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Application of commercial PEBA-based membranes in separation of sulfur compounds from gasoline by vacuum pervaporation

Katarzyna Rychlewska*, Krystyna Konieczny

Silesian University of Technology, Faculty of Energy and Environmental Engineering, Institute of Water and Wastewater Engineering, 44-100 Gliwice, ul. Konarskiego 18, Poland, emails: katarzyna.rychlewska@polsl.pl (K. Rychlewska), krystyna.konieczny@polsl.pl (K. Konieczny)

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ABSTRACT

The aim of this work was to investigate the potential applicability of PEBA-based membranes in the pervaporation (PV) separation process of sulfur compounds from gasoline. The commercial PEBA membrane (PERVATECH, the Netherlands) was used in the vacuum PV separation process of model hydrocarbon/thiophene mixtures to examine their selectivity and transportation properties. Interactions between typical C_8 hydrocarbon present in gasoline (n-octane, octene and isooctane) and the membrane were studied. The influence of PV process parameters on the efficiency of thiophene removal was also examined. The experiments with chosen C_8 hydrocarbons confirmed that the structure of a component and the resulting differences in the affinity toward the PEBA membrane and physico-chemical properties can influence the membrane performance. Total fluxes for n-octane/thiophene, isooctane/thiophene and octene/thiophene mixtures were equal to 1.33, 1.62 and 1.77 kg m^{-2} h^{-1} at 30°C, with the corresponding separation factor values of 7.00, 6.42 and 5.46, respectively.

Keywords: PEBA membranes; Desulfurization; Vacuum pervaporation; FCC gasoline

1. Introduction

Directive 2009/30/EC of the European Parliament and of the Council of 23 April 2009, and other worldwide specifications demand a reduction of the sulfur content in gasoline and diesel fuels to 10 ppm [1]. Many governments have implemented more stringent regulations that aim at reducing the sulfur content in transportation fuels. It is driven by commitment to improve the environment and air quality as combustion of sulfur compounds present in gasoline is a source of sulfur oxides (SO_x) emissions. Furthermore, the presence of SO_x in engine exhaust gases reduces catalytic converters activity and, indirectly, contributes to higher emissions of NO_x (mono-nitrogen oxides, NO and NO_2) and volatile organic compounds.

Pervaporation (PV) is a membrane technique in which the separation process of mixtures of liquids is based on the difference in the affinity of a specific compound or a class of compounds to the membrane. A solution-diffusion model is commonly used to explain the mass transfer across the dense membrane. The model parameters assume that the dissolution of a substance in the membrane material is a necessary prerequisite for the PV process to occur [2,3]. Good interaction between the removal component and the membrane material is essential for the appropriate separation. After selective dissolution of removal components in the separation layer, the diffusion through the membrane takes place. Permeated compounds are then desorbed and removed on the downstream side of a membrane module.

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* Corresponding author.

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The chemical potential gradient, which is the driving force for the pervaporative separation, is maintained by a continuous removal of the permeated compounds from the membrane [3,4].

Gasoline is a multi-component mixture, which consists of hundreds of compounds. Typically, five hydrocarbon groups, that is, alkanes, isoalkanes, cycloalkanes, aromatics and alkenes, can be found. These hydrocarbon groups demonstrate dissimilar affinity toward the membrane material and have various physico-chemical properties, and for that reason, they can exhibit varied influence on membrane's performance. It has already been shown [5–8] that during the pervaporative separation process, the permeation of one component through the membrane depends significantly on the mixture composition. In other words, the transportation of a compound across the membrane is driven by its own physico-chemical properties as well as by physico-chemical properties of other molecules present in the feed.

To ensure efficient process performance, the separation film material needs to show high affinity toward removed components [4]. Complex and varied content of chemicals in gasoline makes any separation process challenging. Factors like solubility parameter, membrane polarity and physical and chemical properties of the separated compounds provide information about interactions between gasoline components and typical polymers, and can be used in the preliminary selection of membrane materials [9–11]. Typical values of solubility parameters of polymers, hydrocarbon and sulfur species present in gasoline are listed in Table 1.

