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Research Paper

Investigation of the Influence of Operating Conditions on the Removal of 2-ethyltoluene from Water Using PDMS Pervaporation Membrane

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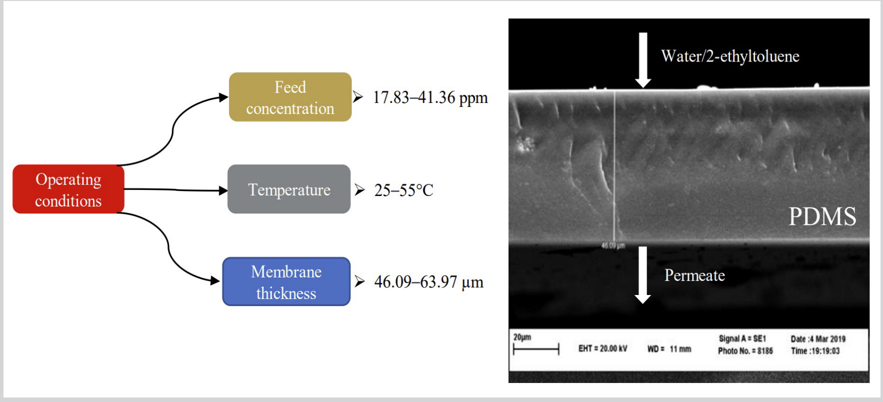
Highlights

- PDMS pervaporation membrane was used for the removal of 2-ethyltoluene from water.
- The effect of VOC concentration, temperature, and membrane thickness on the separation process was investigated.
- Increasing the concentration and temperature led to higher removal efficiency.

Graphical abstract



Abstract

Due to environmental issues, the separation of volatile organic compounds (VOCs) from water is of great importance. Hence, in the current study, polydimethylsiloxane (PDMS) pervaporation membrane was prepared to remove the 2-ethyltoluene from water and characterized using analysis methods such as scanning electron microscopy (SEM). The effect of feed concentration, membrane thickness, and feed temperature on the flux of permeation and the separation factor was investigated via pervaporation experiments. As the concentration of 2-ethyltoluene augmented from 17.83 to 41.36 ppm, the 2-ethyltoluene permeation flux was enhanced by approximately 413%, whereas the water permeation flux and the separation factor were raised by about 9% and 104%, respectively. Moreover, the swelling degree and pervaporation separation index (PSI) increased by almost 191% and 139%, respectively. An augmentation in water flux, 2-ethyltoluene flux, and PSI, by about 33%, 26%, and 27 %, respectively, occurred as a result of increasing the feed temperature from 25 to 55 °C. However, the separation factor was decreased by almost 4%.

1. Introduction

In recent years, the separation of isomeric, azeotropic, and close-boiling liquid mixtures has been investigated by pervaporation [1–3]. This process is highly selective, economical, and flexible in operation conditions [4]. The application of pervaporation is in the purification of chemicals such as separation of organic mixtures, organic solvents dehydration, and organic components recovery from aqueous solution [5, 6]. In pervaporation, the membrane permeability is evaluated by the solubility and diffusivity of the component that permeates the membrane [3]. In the pervaporation process, feed components are sorbed at the upstream side of the membrane and diffuse through the membrane; then, after desorption of the components at the other side of the membrane, evaporated permeate is collected by a condenser [5, 7].

Ethyltoluenes are organic compounds that are used in chemical and petrochemical industries [8, 9]. 2-ethyltoluene is categorized as a volatile organic compound (VOC) that exists in urban, industrial, and residential regions [10] in several applications such as air fresheners, decor, and toys [11].

With population growth and the shortage of freshwater resources, the treatment of water and wastewater has been found much importance in many world regions [12]. Although some methods such as carbon adsorption and air stripping are used to separate the VOCs from aqueous solutions, these methods are economic only at low concentrations of VOCs; so pervaporation

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is a suitable choice for removal of VOCs due to economic and technical reasons [13].

