



## Aqueous immiscibility of cholinium chloride ionic liquid and Triton surfactants



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### ARTICLE INFO

#### Article history:

Received 28 May 2015

Received in revised form 18 July 2015

Accepted 21 July 2015

Available online 29 July 2015

#### Keywords:

Aqueous biphasic systems

Ionic liquids

Cholinium chloride

Non-ionic surfactants

Triton

### ABSTRACT

The immiscibility windows of aqueous solutions containing the ionic liquid cholinium chloride ( $N_{1112}OHCl$ ) and the non-ionic surfactants Triton X-100 and Triton X-102 have been determined by the cloud point method at temperatures ranging from  $T = (298.15 \text{ to } 333.15) \text{ K}$ . The experimental values have been correlated by using two well-known equations. The tie-lines have been ascertained by means of density and refractive indices measurement, and the experimental data have been modeled by the Othmer–Tobias, Bancroft and Setschenow equations. The use of cholinium chloride involves greater demixing capacity than other imidazolium-based ionic liquids.

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### 1. Introduction

Over the last years, environmental concerns have highlighted the extensive and increasing importance of implementing industrial processes using greener solvents. Hence, a key target for enhancing competitiveness of the chemical industry is to reduce the environmental impact when manufacturing high value products. In particular, the replacement of volatile organic compounds with non-flammable and tuneable ionic liquids [1] has set the pace for the achievement of truly revolutionary processes. The last discovery involving these salts allows envisaging the prominence that these solvents may have in the near future: a new IL-based rechargeable battery system affording charging times of around one minute [2]. Currently, these molten salts are already used at an industrial scale in companies such as BASF, Institut Français du Pétrole, Degussa, Linde, Pionics and G24i, and the annual production of some of them (mostly belonging to imidazolium family) exceeds the ton per year [3,4]. However, these promising expectations can be jeopardized when bearing in mind the toxicity and persistence of some cations like the imidazolium. In this sense, the use of more biocompatible ionic liquids like those based on the cholinium cation is the subject of more and more studies focused on a diversity of topics that range from fundamentals to the demonstration of their low environmental impact or their

biocompatibility with enzymes [5,6]. These features have furthered their application in separation processes.

Very often, conventional (liquid + liquid) extraction strategies involve the use of volatile and toxic organic solvents. Thus, the emergence of this kind of biocompatible ionic liquids opens up new roads in the development of more environmentally friendly aqueous biphasic systems (ABS) [7,8]. This separation method consists in the phase segregation of an aqueous solution containing one hydrophilic compound when a certain amount of another hydrophilic compound is added. Traditionally, the most common combination was a polymer and a salt. However, since 2003, when Rogers *et al.* [9] reported the ability of ionic liquids to trigger phase disengagement, many authors have applied this kind of systems for the separation of a variety of biomolecules like enzymes [10,11], antioxidants [12] or alkaloids [13], among others. Several reasons justify the interest in applying ionic liquid-based ABS in the extraction of this type of molecules, *e.g.* short periods of time are required for phase disengagement, low energy demand or the possibility to work at mild operating conditions [14].

Another step towards the building of more competitive ionic liquid-based ABS could be the use of non-ionic surfactants, since they provide more advantages like a low interface tension, low cost (non-ionic surfactants are inexpensive), greater immiscibility region and negligible flammability and volatility [15]. In this sense, we have recently reported for the first time the ability of imidazolium-based ionic liquids to promote phase splitting in aqueous solutions of surface active compounds [16]. Among the

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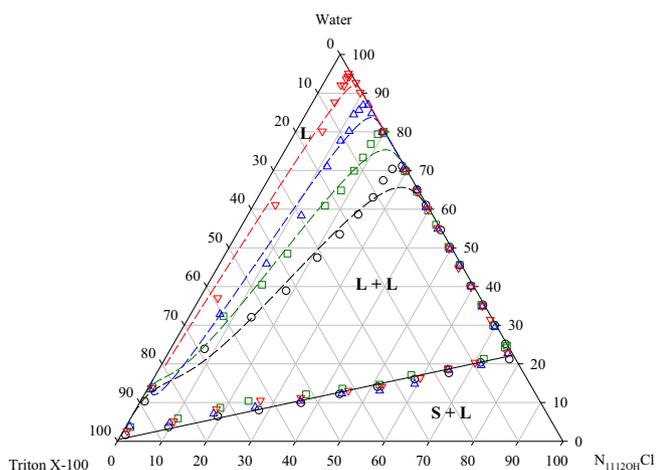
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**TABLE 1**  
Purities, provenance and characteristics of chemicals.<sup>a</sup>

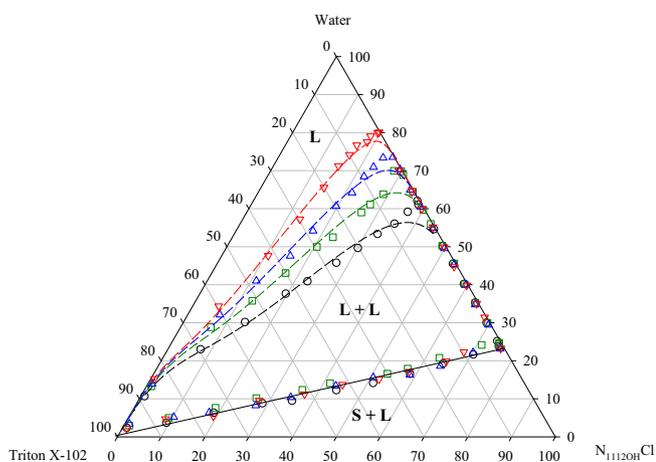
Compound	Chemical structure	Supplier	Mass fraction purity	HLB <sup>b</sup>	CMC (ppm) <sup>b</sup>
Triton X-100 $n = 9.5$ Triton X-102 $n = 12$		Sigma-Aldrich	0.98 0.98	13.4 14.4	189 267
N <sub>11120H</sub> Cl			0.97		

<sup>a</sup> Milli-Q water was used in all the experiments.

