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Application of poly(ether-*block*-amide) membranes to gasoline desulfurization by pervaporation

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ABSTRACT

The aim of this research was to recognize the applicability of poly(ether-*block*-amide) membrane to gasoline desulfurization by means of vacuum pervaporation. PEBAX 2533 and PEBAX 2533/5533 homogenous membranes were prepared by solvent evaporation – phase inversion technique. The influence of process parameters such as feed temperature and feed concentration on thiophene removal from simulated *n*-octane/thiophene mixture was also examined. Total average flux for the simulated gasoline at 35°C and thiophene concentration of 0.07 wt. % was equal to 0.56 kg/m^2 h and it increased to 1.29 kg/m^2 h with temperature increase up to 50°C. The corresponding separation factor β values were equal to 7.06 and 5.99, respectively. The change of sulfur concentration in tested range of 0.05–0.13 wt. % had insignificant impact on the permeation flux. Furthermore, the separation factor was also not influenced by the variation of sulfur compound level in the feed. Considering components permeances and intrinsic membrane selectivity results, PEBA membranes were found to be thiophene selective ($\alpha_{T/O} > 1$).

Keywords: Desulfurization; Gasoline; PEBA; Vacuum pervaporation

1. Introduction

Poly(ether-*block*-amide) (PEBA) copolymer (Fig. 1), which belongs to the group of thermoplastic elastomers, comprises of rigid linear chain of polyamide (PA) interspaced with soft polyether (PE) segments.

Polyamide crystalline domains provide mechanical strength, while the polyether amorphous domains offer high permeability due to the high chain mobility of the ether linkage. Different types of polyamide, that is, nylon 6, nylon 66, nylon 11, nylon 6/11, nylon 12, and nylon 6/12 can be used to synthesize PEBA. The available polyethers include poly (ethylene glycol), poly(propylene glycol), and poly (tetramethylene ether glycol).

The selection of proper PEBA copolymer can enhance the selectivity of membranes and improve the separation efficiency. The properties of PEBA copolymer are affected by the chemical nature and the ratio of PA to PE segments. Different grades of PEBA polymers are commercially available, and they generally have excellent mechanical strength and good chemical resistance. The properties of each segment decide on the overall properties of the PEBA copolymers.

Because of the micro-biphasic structure, PEBA copolymers offer many properties that are not readily available in the constituent polymer. PEBA not only has favorable membrane-forming properties but also good chemical resistance to acidic, basic, and organic solvents and high thermal and mechanical stability.

In the last decade, researchers have studied the possibility of application of PEBA membranes made of various grades of PEBA copolymer in different membrane-based separation processes.

Pure PEBA-based membranes [1–3], as well as ones incorporated with performance-enhancing fillers [4–6], are known

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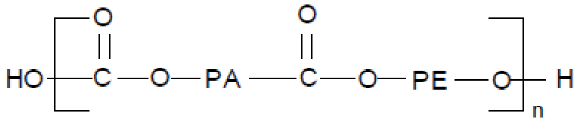

$$\mathrm{HO}\left[\overset{\overset{\displaystyle \mathrm{O}}{\|}}{\mathrm{C}}-\mathrm{O}-\mathrm{PA}-\overset{\overset{\displaystyle \mathrm{O}}{\|}}{\mathrm{C}}-\mathrm{O}-\mathrm{PE}-\mathrm{O}\right]_n\mathrm{H}$$

Fig. 1. Chemical structure of poly(ether-*block*-amide).

to have high potential, when applied in separation of acetone-butanol-ethanol from fermentation broth. Ding et al. [7] investigated the applicability of PEBA 2533 and metal-organic framework (ZIF-8) incorporated mixed-matrix PEBA 2533 membranes (MMMs) for phenol removal. Both membranes exhibited very good performance and had a great potential in separation and recovery of phenol from its aqueous solution. Choudhari et al. [8] prepared composite poly(ether-*b*-amide) membranes by dispersing different nanomaterials, that is, graphene, graphene oxide, and molybdenum disulfide (MoS_2) in PEBA 2533 matrix. Prepared MMMs were applied in the pervaporation separation of butyric acid produced by anaerobic digestion of perennial grass. Among all the tested membranes PEBA–graphene membrane showed the best performance with butyric acid flux of 24.3 g/m^2 h and separation factor of 21. Pervaporation with poly(ether-*b*-amide) membranes was also applied in odorous diacetyl and *S*-methylthiobutanoate removal [9]. The highest observed organic flux and separation factors for diacetyl/water and *S*-methylthiobutanoate/water mixtures were 1.6 g/m^2 h and 40 and 4.6 g/m^2 h and 110, respectively. Tubular, thermal cross-linked-PEBA/ceramic membranes were also applied in separation of an aromatic/aliphatic mixture, that is, toluene/heptane – the highest separation factor of PEBA hybrid membrane filled with graphite was 10.4, while the permeate flux was 29 g/m^2 h [10].