The aim of the work was to investigate the interactions between typical C_8 hydrocarbons present in gasoline, that is, *n*-octane, octene and isooctane, and the applicability of PEBA membrane material. Also, the influence of hydrocarbon structure on the transportation process of feed components across the membrane and the desulfurization process efficiency was tested.

2. Experimental

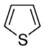
2.1. Materials

In order to prepare model, mixtures simulating gasoline, thiophene (99.5%, extra pure, benzene free, Acros Organics), *n*-octane (pure p.a., Chempur), octene (pure p.a., Acros Organics) and isooctane (pure p.a., Chempur) were used. Physical and chemical characteristics of investigated compounds are listed in Table 2. Composite membranes consisting of a porous support with a thin and dense poly(ether-block-amide) separation layer were obtained from PERVATECH, the Netherlands.

Table 1
Solubility parameters of membrane materials, typical hydrocarbon and typical sulfur species present in gasoline [9,11,12]

Membrane material	δ ((MPa)$^{1/2}$)	Sulfur species	δ ((MPa)$^{1/2}$)	Hydrocarbon	δ ((MPa)$^{1/2}$)
EC	21.10	Thiophene	20.0	*n*-pentane	14.4
PEG	20.10	2-methyl thiophene	19.6	Isopentane	13.8
PVC	26.49	3-methyl thiophene	19.5	*n*-hexane	14.9
PVP	20.56	Dimethyl thiophene	19.3	*n*-heptane	15.3
PVB	23.12	Trimethyl thiophene	19.2	*n*-octane	15.5
PDMS	21.01	Diethyl thiophene	19.2	Isooctane	14.2
PU	20.98	Triethyl thiophene	19.0	Octene	18.7
CA	25.06	Thioether	16.8	Cyclopentane	16.6
PAN	26.61	Dimethyl sulfone	29.8	Cyclohexane	16.7
PTFEP	15.38	Sulfide	16.9	Benzene	18.7
PUU	20.98	Disulfide	17.4	Toluene	18.2
PI	32.30	*n*-butyl sulfide	28.1	*m*-xylene	18.2
CTA	24.55	*n*-butyl mercaptan	18.4	*o*-xylene	18.5
PEBA	19.51	Benzyl mercaptan	21.1	*p*-xylene	18.1

Table 2
Physico-chemical characteristics of investigated compounds

Compound		Structural formula	Molecular weight (g mol^{-1})	Bp (°C)	Vapor pressure (hPa)
Hydrocarbon	*n*-octane	$CH_3(CH_2)_6CH_3$	114.23	125	14.7
	Octene	$CH_3(CH_2)_5CH{=}CH_2$	112.21	121	20
	Isooctane	$CH_3C(CH_3)_2CH_2CH(CH_3)CH_3$	114.23	99	55
Sulfur compound	Thiophene		84.14	84	53

2.2. *Vacuum pervaporation experiments*

The laboratory-scale PV setup equipped with a 9-cm-diameter membrane cell was used in experiments. The membrane's effective area was 55 cm^2. To prevent damage, the membrane was kept on a highly porous stainless steel support. The gasoline model mixture was circulated to the upstream side of the membrane cell. Vapors of permeate were condensed in glass cold traps immersed in Dewar flasks filled with liquid nitrogen, while the retentate was pumped back to the feed tank. The cold traps were then brought to room temperature in order to measure the weight of the collected permeate. Feed flow rate was kept at a constant level and maintained using the recirculation pump. The membrane upstream side was kept at atmospheric pressure while in the downstream side the vacuum was created using a vacuum pump (Alcatel, model Pascal 2015 SD) to provide a pressure gradient across the membrane. Frozen permeates were collected at pre-determined time intervals. During the process, the feed temperature was maintained at a desired level by a thermostat (model Haake DC 30, Thermo Electron Co., USA) and controlled through a digital thermometer device (Greisinger Electronics, GTH 1100/2 DIF). The total downstream pressure was measured by an electronic vacuum gauge (DVR2, Vacuubrand, Germany).