For this purpose, it is better to use a hydrophobic pervaporation membrane such as polydimethylsiloxane (PDMS) [4]. PDMS membrane offers many advantages, such as good operational stability [14]; high permselectivity, and diffusivity of organics, which make it an attractive candidate for the removal of VOCs [13]. Besides, PDMS has proper chemical, thermal, and mechanical stability [12].

The effects of operating conditions on the separation performance of pervaporation membranes have been investigated in many research works. In the study performed by Sampranpiboon et al. [15], the effects of feed temperature and concentration were investigated on the pervaporation separation of ethyl butyrate from water. Based on their results, the separation factor and the permeation flux were enhanced by increasing the concentration of ethyl butyrate. Bakhshi et al. [16] investigated the effects of feed temperature on pervaporation separation of methanol from water. Their results indicated that by increasing the feed temperature, the separation factor was decreased. Gonzalez-Marcos et al. [17] focused on the influence of poly (1-trimethylsilyl-1-propyne) (PTMSP) membrane thickness and feed temperature on the performance of the ethanol-water pervaporation process. Their results indicated that the flux was increased with decreasing the thickness. Increasing the permeability of ethanol and water with the increment of the feed temperature, despite the unchanged selectivity, was another finding of this research group. Hao et al. [18] evaluated the effects of feed temperature and concentration on the pervaporation separation of phenol from water. With respect to their results, a suitable permeation separation factor was obtained for this separation process. They also found that the permeation flux of phenol was increased with the enhancement of its concentration in the feed. In another study, Yi and Wan [19] examined the pervaporative separation of methanol from a multi-component solution at different feed concentrations and temperatures with mixed matrix membranes. Regarding their results, at a specified temperature, with rising the VOC concentration of the feed, the total flux was enhanced. Furthermore, they found that the methanol concentration and feed temperature had a slight effect on the separation factor of the membrane. Matavos-Aramyan et al. [20] used the pervaporation membranes of polyether-block-amide (PEBA) and PEBA/NaX nanozeolite to separate the toluene from water. Their results indicated that by increasing the toluene concentration, the permeation flux was enhanced. Dong et al. [4] investigated the effect of the PDMS layer thickness, operation temperature, and feed concentration on the pervaporation performance of ceramic hollow fiber supported PDMS composite membrane in the recovery of n-butanol from water. Another pervaporation study for dewatering n-butanol through mixed matrix membranes was performed by Wang et al. [21] in which the effect of feed temperature and concentration on the separation performance were investigated. Their research outcome implied rising temperature and water concentration increase total flux while decreasing the separation factor.

Despite the aforementioned research, there is no study in the field on the influence of operating conditions on the pervaporative separation of 2-ethyltoluene from water using the PDMS membrane.

Thus, in the present study, after the preparation of the PDMS pervaporation membrane for the separation of 2-ethyltoluene from water, the obtained membrane was characterized by applying the analysis methods of scanning electron microscopy (SEM) and determination of swelling degree. Furthermore, the effects of the feed concentration and temperature, as well as the membrane thickness on the permeate flux, separation factor, and pervaporation separation index (PSI), were investigated. In addition, the influence of the feed concentration was investigated on the swelling degree.

2. Materials and methods

2.1. Materials

2-ethyltoluene was purchased from Merck, Germany, for the preparation of feed using distilled water. RTV-2 silicone rubber (BISIL 4125 A/B) was provided by Bitex Chemical Industry, Turkey, to prepare the polydimethylsiloxane (PDMS) membrane.

2.2. Membrane preparation

For the preparation of PDMS polymeric solution with the equal mass ratio of the two components of A and B, 1.5 g of A and 1.5 g of B were added to a vial, and the solution was stirred at room temperature by a magnetic stirrer at low speed. Stirring continued for 1h to obtain a homogeneous solution. With the aim of degassing, the solution was allowed to stay immobile for 15 minutes. The solution casting method was used for membrane preparation. At first, the solution was cast onto a flat glass plate. Afterward, the membrane was put in an oven at 100 °C for 30 minutes and then was removed from the plate. The membranes were made in four thicknesses of 46.09, 52.03, 58.41, and 63.97 µm.