In the field of gasoline desulfurization by vacuum pervaporation method, a major progress has already been made in both, basic researches focused on expanding the knowledge as well as in the progressing of this new technology beyond the laboratory test scale [11–15]. There are also some publications reporting on desulfurization potential of PEBA membranes. Liu et al. employed PEBA/PVDF membranes in pervaporative separation of thiophene from its binary mixture with *n*-heptane [16]. Yu et al. [17] prepared PEBA 2533 mixed-matrix membranes filled with polydopamine (PDA)-coated graphene nanosheets and Ag nanoparticles loaded with PDA-coated graphene nanosheets, which appeared to be highly resistant to disadvantageous intensive swelling, not only because of the presence of rigid PA blocks, but also due to coating of graphene nanosheets with PDA.

The aim of this work was to prepare PEBA-based membranes by as fast, cheap, and least complicated method as possible from two different grades of poly(ether-*b*-amide) copolymer and to test such prepared membranes in process of a sulfur-containing organic compound separation from its binary mixture.

2. Experimental

2.1. Materials

In order to prepare simulated, binary *n*-octane/thiophene mixture representing gasoline, thiophene (99.5%, extra pure, benzene free), supplied by Acros Organics, USA, and *n*-octane (99.5% purity) (99%), supplied by Chempur, Poland, were used. PEBAX 2533 and PEBAX 5533 polymers were kindly supplied by ARKEMA, France. Solvent for membrane's preparation, *n*-butanol (99.5% purity), was purchased from Avantor Performance Materials Poland S.A.

2.2. Membrane preparation by solvent evaporation phase inversion technique

Membranes applied during discussed studies were prepared by solvent evaporation phase inversion technique. A certain amount of polymers was dissolved in *n*-butanol at high temperature (90°C) with vigorous stirring conditions to form a 5 wt. % homogeneous polymer solution. In order to obtain a homogeneous solvent–polymer solution, the mixture was stirred and treated with ultrasounds, which also enabled the removal of accidentally trapped air bubbles. The solution was next spin-coated on a Teflon plate. The resulting PEBA membrane was dried at room temperature in ambient air to allow the solvent to evaporate. The membranes were stored in a dry, dust-free environment.

For the preparation of poly(ether-*b*-amide) membranes, solid polymers PEBAX 2533 and PEBAX 5533 characterized by different ratio of polyether to polyamide blocks were used. PEBAX 2533, built predominantly of PE blocks, is easy to process even at room temperature. On the other hand, PEBAX 5533, which contains more PA blocks, requires high temperature during both, solution preparation and at the membrane casting stage, which make the preparation of membranes with reproducible properties difficult. Hence, PEBAX 5533 was used in a mixture with PEBAX 2533 polymer in a percentage ratio of 70:30. The average thickness of prepared homogenous membranes PEBAX 2533 and PEBAX 2533/5533 was 33.2 and 32.6 μm, respectively.

As shown in Fig. 2, membranes were characterized by a compact, homogeneous, and nonporous structure.

2.3. Pervaporation experiments

The laboratory test unit (Fig. 3) was operated with a continuous recirculation of the retentate to the feed tank. A flat sheet membrane module with an effective separation area of 54 cm^2 was operated in the cross-flow mode. The membrane was placed on a porous, sintered, stainless steel plate. A pump was used to recirculate the liquid feed from the feed tank to the membrane cell, where the feed came into direct contact with the membrane active layer. The temperature of the feed stream was controlled by a Greisinger Electronic GTH 1100/2 DIF digital thermometer (NiCr-Ni). During the experiments, the permeate-side pressure was assured by a vacuum pump (Alcatel Pascal 2015 SD) and it was kept at a constant level of 100 Pa. Electronic vacuum gauge (Vacuubrand DRV2) was used to monitor the downstream pressure. The condenser system consisted of a cooling trap refrigerated in the liquid nitrogen (−196°C).

Pervaporation yield was evaluated based on total (J_m) permeate flux and partial (J_i) fluxes of mixture components, which were calculated according to Eqs. (1) and (2) as follows [18,19]:

$$J_m = \frac{\Delta m_p}{S_m \times \Delta t} \tag{1}$$

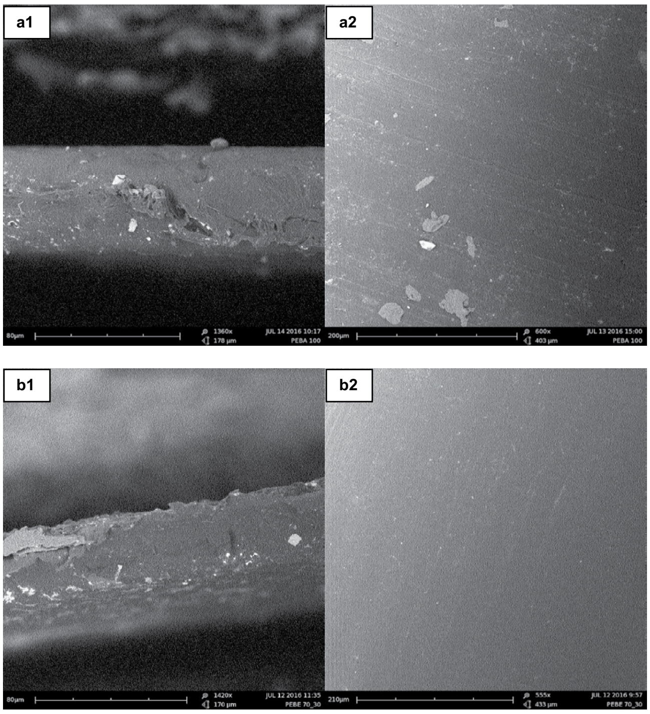

Fig. 2. SEM images of prepared homogeneous membranes: 1 – membrane cross-section; 2 – membrane surface; a – PEBA 2533; b – PEBA 2533/5533.

