

Liquid-Liquid Extraction in a Microextractor: A Laboratory Examination and Thermodynamic Modeling of N-Hexane + Benzene + Sulfolane System

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ABSTRACT: *This study aimed at investigating liquid-liquid extraction of the three-component n-hexane + benzene + sulfolane system in a micro extractor. Experiments were carried out in a microtube with a diameter of 800 μ m using a T-shaped micromixer at a residence time of 15s. Temperature and the ratio of solvent (sulfolane) to feed (95% n-hexane + 5% benzene) investigated as operational variables. The temperature was investigated at (313.15, 323.15, and 333.15) K, and the solvent to feed ratio was investigated in five states including (0.33, 0.50, 1.00, 2.00, and 3.00) mL/mL. The results of experimental design and statistical analysis showed that operational variables had a significant impact on the distribution coefficient and selectivity. It was found that distribution coefficient and selectivity reached their highest levels at (313.15 and 323.15) K, respectively. In addition, in the low volumetric solvent to feed ratio (0.33ml/ml), the highest levels of distribution coefficient and selectivity were been obtained. Finally, the results obtained for liquid-liquid extraction of n-hexane + benzene + sulfolane were assessed using NRTL and UNIQUAC models, and the results confirmed the high accuracy of both models.*

KEYWORDS: *Liquid-liquid extraction; Microtube; Optimization; Thermodynamic modeling; NRTL; UNIQUAC.*

INTRODUCTION

Extraction is an important operational process in chemical technology. In most cases, this process involves two stages. The first stage is the distribution of phases in each other, which involves the extraction of the desired functional component from a liquid phase to an organic phase or an organic phase to a liquid phase by mixing, and the second stage is the separation of the mixture from the liquid and organic phase in order to extract the desired component [1, 2]. Liquid-liquid extraction is an important

and common operating unit in the industry that is widely used in process industries such as oil, food, hydrometallurgy, and chemical industry. Short contact and high efficiency are required for many extraction processes, and the development of this process is required for obtaining more efficient equipment [3, 4].

In the recent decade, several studies in the field of chemical engineering have investigated this topic [3, 5-10]. Pahlavanzadeh *et al.* experimentally investigated

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1021-9986/2021/2/657-666

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liquid-liquid extraction of 2, 3-butanediol from fermentation broth [5]. In another study, *Ghanadzadeh et al.* examined the laboratory results of LLE for a Methylcyclohexane+ Methanol+ Ethyl Benzenesystem [6].

Gomez et al. conducted an experimental measurements of equilibrium data (liquid-liquid) of a 3-component hexane+ benzene+ ethyl pyridinium ethyl sulphate at 283.15K and 293.15K under atmospheric pressure and calculated the corresponding selectivity and distribution coefficients for each item [7]. *Singh et al.* compared liquid-liquid extraction in conventional commercial extractors and microchannels using a standard fuzzy system with a low surface tension [8]. In addition, *Kurt et al.* studied laboratory characteristics of a liquid-liquid extraction system on a micro scale using a slug flow pattern at different residence times; it was concluded that, compared with the direct capillary method at a similar time, the efficiency of this method was 20% higher [9]. In another study, *Gursel et al.* developed a milli-scale liquid-liquid extraction setup and a fuzzy separators [10]. Finally, *John et al.* introduced a new method for liquid-liquid extraction via the application of ultrasound in microchannel tubes, in which the microchannel tubes were directly in contact with the ultrasound and converter without the need for an intermediate fluid [3].

In recent decade, the use of microreactors has been considered as a promising new technology in chemistry and chemical engineering because it has several advantages such as precise control over the location of the reaction via the utilization of micro spaces, utilization of spatial surfaces in every sizes, smaller length of penetration, the increase in mixing effects via cyclic circulation, and the increase in the amount of heat and mass transfer [3]. In recent decade, many studies have been conducted in this field [2, 11-17].

Okubo et al. studied a new microchannel setup that was designed to reach an effective liquid-liquid extraction system using a micromixer [2]. *Xu et al.* examined the enhancement of the mass transfer of the liquid-liquid distribution system on a micro scale using droplet flow in two types of microchannels [11]. Furthermore, *Dessimoz et al.* studied two-phase liquid-liquid flow patterns and their mass transfer characteristics in a rectangular glass microreactor [12]. In addition, *Su et al.* studied a new method for liquid-liquid two-phase mass transfer by a stimulating gas in a microchannel [13].

