из водных растворов. Важным преимуществом проведения подобного процесса является относительно небольшой расход хладагента, поскольку отвод теплоты из раствора осуществляется в результате изменения агрегатного состояния хладагента. Использование контактной кристаллизации позволяет также значительно упростить аппаратурное оформление процесса.*

Ключевые слова: *кристаллизация, контактное охлаждение, испаряющиеся хладагенты, водные растворы*

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INTRODUCTION

It is known that crystallization process is widely used to isolate substances from various solutions, purify substances from impurities by their recrystallization, and concentrate dilute solutions by freezing solvents [1–4]. The application of this process is not limited to the chemical industry. Crystallization is also used with great success in the food, pharmaceutical, petrochemical, and construction industry, and also in the production of radio electronics [5–13]. In most cases, this process is performed by cooling the solutions and melts. Evaporation, vacuum evaporation crystallization, and crystallization with salting-out agents are less commonly used.

In the case of crystallization by cooling, devices equipped with various cooling elements (e.g., jackets, coils, tubes, and hollow disks) are usually used. Heat removal from the crystallizing substance occurs through the heat transfer walls of the above-mentioned devices [14]. Crystallization process is sometimes carried out by the direct contact of solutions with refrigerants [2, 4, 15, 16].

One of the significant advantages of the latter process is the absence of heat transfer surfaces to remove heat flux. This greatly simplifies the design of the apparatus and also removes the issue of encrustation. The possibility of achieving a more extended interphase surface, in contrast to the conventional contactless method of fractional crystallization, should be also attributed to the advantages of this process. Mixing of mass flows in a contact crystallizer is usually quite intensive. Carrying out the crystallization in this manner allows the process to occur at a relatively low-temperature difference between the incoming and crystallizing mixture refrigerant (approximately 0.5–2.0°C). This makes it possible not to be limited to expensive steel when choosing structural materials for crystallizers and to manufacture such devices from materials with low thermal conductivity (e.g., polymers, glass, and ceramics). In general, all of these advantages lower capital expenditures and operating costs.

When choosing a contact crystallization method, one should take into account the possibility of contamination of the target product with a refrigerant, which is a significant disadvantage of this process [2, 4]. Considering this, the stage of separating the spent refrigerant from the crystalline phase is necessary for this process.

It should be noted that various types of refrigerants can be used in the process of contact crystallization, differing from each other in thermophysical properties and state of aggregation. Chilled liquids, liquefied and nonliquefied gases can be used. The refrigerants entering the crystallizer upon contact with the crystallizing mixture pass into a gaseous state (evaporate) [2]. Contact crystallization can also be carried out by mixing the initial mixture (solution) with a highly supercooled solvent [17].

The contact crystallization process can occur both in continuous and dispersed phases. [2, 4, 16]. In this case, liquid refrigerants that do not mix with the original solution are used. The dispersion of cooled solution in the form of drops in the mass flow of the refrigerant forms a dispersed phase. When the refrigerant is dispersed in the crystallizing solution, the phase becomes continuous. Depending on the task that contact crystallization process needs to solve, it can be implemented in a batch or continuous mode. When choosing the contact crystallization method, it should be taken into account that with a small difference in the densities of the refrigerant, mother liquor, and crystalline phase, difficulties often arise in separating the resulting suspension, which can lead to contamination of the separation products with a refrigerant.

The use of gaseous refrigerants greatly facilitates their separation from suspensions. However, currently, they are rarely used in the industry because of their high consumption due to their low heat capacity.

The prospect of the industrial application of evaporating refrigerants in crystallization processes continues to be relevant. On one hand, they can significantly intensify the crystallization process because a significant amount of heat is absorbed during their evaporation, and on the other hand, there are no problems in separating the resulting refrigerant vapor from the suspension. Currently, this crystallization process is used mainly for desalination of water by freezing and concentrating dilute aqueous solutions [2, 5, 16]. This article presents the results of studies on contact crystallization concerning the isolation of potassium nitrate, carbamide, sodium iodide, and sucrose from their aqueous solutions.