The impact of sulfur compound concentration in the model gasoline on the PV performance in the PEBA-based membrane was investigated in the range of 0.05–0.13 wt.%. The influence of operating temperature ranging from 30°C to 50°C on membrane's performance was also examined. All experiments were conducted using one piece of membrane, which has shown high stability. Small amount of permeates were collected at set time intervals to simulate a continuous process and keep feed concentration at a constant level.

SRI 8610C instruments gas chromatograph equipped with a dual FID/FPD (flame ionization detector/flame photometric detector) and a Restek DB-1 MTX capillary column (0.5 µm, 0.53 mm × 60 m) was used for feed and permeate samples analyses. The retention time for thiophene was 3.38 ± 0.01 min. The relative standard deviation (RDS, %) for *n*-octane/thiophene, octene/thiophene and isooctane/thiophene were equal to 1.14%, 1.87% and 1.25%, respectively.

The PV process yield was evaluated using the permeation flux J_m that can be determined as total permeate flux and partial flux of individual components (1):

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

where m_p is the permeate weight, kg; S_m is the membrane effective area, m^2; and t is the time, h.

To evaluate the efficiency and selectivity of the sulfur compound separation process, the dimensionless separation factor β was calculated (2):

$$\beta = \frac{w_T^p / w_H^p}{w_T^f / w_H^f} \tag{2}$$

where w^p_T and w^p_H are the weight fractions of thiophene and hydrocarbon, respectively, in the permeate; w^f_T and w^f_H are the weight fractions of thiophene and hydrocarbon, respectively, in the feed.

3. Results and discussion

3.1. *Influence of operational feed temperature*

The experimental results have shown positive correlation between the operating temperature and both total (Fig. 1) and partial fluxes of model gasoline components (Fig. 3). During the experiments, a typical PV trade-off phenomena between flux and selectivity has occurred, as previously reported in [12–16]. As the feed temperature increased, the total flux increased but it resulted in the decline in the sulfur separation factor (Fig. 2).

Results presented in Figs. 1 and 2 have demonstrated that temperature had a more substantial impact on the PV yield than on the separation efficiency. The trend is that the total flux has increased significantly while the separation factor has decreased at a lower rate. Partial fluxes of sulfur compounds have also been increasing with the feed temperature (Fig. 3) but a more significant increase in the hydrocarbon flux lead to lower values of the thiophene separation factor.

Previous research [3,4] has established that the operational temperature is a parameter of great importance for the PV process. Above-mentioned relationships are thought to be a consequence of two simultaneously occurring phenomenon. First, the operating temperature affects the driving force for the mass transfer, that is, 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 as the downstream pressure remains practically unaltered. Secondly, the temperature rise additionally enhances the swelling of the membrane active layer, which, on one hand, is a "natural" consequence of feed

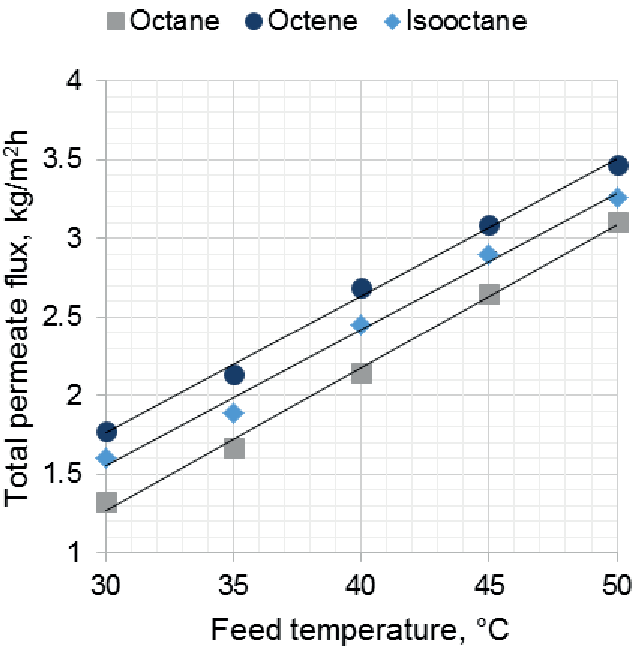


Fig. 1. Influence of feed temperature on the total permeation flux at feed concentration of 0.07 wt.% and permeate pressure of 100 Pa.