In another study, they also studied the effect of surface properties on the flow profile and mass transfer of two non-intermolecular fluids in mutually opposite T-shaped microchannels [14]. Moreover, *Jovanovic et al.* investigated the applications of extraction in spiral, bubble, parallel, and circular flow regiments in a Y-shaped microreactor mixer system [15]. Furthermore, *Matsuo et al.* assessed high purification of silica in a microchannel using solvent extraction [16]. Moreover, *Dareker et al.* conducted laboratory experiments on liquid-liquid extraction in two different types of microchannels using a water+ Zn+ D2EHPA [17].

In this study, a laboratory study and thermodynamic modeling were performed on the data of a three-component n-hexane+ benzene+ sulfolane system. In order to increase the intensity of isolation and decrease the separation time, a T-shaped micromixer and then a microtube were used. Full factorial design was used to determine optimal conditions (feed to solvent ratio and process temperature). Then the data obtained from organic and water phase separation was modeled using NRTL and UNIQUAC models. In order to increase the accuracy of the presented models, the inter-molecular interaction parameters were considered as a function of temperature.

EXPERIMENTAL SECTION

Materials

In order to perform liquid-liquid extraction, n-hexane $\geq 98.5\%$, benzene $\geq 99.8\%$, and sulfolane $\geq 99.0\%$ were purchased from Merck Company. The purity of the material was investigated by the Gas Chromatography (GC) Agilent, model 6890N. In all the stages, deionized water was used for washing. A mixture of n-hexane (95wt%) and benzene (5wt%) was used as the feed and sulfolane was used as the solvent to remove benzene from n-hexane. Detailed specifications of chemicals used in this work were shown in Table 1.

Instruments

A microtube with an internal diameter of 800 μm and a volume of 1 cm^3 was used for the liquid-liquid extraction. Using a peristaltic pump, model BT100-1F, the n-hexane+ benzene mixture on one side and the sulfolane solvent on the other side were introduced into a T-shaped micromixer with a diameter of 800 μm and after mixing were introduced into the microtube (see Fig.1). The flow rate

Table 1: Specifications of the Chemicals.

Chemical name	CAS #	Source	Purity (wt %) ^a	GC analysis (wt %) ^b
n-Hexane	110-54-3	Merck	≥ 98.5 %	≥ 98.6 %
Benzene	71-43-2	Merck	≥ 99.8 %	≥ 99.6 %
Sulfolane	126-33-0	Merck	≥ 99.0 %	≥ 99.0 %

^a Obtained by the supplier. ^b Experimentally determined by gas chromatography.