DESCRIPTION OF THE CRYSTALLIZATION PROCESS INSTALLATION

A schematic diagram of the contact crystallization unit is shown in Fig. 1. Here the crystallizer Cryst (crystallization stage) continuously receives the initial solution in the amount F with the concentration of the dissolved substance x_F at the temperature t_F, which gradually cools down to the temperature t_{fr} below the point of its saturation. As a result, a crystalline phase of the target product is formed in solution. Liquid refrigerant, which is supplied to the crystallizer in the amount of G_{R}, is in contact with the cooled solution. This leads to gradual evaporation of the refrigerant, followed by cooling of the solution in the crystallizer. The suspension formed in the crystallizer consists of the crystalline phase C of the composition x_C and the mother liquor M of the composition x_M. Further, the $C+M$ suspension is sent to Sep filtration (separation stage), where the crystalline phase is extracted in the amount of S from the mother liquor L. Of note, the suspension can be separated both by filtration and centrifugation [18].

In the process under consideration, a vapor compression refrigeration unit is used, in the circuit of which the G_{R} refrigerant circulates. In this case, crystallizer Cryst, which is continuously supplied with liquid refrigerant, is the evaporator of the refrigeration unit. The vapors of the G_{R} refrigerant, which are in a saturated state, are removed from the crystallizer and then they enter the Com compressor to compress them from pressure p_1 to pressure p_2. In this case, the temperature of the compressible vapors increases from Θ_1 to Θ_2, and the heat content

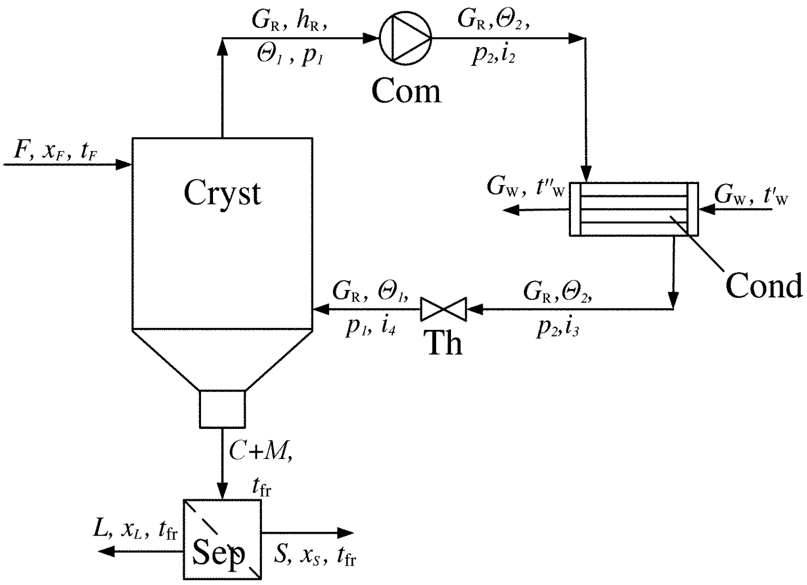


Fig. 1. Schematic diagram of contact crystallization with an evaporating refrigerant for the extraction of substances from aqueous solutions.

increases from i_1 to i_2. The change in the parameters of the refrigerant in the pressure–enthalpy diagram is shown in Fig. 2. In this diagram, the arrow *1–2* corresponds to the process of compressing the refrigerant by the compressor from pressure p_1 to p_2. Then, the compressed vapor of the G_R refrigerant enters the condenser of the Cond refrigeration unit, where it is cooled by the G_W water stream. In this case, the heat content of the refrigerant decreases from i_2 to i_3 (arrow *2–3*). A conventional shell and tube heat exchanger can be used as a condenser. Before supplying refrigerant to the crystallizer, it is necessary to reduce its pressure. Thus, it in condensed form passes through the throttle valve Th, which reduces the refrigerant pressure from p_2 to p_1. Then, the refrigerant returns to the crystallization stage Cryst in a liquid state. In the state diagram of the refrigerant, the arrow *3–4* shows the change in the parameters of the G_R flow when it is throttled. Of note, the throttling enthalpy remains constant $i_3 = i_4$ The arrow *4–1* corresponds to the evaporation of refrigerant at pressure p_1. In this case, the enthalpy of the refrigerant increases from i_4 to i_1.

