Vegetable Oils Deacidification by Solvent Extraction: Liquid−Liquid Equilibrium Data for Systems Containing Sunflower Seed Oil at 298.2 K

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Liquid−liquid equilibrium experimental data for refined sunflower seed oil, artificially acidified with commercial oleic acid or commercial linoleic acid and a solvent (ethanol + water), were determined at 298.2 K. This set of experimental data and the experimental data from Cuevas et al.,[1] which were obtained from (283.2 to 333.2) K, for degummed sunflower seed oil-containing systems were correlated using NRTL and UNIQUAC models with temperature-dependent binary parameters. The deviation between experimental and calculated compositions presented average values of (1.13 and 1.41) % for NRTL and UNIQUAC equations, respectively, indicating that the models were able to correctly describe the behavior of compounds under different temperature and solvent hydration.

Introduction

In recent years, our research group has worked on phase equilibrium of vegetable oils to improve the oil deacidification technique by liquid−liquid extraction.[2−4]

The liquid−liquid extraction technique has been considered as an alternative to the traditional processes of vegetable oil refining such as chemical and physical processes.[5] Phase equilibrium data are necessary not only to design and correctly operate the extractor but also to design the equipment used to recover the residual solvent from the oil phase and fatty compounds from the extract phase.

In the present paper, liquid−liquid equilibrium data are reported for systems composed of refined sunflower seed oil, oleic or linoleic acids, ethanol, and water, determined at (298.2 ± 0.1) K. The experimental data presented in this work combined with data previously published by Cuevas et al.[1] (experimentally determined from (283.2 to 333.2) (± 0.1) K) were favorably correlated using the NRTL and UNIQUAC equations with temperature-dependent binary parameters.

The adjusted interacting parameters enable the simulation of liquid−liquid extractors in the deacidification process of vegetable oils.

Experimental Section

The solvents used as extractants were absolute ethanol, from Merck (Germany), with purity higher than 99.5 %, and aqueous solvents with different water mass fractions, (6.32, 12.84, and 18.95) %, prepared by diluting deionized water (Millipore, Milli-Q, Bedford, MA, USA) into absolute ethanol. The values of molar masses, area, and volume parameters of ethanol and water were calculated and published in a previous paper.[1]

The refined sunflower seed oil used in this work was kindly supplied by Cargill (São Paulo/SP, Brazil). The fatty acid composition of the refined vegetable oil studied in this work is presented in Table 1. This composition was determined by gas chromatography of fatty acid methyl esters, as described elsewhere.[1] The expected triacylglycerol composition of refined sunflower seed oil was estimated by the algorithm suggested by Antoniosi Filho et al.[6]

Table 1. Fatty Acid Mole Fractions *x* and Mass Fractions *w* of the Refined Sunflower Seed Oil

fatty acid	symbol		M^b / g·mol^{-1}	100*x*	100 *w*
palmitic	P	C16:0[a]	256.43	7.14	6.54
stearic	S	C18:0	284.49	4.05	4.11
oleic	O	C18:1	282.47	24.56	24.80
linoleic	Li	C18:2	280.45	62.11	62.27
linolenic	Le	C18:3	278.44	0.88	0.89
arachidic	A	C20:0	312.54	0.30	0.33
gadoleic	Ga	C20:1	310.52	0.31	0.35
behenic	Be	C22:0	340.59	0.65	0.71

[a] In C*x*:*y*, *x* = number of carbons and *y* = number of double bonds. [b] *M* = molar mass.

These results of fatty acid and triacylglycerol composition make it possible to calculate the average molar mass of oil as well as the volume and area parameter values (r'_i and q'_i), required by the UNIQUAC model. The values obtained were 876.38 g·mol^{-1}, 0.04395, and 0.03560, respectively.

Commercial oleic and linoleic fatty acids were purchased from Merck (Germany) and Fluka (Switzerland), respectively. Fatty acid compositions, molar mass values, and volume and area parameters (r'_i and q'_i) of these acids were previously published and presented the following values: commercial oleic acid[7] (278.96 g·mol^{-1}, 0.04513, and 0.03714, respectively) and commercial linoleic acid[3] (279.18 g·mol^{-1}, 0.04484, and 0.03685, respectively).

