

# Scientific and Engineering Principles of Membrane Gas Separation Systems Development

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Received November 10, 2015

**Abstract**—A key part in development of membrane gas separation is assigned to membrane materials, their properties, such as the separation selectivity and specific performance with respect to individual components of gas mixtures. At the same time, the role of a particular process and its features in the separation of gas mixtures are insufficiently accounted for and are often not considered at all. In this paper, the authors have tried to formulate the engineering principles of membrane systems development based on a specific task taking into consideration the dialectical relationship between the membrane properties and optimal process parameters, such as the mixture composition, pressure, hydrodynamic conditions, and the process organization.

**Keywords:** membrane technology, gas separation, membrane, permeability, separation factor, process parameters

**DOI:** 10.1134/S0040579517010092

## INTRODUCTION

The membrane separation of gas mixtures on a production scale has firmly won its niche in a wide variety of traditional methods, such as physical and chemical absorption, adsorption (simple and short-cycle), and rectification, including low temperature and others [1–7].

Prospects for the use of membrane gas separation are determined first of all by the following advantages over other methods:

- (1) simple instrumentation;
- (2) relatively low energy consumption;
- (3) possible full process automation;
- (4) highly reliable membrane performance with retention of constant characteristics;
- (5) a real, not declared reagentless nature, etc.

All of the above defines the requirements for membrane or, rather, membrane system design for specific areas of the effective application of this method.

## FUNDAMENTAL THEORY AND SEPARATION MECHANISM

Depending on the membrane structure and its mass-transfer mechanism, all gas-separation membranes may be roughly classified into two types, i.e., porous and nonporous. The latter type has a separation (or selective) layer formed as a uniform matrix.

Depending on the energy interaction between the permeating gas and the membrane material (mainly

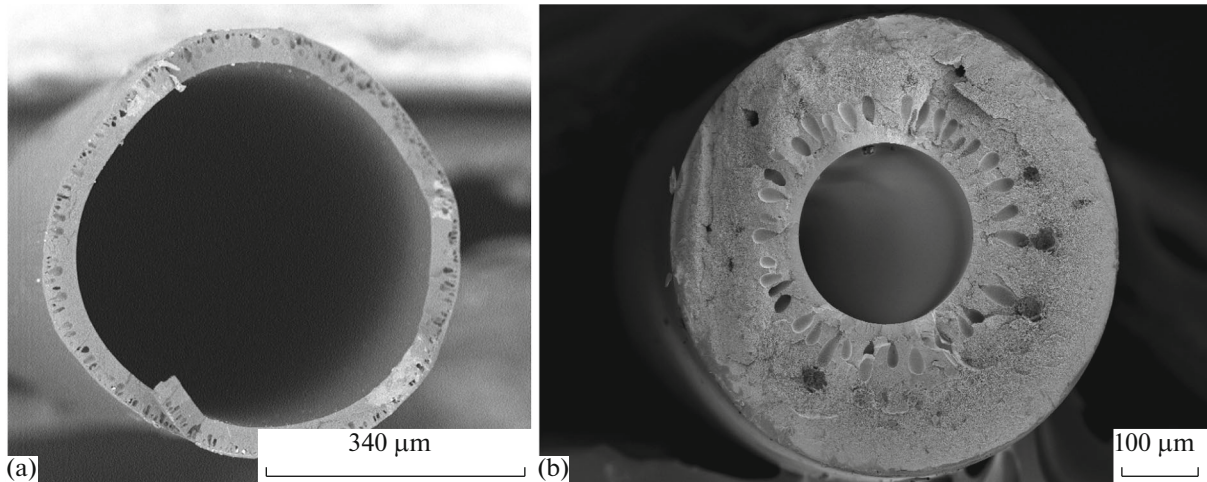
with its selective layer), we can define the following four types of membranes or rather membrane systems [3]:

- (1) porous gas-diffusion membrane systems;
- (2) porous sorption-diffusion membrane systems;
- (3) nonporous sorption-diffusion membrane systems;
- (4) nonporous reaction-diffusion membrane systems.