CALCULATION METHODS

To calculate the yield of the crystalline and liquid phases when carrying out the considered separation process, the same dependencies can be used as in the usual crystallization process. Thus, the yield of the crystalline phase can be established using the dependence [2]

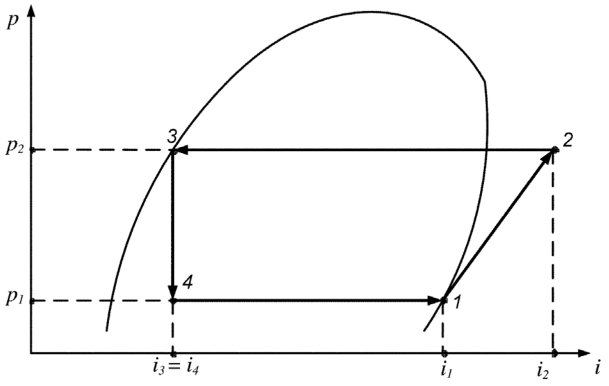


Fig. 2. Changes in the parameters of the refrigerant in the *p–i* state diagram during contact crystallization.

$$C = F\frac{x_F - x_M}{x_C - x_M} \qquad (1)$$

where x_C and x_M are the concentrations of solute in the crystalline phase *C* and mother liquor *M*.

The heat balance equation for the crystallization stage Cryst has the form

$$Fc_F t_F + Cr_{\text{sub}} + G_R i_4 = Cc_C t_{\text{fr}} + Mc_M t_{\text{fr}} + G_R i_1 \qquad (2)$$

where c_C and c_M are the heat capacities of the crystalline phase of substance *C* and mother liquor *M*; r_{sub} is the heat of crystallization of the substance; i_4 and i_1 are the enthalpies of the refrigerant at the inlet and outlet of the crystallizer.

Using equation (2), it is possible to determine the amount of removed heat Q_{cool} and the flow rate of the refrigerant G_{R} at the crystallization stage Cryst:

$$Q_{\mathrm{cool}} = G_{\mathrm{R}}(i_1 - i_2) = Fc_F t_F + C(r_{\mathrm{sub}} - c_C t_{\mathrm{fr}}) - Mc_M t_{\mathrm{fr}} \qquad (3)$$

$$G_{\mathrm{R}} = \frac{Q_{\mathrm{cool}}}{i_1 - i_4} = \frac{Fc_F t_F + C(r_{\mathrm{sub}} - c_C t_{\mathrm{fr}}) - Mc_M t_{\mathrm{fr}}}{i_1 - i_4} \qquad (4)$$

The amount of removed heat energy Q_{cond} from the condensing compressed heat carrier in the condenser Cond can be determined using the dependence

$$Q_{\mathrm{cond}} = G_{\mathrm{R}}(i_2 - i_3) \qquad (5)$$

where i_2 and i_3 are enthalpies of the refrigerant at the inlet and outlet of the condenser.

The power consumed by the turbocharger for the compression of refrigerant vapors from pressure p_1 to pressure p_2 is determined [19]

$$N_{\mathrm{real}} = \frac{G_{\mathrm{R}}(i_2 - i_1)}{\eta_{\mathrm{A}}\eta_{\mathrm{M}}} \qquad (6)$$

where η_{A} and η_{M} are the adiabatic and mechanical efficiency coefficients of the compressor.