Cuevas et al.[1] reported the molar mass values for degummed sunflower seed oil and for free acidity naturally present in this oil, which were 279.80 g·mol^{-1} for free acidity and 873.12 g·mol^{-1} for degummed oil.

There is a similarity between the molar mass values for the compounds used in these different reports, thus the value of

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Table 2. Liquid−Liquid Equilibrium Data for the System Refined Sunflower Seed Oil (1) + Commercial Oleic Acid (2) + Commercial Linoleic Acid (3) + Ethanol (4) + Water (5), at (298.2 ± 0.1) K

	OC[b]					OP[c]					AP[d]				
100 w_5^{solv} [a]	100 w_1	100 w_2	100 w_3	100 w_4	100 w_5	100 w_1	100 w_2	100 w_3	100 w_4	100 w_5	100 w_1	100 w_2	100 w_3	100 w_4	100 w_5
0	49.88	0.00	0.00	50.12	0.00	87.75	0.00	0.00	12.25	0.00	4.88	0.00	0.00	95.12	0.00
	49.10	1.03	0.00	49.87	0.00	86.49	0.89	0.00	12.62	0.00	5.41	1.21	0.00	93.38	0.00
	47.94	2.08	0.00	49.98	0.00	84.80	1.78	0.00	13.43	0.00	6.08	2.35	0.00	91.57	0.00
	46.84	3.21	0.00	49.95	0.00	83.07	2.75	0.00	14.18	0.00	6.80	3.59	0.00	89.61	0.00
	44.79	5.30	0.00	49.91	0.00	77.67	4.82	0.00	17.51	0.00	10.67	6.15	0.00	83.17	0.00
	39.75	10.21	0.00	50.04	0.00	63.31	9.61	0.00	27.07	0.00	21.36	10.95	0.00	67.69	0.00
	50.02	0.00	0.00	49.98	0.00	88.06	0.00	0.00	11.94	0.00	5.85	0.00	0.00	94.15	0.00
	48.97	0.00	1.14	49.89	0.00	86.25	0.00	0.99	12.76	0.00	6.99	0.00	1.38	91.63	0.00
	48.01	0.00	2.07	49.92	0.00	85.45	0.00	1.77	12.78	0.00	7.56	0.00	2.47	89.97	0.00
	46.83	0.00	2.99	50.17	0.00	83.13	0.00	2.76	14.11	0.00	8.31	0.00	3.39	88.30	0.00
	44.72	0.00	5.16	50.11	0.00	79.45	0.00	4.62	15.93	0.00	9.47	0.00	6.33	84.20	0.00
	40.03	0.00	10.01	49.96	0.00	65.22	0.00	9.44	25.34	0.00	18.56	0.00	11.04	70.40	0.00
6.32	50.01	0.00	0.00	46.83	3.16	93.19	0.00	0.00	6.20	0.61	0.89	0.00	0.00	91.79	7.32
	49.06	1.01	0.00	46.77	3.16	91.49	1.00	0.00	6.97	0.54	0.65	1.07	0.00	90.30	7.98
	47.87	2.16	0.00	46.81	3.16	89.99	2.11	0.00	7.18	0.71	0.93	2.21	0.00	89.72	7.14
	46.90	3.16	0.00	46.78	3.16	88.71	2.91	0.00	7.76	0.63	1.20	3.28	0.00	88.25	7.27
	44.89	5.04	0.00	46.90	3.17	86.10	4.97	0.00	8.09	0.84	1.74	5.42	0.00	85.95	6.89
	39.61	10.22	0.00	46.99	3.17	78.30	10.10	0.00	10.80	0.80	3.21	10.55	0.00	80.17	6.07
	29.54	20.50	0.00	46.80	3.16	57.07	19.96	0.00	20.56	2.41	12.22	20.79	0.00	61.73	5.26
	49.74	0.00	0.00	47.08	3.18	94.38	0.00	0.00	5.10	0.52	1.35	0.00	0.00	92.74	5.91
	48.97	0.00	0.98	46.88	3.16	93.04	0.00	0.88	5.60	0.48	1.43	0.00	0.98	91.58	6.01