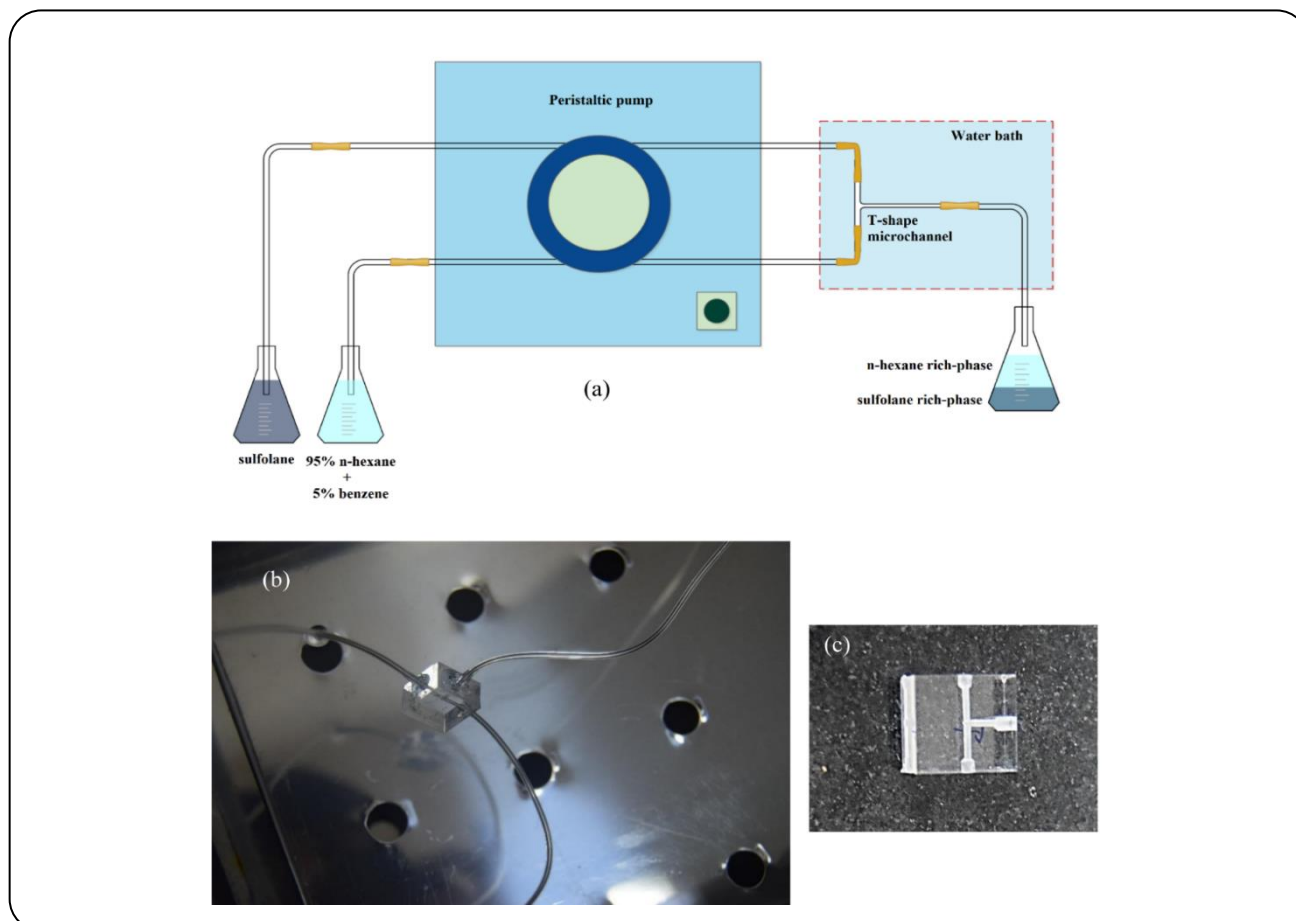


Fig. 1: a) Schematic diagram for extraction method in the microextractor; b) photograph of the microtube using a T-shaped micromixer in the bain-marie bath; c) photograph of T-shaped micromixer.

was set so that the residence time for all the experiments was 15s. A bain-marie bath (with an uncertainty of 0.2 K) was used to control the temperature. After taking the mixture out of the microtube, a separator funnel separated two phases of n-hexane-rich phase and sulfolane-rich phase, and a gas chromatography device, Agilent, model 6890N, was used to determine the composition of the components in each of the phases. The GC (Agilent 6890N model) equipped with a capillary column, DB-WAX (30 m×0.25 mm) and a flame ionization detector (FID). Helium gas as the carrier gas and an auxiliary gas was applied

for FID. The oven temperature of sample was 40-230°C and the injection temperature was 250°C.

Full factorial design of experiments (procedure method)

Sulfolane was used as a solvent to isolate benzene from n-hexane. Experiments were conducted in different conditions to determine the optimal operating conditions. The studied variables included sulfolane to feed volumes ratio (n-hexane and benzene mixture) and operating temperature. Table 2 shows the values of each of these two variables. Using full factorial design, the order

Table 2: Operating parameters and their values for the separation of benzene from n-hexane by sulfolane solvent ^a

Factor	Symbol	Levels
Solvent to feed ratio, ml/ml	S_R	0.33, 0.50, 1.00, 2.00, 3.00
Temperature, K	T	313.15, 323.15, 333.15

^a Standard uncertainties u are $u(S_R) = 0.01$ mL/mL and $u(T) = 0.2$ K.