<sup>b</sup> HLB: hydrophilic lipophilic balance; CMC: critical micellar concentration. HLB and CMC were obtained from the supplier.



**FIGURE 1.** Phase diagrams for the systems {Triton X-100 (1) + N<sub>11120H</sub>Cl (2) + H<sub>2</sub>O (3)} at  $T = 298.15$  K (○),  $T = 313.15$  K (□),  $T = 323.15$  K (△),  $T = 333.15$  K (▽) and  $P = 101.33$  kPa. Symbols represent experimental values, solid lines are guides to the eye and dashed lines refer to the model.



**FIGURE 2.** Phase diagrams for the systems {Triton X-102 (1) + N<sub>11120H</sub>Cl (2) + H<sub>2</sub>O (3)} at  $T = 298.15$  K (○),  $T = 313.15$  K (□),  $T = 323.15$  K (△),  $T = 333.15$  K (▽) and  $P = 101.33$  kPa. Symbols represent experimental values, solid lines are guides to the eye and dashed lines refer to the model.

possible surfactants, Triton X family is widely applied in the biotechnological sector (enzyme purification, pollutants solubilization agent in bioremediation, etc.) [17,18], and has thus been selected for the present work.

**TABLE 2**  
Phase equilibria of {Triton X-100 (1) + N<sub>11120H</sub>Cl (2) + H<sub>2</sub>O (3)} at  $T = (298.15$  to  $333.15)$  K and  $P = 101.33$  kPa.<sup>a</sup>

$T = 298.15$ K		$T = 313.15$ K		$T = 323.15$ K		$T = 333.15$ K	
100 $w_2$	100 $w_1$	100 $w_2$	100 $w_1$	100 $w_2$	100 $w_1$	100 $w_2$	100 $w_1$
(Liquid + liquid) equilibria							
74.51	0.32	74.89	0.90	70.02	0.51	68.01	0.57
69.71	0.28	69.37	0.68	64.47	0.75	64.38	0.61
64.44	0.31	64.07	0.68	59.32	0.65	59.42	0.71
59.21	0.61	59.38	0.49	54.12	0.44	54.17	1.03
54.12	0.39	54.02	0.39	49.88	0.57	49.49	0.62
49.55	0.31	49.18	0.59	44.47	0.65	44.34	0.51
45.29	0.12	43.53	0.48	39.01	0.49	39.74	0.31
38.74	0.21	39.89	0.44	34.83	0.24	35.01	0.50
34.71	0.22	34.98	0.54	29.03	0.65	29.52	0.51
29.83	0.23	29.59	0.58	19.51	0.55	19.47	0.61
28.36	0.52	20.02	0.38	14.71	0.54	9.46	0.41
26.53	3.09	19.72	0.24	12.84	0.17	7.68	31.23
25.95	6.63	18.75	1.86	12.12	29.55	7.20	0.10
25.87	11.11	18.47	4.69	11.95	7.90	6.68	56.28
24.75	16.63	18.45	8.19	11.73	1.39	5.97	13.92
23.19	23.32	18.16	11.92	11.60	17.40	4.97	0.68
21.16	31.40	17.80	17.37	11.45	2.93	4.95	7.41
18.43	42.68	16.15	22.96	11.27	11.06	4.92	3.16
14.04	53.91	13.92	37.57	10.82	4.72	4.61	1.16
7.64	68.50	12.23	47.31	10.52	43.59	4.52	1.80
0.97	88.71	7.65	60.02	6.61	60.55	4.35	0.58
		0.95	85.15	0.74	85.92	4.13	3.79
						0.78	85.47
(Solid + liquid) equilibria							
77.41	1.42	75.14	0.19	76.27	1.01	76.09	1.06
71.34	8.31	71.60	7.11	71.84	8.52	70.08	9.58
65.61	16.76	64.89	16.47	64.88	16.58	64.89	16.36
58.71	25.22	57.42	25.48	59.30	25.87	59.79	23.84
51.39	34.53	51.54	33.83	52.34	34.60	52.32	33.62
43.78	44.03	43.72	42.68	44.24	43.45	45.31	41.61
36.27	53.82	36.27	51.58	35.84	53.76	35.55	53.21
27.74	64.22	24.21	65.37	26.53	64.67	26.85	62.63
19.33	74.23	18.77	72.58	18.06	74.81	18.09	73.60
9.67	86.67	10.63	83.46	9.38	85.88	10.07	84.78
1.06	97.33	1.07	95.25	0.82	95.29	0.88	96.51

<sup>a</sup> Standard uncertainties are  $u_c(w) = \pm 0.02$ ,  $u(T) = \pm 0.01$  K;  $u(P) = \pm 2$  kPa.

Then, in view of the above, the immiscibility regions for the systems (Triton X-100 or Triton X-102 + N<sub>11120H</sub>Cl + H<sub>2</sub>O) have been determined at several temperatures, and the experimental data were correlated with known equations. The tie-lines were also ascertained in order to deeper characterize the extraction capacity. The use of models like Othmer–Tobias, Setschenow and Bancroft helped to elucidate the consistency of the experimental tie-line data.

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