	47.99	0.00	2.06	46.79	3.16	91.02	0.00	1.99	6.48	0.51	1.52	0.00	2.15	90.44	5.89
	47.05	0.00	2.99	46.81	3.16	89.78	0.00	2.97	6.73	0.52	1.65	0.00	3.29	89.16	5.90
	44.93	0.00	5.21	46.71	3.15	86.43	0.00	5.19	8.03	0.35	1.97	0.00	5.74	86.43	5.86
	39.57	0.00	10.22	47.04	3.18	78.14	0.00	9.96	11.33	0.57	3.52	0.00	11.01	79.89	5.59
	29.11	0.00	20.89	46.83	3.16	56.31	0.00	19.95	22.28	1.45	11.90	0.00	21.85	61.84	4.41
12.84	50.09	0.00	0.00	43.50	6.41	96.02	0.00	0.00	3.63	0.35	0.34	0.00	0.00	87.85	11.81
	49.03	1.00	0.00	43.55	6.42	94.77	1.07	0.00	3.64	0.52	0.36	0.85	0.00	87.04	11.76
	48.01	2.02	0.00	43.55	6.42	93.53	2.28	0.00	3.59	0.61	0.36	1.63	0.00	86.38	11.63
	46.98	3.02	0.00	43.58	6.42	92.46	3.30	0.00	3.56	0.69	0.39	2.37	0.00	85.90	11.34
	44.96	5.12	0.00	43.51	6.41	88.22	6.13	0.00	4.39	1.26	0.41	4.45	0.00	86.30	8.84
	39.78	10.22	0.00	43.58	6.42	80.59	12.12	0.00	5.45	1.85	0.53	9.00	0.00	82.33	8.15
	28.67	21.30	0.00	43.60	6.42	60.30	23.69	0.00	14.56	1.45	2.93	20.12	0.00	68.85	8.10
	50.03	0.00	0.00	43.56	6.42	95.76	0.00	0.00	3.77	0.46	0.39	0.00	0.00	88.08	11.53
	48.94	0.00	1.05	43.59	6.42	94.44	0.00	1.07	4.09	0.41	0.41	0.00	0.79	86.91	11.88
	47.74	0.00	2.27	43.57	6.42	93.02	0.00	2.04	4.33	0.61	0.53	0.00	1.44	86.43	11.60
	46.98	0.00	3.01	43.58	6.42	91.25	0.00	3.19	4.96	0.61	0.70	0.00	2.56	85.02	11.72
	44.70	0.00	5.32	43.56	6.42	87.46	0.00	6.20	5.08	1.26	0.77	0.00	4.67	85.72	8.84
	39.89	0.00	9.98	43.69	6.44	81.06	0.00	11.55	5.54	1.85	0.90	0.00	9.51	81.06	8.53
	29.94	0.00	20.00	43.63	6.43	65.13	0.00	22.48	10.93	1.45	2.91	0.00	20.12	68.86	8.10
18.95	49.92	0.00	0.00	40.59	9.49	96.22	0.00	0.00	3.11	0.67	0.07	0.00	0.00	80.48	19.44
	47.36	2.57	0.00	40.58	9.49	91.32	3.77	0.00	4.11	0.80	0.09	1.67	0.00	81.12	17.12
	44.86	5.14	0.00	40.52	9.47	86.62	7.28	0.00	5.07	1.03	0.10	3.37	0.00	79.47	17.06
	39.88	10.12	0.00	40.52	9.47	76.70	14.42	0.00	7.58	1.29	0.11	6.73	0.00	76.18	16.98
	28.96	21.04	0.00	40.53	9.48	57.87	26.91	0.00	11.69	3.53	0.11	16.23	0.00	66.55	17.10
	49.98	0.00	0.00	40.54	9.48	96.36	0.00	0.00	2.84	0.81	0.08	0.00	0.00	81.27	18.65
	47.51	0.00	2.52	40.50	9.47	92.86	0.00	3.35	2.92	0.87	0.09	0.00	1.89	79.64	18.38
	45.01	0.00	5.01	40.50	9.47	88.62	0.00	6.87	3.55	0.96	1.20	0.00	3.46	76.66	18.68
	39.87	0.00	10.10	40.55	9.48	79.52	0.00	13.89	4.99	1.60	0.25	0.00	7.23	74.66	17.86
	29.98	0.00	20.02	40.52	9.47	60.41	0.00	24.99	11.84	2.75	0.82	0.00	16.87	64.05	18.26

[a] w_5^{solv} = mass fraction of water in the alcoholic solvent. [b] OC = overall composition. [c] OP = oil phase. [d] AP = alcoholic phase.