2.3. Scanning electron microscopy (SEM)

The membranes were characterized using scanning electron microscopy (LEO 1450 VP, Germany) to investigate their morphology. For the cross-sectional views, all the samples were fractured in liquid nitrogen to obtain a smooth cross-section.

2.4. Pervaporation experiments

A membrane set-up including a feed tank, a circulation pump, a vacuum pump, a membrane cell, and a cold trap was used for performing the pervaporation experiments. The flat and circular membrane was fixed in the stainless-steel membrane cell with an effective membrane area of 15.89 cm^2. A vacuum pump kept the downstream pressure at 15 mmHg. It took about 15 minutes to reach the system's steady state. The pervaporation operation was run for 1h, and each experiment was repeated three times with the average results being reported. The tests were performed at four different temperatures of 25, 35, 45, and 55 °C and four various 2-ethyltoluene concentrations of 17.83, 25.86, 34.41, and 41.36 ppm in water. After collecting the permeate using the cold trap, the permeation rate was calculated by measuring the weight of the permeate. The concentration of components in the permeate and feed was determined by a UV-spectrophotometer (SP-UV 300SRB instrument, Germany). The pervaporation membrane performance was investigated using three parameters of permeation flux (J), separation factor (α), and PSI as follows [20]:

$$J = \frac{W}{A \times t} \tag{1}$$

$$\alpha = \frac{Y/(1-Y)}{X/(1-X)} \tag{2}$$

$$PSI = J(\alpha - 1) \tag{3}$$

where W (g) is the permeate sample mass that accumulated in a period of t (h), and A (m^2) is the membrane effective area. X and Y are the weight fractions of 2-ethyltoluene in the feed and permeate, respectively. The values for water flux and 2-ethyltoluene flux were determined using Equation 1. Subsequently, the total flux was calculated by summing these two values.

2.5. Swelling test

For the swelling test, the weight of a dry and clean membrane sample was measured, and then the sample was placed in the 2-ethyltoluene/water solution for 72 h. After achieving the equilibrium swelling, the surface of the sample was dried with tissue papers and its weight was measured in this situation. The swelling degree (SD) of the membrane sample was calculated by the following equation [22]:

$$SD(\%) = \frac{W_{wet} - W_{dry}}{W_{dry}} \times 100 \tag{4}$$

where W_{dry} (g) is the dry membrane weight and W_{wet} (g) is the weight of the membrane after swelling. The test was performed in four various 2-ethyltoluene concentrations of 17.83, 25.86, 34.41, and 41.36 ppm in water.

3. Results and discussion

3.1 Morphology of the membranes

SEM surface images of the PDMS membranes with different thicknesses are shown in Fig. 1. Regarding Fig. 1, the surface of the membranes is completely dense and uniform. Fig. 2 illustrates the cross-sectional images of the membranes. As can be seen in Fig. 2, the membranes have thicknesses of 46.09, 52.03, 58.41, and 63.97 µm and dense structure.

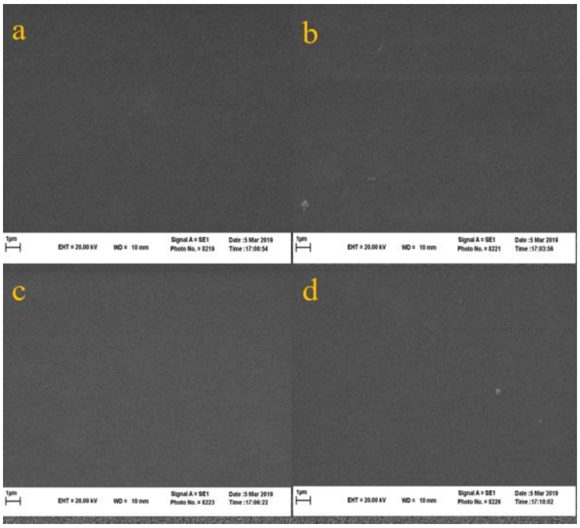

Fig. 1. SEM surface images of the membranes with different thicknesses: a) 46.09 µm, b) 52.03 µm, c) 58.41µm, and d) 63.97µm