Table 3: Experimental design using full factorial design method and liquid-liquid extraction data for three-component n-hexane (1)+ benzene (2)+ sulfolane (3) system in a microtube with an internal diameter of 800 μ m at a residence time of 15s at pressure $P = 101.2$ kPa ^a.

No.	Manipulated variables		Overall composition			n-hexane rich-phase			sulfolane rich-phase			Responses	
	S_R , mL/mL	T , K	w_1	w_2	w_3	w_1^I	w_2^I	w_3^I	w_1^{II}	w_2^{II}	w_3^{II}	D_2	S
1	3.00	323.15	0.1406	0.0074	0.8520	0.8242	0.0141	0.1617	0.0373	0.0064	0.9563	0.45	10.01
2	3.00	333.15	0.1406	0.0074	0.8520	0.6917	0.0139	0.2944	0.0574	0.0064	0.9362	0.46	5.56
3	0.33	333.15	0.5816	0.0306	0.3878	0.9387	0.0344	0.0269	0.0181	0.0246	0.9573	0.72	37.13
4	0.50	313.15	0.4871	0.0256	0.4872	0.9097	0.0294	0.0609	0.0228	0.0215	0.9557	0.73	29.18
5	2.00	323.15	0.1979	0.0104	0.7917	0.8489	0.0174	0.1337	0.0342	0.0087	0.9571	0.50	12.35
6	0.50	323.15	0.4871	0.0256	0.4872	0.9321	0.0303	0.0376	0.0187	0.0207	0.9606	0.68	34.10
7	2.00	313.15	0.1979	0.0104	0.7917	0.7564	0.0165	0.2271	0.0501	0.0088	0.9411	0.53	8.06
8	2.00	333.15	0.1979	0.0104	0.7917	0.7888	0.0181	0.1931	0.0478	0.0085	0.9437	0.47	7.72
9	0.50	333.15	0.4871	0.0256	0.4872	0.8991	0.0299	0.0710	0.0273	0.0209	0.9518	0.70	23.00
10	1.00	323.15	0.3275	0.0172	0.6552	0.8906	0.0225	0.0869	0.0272	0.0144	0.9584	0.64	21.00
11	3.00	313.15	0.1406	0.0074	0.8520	0.6821	0.0130	0.3049	0.0587	0.0066	0.9347	0.50	5.86
12	1.00	333.15	0.3275	0.0172	0.6552	0.8610	0.0254	0.1136	0.0387	0.0128	0.9485	0.50	11.23
13	0.33	323.15	0.5816	0.0306	0.3878	0.9658	0.0341	0.0001	0.0121	0.0254	0.9625	0.75	59.54
14	1.00	313.15	0.3275	0.0172	0.6552	0.8211	0.0213	0.1576	0.0384	0.0149	0.9467	0.70	14.92
15	0.33	313.15	0.5816	0.0306	0.3878	0.9505	0.0333	0.0162	0.0141	0.0265	0.9594	0.79	53.59

^a Given are the solvent to feed ratio S_R , temperature T , weight fraction of water w_1 , weight fraction of acetone w_2 , weight fraction of ethyl acetate w_3 , distribution coefficient D_2 and selectivity S , Standard uncertainties u are $u(S_R) = 0.01$ mL/mL and $u(T) = 0.2$ K, $u(w_1) = u(w_2) = u(w_3) = 0.0002$ and $u(P) = 0.9$ kPa.

of the experiments was determined by Design Expert 7.1.3. Taking into account the number of different levels of variables, that included five levels for the solvent to feed volume ratio (0.33, 0.50, 1.00, 2.00, and 3.00) mL/mL and three levels for the temperature (313.15, 323.15, and 333.15) K, the number of experiments was $5 \times 3 = 15$. Table 3 presents the order of the experiments, which was determined randomly by the design of experiments.