RESULTS AND DISCUSSION

The considered crystallization process was analyzed using the separation of KNO_3, $(NH_2)_2CO$, NaI, and sucrose from their aqueous solutions as an example. All of these substances do not form crystalline hydrates during crystallization. In the diagrams of their equilibrium with water, there are eutectic points, the parameters of which are provided in the table.

The separation process of the abovementioned binary systems was performed using isobutane as a refrigerant. The vapor pressure of isobutane after throttling was $p_1 = 1$ atm, and their compression in the compressor was carried out in the range up to $p_2 = 2$–5 atm depending on the cooling temperature of the solutions in the crystallizer t_{fr}.

As a result of the analysis, it was determined that cooling the solution to lower temperatures t_{fr} at the constant concentration of the initial solution x_F led to a regular increase in the yield of the crystalline phase of the isolated substance and to a corresponding decrease in the yield of the mother liquor (Fig. 3).

Of note, a change in the yield of the crystalline phase, in turn, affects the amount of heat released. Thus, a decrease in the solution cooling temperature t_{fr}, and, consequently, an increase in the yield of crystals leads to the corresponding increase in the amount of released thermal energy Q_{cool} (Fig. 4a) and an increase in the consumption of G_{R} refrigerant for the crystallization process (Fig. 4b). The power of compressor N_{real}, which is used to compress the vapor of the intermediate heat carrier, also increases (Fig. 5a) as well as the amount of heat Q_{cond}, which is removed during the condensation of the compressed heat carrier vapor in the condenser of the installation (Fig. 5b).

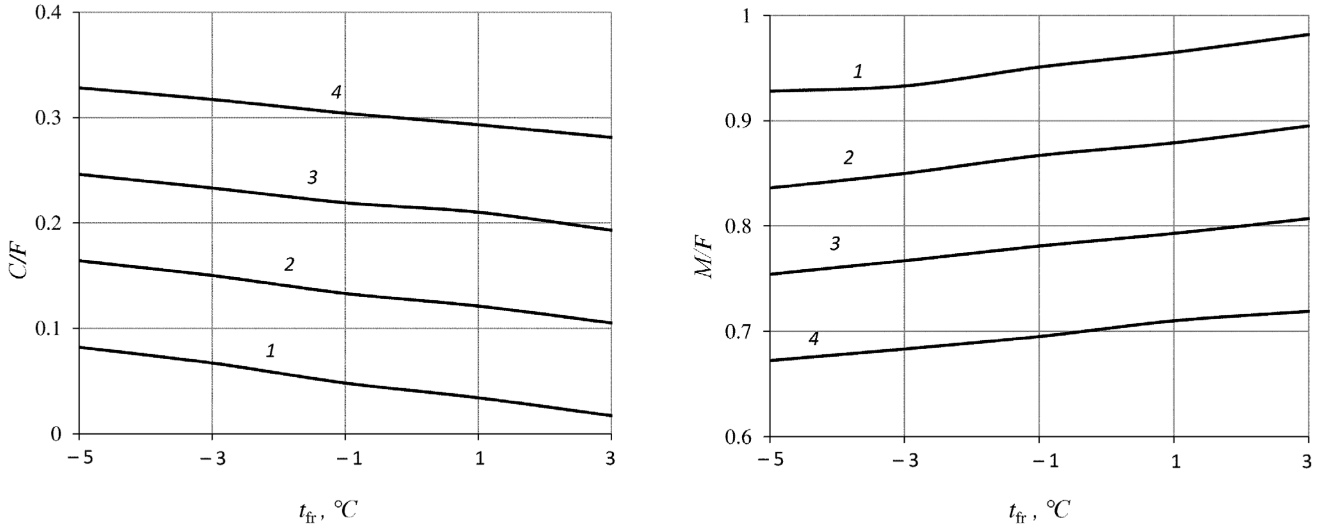
The efficiency of the considered separation process greatly depends on the concentration of the initial solution x_F. Its increase leads to a regular increase in the yield of the crystalline phase C (Fig. 3) and the amount of heat Q_{cool}, which is removed at the crystallization stage (Fig. 4). The flow rate of the refrigerant G_{R} and the compressor capacity N_{real} also increases (Fig. 5).