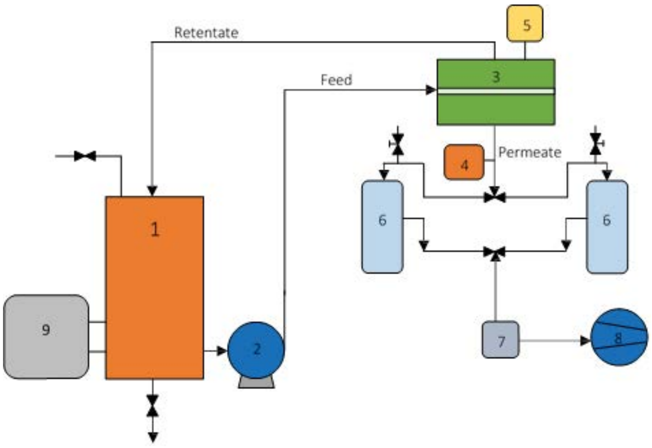

Fig. 3. The scheme of the vacuum pervaporation set-up – 1 – feed tank; 2 – circulation pump; 3 – membrane module; 4 – vacuum gauge; 5 – thermometer; 6 – cooling traps; 7 – buffer tank; 8 – vacuum pump; and 9 – thermostat.

$$J_i = J_m \times w_i^p \tag{2}$$

where Δm_p – permeate mass collected over Δt period, kg; S_m – membrane effective separation area, m^2; Δt – time of sample collection, h; and w_i^p – weight fractions of components in the permeate.

Separation efficiency was evaluated based on the dimensionless separation factor β according to Eq. (3) as follows [18,19]:

$$\beta = \frac{w_\mathrm{T}^p / w_\mathrm{O}^p}{w_\mathrm{T}^f / w_\mathrm{O}^f} \tag{3}$$

where w_T^p, w_O^p – weight fractions of thiophene and octane in the permeate, respectively; w_T^f, w_O^f – weight fractions of thiophene and octane in the feed, respectively.

Overall membrane performance and efficiency of thiophene removal were evaluated using the pervaporation separation index, PSI, kg/m^2 h according to Eq. (4) as follows [19,20]:

$$PSI = (\beta - 1) \times J_m \tag{4}$$

In order to evaluate the intrinsic membrane performance, permeance (P_i/l) of a component i and selectivity α_{ij} were calculated according to Eqs. (5) and (6) [21], respectively:

$$\left(\frac{P_i}{l}\right) = \frac{j_i}{X_i \gamma_i p_i^f - Y_i p_i^p} \tag{5}$$

$$\alpha_{ij} = \frac{\left(\frac{P_i}{l}\right)}{\left(\frac{P_j}{l}\right)} \tag{6}$$

where (P_i/l) – permeance of component i; j_i – molar flux of component i; x_i, y_i – molar fractions of component i in feed and permeate, respectively; γ_i – activity coefficient of component i; p_i^f, p_i^p – saturated vapor pressures of component i in feed (f) and permeate (p); and l – membrane thickness.

The activity coefficient of component i in binary systems was calculated applying UNIQUAC method [22,23] using data given in Table 1.

Saturated vapor pressures of components in tested range of temperatures were calculated according to Antoine equation using Antoine constants given in Table 2 – Eq. (7) as follows [24]:

$$\log p_i^s = A - \frac{B}{C + T} \tag{7}$$

where p_i^s – saturated vapor pressure, hPa; A, B, C – Antoine equation constants; and T – temperature, K.

Table 1
UNIQUAC method parameters used for calculations of activity coefficient [23]

	r	q	q'	α_{ij}	α_{ji}
j-Octane	5.849	4.936	4.936	7.761	148.28
i-Thiophene	2.857	2.140	2.140		

Table 2
Antoine's constants

	A	B	C
n-Octane	6.90940	1349.820	209.385
Thiophene	6.95926	1246.020	221.350

The apparent activation energy was calculated on the basis of the Arrhenius-type relation as follows [25]:

$$J_P = J_0 \exp\left(\frac{-E_{\text{app}}}{RT}\right) \tag{8}$$

where J_P – the total flux or partial flux of individual component, kg/m^2 h; J_0 – pre-exponential factor; E_{app} – the apparent activation energy of pervaporation, kJ/mol; R – the universal gas constant, J/(mol K); T – absolute temperature, K.

Membranes swelling degree S_D was calculated based on Eq. (9) as follows [26]:

$$S_D = \frac{W_W - W_D}{W_D} \tag{9}$$

where W_W – weight of the membrane in equilibrium state, g and W_D – weight of dry membrane, g.