Thermodynamic modeling

In this study, the NRTL [18] and UNIQUAC [19] thermodynamic models were used to calculate the activity

coefficients and then to evaluate the liquid-liquid extraction. Since the data were measured at three different temperatures, the temperature function for the intermolecular interaction parameters was defined as follows:

$$\text{NRTL model: } \tau_{ij} = \frac{\Delta g_{ij}}{RT} = A_{ij} + \frac{B_{ij}}{T} \quad (1)$$

$$(\tau_{ij} \neq \tau_{ji} \text{ and } \tau_{ii} = 0)$$

$$\text{UNIQUAC model: } \tau_{ij} = \exp\left[-\frac{a_{ij}}{R}\right] = \exp\left[A_{ij} + \frac{B_{ij}}{T}\right] \quad (2)$$

$$(\tau_{ij} \neq \tau_{ji} \text{ and } \tau_{ii} = 0)$$

Table 4: r_i , q_i , and q'_i parameters for the substances used in this study ^a.

Component	r_i	q_i	q'_i
n-hexane	4.4998	3.856	3.856
benzene	3.1878	2.400	2.400
sulfolane	4.0385	3.200	3.200

^a Given are the values of volume parameters of pure component r_i , surface area parameters of pure component q_i and the interaction correction factor of pure component q'_i .

where τ_{ij} and τ_{ji} are the energy of interaction between i and j molecules, A_{ij} and B_{ij} are dual interaction parameters, and T is absolute temperature. Table 4 presents the values of r_i , q_i , and q'_i for the materials investigated by the UNIQUAC model in this study.

RESULTS AND DISCUSSION

Laboratory data on liquid-Liquid extraction

As shown in Table 3, all the experiments were, respectively, performed in the micro extractor. Output streams moving from the extractor into a separatory funnel were divided into two phases including n-hexane-rich and sulfolane-rich phase. The samples were then sent to GC to analyze the percentage of the components. In this study, w_i , w_i^I , and w_i^{II} , respectively, were the weighted fraction of i in the total composition, the weight fraction of i in the n-hexane-rich phase, and the weighted fraction of i in the sulfolane-rich phase. Moreover, i was related to the components of the mixture and was equal to 1, 2, and 3 for n-hexane, benzene, and sulfolane, respectively.

Table 3 presents the data on liquid-liquid extraction of the three-component n-hexane+ benzene+ sulfolane system. The distribution coefficient and selectivity are the basic parameters in liquid-liquid extraction that are represented by D_2 and S , respectively, and are defined as follows [20]:

$$D_2 = \frac{w_2^{II}}{w_2^I} \quad (3)$$

$$S = D_2 \frac{w_1^I}{w_1^{II}} \quad (4)$$

Where w_1 , and w_2 , respectively, are the n-hexane and benzene mass fractions, and the superscripts of I and II , respectively, indicate n-hexane-rich and sulfolane-rich phases.

The quality of liquid-liquid extraction data is confirmed by Othmer-Tobias and Hand relationships. These relationships are presented below:

$$\ln \left(\frac{1 - w_1^I}{w_1^I} \right) = a_{OT} \ln \left(\frac{1 - w_3^{II}}{w_3^{II}} \right) + b_{OT} \quad (5)$$

$$\ln \left(\frac{w_2^I}{w_1^I} \right) = a_H \ln \left(\frac{w_2^{II}}{w_3^{II}} \right) + b_H \quad (6)$$

where a_{OT} , b_{OT} are the constants of Othmer-Tobias equation and a_H and b_H are the constants of Hand equation that are obtained by regression of the data [21, 22]. Figs. 2 and 3 show the matching of liquid-liquid extraction data with Othmer-Tobias and hand equations. In addition, the equations constants and R^2 are presented in Table 5. Considering Figs. 2 and 3 and Table 5, it can be concluded that there is a fairly good match between empirical equations and experimental data.

Determination of optimal point and statistical analysis

As stated above, the distribution coefficient and selectivity are two important criteria for checking the performance of the solvents. Using the statistical analysis, the effects of these two parameters were investigated. In this study, the effects of process temperature and the solvent to feed ratio were studied. Table 3 presents the distribution coefficient and selectivity values obtained in different conditions. These variables were tested by the analysis of variance (ANOVA). Table 6 presents the results of this analysis obtained for the distribution coefficient and selectivity. According to the results of these analyses, it is clear that the change in the temperature had a statistically significant effect on the distribution coefficient and selectivity; moreover, the change in the solvent to feed ratio had a very significant effect on the distribution coefficient and selectivity.