The analysis also showed that in addition to the fractionation temperature t_{fr} and concentration x_F, the refrigerant flow rate G_{R} and compressor power N_{real} are greatly affected by pressure p_2, to which the refrigerant vapor is compressed in the compressor. It is characteristic that during the separation of dilute solutions, the value of G_{R} slightly decreases with an increase in pressure p_2, and when fractionating concentrated solutions, it slightly increases (Fig. 6a); the compressor power always increases with an increase in p_2 (Fig. 6b).

Refrigerant consumption G_{R} and compressor power N_{real} can also depend on the physicochemical and thermophysical properties of crystallizing substances and their aqueous solutions [17]. Thus, for example, at the same yield of the crystalline phase for

Parameters of the eutectic point for some binary systems

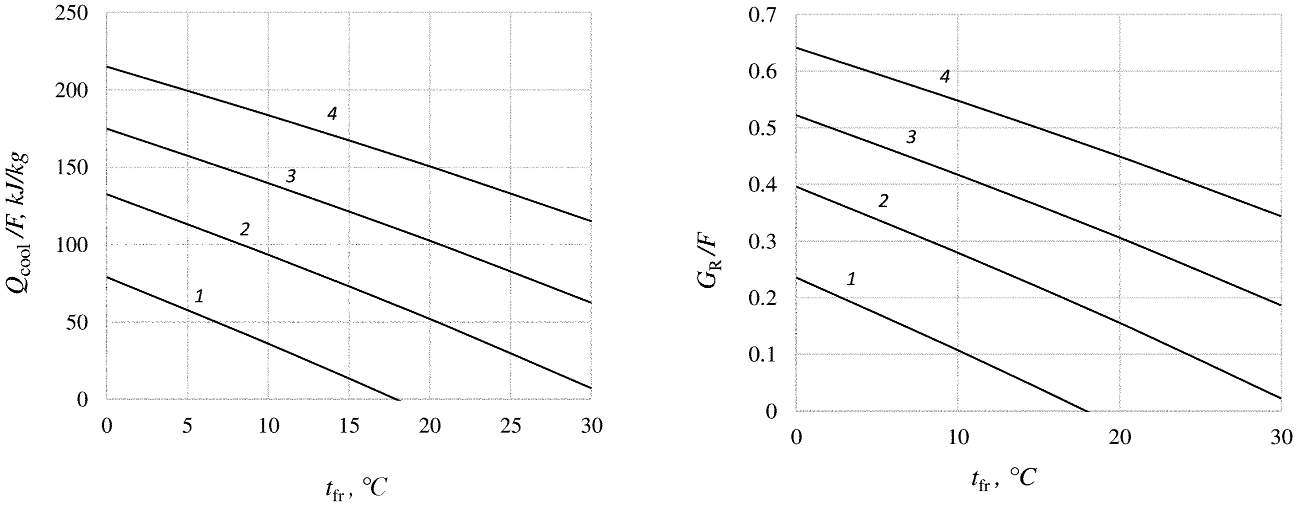
System	x_F, wt %	t_F, °C
KNO_3–H_2O	11	−2
$(NH_2)_2CO$–H_2O	32	−12
NaI–H_2O	47	−32
$C_{12}H_{22}O_{11}$–H_2O	63	−14



a

b

Fig. 3. Influence of the cooling temperature t_{fr} on the yield of the crystal phase (a) and mother solution (b) (system $(NH_2)_2CO–H_2O$; p_2 = 2 atm): (1) x_F = 40% $(NH_2)_2CO$; (2) x_F = 45%; (3) x_F = 50%; (4) x_F = 55%.



a

b

Fig. 4. Dependence of the amount of heat removed from the crystallizer (a) and the refrigerant flow rate (b) on the fractionation temperature ($KNO_3–H_2O$ system; p_2 = 2 atm): (1) x_F = 20% KNO_3; (2) x_F = 30%; (3) x_F = 40%; (4) x_F = 50%.