Each type of membrane system is characterized by a certain type of interaction between gas molecules and structural elements of the membrane matrix. The quantitative characteristics of this interaction are binding energy ( $E$ ) and potential ( $\psi$ ), which in turn depend on the molecular interaction parameters, morphology and molecular nature of the membrane matrix. For example, in gas-diffusion membranes (systems), the binding energy is close to zero ( $E \rightarrow 0$ ); in porous and nonporous sorption-diffusion systems, the binding energy is a function of the heat of sorption (solution), ( $E = f(\Delta H_{\text{sol}})$ ); and, in reaction-diffusion systems, the binding energy is a function of the enthalpy of solution ( $\Delta H_{\text{sol}}$ ) and the heat of the reaction ( $\Delta H_{\text{ch,r}}$ ) – ( $E = f(\Delta H_{\text{sol}}; \Delta H_{\text{ch,r}})$ ).

Potential is the work done in moving a component (gas) unit mass from its volume phase to the field of the membrane material forces, and the potential gradient is a driving force of mass transfer.

The potential in porous sorption-diffusion membranes is significantly impacted by adsorption and capillary effects. In nonporous sorption-diffusion and



**Fig. 1.** Hollow-fiber membranes: (a) for the air separation of air and (b) for helium extraction from natural gas.

reaction-diffusion systems, the transfer potential is impacted by the partial chemical potential and chemical affinity.

In the above-mentioned membrane systems (from porous to nonporous), the increasing binding energy affects the permeability and selectivity (separation factors). The increasing binding energy between the component and matrix reduces the mobility of the gas molecules and, therefore, reduces the permeability. The highest permeability value is inherent to the porous gas-diffusion membrane systems, the binding energy of the gas mixture components for which is close to zero.

Increased binding energy enhances the role of sorption effects. For example, as a rule, the diffusion coefficients of light gases in polymeric membranes are greater, and the sorption (solubility) coefficients are lower than that of heavy gases. Thus, the permeability of heavy components is often higher than that of light components. In membrane systems, with increasing binding energy, an increase in separation selectivity is usually accompanied by a decrease in permeability.

It should be noted that the first (and almost only) example of the successful industrial application of membrane separation using inorganic gas-diffusion microporous membranes is the process of separating uranium isotopes and obtaining enriched  $U^{235}$  (in the form of hexafluoride). The method was developed and introduced in the 1940s–1950s when implementing the nuclear project in both the United States and the Soviet Union. However, currently, almost all industrial membrane gas-separation plants, which are successfully in operation (and under development), use nonporous sorption-diffusion membrane systems. And the vast majority of them are manufactured from polymers of different nature.

## ENGINEERING PRINCIPLES AND PROCESS OBJECTIVES

### *Requirements for Polymeric Membranes and Gas-Separation Units*

Modern industrial polymeric membranes are manufactured either in the form of flat films or hollow fibers, and the latter are successfully displacing the films. This is due to the substantially higher packing density of membranes in the hollow-fiber module (unit) up to  $50 \times 10^3 \text{ m}^2$  of the membrane surface area in  $1 \text{ m}^3$  per unit operating volume. For comparison, in spiral wound units, using film membranes, the packing density does not exceed  $800\text{--}1000 \text{ m}^2/\text{m}^3$ .

In order to increase the mechanical strength and the ability to successfully resist the large transmembrane pressure differential (up to 100 atm) and, at the same time, to achieve high packing density (and, consequently, to reduce specific operating and especially, capital cost) the geometrical dimensions of the hollow fiber are substantially less than the fiber used in baromembrane processes, such as micro- and ultrafiltration. For example, the outer diameter of modern gas-separation hollow-fiber membranes varies in the range of  $100\text{--}500 \mu\text{m}$ , while the thickness of the hollow fiber wall varies in the range of  $20\text{--}150 \mu\text{m}$  (Fig. 1).

In addition, if at the beginning of the industrial application of membrane gas separation the flow rate of processed gas mixtures did not exceed several thousand cubic meters per hour, now, in accordance with modern industrial requirements the feed gas flow rate reaches hundreds of thousands and even millions  $\text{m}^3/\text{h}$  (for example, in the purification of natural and associated gas from acidic components, such as  $\text{CO}_2$ ,  $\text{H}_2\text{S}$ , organic sulfur, etc., with its simultaneous dehydration, the separation of helium concentrate, heavy hydrocarbons, etc.) [3, 5–7].