Fig. 3 shows the effect of the process temperature and the solvent to feed ratio on the distribution coefficient and selectivity when using sulfolane as the solvent. As shown

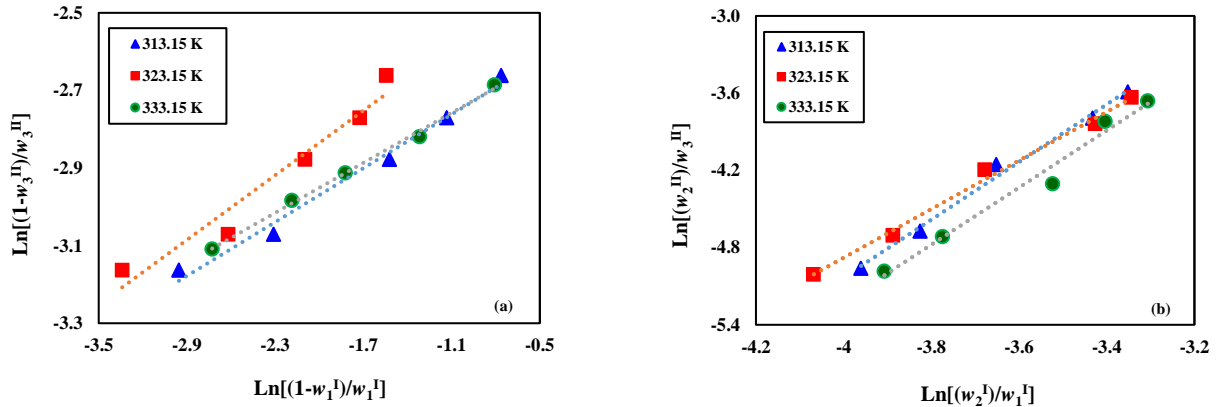


Fig. 2: Determination of the accuracy of the equilibrium data of the n-hexane+ benzene+ sulfolane system; a) the Othmer-Tobias equation; b) Hand equation.

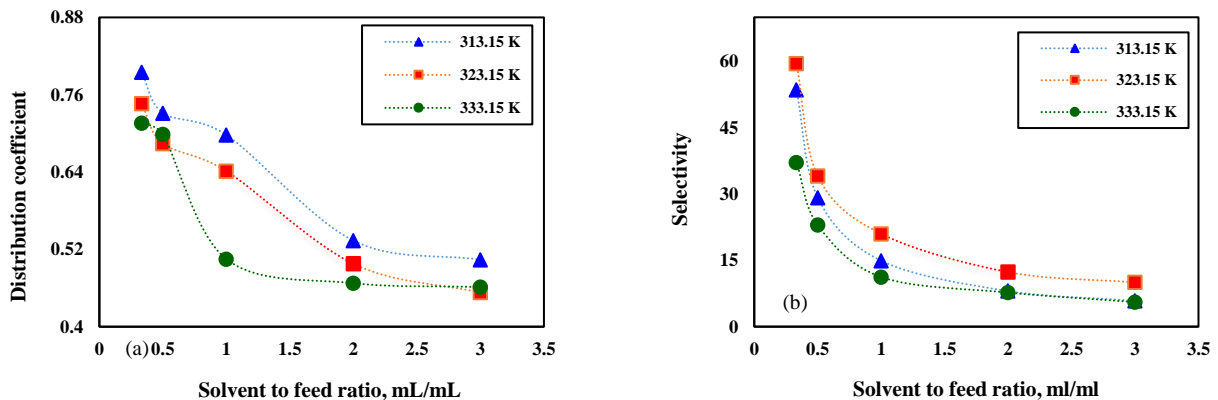


Fig. 3: Effect of sulfolane to feed ratio at different temperatures; a) distribution coefficient; b) selectivity.

in these figures (Figs. 3a and 3b), with increasing the proportion of the solvent, the distribution coefficient and selectivity reduced at all the temperatures. In addition, in most of the solvent-to-feed ratios, the increase in the temperature resulted in a reduction in the distribution coefficient. In addition, the selectivity of benzene by the sulfolane as the solvent reached its maximum value at a moderate temperatures (323.15K). Therefore, the optimum point for reaching the highest distribution coefficient was reached at the temperature of 313.15K and the solvent to feed ratio of 0.33ml/ml. Under these conditions, the distribution coefficient was 0.79. The optimum point for reaching the maximum selectivity was achieved at the temperature of 323.15K and the solvent to feed ratio of 0.33ml/ml. Under these conditions, the selectivity value was 59.54. The GC chromatograms of optimum point for n-hexane rich-phase and sulfolane rich-phase were been shown in Fig. 4.