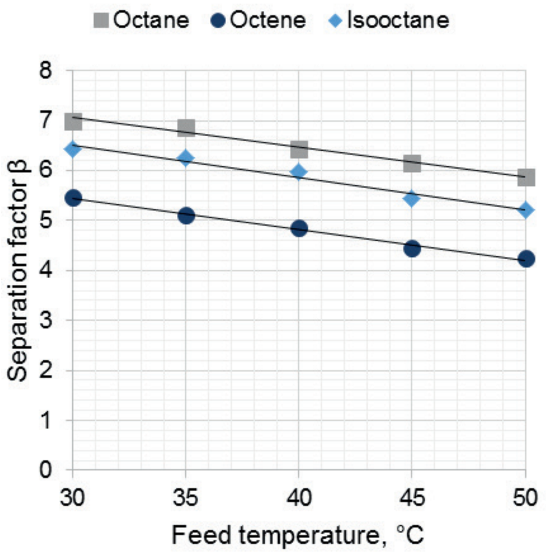


Fig. 2. Influence of operating temperature on the separation factor β at thiophene concentration of 0.07 wt.% and permeate pressure of 100 Pa.

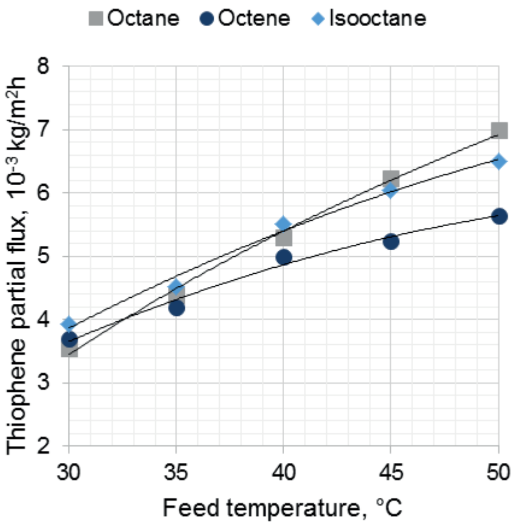


Fig. 3. Influence of feed temperature on thiophene partial flux at feed concentration of 0.07 wt.% and permeate pressure of 100 Pa.

components dissolution and, on the other, can be responsible for the decrease in thiophene separation factor [9,10,12,14]. Nevertheless, promising findings of Yang et al. [15] have demonstrated that self-cross-linking of polyphosphazene membranes during its storage at room temperature can vanquish the trade-off effect. Furthermore, Yang et al. [16] have shown that in the case of polyimide asymmetric membrane application the enrichment factor for sulfur compound was stable in tested range of temperatures (50°C–90°C), while the yield of the PV process was enhanced.

The apparent activation energy, that is, the minimum energy, which is required for the permeation to occur, can be found using the Arrhenius-type plot [17] and the following equation:

$$J_m = J_0 \exp\left(-E_{appa}/RT\right) \tag{3}$$

where J_m is the total flux or partial flux of individual component, kg m^{-2} h^{-1}; J_0 is the pre-exponential factor; E_{appa} is the apparent activation energy of PV, kJ mol^{-1}; R is the universal gas constant J (mol·K)$^{-1}$; T is the absolute temperature, K. This experimentally determined value, which can be found from ln(J_m) vs. 1,000/T plots, provides information about the susceptibility of the flux/permeability to temperature changes [6,10,11]. Relationship between ln(J_m) and 1,000/T for tested hydrocarbons and thiophene are shown in Table 3 and Figs. 4 and 5. Calculated apparent activation energies are listed in Table 4.

According to references [6,17], linearity of obtained relationships and positive values of apparent activation energies for the tested model mixtures components (Table 4) indicates that first, the performance of the PEBA membrane obeyed the Arrhenius law, and secondly, the increase in process temperature enhanced the permeability of feed components and resulted in higher total and partial fluxes.

Lower E_{appa} values for thiophene in comparison with those found for other mixture components (see Table 4) confirmed its higher affinity to the selective layer material of the membrane used (solubility parameter δ equal to 20.0 for thiophene and 19.51 for PEBA). It was found that thiophene's transport across the membrane required less energy [6,11,17].