Therefore, a significant reduction in thickness of the separating (selective) membrane layer is required (and successfully implemented) in order to increase the specific membrane performance. For example, in the 1980s–1990s, it was considered successful to achieve a selective layer 200 nm thick; however, modern membranes 40–50 nm thick are not uncommon. It is hardly possible to obtain these composite membranes with a selective layer made of one polymer applied on a porous substrate made of another polymer.

An alternative technique is asymmetric membranes with a substrate and a selective layer made of the same polymer. The substrate pore dimensions vary (increase) from zero to a few microns at the outlet to the permeation region, which thus provides the lowest possible hydraulic resistance of the membrane itself.

The requirements to the structure of the selective layer is even tougher; it cannot have any microdefects, or rather vacancies larger than 2–3 Å, which is nearly impossible to obtain from a probabilistic assessment (statistically). Therefore, the membrane technology should make a provision for healing microdefects of the selective layer, since the presence of these defects leads to a noticeable decrease in separation selectivity.

Laying and sealing hollow fibers in the membrane module (unit) is a high-technology problem based largely on advances in the sciences. In addition, when choosing a material (polymer) for the membrane and sealant, it is necessary to take into consideration the possibility (probability) of its plasticization [8–12] when in contact with the separated mixture, which accompanied by all negative consequences, such as changes in gas transmission and physicochemical and other characteristics of the membranes and modules during long-term operation (at least 5 years old), etc.

The polymeric membrane, which is designed for separating a particular gas mixture and must be highly productive and highly selective, is the most important. It is desirable that the membrane polymer be already commercially available and relatively inexpensive. The development of a new special membrane polymer from R&D to industrial production requires a huge investment of time and cost. All of these requirements enormously complicate the process of membrane gas separation and modules on their basis. Thus, scientists and engineers have a fair chance to properly beat back this challenge. The technology of gas-membrane separation has closely intertwined both science and engineering, and in some measure art.

#### *Main Characteristics of Membranes for Industrial Gas-Separation Systems*

All polymeric materials used in the creation of gas separating membranes are characterized by two main parameters (properties) that are involved in the dialectical interaction (contradiction), such as the gas permeability factor  $\Lambda$  and the separation factor  $\alpha$ .

Based on the assumption of well-known ideas about the mechanism of gas transfer through a continuous (nonporous) membrane [2, 3, 13]

$$\Lambda_i = D_i \sigma_i, \quad (1)$$

where  $D_i$  is a gas component diffusion coefficient (sorbed and soluted in the membrane material) through the membrane material,  $\text{m}^2/\text{s}$ ;  $\sigma_i$  is a coating efficiency (and solubility) of the gas component of the membrane material,  $\text{mol}/(\text{m}^3 \text{Pa})$ ; and  $\Lambda_i$  is a permeability factor (ideal) of the gas component through the membrane material ( $\text{mol m})/(\text{m}^2 \text{ s Pa})$ .

The literature usually presents data on permeability factors obtained based on experiments on the transfer of clean (individual) gas through the polymer, as well as on the separation (ideal) factors  $\alpha^0$  [5, 6, 14, 15]

$$\alpha_{i,j}^0 = \frac{\Lambda_i}{\Lambda_j}. \quad (2)$$

In terms of gas-mixture separation, it is necessary to account for both the mass-transfer resistance of the membrane itself and external diffusion resistance [3, 13, 16, 17].

Then, the real (observed, effective) permeability factor is as follows:

$$(\Lambda_i)_{\text{eff}} = \frac{\Lambda_i \delta_m}{\Lambda_i (r_1 + r_2) + \delta_m}, \quad (3)$$

$$\frac{\delta_m}{\Lambda_{\text{eff}}} = \frac{\delta_m}{\Lambda_i} + (r_1 + r_2), \quad (3')$$

where  $r_1$  and  $r_2$  are mass-transfer resistance in the boundary (diffusion) layers on the both sides of the membrane and  $\delta_m$  is thickness of the membrane selective layer, [m].