2.4. Permeate and feed samples analyses

Gas chromatograph equipped with a flame ionization detector (SRI 8610C Instruments) and Restek DB-1 MTX capillary column (0.5 µm, 0.53 mm × 60 m) was employed to analyze concentration of thiophene in feed and permeate samples.

3. Results

3.1. Membrane swelling

Due to the fact that the membrane is in contact with the liquid feed, the sorption of the components and the resulting swelling are natural phenomena and they are strictly related to the solution-diffusion model. Too intensive swelling can lead to the degradation of the membrane separation layer. In addition, as the membrane swelling degree increases, the difference between the sorption and diffusion coefficients of the feed components becomes smaller, which can contribute to the drop of the separation selectivity. The sorption of a component with high affinity to the polymer may facilitate the sorption of the component, which is poorly absorbed in a nonswollen membrane [17,26–28].

As expected, the addition of PEBAX 5533 polymer reduced the susceptibility of the PEBA membrane to swelling (Table 3). In the case of pure PEBAX 2533 membranes, the swelling ratio was 25.5%. The 30% addition of PEBAX 5533 polymer allowed to reduce the S_D value of the PEBA 2533/5533 membrane to 20.5% by weight.

3.2. Influence of feed temperature

The influence of process temperature on the efficiency of separation of the sulfur compound from gasoline using prepared homogeneous PEBA 2533 and PEBA 2533/5533 membranes was checked in the temperature range of 30°C–50°C. The dependences of total and partial fluxes, separation factor β, permeability, and intrinsic selectivity on the temperature are illustrated in Figs. 4–7.

As demonstrated by the experiments, regardless of the polymer of which the membrane was prepared, the total flux of permeate J_m (Fig. 4), as well as partial fluxes of individual components (Fig. 5) through homogeneous membranes, increased with increasing feed temperature. At the same time, for all membranes, an increase in the process temperature entailed a decrease in the value of the separation factor β (Fig. 4) despite the fact that the partial thiophene flux increased with the temperature.

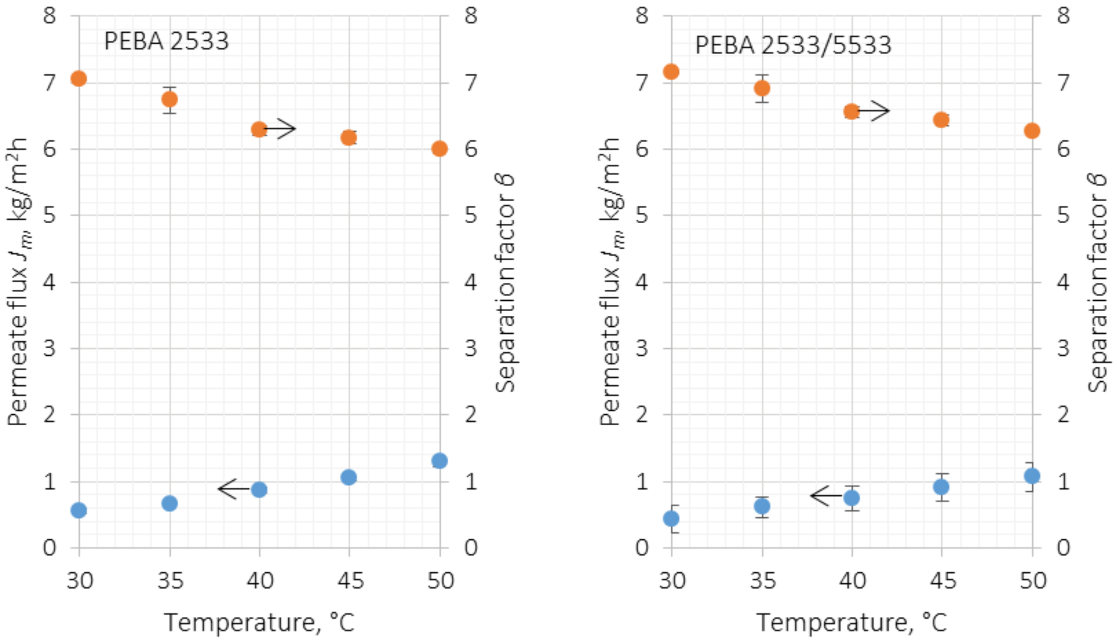

Fig. 4. The impact of operating temperature on the total permeate flux and separation factor at thiophene feed concentration of 0.07 wt. % and permeate pressure of 100 Pa.