NaI, the energy consumption for performing contact crystallization is 1.5–2.0 times higher than that for the crystallization of sucrose.

CONCLUSIONS

The analysis of data from previous studies and our research showed that contact crystallization using evaporating refrigerants can be successfully applied to isolate various substances from their aqueous solutions. It is especially advantageous to use it when the crystallization process is carried out at low temperatures. When using it, interfacial heat transfer is considerably intensified, and the instrumentation of the separation process is simplified.

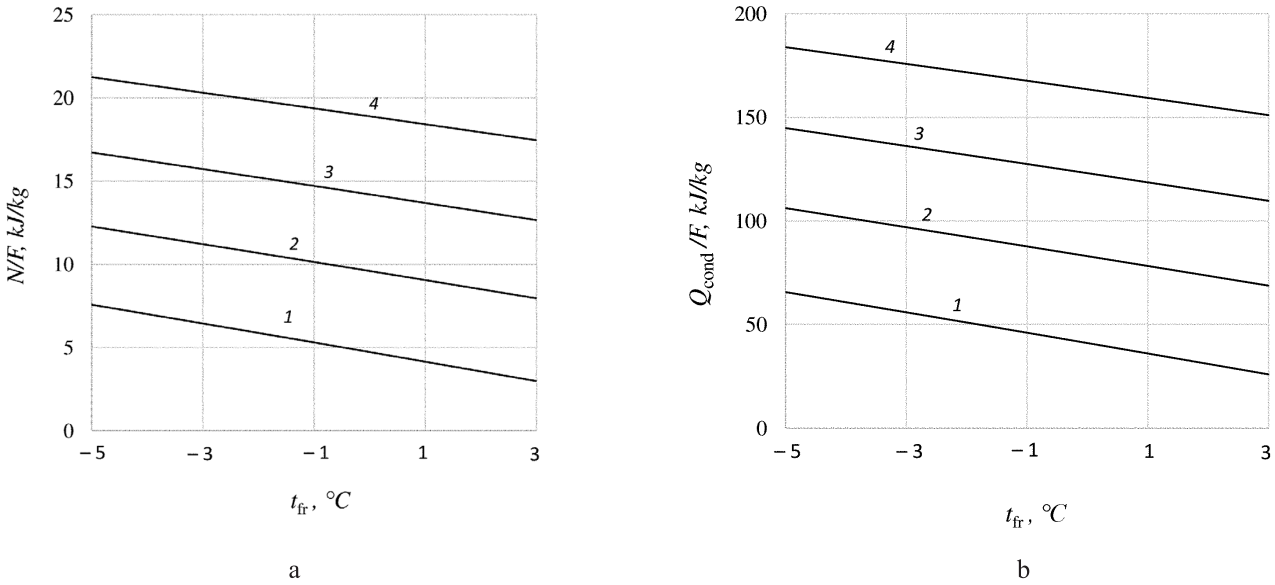


Fig. 5. Dependence of the compressor power consumption (a) and the amount of heat removed during the condensation of refrigerant vapor (b) on the fractionation temperature (system $(NH_2)_2CO–H_2O$; p_2 = 2 atm): (1) x_F = 40% $(NH_2)_2CO$; (2) x_F = 45%; (3) x_F = 50%; (4) x_F = 55%.