It follows from this equation that the real (observed) permeability factor varies with the membrane thickness, and the mass-transfer resistance at the membrane surface is decisive at high values of the ideal separation factor ( $\alpha^0$ ) and a small thickness of the membrane selective layer. This just the case that corresponds to the requirements of the membranes intended for industrial gas media separation. Impact of these factors, as well as the gas velocity in the discharge channel on the external diffusion resistance is well illustrated by the authors' experimental data [18, 19] (Figs. 4, 5). The ideal separation factor of a sulfur dioxide–nitrogen pair is  $\alpha_{\text{SO}_2/\text{N}_2}^0 = 700$ , and the sulfur dioxide–oxygen pair is  $\alpha_{\text{SO}_2/\text{O}_2}^0 = 200$ .

The specific gas flow through the membrane [ $\text{mol}/(\text{m}^2 \text{ s})$ ] is determined by the value of the driving force of mass transfer and may be represented as follows:

$$j_i = \frac{(\Lambda_i)_{\text{eff}}}{\delta_m} (\Delta p_i)_{\text{avg}}, \quad (4)$$

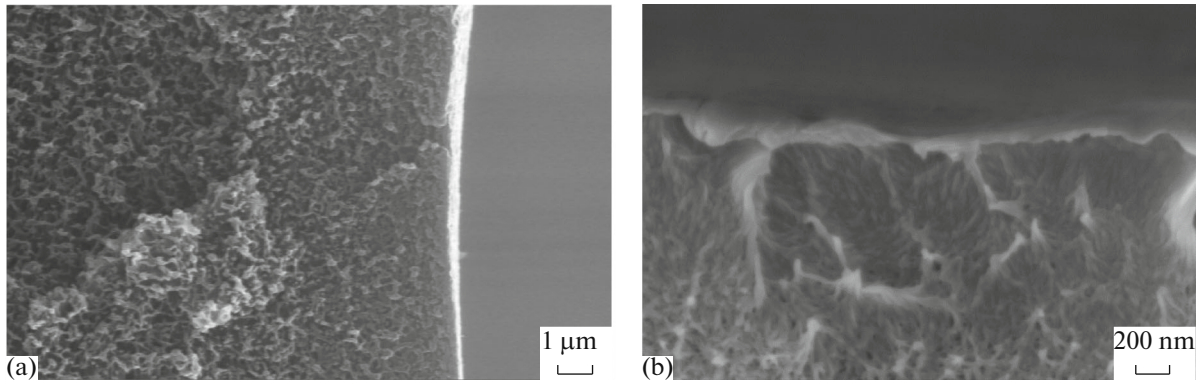


Fig. 2. Nonporous membrane selective layer (a) for air separation and (b) for helium extraction from natural gas.

where  $(\Delta p_i)_{\text{avg}}$  is the driving force of mass transfer, which is equal to the average differential of partial pressures of the component on both sides of the membrane.

An analysis of Eqs. (3) and (4) shows that separation efficiency depends not only on the permeability factors and thickness of the selective layer of the membrane, but also on both the composition of a feed gas mixture and separating flows (retentate and permeate), that is, on the driving force of mass transfer [19].

#### *Principles of the Separation Process Organization*

There are two main methods of separation, such as the so-called compressor and vacuum methods.

According to the first method, the feed gas mixture is fed to separation at elevated pressure, the permeate is removed from the membrane unit at normal pressure. This method is used normally, if a less permeable component to be separated (in the retentate) from the mixture as a target product, for example, in preparation of highly concentrated (up to 99.5 vol. %) nitrogen from air, in the purification of natural gas from carbon dioxide and hydrogen sulfide with its simultaneous dehydration, in the separation of hydrogen from ammonia synthesis purge gas and other petrochemical processes, etc. [3, 7, 20–22].

According to the vacuum method, the feed gas mixture is fed to separation by a blower under pressure slightly above atmospheric. A vacuum pump maintains low pressure in the permeate media. This method is used mainly to obtain a concentrated oxygen stream (50–55 vol %) from air.

A combined compressor-vacuum method is also possible, which can be used for deep recovery from the gas (in the permeate) of a high permeability but low concentrated component, which content in such a mixture usually varies in the range of about 0.03–0.4 vol %, such as helium from natural gas. This method provides the highest average value of the driving force of mass transfer and, at a low volume of extraction (a ratio of

the permeate flow rate to the initial flow), we can obtain a helium-enriched flow [23].