Diverse activation energies with the structure of C_8 hydrocarbon have been obtained, and the activation energy of octene was lower when compared with those of isooctane and *n*-octane. E_{appa} values of tested hydrocarbons were in the

Table 3
Ln(J_m) vs. 1,000/T correlation

Compound	Mixture	Pre-exponential factor J_0	Ln(J_m) vs. 1,000/T correlation	Correlation coefficient R^a
n-octane	*n*-octane/thiophene	1.59E + 09	$y = -4.2379x + 21.1886$	0.9958
Octene	Octene/thiophene	1.24E + 08	$y = -3.3752x + 18.6356$	0.9872
Isooctane	Isooctane/thiophene	2.16E + 08	$y = -3.5767x + 19.1905$	0.9901
Thiophene	*n*-octane/thiophene	3.93E + 05	$y = -3.3307x + 12.8819$	0.9890
	Octene/thiophene	7.72E + 03	$y = -2.1223x + 8.9514$	0.9543
	Isooctane/thiophene	1.59E + 04	$y = -2.30983x + 8.6769$	0.9676

[a] Significance level $\alpha = 0.05$.

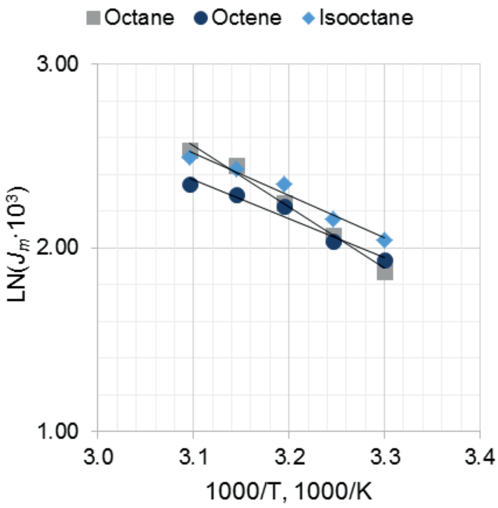


Fig. 4. Arrhenius-type relationship for hydrocarbon's partial fluxes at thiophene concentration of 0.07 wt.% and permeate pressure of 100 Pa.

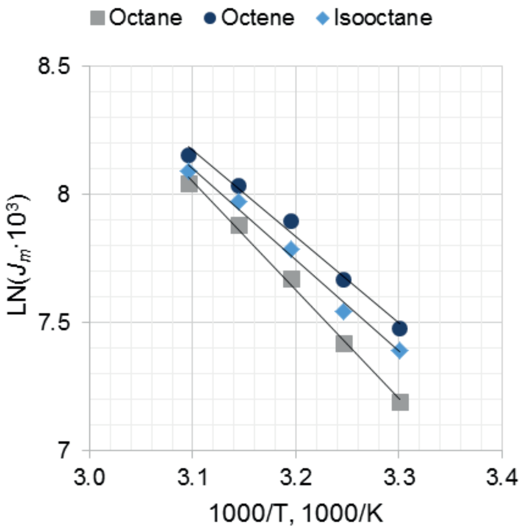


Fig. 5. Arrhenius-type relationship for the thiophene partial fluxes at thiophene concentration of 0.07 wt.% and permeate pressure of 100 Pa.

Table 4
Calculated apparent activation energies of individual components

Compound	Mixture	E_{appa} (kJ mol^{-1})
n-octane	*n*-octane/thiophene	34.23 ± 0.16
Octene	Octene/thiophene	28.06 ± 0.22
Isooctane	Isooctane/thiophene	29.74 ± 0.21
Thiophene	*n*-octane/thiophene	27.69 ± 0.20
	Octene/thiophene	17.64 ± 0.26
	Isooctane/thiophene	19.20 ± 0.24