Thermodynamic modeling of data on liquid-liquid extraction

To determine the parameters of dual interaction, the models were optimized. To this end, the objective function presented in Equation 7 was minimized. This equation represents the mean difference of square mass fractions that were measured and calculated using NRTL and UNIQUAC models for the components in organic and liquid phases:

$$OF = 100 \times \quad (7)$$

$$\frac{1}{3D} \sum_{(j=1)}^D \sum_{(i=1)}^3 \left(\left(w_{ij}^{I,exp.} - w_{ij}^{I,calc.} \right)^2 + \left(w_{ij}^{II,exp.} - w_{ij}^{II,calc.} \right)^2 \right)$$

To compare the accuracy of the examined models, the root mean square deviation (RMSD) was used, which is shown in Equation 8:

Table 5: The coefficients of the Othmer-Tobias and hand equations and R^2 for the system investigated in this study ^a.

T, K	Othmer-Tobias			Hand		
	a_{OT}	b_{OT}	R^2	a_H	b_H	R^2
n-hexane (1) + benzene (2) + sulfolane (3)						
313.15	0.2313	-2.5074	0.9858	2.2401	3.9290	0.9909
323.15	0.2776	-2.2802	0.9497	1.8899	2.6803	0.9933
333.15	0.2149	-2.5212	0.9963	2.2185	3.6529	0.9786

^a Given is the temperature T, Standard uncertainty u is $u(T) = 0.2$ K.

Table 6: Analysis of variance (ANOVA) for the distribution coefficient and selectivity of benzene by sulfolane solvent ^a.

Source	SS	DF	MS	F-Value	P-Value	Level of significance
Distribution coefficient (D_2)						
Model	0.1979	6	0.0330	25.14	0.000	highly significant
Linear	0.1979	6	0.0330	25.14	0.000	highly significant
S_R	0.1807	4	0.0452	34.43	0.000	highly significant
T	0.0172	2	0.0086	6.57	0.021	significant
Error	0.0105	8	0.0012			
Model	0.2084	14				
Selectivity (S)						
Model	4063.0	6	672.67	40.92	0.000	highly significant
Linear	4036.0	6	672.67	40.92	0.000	highly significant
S_R	3761.7	4	940.43	57.22	0.000	highly significant
T	274.3	2	137.14	8.34	0.011	significant
Error	131.5	8	16.44			
Total	4167.5	14				

^a Given are the solvent to feed ratio S_R and temperature T, Standard uncertainties u are $u(S_R) = 0.01$ mL/mL and $u(T) = 0.2$ K