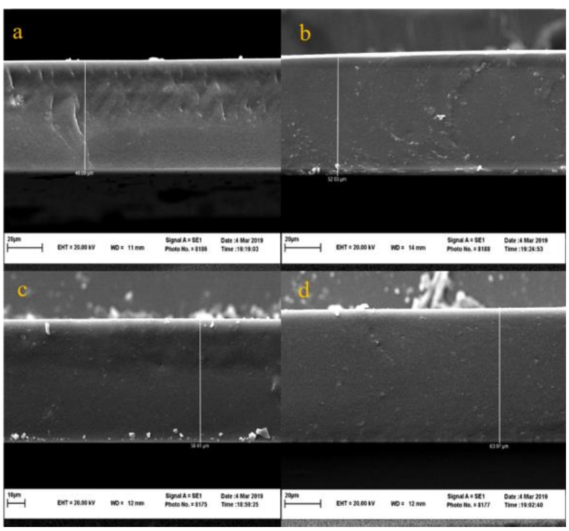

Fig. 2. SEM cross-sectional images of the membranes with different thicknesses: a) 46.09 µm, b) 52.03 µm, c) 58.41µm, and d) 63.97µm

3.2. Effect of feed concentration

The swelling degree of the PDMS membrane with a thickness of 52.03 µm was evaluated in the 2-ethyltoluene concentrations of 17.83, 25.86, 34.41, and 41.36 ppm at 25°C.

In the feed solution, if the polarity difference between the membrane and the target substance is lower than that of the membrane and the other substance in the feed, the membrane absorbs the target substance more and swells more by that substance [23]. The polarity difference between the PDMS membrane and 2-ethyltoluene is less than that of the PDMS membrane and water. Therefore, the PDMS membrane absorbs 2-ethyltoluene more and swells more by this component.

By raising the 2-ethyltoluene concentration from 17.83 to 41.36 ppm, a further amount of 2-ethyltoluene was sorbed by the membrane, and as can be seen in Table 1, the swelling degree of the membrane was augmented from 1.40 to 4.08.

Table 1
Swelling degree of the PDMS membrane for the various 2-ethyltoluene concentrations in the feed (thickness: 52.03 µm, temperature: 25°C)

2-Ethyltoluene concentration (ppm)	Degree of swelling (%)
17.83	1.40
25.86	2.63
34.41	3.48
41.36	4.08

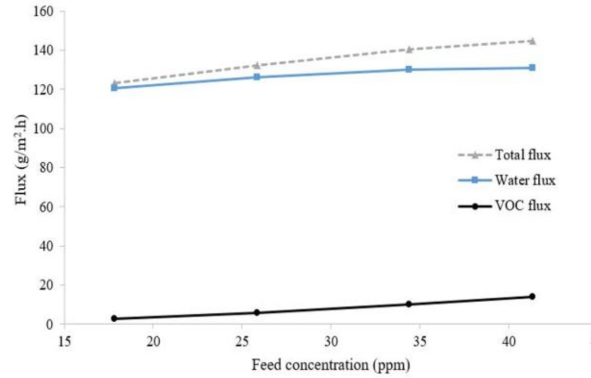

Fig. 3. The effect of feed concentration on 2-ethyltoluene, water, and total fluxes (membrane thickness: 52.03 µm, feed temperature: 25 °C)

