



Separation of azeotropic mixture acetone + hexane by using polydimethylsiloxane membrane



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ABSTRACT

We report on possibilities of separation of the azeotropic mixture of acetone + hexane *via* sorption and pervaporation techniques using polydimethylsiloxane (PDMS) membrane. Acetone + hexane mixtures were studied at the temperature of 25 °C over the whole concentration range. The equilibrium and preferential sorption experiment were performed. Furthermore, the original physicochemical properties of acetone + hexane mixtures such as the excess surface tensions were determined and correlated using the Redlich-Kister equation. Determined differences in hexane and acetone sorption in PDMS, as well as in Flory-Huggins interaction parameters, are consistent with the differences in Hildebrand solubility parameters of pure liquids compared to PDMS, i.e. hexane and PDMS have identical value (14.9) which corresponds to higher sorption and higher rate of interaction with PDMS exhibited by hexane compared to acetone with 19.6. The pervaporation experiments confirmed that PDMS is able to separate this liquid mixture at the azeotropic point composition.

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1. Introduction

Solvents and their mixtures are extensively used in chemical processes to aid in mass and heat transfer, and to facilitate separations and purifications [1]. They are also important components of various cleaning agents, adhesives, paints, varnishes, or coatings in many processes in chemical industry. Therefore, a detailed knowledge of solvents properties contributes to the goal to minimise their negative impact on environment and human health. Determination and understanding of the physicochemical properties of solvents and their mixtures are essential for (i) the correct process design and operation conditions and (ii) for an adequate product formulation with respect to meet the stringent regulations [1].

Finding a suitable separation method in case of effective use/reuse of solvents is very important with respect to volumes used in industrial applications. The minimisation of their environmental impacts is essential also for the improvement of current technologies for solvent regeneration. However, the classical separation methods such as freeze-drying or distillation process, commonly used for the solvents separation are very often energy-consuming. Other limitations can arise from the physicochemical

properties e.g. thermal stability or vapour–liquid equilibria especially in cases of azeotropic mixtures. Azeotrope is a mixture which exhibits the same compositions of equilibrium vapour and liquid phases. Distillation, one of the primary tools that chemists and chemical engineers use to separate mixtures into their constituents, is not possible in this case. Other methods of separation of azeotropic mixtures are therefore topics of considerable interest. If the process can be replaced by the selective membrane separation, these difficulties might be overcome. Very promising alternative represents for instance pervaporation. Compared to distillation, pervaporation is less energy demanding, does not require high temperatures and can be performed at room temperature [2–5].

In this work, we focused on acetone–hexane binary liquid mixtures. Interestingly, the knowledge of the physicochemical properties of these widely used solvents has still some limitations [6–12]. The primary purpose of this work was to find the possibility of using the polydimethylsiloxane (PDMS) to membrane separation of the title mixture, mainly of the azeotropic composition. Although the pervaporation of mixture acetone + hexane has been already researched [13,14], a possibility of separation of the acetone–hexane azeotropic mixture (at 36 mol% of hexane) by PDMS has not been sufficiently explored. We see a good chance for that because the physicochemical properties are in close

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Nomenclature

α^{gl}	vapour-liquid equilibrium separation factor	p^s	vapour pressure, kPa
α^{sb}	sorption equilibrium separation factor	Q_m	the mass swelling degree, $g\ g_p^{-1}$
α^{pervap}	pervaporation separation factor	x_A^b	molar fraction of acetone in bulk liquid
γ	surface tension, $mN\ m^{-1}$	x_A^s	molar fraction of acetone in liquid sorbed into membrane
γ^E	excess surface tension, $mN\ m^{-1}$	x_H^b	molar fraction of hexane in bulk liquid
ρ^b	density of liquid, $g\ cm^{-3}$	x_H^s	molar fraction of hexane in liquid sorbed into membrane
ρ_p	density of dry polymer, $g\ cm^{-3}$	$x_{H,0}^s$	molar fraction of hexane in bulk initial solution
μ	dipole moment, D	x_H	molar fraction of hexane in liquid in VLE
η	viscosity, mPa s	y_H	molar fraction of hexane in vapour in VLE
Ω_H	preferential sorption of hexane, $mol\ g_p^{-1}$	w_P^{ter}	weight fraction of polydimethylsiloxane in ternary system
J	pervaporation flux, $g\ m^{-2}\ h^{-1}$	w_H^{ter}	weight fraction of hexane in ternary system
m	mass of swollen polymer, g	w_A^{ter}	weight fraction of acetone in ternary system
m_0	mass of dry polymer, g		
M_A	molar mass of acetone, $g\ mol^{-1}$		
M_H	molar mass of hexane, $g\ mol^{-1}$		
n_D	refraction index		
n_0	the number of moles of the initial solution per 1 g of dry membrane, $mol\ g_p^{-1}$		
n_A^s	the mole amount of acetone sorbed into membrane, $mol\ g_p^{-1}$		
n_H^s	the mole amount of hexane sorbed into membrane, $mol\ g_p^{-1}$		
n^s	the total mole amount of liquid sorbed into membrane, $mol\ g_p^{-1}$		