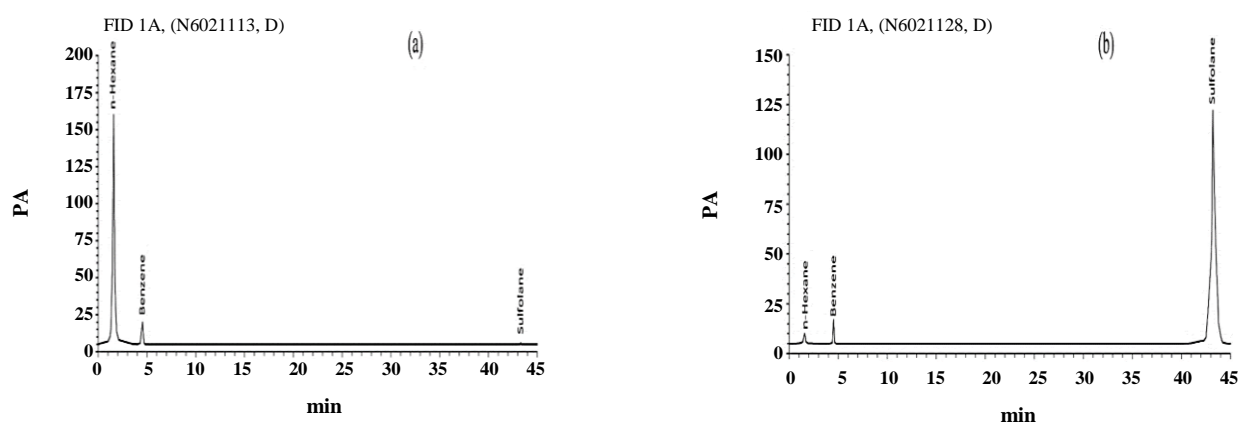


Fig. 4: The GC chromatograms of optimum point ($S_R = 0.33$ ml/ml and $T = 323.15$ K); a) n-hexane rich-phase sample; b) sulfolane rich-phase sample.

Table 7: Parameters of intermolecular interaction between n-hexane+ benzene+ sulfolane.

Thermodynamic Model	Component		Binary interaction parameter			
	<i>i</i>	<i>j</i>	A_{ij}	B_{ij}, K	A_{ij}	B_{ij}, K
NRTL	n-hexane	benzene	14.4081	17.97793	-3.0382	17.03327
	n-hexane	sulfolane	-2.6057	-4.0185	1.05165	-22.7162
	benzene	sulfolane	4.92613	2.111688	-0.7463	27.43577
UNIQUAC	n-hexane	benzene	-27.4171	-7.3625	1.1074	3.0288
	n-hexane	sulfolane	0.6187	29.14473	-0.0015	-33.8066
	benzene	sulfolane	-27.327	-4.30235	1.52276	-9.05435

Table 8: Accuracy of NRTL and UNIQUAC models for n-hexane + benzene + sulfolane system ^a.

T (K)	NDP	RSMD		D_2	<i>S</i>
		NRTL	UNIQUAC		
313.15	5	0.0090	0.0114	0.65	22.32
323.15	5	0.0089	0.0112	0.60	27.40
333.15	5	0.0081	0.0104	0.57	16.93

^a Given are the temperature *T*, number of data points *NDP*, distribution coefficient D_2 and selectivity *S*, Standard uncertainty *u* is $u(T) = 0.2 K$.

$$MSD = \left(\frac{1}{3D} \sum_{j=1}^D \sum_{i=1}^3 \left(w_{ij}^{I,exp.} - w_{ij}^{I,calc.} \right)^2 + \left(w_{ij}^{II,exp.} - w_{ij}^{II,calc.} \right)^2 \right)^{1/2} \quad (8)$$

Where *D* is the total number of tie lines, superscripts *i* and *j*, respectively, are the desired components and tie lines, and *exp.* and *calc.* are the measured and calculated components.

After optimizing the data using to the objective function (Equation 7), intermolecular interaction parameters were obtained for NRTL and UNIQUAC thermodynamic models. Table 7 shows the values of these parameters for both thermodynamic models. After determining the interaction parameters between the molecules in the thermodynamic models, the models were applied to all available data and the accuracy of each thermodynamic model was determined. Table 8 shows the mean root mean square deviation (RSMD) of three-component systems consisting of n-hexane+ benzene+ sulfolane for NRTL and UNIQUAC models separately. Considering the results of Table 8, it is clear that the accuracy of the thermodynamic models is UNIQUAC << NRTL.

CONCLUSIONS

In this study, liquid-liquid extraction of the three-component n-hexane + benzene + sulfolane system in a microtube with a diameter of 800μm was been investigated. Experiments were carried out at different temperatures and different solvent to feed ratios. The results of the experiments showed that the studied parameters (temperature and solvent to feed ratio) were very effective on the distribution coefficient and selectivity. The results showed that the distribution coefficient at 313.15K and selectivity at 323.15K reached the highest values. In addition, in the solvent to feed volume ratio of 0.33ml/ml, the highest values of the distribution coefficient and selectivity were obtained. Finally, this study investigated the matching between NRTL and UNIQUAC thermodynamic models applied on fluid extraction data. The RSMD values for the NRTL and UNIQUAC models were very low and the accuracy of NRTL model was much higher than the accuracy of UNIQUAC model.

Received : Aug. 7, 2019 ; Accepted : Nov. 18, 2019

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