876.38 g·mol^{-1} for sunflower seed oil (average molar mass of the refined oil) and 279.18 g·mol^{-1} for free fatty acids present in degummed oil (average molar mass of the commercial linoleic acid) were adopted for the estimation procedure.

The experimental procedure and validation tests to determine the tie lines and to characterize oil and alcoholic phases can be found in a previous work.[7]

Regarding the modeling approach, the data set from this work (determined at (298.2 ± 0.1) K) and the data previously published by Cuevas et al.[1] (determined from (283.2 to 333.2) (± 0.1) K) were correlated using UNIQUAC and NRTL equations with temperature-dependent binary interaction parameters. The temperature dependencies of NRTL parameters (α_{ij}, $A_{0,ij}$, $A_{0,ji}$, $A_{1,ij}$, and $A_{1,ji}$) and UNIQUAC parameters ($A_{0,ij}$, $A_{0,ji}$, $A_{1,ij}$, and $A_{1,ji}$) can be visualized in the reports of Silva et al.,[8] Follegatti-Romero et al.,[9] and Cuevas et al.,[1] where α_{ij} represents the nonrandomness parameter of the mixture, meaning that the components have a distribution pattern dictated by the local composition, and $A_{0,ij}$, $A_{0,ji}$, $A_{1,ij}$, and $A_{1,ji}$ represent the characteristic energy parameters of *i* and *j* interactions.

Results

In the present work, the following notations represent the components used in the experiments: sunflower seed oil (1) (representing refined or degummed oil), oleic acid (2), linoleic acid (3) (representing commercial linoleic acid added to refined oil and free acidity naturally present in degummed oil), ethanol (4), and water (5).

Table 2 presents the overall experimental composition of the mixtures and the corresponding tie lines for the pseudoternary and pseudoquaternary systems composed by refined sunflower

Table 3. NRTL Parameters

pair ij[a]	$A_{0,ij}$ K	$A_{0,ji}$ K	$A_{1,ij}$	$A_{1,ji}$	α_{ij}
12	−271.92	−146.35	0.00	0.00	0.70
13	−2048.70	−127.19	$-4.74\cdot10^{-2}$	$9.00\cdot10^{-2}$	0.55
14	284.66	1464.40	$-7.33\cdot10^{-3}$	$1.95\cdot10^{-2}$	0.48
15	−1149.80	4109.50	$-2.03\cdot10^{-3}$	$-2.03\cdot10^{-2}$	0.11
23	0.00	0.00	0.00	0.00	0.00
24[b]	4800.00	−170.55	0.00	0.00	0.23
25[b]	1006.70	4210.60	0.00	0.00	0.10
34	5000.00	−2020.20	$-8.96\cdot10^{-2}$	$1.89\cdot10^{-1}$	0.22
35	2546.10	1400.40	$-5.21\cdot10^{-3}$	$-7.93\cdot10^{-4}$	0.10
45[b]	−10.98	−173.64	0.00	0.00	0.15

[a] Sunflower seed oil (1), commercial oleic acid (2), commercial linoleic acid (3), ethanol (4), water (5). [b] Parameters taken from Rodrigues and Meirelles.[7]

Table 4. UNIQUAC Parameters

pair ij[a]	$A_{0,ij}$ K	$A_{0,ji}$ K	$A_{1,ij}$	$A_{1,ji}$
12	248.42	−213.41	0.00	0.00
13	177.00	−189.87	0.00	0.00
14	720.10	−260.51	−436.55	195.28
15	1028.50	−71.94	$2.19\cdot10^{-2}$	$-1.33\cdot10^{-2}$
23	0.00	0.00	0.00	0.00
24[b]	67.64	−88.95	0.00	0.00
25[b]	191.68	157.03	0.00	0.00
34	−114.91	75.83	$1.24\cdot10^{-2}$	$1.33\cdot10^{-2}$
35[c]	271.18	−117.07	0.00	0.00
45[b]	337.46	−279.92	0.00	0.00

[a] Sunflower seed oil (1), commercial oleic acid (2), commercial linoleic acid (3), ethanol (4), water (5). [b] Parameters taken from Rodrigues and Meirelles.[7] [c] Parameters taken from Rodrigues et al.[3]