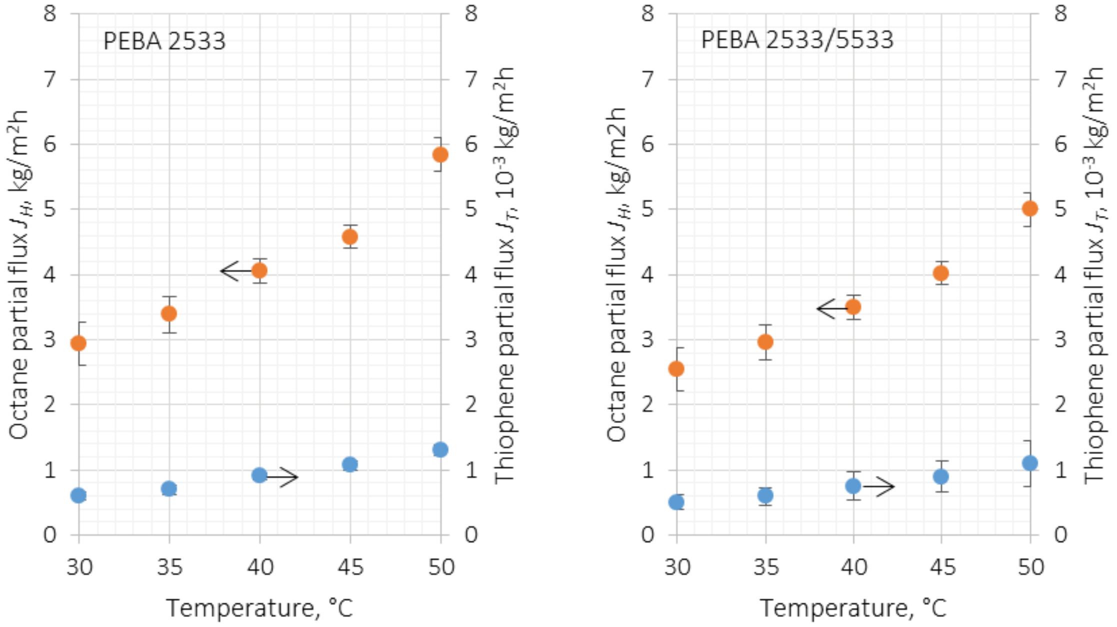

Fig. 5. The impact of operating temperature on the partial fluxes of components at thiophene feed concentration of 0.07 wt. % and permeate pressure of 100 Pa.

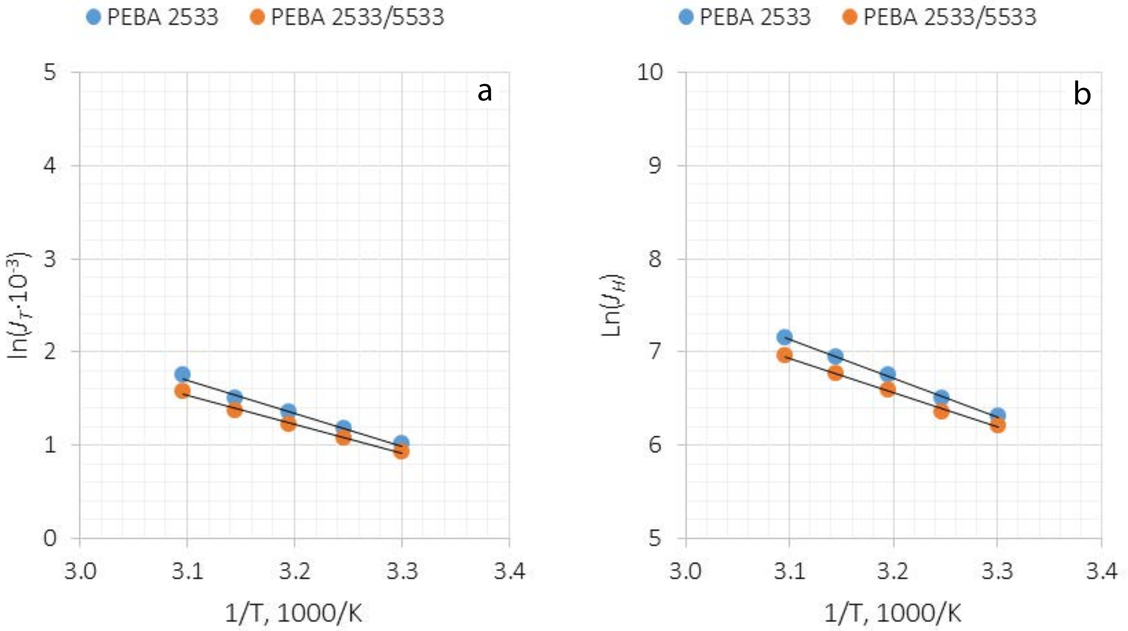

Fig. 6. Arrhenius-type relation on components fluxes for PEBA membrane at feed concentration of 0.07 wt. % and permeate pressure of 100 Pa; (a) thiophene and (b) octane.

A typical for pervaporation, so-called *trade-off phenomenon* between flux and selectivity, was observed. Taking into account the solution-diffusion model [19,29], the relationship might be a consequence of two simultaneously occurring phenomena. First, operating temperature affects the driving force for the mass transfer – the increase in the temperature results in higher partial vapor pressure of components in the upstream side of the membrane and provides a greater driving force for the permeation of components since the downstream pressure remains practically unaltered. Second, in higher temperature, the swelling of membrane's separating layer, resulting of the dissolution of feed components, is thought to be enhanced and it can be responsible for the decrease in thiophene separation factor [11,17].