following order: octane > isooctane > *n*-octane. The observed trend in the case of *n*-octane and octene is consistent with the solubility parameter theory, which confirms that this theory can be used for membrane material pre-selection. δ values for hydrocarbons listed in Table 1 (15.5 for *n*-octane and 18.7 for octene) indicate that octene exhibits the highest affinity toward the tested membrane material (19.51 for PEBA). This and higher apparent activation energy of octene can be the reason of the lowest separation factor values obtained in the case of octene/thiophene mixture. Results of sulfur compound removal from isooctane/thiophene solution demonstrated that apart from the solubility parameter, the structure of main components and its physico-chemical properties are also very important factors. Despite its lowest affinity for PEBA membrane (δ = 14.2 for isooctane), the activation energy of chosen isoalkane was lower when compared with E_{appa} for *n*-octane. Furthermore, a branched chain of isooctane, which hampered its diffusion, resulted in lower fluxes (in comparison with octene) despite its higher vapor pressure and lower boiling point (Table 2).

Because of higher activation energy obtained for hydrocarbons, their fluxes were more sensitive to temperature changes in comparison with thiophene [6,11]. The partial flux of hydrocarbons across the membrane was influenced by the temperature to a greater extent in comparison with sulfur compounds. The separation factor decreased (Fig. 2) because of the change in the hydrocarbon to thiophene flux ratio [10,11].

3.2. Influence of sulfur concentration in feed

The impact of sulfur compound concentration in model gasoline on the PV process performance through PEBA-based membranes at 30°C was investigated in the range of 0.05–0.13 wt.%.

Fig. 6 shows the influence of feed concentration on the total permeation flux for all binary hydrocarbon/thiophene mixtures at the feed temperature of 30°C. The change of sulfur concentration in the tested range produced insignificant impact on the total flux for all tested mixtures. As it can be

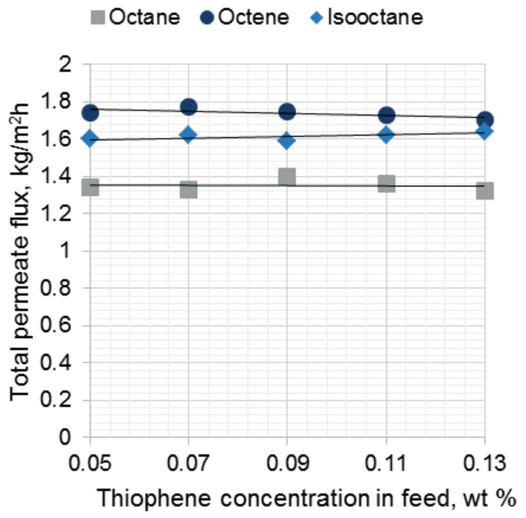


Fig. 6. Influence of feed concentration on the total permeate flux at feed temperature of 30°C and permeate pressure of 100 Pa.

seen in Fig. 7, the selectivity was also not influenced by the variation of sulfur compound levels in the feed. Nonetheless, partial fluxes of sulfur compounds were proportional to the sulfur concentration (Fig. 8), which leads to nearly unchanged values for the thiophene separation factor.

The results are consistent with data obtained by other researchers [13,18–20]. The above-presented relationship may be a consequence of the very narrow range of thiophene's concentration and the fact that the thiophene concentration in comparison with hydrocarbons was very low. Taking into account the solution–diffusion theory, the separation attained during the PV process is determined by the initial two stages, that is, sorption and diffusion of mixture components. It is suspected that the concentration of sulfur compounds was beyond the range, where the effect of swelling caused by thiophene surpasses the swelling phenomena resulted from hydrocarbons' presence [10,11]. Previously obtained results on the separation of hydrocarbon/thiophene mixtures using the poly(dimethylsiloxane)-based membranes [21] seem to confirm the above explanation. With the increase in the sulfur content in the feed from 0.8 to 1.6 wt.%, an increase in the total permeate flux was observed. Simultaneously, the increase in total permeation flux resulted in the enrichment factor decrease.