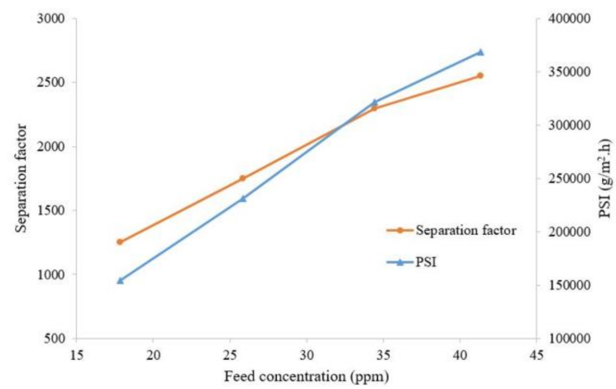

Fig. 4. The effect of feed concentration on the separation factor and PSI (membrane thickness: 52.03 µm, feed temperature: 25 °C)

In the pervaporative separation of 2-ethyltoluene from water, the effect of 2-ethyltoluene concentration in the feed on the separation factor and flux was investigated using the PDMS membrane with a thickness of 52.03 µm at room temperature. As mentioned earlier, when the feed concentration increased, the PDMS membrane swelled more, and the resultant higher mobility of polymer chains increased the diffusion rate of components [23].

As shown in Fig. 3, when the concentration of 2-ethyltoluene increased from 17.83 to 41.36 ppm, the 2-ethyltoluene permeation flux was enhanced from 2.69 to 13.79 g/m^2.h, whereas the water permeation flux was risen from 120.60 to 130.89 g/m^2.h. With an increment in the concentration of 2-ethyltoluene in the feed, further augmentation was observed in the 2-ethyltoluene permeation flux compared to the water permeation flux. Consequently, as can be seen in Fig. 4, an increment of 2-ethyltoluene concentration from 17.83 to 41.36 ppm increased the separation factor from 1251.19 to 2547.44. In the study performed by Sampranpiboon et al. [15], the separation factor was enhanced by increasing the concentration of ethyl butyrate. As shown in Fig. 4, by increasing the 2-ethyltoluene concentration from 17.83 to 41.36 ppm in the feed, PSI was significantly improved from 154144.91 to 368433.63 g/m^2.h due to the simultaneous effect of separation factor and flux on the membrane performance.

3.3 Effect of temperature

The effect of feed temperature on 2-ethyltoluene separation by pervaporation process was investigated using the membrane with a thickness of 58.41 µm at the feed concentration of 34.41 ppm. Fig. 5 demonstrates that raising the feed temperature from 25 to 55°C led to the improvement of the total flux as well as the flux of water and 2-ethyltoluene from 116.38 to 154.13 g/m^2.h, 107.63 to 143.12 g/m^2.h, and 8.75 to 11.01g/m^2.h, respectively. The enhanced movement of polymer chains in the amorphous regions induced by temperature increase augmented the free volumes of the membrane; therefore, more molecules passed through the membrane, and the diffusion rates were improved [16]. Furthermore, as the temperature increased, the driving force of mass transfer was enhanced [24]. This force is based on the difference in the partial vapor pressure of components across the membrane [25]. The components' partial vapor pressure on the permeate side stays approximately constant with increasing the feed temperature, whereas in the feed section, augmenting the temperature results in higher partial pressure of the components and consequently increases the driving force of mass transfer [24].

With respect to Fig. 5, 2-ethyltoluene flux was improved much lower than water flux. The reason is based on the dependence of components flux to feed temperature. For this purpose, the following Arrhenius expression is applied [4]:

$$J = J_0 \exp(\frac{-E_J}{RT}) \tag{5}$$

where J ($g/m^2.h$) is the permeation flux, J_0 ($g/m^2.h$) is the constant parameter, E_J (kJ/mol) is the average activation energy, R (kJ/K.mol) is the gas constant, and T (K) is the temperature. E_J can be defined as a compounded parameter characterizing heat of sorption of the permeants' molecules in the membrane and diffusion of the molecules through the membrane with respect to the solution-diffusion model [17]. This parameter is determined by the slope of the Arrhenius plot. Different parameters such as the size of permeant molecules, the polarity of permeation components, the nature of the membrane, and the affinity between the membrane and the permeants result in different activation energies [19]. Fig. 6 indicates the Arrhenius plot of 2-ethyltoluene and water fluxes against the reciprocal of temperature. The activation energies for the permeation of water and 2-ethyltoluene were obtained as 7.45 and 6.04 kJ/mol, respectively. According to the fact that the flux of the component with higher activation energy is more susceptible to temperature variation [4], the temperature dependence of water flux was more than that of 2-ethyltoluene flux because of the higher activation energy for water, and as a result, the separation factor was reduced with enhancing the temperature. Thus, as can be seen in Fig. 7, the separation factor was reduced from 2290.29 to 2199.35 with the augmentation of feed temperature from 25 to 55°C. However, PSI rose from 266423.14 to 338814.30 $g/m^2.h$ with the increase of feed temperature from 25 to 55°C due to the significant increase of total flux.

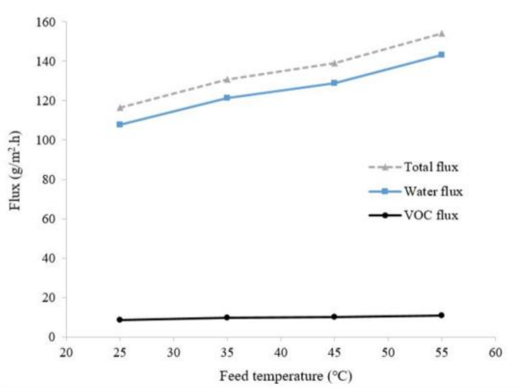

Fig. 5. The effect of feed temperature on 2-ethyltoluene, water, and total fluxes (feed concentration: 34.41 ppm, membrane thickness: 58.41 µm)

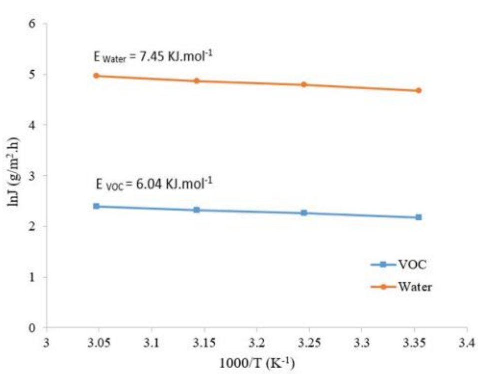

Fig. 6. Arrhenius plots of components flux

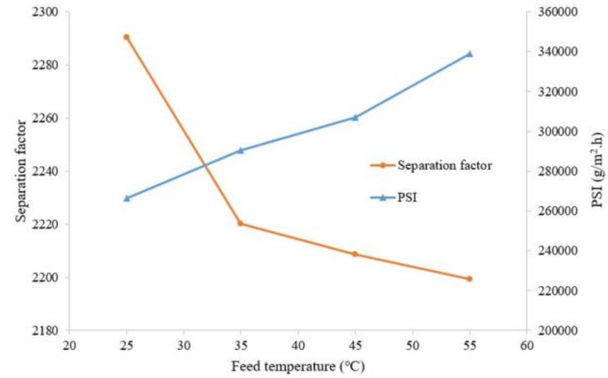

Fig. 7. The effect of feed temperature on the separation factor and PSI (feed concentration: 34.41 ppm, membrane thickness: 58.41 µm)

3.4 Effect of membrane thickness

The mass transfer resistance of a pervaporation membrane is proportional to its thickness [5, 26]. Since there is an inverse proportion between permeation flux and the membrane thickness, thin membranes are commonly used to reach high fluxes; however, the limitations imposed by either the fabrication methods or the mechanical resistance of the membrane allow for the reduction of membrane thickness to a certain limit [26]. Hence, the effect of the membrane thickness on permeation flux and separation factor was investigated using the membranes with thicknesses of 46.09, 52.03, 58.41, and 63.97 µm.