Based on investigated temperature-dependences of partial fluxes, the apparent activation energy E_{app} of both

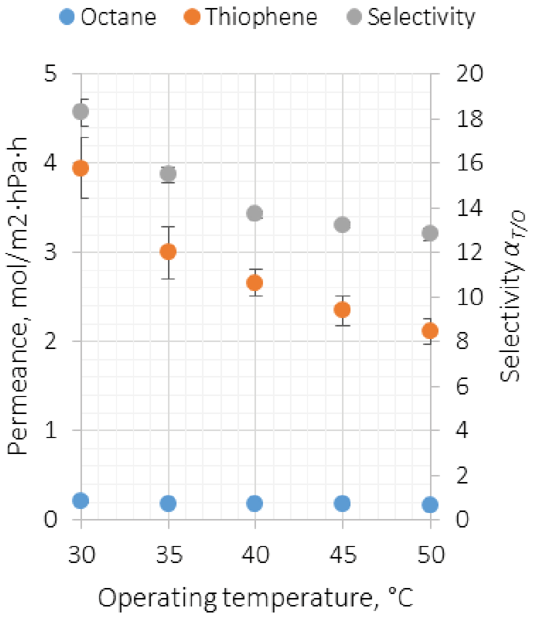

Fig. 7. The impact of operating temperature on thiophene and octane permeances at thiophene feed concentration of 0.07 wt. % and permeate pressure of 100 Pa.

components' transport through homogeneous membranes was determined based on the modified Arrhenius-type relationship (Eq. (15)) [25].

Dependences of ln(J_i) versus inverse absolute temperature 1/T for n-octane and thiophene are shown in Fig. 6. Calculated values of the apparent activation energy E_{app} are collected in Table 4.

Regardless of the type of PEBAX polymer grade, values of apparent activation energy E_{app} of tested compounds were arranged in the following order: n-octane > thiophene.

By analyzing the data presented in Table 4, it can be seen, that in the case of thiophene and n-octane, the apparent activation energy values were similar for both tested membranes. Nevertheless, E_{app} for both components were slightly higher in case of PEBA 2533/5533, which most likely resulted of the higher content of rigid polyamide segments that decreased membranes sorption capacity. Those results could explain lower permeate fluxes obtained during separation with PEBA 2533/5533 membranes.

Table 3
Determined swelling degrees S_D for prepared PEBA-based membranes

Membrane	W_D (g)	W_W (g)	S_D (%)
PEBA 2533	0.1217	0.1554	25.5 ± 0.8
PEBA 2533/5533	0.1167	0.1407	20.5 ± 1.2

Table 4
Calculated activation energies of individual components

Component	E_{app} (kJ/mol)	
	PEBA 2533	PEBA 2533/5533
n-Octane	33.5 ± 0.2	34.6 ± 0.1
Thiopene	26.4 ± 0.2	29.2 ± 0.2

Furthermore, the decrease in thiophene separation factor β with temperature increase could be caused by lower, in comparison with n-octane, E_{app} values determined for thiophene. Partial fluxes of n-octane, due to the higher E_{app}, were more susceptible to increment with the feed temperature increase, hence thiophene separation factor β declined due to the unfavorable changes in ratio between n-octane and thiophene fluxes. Corresponding trends were also reported by other researchers [16,30,31].

It was also found that with increasing temperature of the mixture, the $\alpha_{T/O}$ of all membranes decreased (Fig. 7). This was due to the fact that the activation energy of n-octane molecules transport was higher than the activation energy found for thiophene molecules.

In regard to components permeances and intrinsic membrane selectivity results, prepared PEBA membranes were thiophene selective (Fig. 7). Moreover, while partial fluxes increase with temperature, permeances values exhibited a reverse temperature dependency. Baker et al. [21] state that such relationship indicates on the increase in permeate flux with increasing temperature, which results exclusively of the enhanced driving force, that is, the increase of vapor pressure on the feed side of the membrane.

3.3. Influence of thiophene concentration

A series of PV tests was also carried out to investigate the performance and separation properties of prepared PEBA membranes and to determine the efficiency of thiophene removal, depending on its concentration. The effect of the contaminant concentration in the feed on the pervaporation performance at 30°C was investigated in the range of 0.05–0.13 wt. %. Obtained results are presented in Figs. 8–10.

As can be seen in Fig. 8, the change in sulfur compound concentration in the tested range had a negligible effect on the efficiency of the separation process. Irrespective of the content of thiophene in the mixture, the permeate flux and the separation factor β were at a constant level.

The obtained dependences are consistent with a part of the results presented in the literature by other researchers [32,33]. This may be due to the narrow range of studied thiophene concentrations and the fact, that the concentration of thiophene was very low, when compared with the hydrocarbon concentration. Considering the dissolution-diffusion theory, the effect of separation in the pervaporation process is determined by the first two stages, that is, the sorption and diffusion of the mixture components through the membrane. Most probably, the concentration of sulfur compound was beyond the range in which the membrane swelling caused by the thiophene dissolution in the membrane exceeded the swelling phenomena resulting of the presence of octane [11,14].