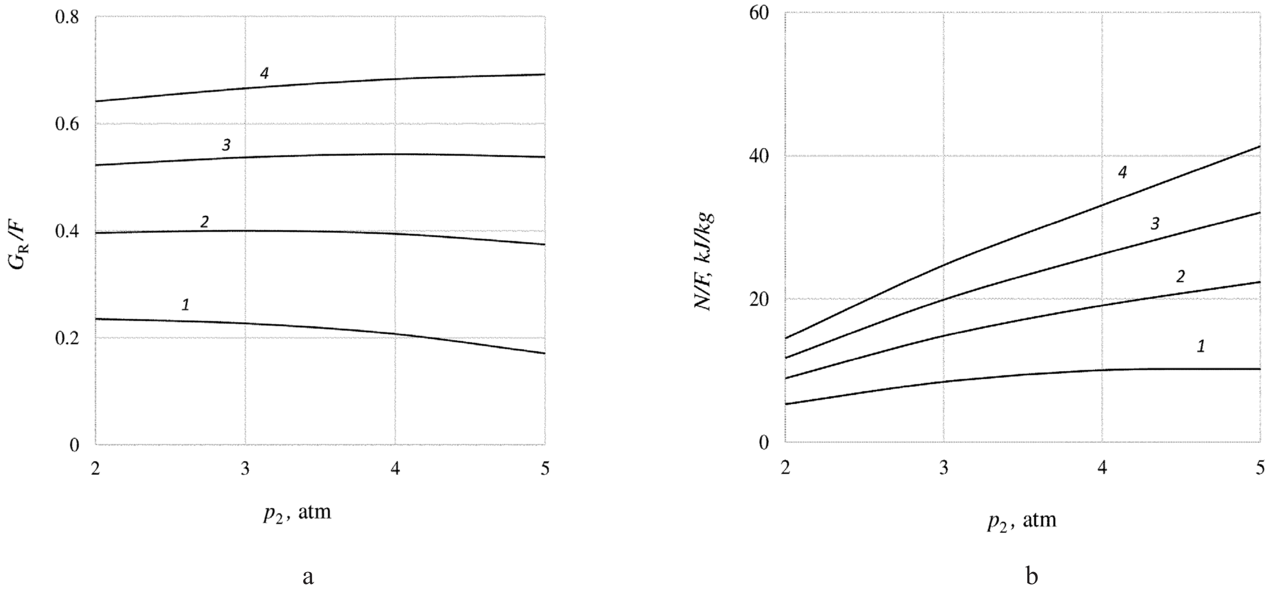


Fig. 6. Dependence of the refrigerant flow (a) and compressor power (b) on the refrigerant pressure at the compressor outlet (NaI–H_2O system; t_{fr} = −28°C): (1) x_F = 60% NaI; (2) x_F = 63%; (3) x_F = 66%; (4) x_F = 69%.

Key
Cryst – crystallizer;
Sep – filter (separation);
Com – turbocharger;
Th – throttle valve;
Cond – refrigerant condenser;
F – mass flow of the initial solution;
C – output of the crystal phase;
M – output of the stock solution;
S – mass flow of the extracted crystal phase;
L – mass flow of the removed uterus;
G_R – refrigerant consumption;

G_W – water consumption;
x_F – concentration of the initial solution;
x_S – concentration of the extracted substance in the crystal phase;
x_L – concentration of the substance in the mother liquor;
t_{fr} – cooling temperature of the solution (fractionation);
Θ_1 – temperature of the refrigerant at the compressor input;
Θ_2 – temperature of the refrigerant compressed in the compressor;
t'_W – water temperature at the condenser input;
t''_W – water temperature at the condenser output;
p_1 – pressure of the refrigerant at the compressor input;
p_2 – pressure of the refrigerant compressed in the compressor;
h_R – enthalpy of the refrigerant at the compressor input;
i_2 – enthalpy of the refrigerant compressed in the compressor;
i_3 – enthalpy of the refrigerant at the condenser output;
i_4 – enthalpy of the refrigerant at the crystallizer input.

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Authors' contribution

G.A. Nosov – research planning, analysis of scientific works, scientific editing, preparation of the list of references;

M.E. Uvarov – selection of model systems, conducting the study, writing the text of the article, technical editing, design of the list of references.

The authors declare no conflicts of interest.

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