List of abbreviations

PDMS	polydimethylsiloxane
A	acetone
H	hexane
RK	Redlich-Kister equation
VLE	vapour-liquid equilibrium

relation to transport mechanisms of penetrants in polymers. Thus, for the first time, a detailed study the surface tension of acetone-hexane mixtures and its description using Redlich–Kister equation at the temperature of 25 °C and atmospheric pressure over the whole concentration range is reported. Furthermore, equilibrium and preferential sorption experiments were performed and the sorption behaviour of polydimethylsiloxane (PDMS) in these binary liquid mixtures was examined. In addition, pervaporation, a well-established membrane separation technique, confirmed PDMS ability to separate azeotropic mixture.

1.1. Acetone + hexane binary liquid mixture

Although acetone and hexane are quite dissimilar in structure and properties, they are miscible in the whole concentration range. As can be seen from Table 1, acetone and hexane differ in number of C-atoms, polarity, dipole moment and surface tension. However, their viscosities are almost identical.

Acetone, the simplest ketone, is a colourless, flammable liquid, used for instance as a solvent in the pharmaceutical industry, as a denaturant in denatured alcohol, or as an excipient in some pharmaceutical drugs. Its central carbonyl group guarantees the polar character of liquid. Dipole-dipole interactions between neighbouring molecules are responsible for the liquid state of acetone. Contrarily, hexane is a non-polar colourless liquid linear C₆-alkane used in the formulation of glues for shoes, leather and roofing products, for cleansing and degreasing, in textile manufacturing, or to extraction in soyfood processing. Its surface tension not only determines the quality of many products of various industries producing coatings, paints, detergents, cosmetics and agrochemicals, but influences also some significant stages of their processing like catalysis, adsorption, distillation, extraction, electrochemical reactions, operation of biological membranes, flotation, lubrication, and other processes where adhesion and detergency play important roles [21–23].

In most cases the surface tension of liquid mixture cannot be calculated according to a simple mixing rule (ideal mixture). This fact is expressed by the excess property:

Table 1
Comparison of acetone and hexane.

	Acetone		n-hexane	
Polarity	Polar		Non-polar	
Functional group	C=O		–	
M ($g\ mol^{-1}$)	58.08		86.18	
Number of C-atoms	3		6	
Property	Value	Literature	Value	Literature
ρ ($g\ cm^{-3}$)	0.78547	[6]	0.65489	[6]
γ ($mN\ m^{-1}$)	23.8	This work	18.1	This work
	23.1	[7]		
	22.42	[8]	17.94	[16]
	24.01	[9]		
η (mPa s)	0.308	[10]	0.311	[17]
			0.298	[18]
n_D	1.357	[11]	1.371	[19]
			1.373	[18]
p^s (kPa)	30.81	[12]	20.07	[20]
μ (D)	3.68	[15]	–	

$$\gamma^E = \gamma - (x_H \cdot \gamma_H + x_A \cdot \gamma_A) \quad (1)$$

where γ is experimental value of surface tension of liquid mixture, γ^E is value of excess property, x is molar fraction of hexane (H) or acetone (A). To describe the excess property as a function of the mixture composition the fourth-order Redlich-Kister equation was used:

$$\gamma^E = x_H \cdot x_A \sum_0^2 A_i \cdot (x_A - x_H)^i \quad (2)$$

1.2. Acetone + hexane + polydimethylsiloxane ternary system

Polydimethylsiloxane (PDMS), a commercially available type of silicone rubber used in a wide range of applications, is low-cost, isotropic, well processable, chemically inert, and thermally-stable gas permeable polymer. Such properties make PDMS attractive for the development of microelectromechanical systems, for

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