Fig. 8 shows the permeation flux as a function of the membrane thickness. The pervaporation results were collected at room temperature and the 2-ethyltoluene concentration of 34.41 ppm. As shown in Fig. 8, by increasing the membrane thickness from 46.09 to 63.97 µm, the flux of 2-ethyltoluene and water was decreased from 11.77 to 7.74 $g/m^2.h$ and 158.07 to 86.62 $g/m^2.h$, respectively. With increasing the thickness, the molecules have to diffuse through a longer path, the diffusion is retarded, and thus the flux is reduced [27]. Furthermore, the variation of water flux was more than that of VOC flux, because the restriction of water transport occurs by the membrane, whereas VOC transport is partly restricted by the liquid boundary-layer resistance, therefore by increasing the membrane thickness, the separation factor, and the concentration of VOC in permeate were enhanced [26]. As can be seen in Fig. 9, by augmenting the membrane thickness from 46.09 to 63.97 µm, the separation factor was enhanced from 2068.08 to 2615.96. according to the results, the effect of the flux was more pronounced than that of the separation factor. Therefore, by increasing the membrane thickness from 46.09 to 63.97 µm, the PSI was reduced from 351089.83 to 246748.52 $g/m^2.h$.

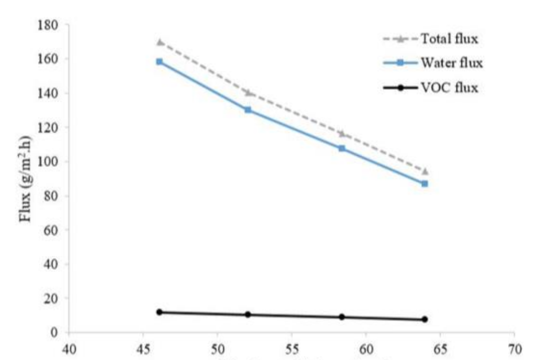

Fig. 8. The effect of membrane thickness on 2-ethyltoluene, water, and total fluxes (feed concentration: 34.41 ppm, feed temperature: 25 °C)

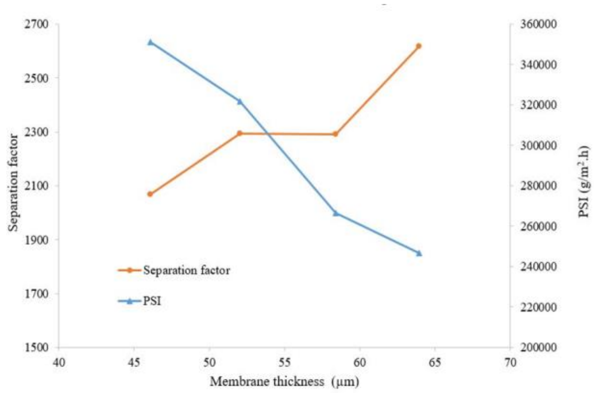

Fig. 9. The effect of membrane thickness on the separation factor and PSI (feed concentration: 34.41 ppm, feed temperature: 25 °C)

4. Conclusion

In this study, the pervaporative separation of 2-ethyltoluene from water using a PDMS membrane was investigated. When the concentration of 2-ethyltoluene in the feed was augmented, 2-ethyltoluene permeation flux, separation factor, PSI, and swelling degree were enhanced significantly. An increment in the feed temperature resulted in the augmentation of water flux, 2-ethyltoluene flux, and PSI, whereas the separation factor was reduced. The effect of membrane thickness on the permeation flux and separation factor was also investigated, and based on the obtained results, by increasing the membrane thickness, 2-ethyltoluene permeation flux, water permeation flux, and PSI were decreased, and the separation factor was enhanced. The obtained results indicated that the PDMS membrane separated 2-ethyltoluene from water successfully.

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CRedit authorship contribution statement

F. Fardoust: Conceptualization; Data curation; Formal analysis; Investigation; Methodology; Writing – review & editing; Visualization.
M. Cheraqi: Data curation; Formal analysis; Investigation; Methodology; Writing – review & editing; Visualization.
M. Doaei: Writing – original draft; Visualization; Validation; Writing – review & editing.

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Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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