It should be noted that the properties of gas-separation membranes should comply with the requirements of a particular industrial gas-mixture separation process. This thought seems simple (trivial), but at the dawn of the membrane technology industrial application (gas separation), a membrane and corresponding equipment initially intended for, e.g., air separation, were developed, then used with certain success to separate other gaseous mediums.

Thus, for example, an asymmetric film membrane made of polyvinyltrimethylsilane (PVTMS) was developed in the Soviet Union in the 1970s–1980s, and devices on its basis were used to obtain an oxygen- (or nitrogen-) enriched flow from air, and then to separate hydrogen from purge gases, for biogas purification, to control gaseous medium in agricultural storage facilities, etc. [3, 5–7].

The same thing occurred with an industrial membrane of the famous Monsanto Company [3, 5–7], which was developed and widely used to extract hydrogen from purge gas, then for air separation. However, this approach is justified mainly for the permeate gas separation of a fast permeating (target) component, the permeability of which is substantially higher than that of the mixture components; both separated and hardly permeated components are transferred selectively, mainly due to the difference in the diffusion coefficients of the membrane material, while the separation factor is defined by the relationship

$$\alpha_{i,j}^0 = \frac{\Lambda_i}{\Lambda_j} \approx \frac{D_i}{D_j}. \quad (5)$$

At the same time, the coating efficiency of the membrane material components differs slightly  $\sigma_i \approx \sigma_j$  [3, 13].

The example of gas mixtures, the separation of which is appropriate (justified) in air as an enriched both oxygen and nitrogen product; hydrogen extraction from its nitrogen or methane mixtures;

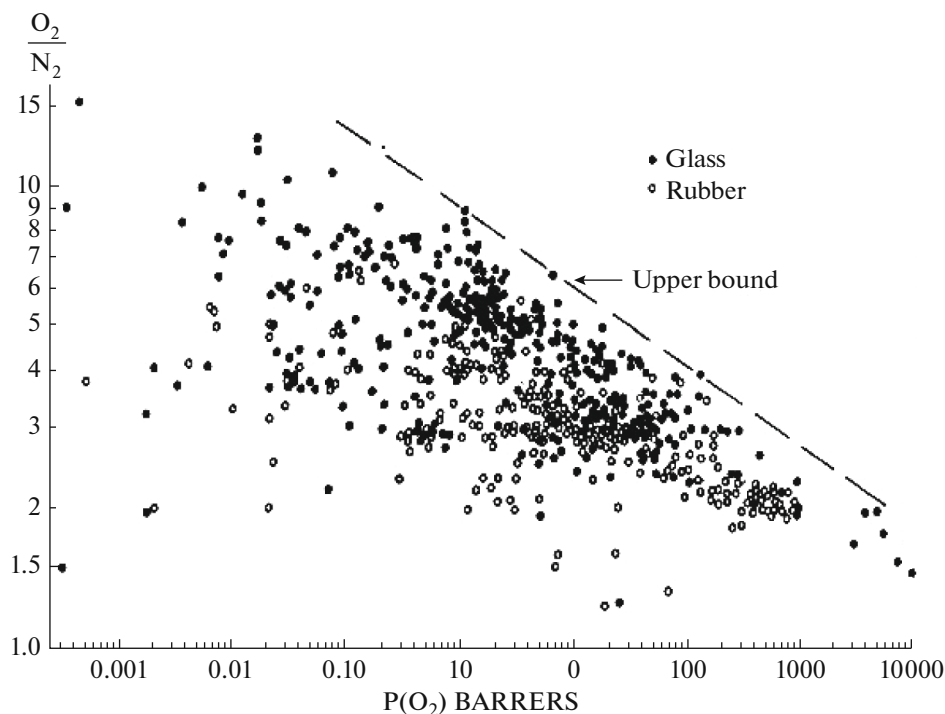


Fig. 3. Robson's diagram for an oxygen-nitrogen pair.

helium extraction (concentration) from natural gas; the separation of radioactive gas, etc. [3, 5–7].

A fundamentally different scientific and engineering approach is needed in the development of membranes for separation of gas components separation, which have a molecular weight much higher than the mass of the main mixture component; hence,  $D_i \ll D_j$ .