Nevertheless, the partial fluxes of thiophene were proportional to its concentration in the feed (Fig. 9), which resulted in constant values of the separation factor. The analysis of results shown in Fig. 10 leads to the conclusion, that the effect of the mixture composition on the permeance of the components was small, and the resulting selectivity $\alpha_{T/O}$

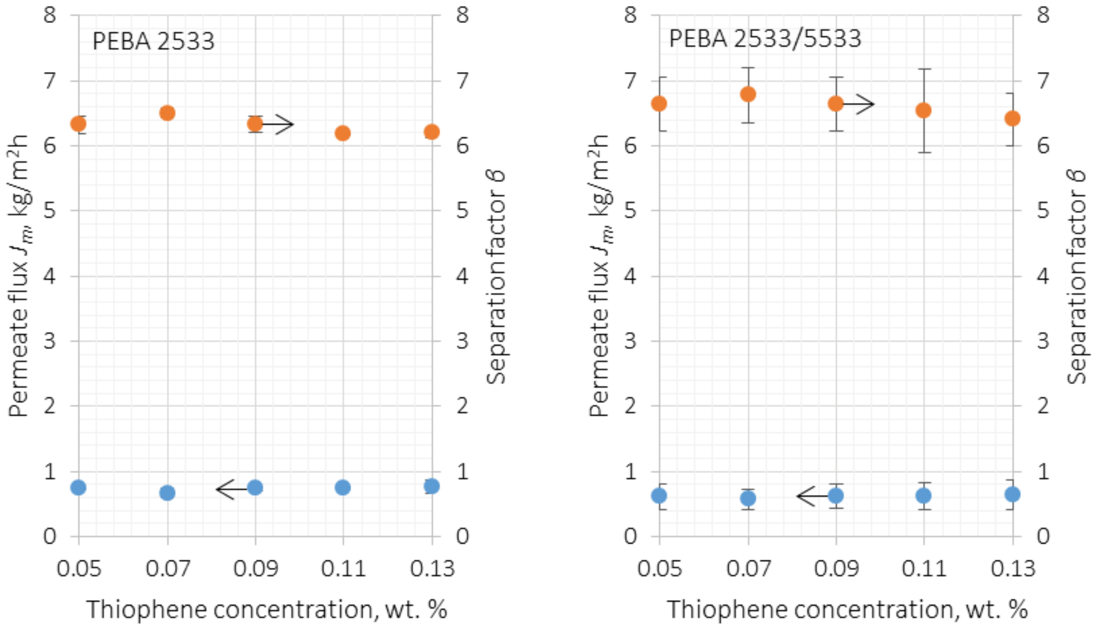

Fig. 8. The influence of thiophene concentration in feed on the total permeate flux and separation factor at temperature of 35°C and permeate pressure of 100 Pa.

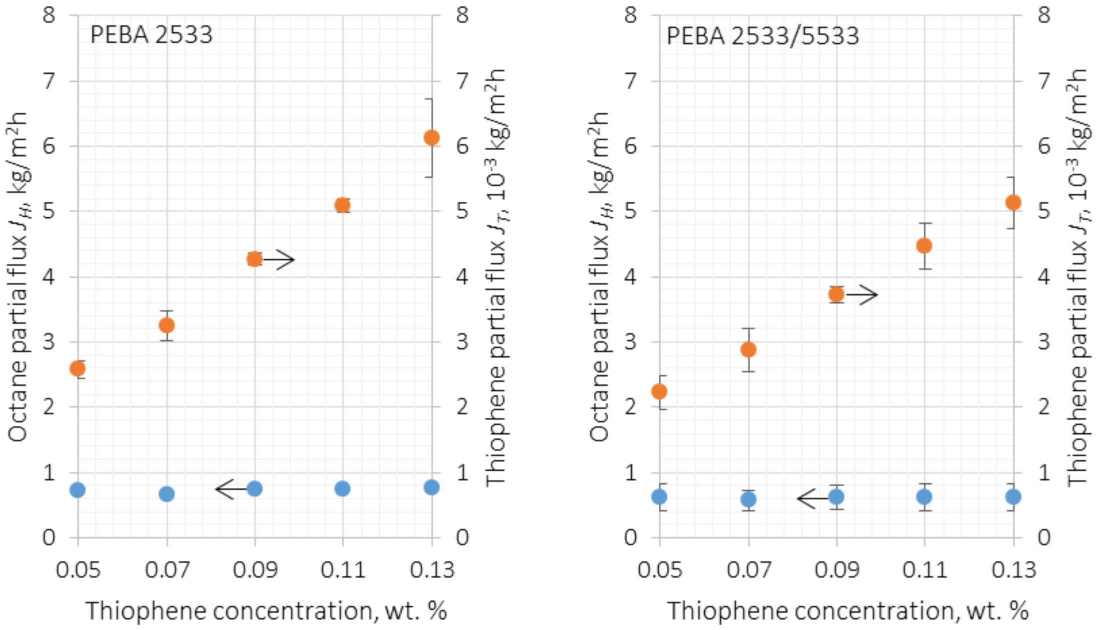

Fig. 9. The influence of thiophene concentration in feed on thiophene and octane partial fluxes at temperature of 35°C and permeate pressure of 100 Pa

was practically constant in the tested concentration range. Both mentioned relationships proved that process was poorly sensitive to fluctuations of removed components concentration in the feed at the investigated range.