Table 5. Mean Deviations between the Experimental and the Calculated Compositions in Both Phases (Δw)

system	100 w_5^{solv} [c]	T K	100 Δw[d] UNIQUAC	NRTL
refined sunflower seed oil[a]	0	298.2	2.38	0.60
	6.32	298.2	1.87	1.44
	12.84	298.2	1.38	0.72
	18.95	298.2	1.18	0.91
degummed sunflower seed oil[b]	0.12	283.2	0.71	0.92
	5.72	283.2	0.58	0.34
		298.2	0.54	0.91
		333.2	0.80	3.46
	18.34	283.2	0.22	0.64
		298.2	0.20	0.95
		333.2	0.47	1.73
	29.13	298.2	0.15	0.77
		333.2	0.37	1.56
global deviation of the correlation			1.41	1.13

[a] Data taken from Table 2. [b] Data taken from Cuevas et al.[1] [c] w_5^{solv} = mass fraction of water in the alcoholic solvent. [d] Calculated according to Cuevas et al.[1]

seed oil + oleic or linoleic acid + ethanol + water at (298.2 ± 0.1) K. All compositions are expressed as mass percentages.

Tables 3 and 4 present the adjusted parameters of the NRTL and UNIQUAC models. These thermodynamic equations were used to correlate the experimental data set obtained from the systems containing refined sunflower seed oil, oleic or linoleic acids, and mixed solvent at (298.2 ± 0.1) K, to those obtained from the systems containing degummed sunflower seed oil, free fatty acids (considered as commercial linoleic acid), and mixed solvent, from (283.2 to 333.2) (± 0.1) K. Average deviations between experimental and calculated compositions in both phases were calculated according to the procedure presented elsewhere[1] and are shown in Table 5.

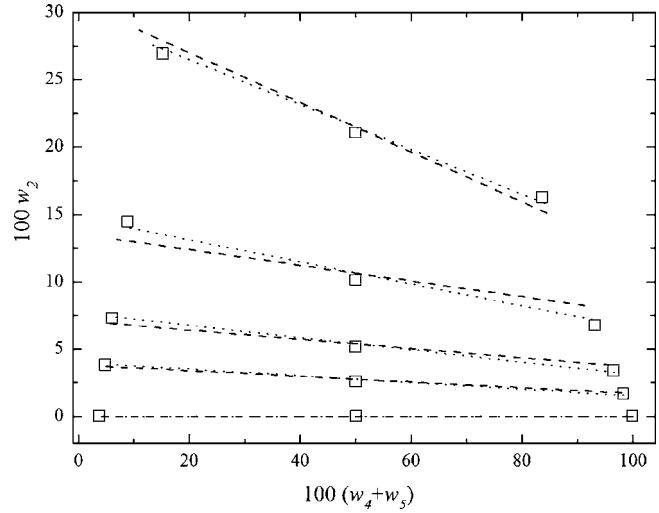

Figure 1. System of refined sunflower seed oil (1) + commercial oleic acid (2) + aqueous solvent [ethanol (4) + water (5), where w_5^{solv} = 18.95 %], at (298.2 ± 0.1) K. □, experimental; - - -, NRTL model; ·····, UNIQUAC model.

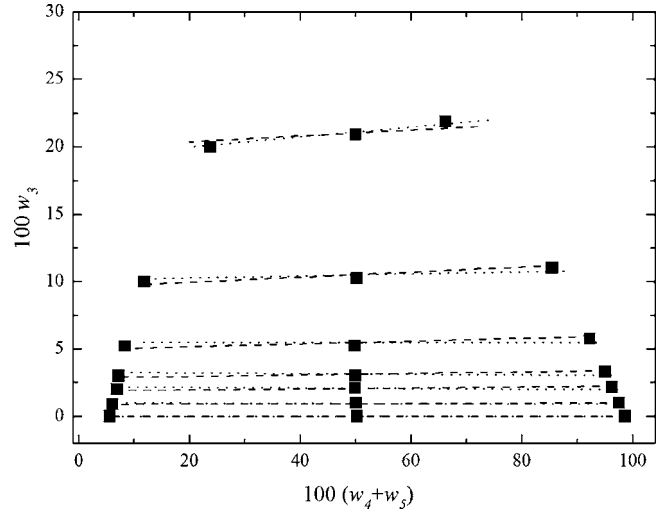

Figure 2. System of refined sunflower seed oil (1) + commercial linoleic acid (3) + aqueous solvent [ethanol (4) + water (5), where w_5^{solv} = 6.32 %], at (298.2 ± 0.1) K. ■, experimental; - - -, NRTL model; ·····, UNIQUAC model.