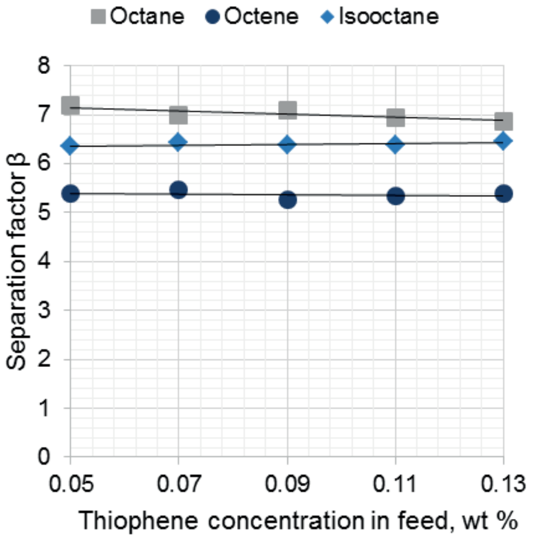


Fig. 7. Influence of feed concentration on the separation factor at feed temperature of 30°C and permeate pressure of 100 Pa.

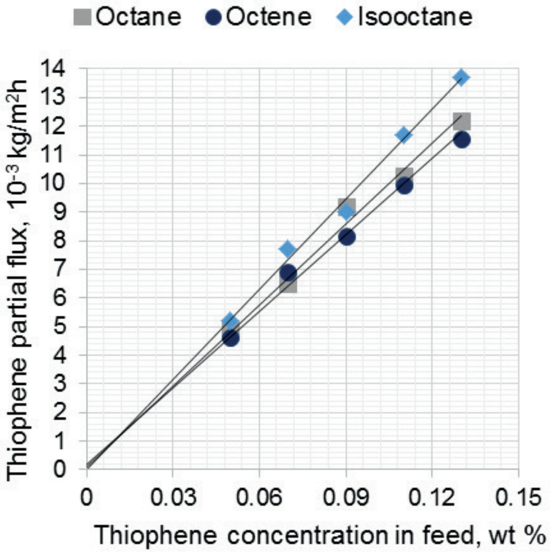


Fig. 8. Influence of feed concentration on the partial flux of thiophene at feed temperature of 30°C and permeate pressure of 100 Pa.

4. Conclusions

Conducted experiments with chosen C_8 hydrocarbons confirmed that the structure and physico-chemical properties of a component and the resulting differences in the affinity toward the PEBA membrane can influence membrane's performance. As demonstrated in Figs. 2 and 7, separation factor results for tested hydrocarbon/thiophene mixtures increased in the following order: octane/thiophene < isooctane/thiophene < *n*-octane/thiophene. As it can be seen in Figs. 1 and 6, the total permeation flux in case of alkene/thiophene mixture, with octene as the representative olefin hydrocarbon, was higher than that obtained for *n*-alkane/thiophene and isoalkane/thiophene mixtures, which were represented by *n*-octane and isooctane, respectively.

The branched chain of isooctane, which hampered its diffusion, resulted in lower fluxes (in comparison with octene) despite its higher vapor pressure and lower boiling point (Table 2). The presence of a double bond in the octene molecule resulted in its higher affinity to the PEBA membrane [10], and it was the reason of a less efficient separation process. According to data presented in references [7,8], olefin, along with aromatics, contributes to the higher swelling of a separation layer. The above-mentioned swelling phenomena results in the higher flexibility of polymer structure and, in consequence, in the rise of free volume available for the diffusion [3,4]. On the other hand, as it was shown in Figs. 3 and 8, the variations in hydrocarbons' content had also a small impact on the partial flux of thiophene across the PEBA membrane. It is in line with the effect of interactions between the permeating molecules on membrane's performance reported also for other type of membranes in references [6–8,22]. Based on the obtained results and those presented in references, it can be assumed that in the case of binary and more complex organic-organic mixtures, the component presented "in excess" has the dominant influence on the separation efficiency.

Differences in chemical affinity, structure and physico-chemical properties of the investigated hydrocarbons led to various selectivities toward the PEBA membrane and diversity in hydrocarbon and thiophene activation energy levels for all tested mixtures. Partial fluxes of thiophene were proportional to the concentration of thiophene in the feed. However, the variation of thiophene concentration had a negligible influence on the total flux and selectivity in case of all model mixtures. During the experiments, a well-documented PV trade-off phenomena between the flux and selectivity occurred [12–16]. Namely, as the feed temperature increased, the total flux increased but at the same time, the sulfur separation factor has declined.

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