Equation (2) can be written as

$$\alpha_{i,j}^0 = \frac{\Lambda_i}{\Lambda_j} \approx \frac{\sigma_i}{\sigma_j}. \quad (6)$$

The following are examples of application of these membrane systems [3, 5–7, 20–22]: natural and process gas acid purification, such as  $\text{CO}_2$ ,  $\text{H}_2\text{S}$ ,  $\text{COS}$ ,  $\text{CS}_2$ , and  $\text{SO}_2$ ; the extraction of higher hydrocarbons from natural and associated gas; biogas cleaning; and the regulation of gaseous medium in closed volumes.

In this case, since the content of contaminants in these mixtures may be very small (0.1–0.5%), the requirements for selectivity of this type of membrane are extremely rather stringent; the value of the ideal separation factor should be large, i.e., 50–500 or higher; in other words, the membrane should be substantially impermeable for the main low-permeability component and the polymer membrane material should be chemically, physically, and mechanically resistant to the separated environment. Examples of the development of these membrane systems are rare, but they are things of the future.

Obviously, the separation of these mixtures should be reasonably carried out by a mixed compression-vacuum method, i.e., under elevated pressure in the delivery channel and under a vacuum in the drainage hole.

In the estimation of these systems, a real separation factor is much less than the ideal one ( $\alpha^{\text{eff}} \ll \alpha^0$ ) and a failure to take into account external diffusion resistances leads to a significant distortion of the results [3, 13, 18, 19].

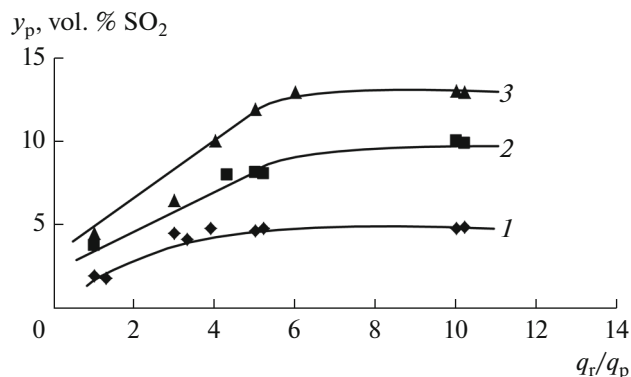
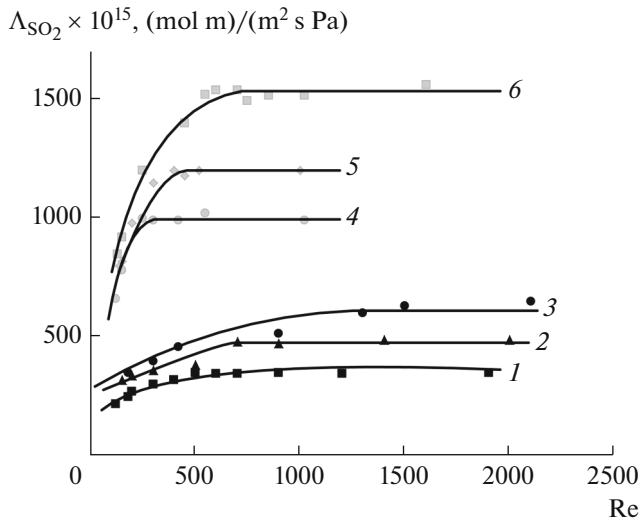
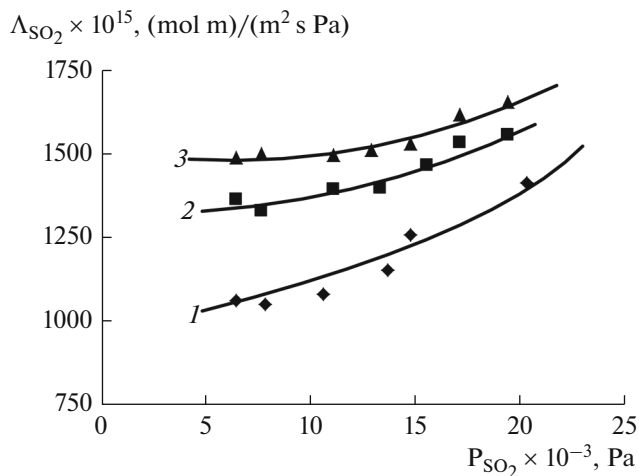


Fig. 4. Dependence of  $\text{SO}_2$  concentration on the ratio of the retentate and permeate flow  $q_r/q_p$ . The  $\text{SO}_2$  concentration in the feed mixture is  $y_f = 1.5$  vol %. Pressure in the delivery channel is (1) 0.3; (2) 0.5; (3) 1.0 MPa. Permeate pressure is atmospheric. Temperature is 295 K.