In this work, some interaction parameters (24, 25, 35, and 45) were obtained from previous reports,[3,7] and the interaction parameters between oleic and linoleic acid (23) were considered null.

Figures 1 and 2 show the experimental points and the tie lines calculated using the molecular models for the systems composed of refined sunflower seed oil + commercial oleic acid + ethanol with 18.95 % water, at (298.2 ± 0.1) K, and refined sunflower seed oil + commercial linoleic acid + ethanol with 6.32 % of water, at (298.2 ± 0.1) K, respectively. The equilibrium diagram was plotted in rectangular coordinates. To represent the pseudoquaternary system in rectangular coordinates, ethanol + water were admitted to be a mixed solvent. In these figures, the mass fraction composition of the vegetable oil can be obtained by difference. Both thermodynamic models studied are able to accurately describe the phase compositions for the systems investigated.

Figure 3 presents the performance of both sets of parameters obtained herein to describe the phase equilibrium for systems

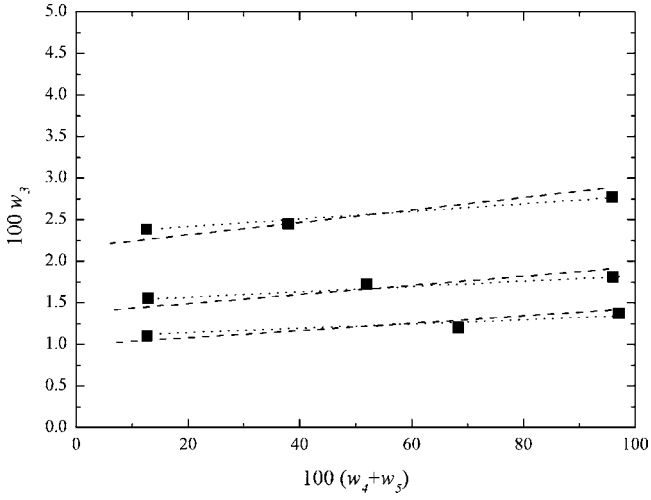

Figure 3. System of degummed sunflower seed oil (1) + commercial linoleic acid (3) + aqueous solvent [ethanol (4) + water (5), where $w_5^{solv} = 5.72$ %], at (333.2 ± 0.1) K. ■, experimental;[1] - - -, NRTL model; •••••, UNIQUAC model.

composed of degummed sunflower seed oil, a naturally free fatty acid-containing oil, ethanol, and water, at (333.2 ± 0.1) K.[1] The analysis of this figure indicates that the adjusted parameters describe reasonably well the partitioning of free fatty acids and the compositions of the alcohol and oil phases.

In Table 5, which presents the average deviations between experimental and calculated compositions in both phases, it can be observed that the lower deviations are related to systems with high water content in the solvent. In addition, it is possible to note that the NRTL model presented larger deviations for systems determined at higher temperatures, regardless of water level in the alcoholic extractant.

The estimated NRTL and UNIQUAC models parameters are considered representative and consistent, considering factors such as the complexity of the studied systems and sources of vegetable oils (refined and degummed sunflower seed oils) and of fatty acids (commercial linoleic acid and free acidity in degummed oil) with different fatty acid compositions.

Conclusions

In the presented paper, experimental equilibrium data for refined sunflower seed oil-containing systems were measured at (298.2 ± 0.1) K. These data, associated with equilibrium data previously determined from (283.2 to 333.2) (± 0.1) K, were correlated by NRTL and UNIQUAC models with temperature-dependent binary interaction parameters that represented the phase equilibrium reasonably well. The good descriptive quality of the thermodynamic models was indicated by the low global deviation values between calculated and experimental data, (1.13 and 1.41) %, for NRTL and UNIQUAC equations, respectively.

The estimated parameters of the thermodynamic models are representative, and based on them it is possible to model and simulate, with reasonable accuracy, liquid−liquid extractors for vegetable oil deacidification. In fact, the results shown in this paper corroborate the efforts of our research group to improve the vegetable oil deacidification technique by liquid−liquid extraction using ethanol as solvent.

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