3.4. Comparison of overall efficiency

Analyzing the values of the PSI index (Fig. 11) calculated for tested homogeneous membranes and partial fluxes of the selected sulfur compound (Figs. 5 and 9), it can be seen that the efficiency of separation was arranged in the following order: PEBA 2533 < PEBA 2533/5533.

The test results obtained for homogenous membranes of PEBA type were comparable with results obtained during tests with commercial membranes made of the same polymer, in terms of separation efficiency evaluated based on β [34,35], which showed that membranes produced by a simple method in the laboratory conditions could be successfully

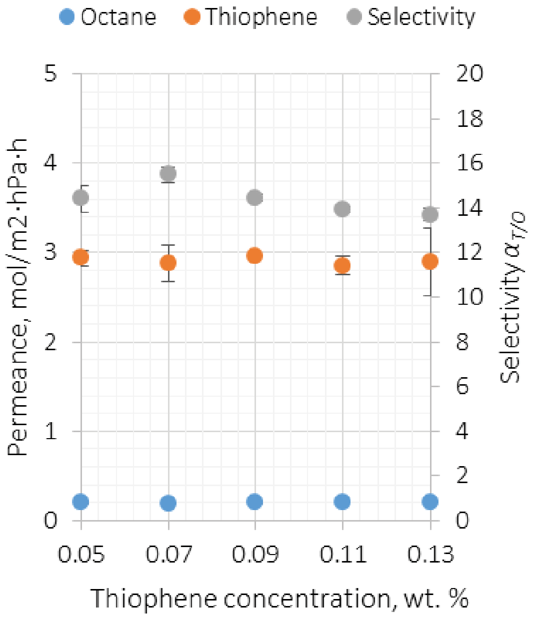

Fig. 10. The influence of thiophene concentration in feed on thiophene and octane permeances and selectivity at temperature of 35°C and permeate pressure of 100 Pa.

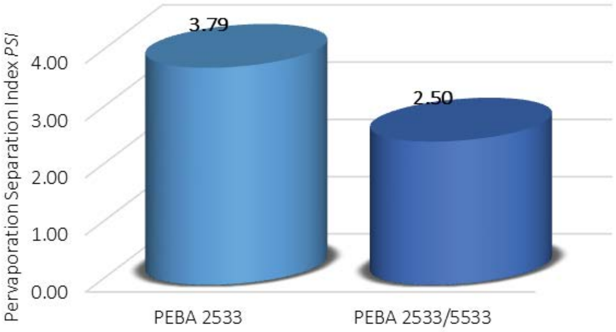

Fig. 11. PSI index values for tested homogenous PEBA membranes.

used for research purposes. Lower permeate fluxes in comparison with commercial membranes resulted of the higher thickness of homogeneous membranes. The worse process yield, not compensated by the noticeably higher separation efficiency evaluated, based on the separation factor β value, suggests that, although homogeneous membranes are very useful in evaluating the selectivity of selected polymers toward compounds of interest, their commercial application may be economically unjustified.

4. Summary

- Typical for pervaporation process *trade-off* phenomena was observed – with the increase of temperature an increase in the total permeate flux was observed in the case of the tested membranes, with a simultaneous decrease of the thiophene separation factor.
- The experiments showed that in the studied range, the concentration of thiophene in separated mixtures had no effect on the efficiency of its removal, which indicated that process was less sensitive to fluctuations of removed components concentration in the feed.
- Prepared PEBA membranes were thiophene selective since calculated $\alpha_{T/O}$ were higher than 1.
- The performance of prepared membranes of PEBA type was comparable with those exhibited by commercial membranes based on the same polymers in terms of separation efficiency evaluated based on β.
- Lower permeate fluxes not compensated by the noticeably higher separation factor values suggest that, although homogeneous membranes are very useful in evaluating the selectivity of selected polymers toward compounds of interest, their commercial application may be economically unjustified.

Symbols

Δm_p — Permeate mass collected over Δt period, kg
S_m — Membrane effective area, m^2
Δt — Time of sample collection, h
w_i^p — Weight fractions of components in the permeate
w_T^p, w_O^p — Weight fractions of thiophene and octane in the permeate, respectively
w_T^f, w_O^f — Weight fractions of thiophene and octane in the feed, respectively
(P_i/l) — Permeance of component *i*
j_i — Molar flux of component *i*
x_i,y_i — Molar fractions of component *i* in feed and permeate, respectively
γ_i — Activity coefficient of component *i*
p_i^f, p_i^p — Saturated vapor pressures of component *i* in feed (*f*) and permeate (*p*)
l — Membrane thickness
p_i^s — Saturated vapor pressure, hPa
A, *B*, *C* — Antoine equation constants
J_P — The total flux or partial flux of individual component, kg/m^2 h
J_0 — Pre-exponential factor
E_{app} — The apparent activation energy of pervaporation, kJ/mol
R — The universal gas constant, J/(mol K)
T — Absolute temperature, K
S_D — Membrane swelling degree, %
W_W — Weight of the membrane in equilibrium state, g
W_D — Weight of dry membrane, g

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