**Fig. 5.** Dependence of the permeability coefficient on the flow velocity in the high pressure channel. (1–3)  $SO_2$  (1.7%)– $N_2$ ; (4–6)  $SO_2$  (1.8%)– $O_2$ . Permeate pressure is atmospheric: (1, 4) 0.3; (2, 5) 0.5; (3, 6) 1.0 MPa.



**Fig. 6.** Dependence of the permeability coefficient on the  $SO_2$  partial pressure in the  $SO_2$ – $O_2$  mixture under pressure of 0.3 MPa, flow rate of 1 m/s, flow channel height is: (1) 0.5; (2) 1.0; (3) 1.5 mm.

## CONCLUSIONS

Principles of membrane gas separation are effective for all types of membranes, both polymeric, based on inorganic materials (metals, alloys, etc.) and so-called mixed (polymers and inorganic inclusions) and reaction-diffusion membrane systems.

Fields and objects of industrial applications of membrane gas-separation systems are expanding. For already common technologies of air separation and hydrogen extraction, new technologies are being developed and applied in industrial processes, such as

the purification of natural gas from  $CO_2$  and  $H_2S$  with its simultaneous dehydration, the extraction of heavy hydrocarbons ( $C_2$ – $C_{5+}$ ) from natural and associated gas, the separation of nitrogen from methane mixtures, air drying, the recovery of helium from natural gas, the separation of radioactive gases, and many others.

The combination of membrane and other separation processes (absorption, adsorption, distillation, cryogenics) seems promising, which makes it possible to realize the advantages of each of the processes in order to reduce unit expenses and operating cost.

However, the development of membrane gas separation, including our country, has been prevented by some circumstances that the authors consider their duty to note:

(1) a weakened relationship between research organizations and industry (business), which is already weak in our country;

(2) the relationship between the worlds of academia, applied science, and industry is broken;

(3) the production of membranes and respective equipment is in the hands of a few companies, mainly foreign, which, when advertising their products, hardly publish experimental data on the characteristics of their products in real operation. This policy consists of isolation from the scientific and engineering community and harms the wider dissemination of their own products, but this is a reality of a modern monopolies.

The authors hope that this article will help researchers and developers to evaluate and take into account many aspects of the membrane gas-separation technology, as well as to attract business and government attention to the advantages of this innovative technology.

Examples of successful cooperation between science and business in this field in Russia are known, such as the development of membranes and equipment for separating helium from Eastern Siberia natural gas deposits, as well as for air separation.

## NOTATION

$D$	coefficient of the diffusion of the gas component (sorbed or soluted in the membrane material) through the membrane material, $m^2/s$
$E$	binding energy, J/mol
$\Delta H$	enthalpy (heat), J/mol
$P$	partial pressure, Pa
$q$	volume flow, $m^3/s$
$r$	mass transfer resistance in the boundary (diffusion) layers ( $m^2 s Pa$ )/mol
$y$	component volume concentration, vol %
$\alpha$	separation factor

$\delta$	membrane selective layer thickness, m
$\Lambda$	permeability of the gas component through the membrane material (mol m)/(m <sup>2</sup> s Pa)
$\sigma$	coating efficiency (and solubility) of the gas component by the membrane material, mol/(m <sup>3</sup> Pa)
$\psi$	binding potential, J

#### SUBSCRIPTS AND SUPERSSCRIPTS

0	ideal (ratio, factor)
1,2	space respectively before and after the membrane
avg	average
ch.r	chemical reaction
eff	effective factor
$f$	feed mixture
$i, j$	number of components
m	membrane
p	permeate
r	retentate
sol	solution
$\sigma$	sorption (solution)

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Translated by A. Kolesesin