Non-aqueous solvent extraction of indium from an ethylene glycol feed solution by the ionic liquid Cyphos IL 101: speciation study and continuous counter-current process in mixer-settlers

Clio Deferm, Bieke Onghena, Viet Tu Nguyen, Dipanjan Banerjee, Jan Fransaer and Koen Binnemans*

Electronic Supplementary Information (ESI)



Fig. S1 Effect of the LiCl concentration on the mutual solubility for the EG+(5 vol% Cyphos IL 101 in toluene) system (\blacksquare) and the EG+(5 vol% Aliquat 336 in toluene) system (\blacktriangle): (a) solubility of EG in the toluene phase; (b) solubility of toluene in the EG phase; (c) solubility of Aliquat 336 and Cyphos IL 101 in the EG phase.



Fig. S2 Effect of the extractant and LiCl concentration on the mutual solubility for the EG+(Cyphos IL 101 in toluene) system (\blacksquare) and the EG+(Aliquat 336 in toluene) system (\blacktriangle): (a) solubility of EG in the toluene phase, 5 vol% (filled), 10 vol% (half-filled), 20 vol% (unfilled) of extractant; (b) solubility of toluene in the EG phase, 5 vol% (filled), 10 vol% (half-filled), 20 vol% (unfilled) of extractant.



Fig. S3 Raman spectra at of solutions containing 1 M LiCl (2), 2 M LiCl (3) and 3.5 M LiCl (4) in EG (1). λ_{laser} = 532.0 nm.



Fig. S4 Raman spectra of solutions containing 1 M LiCl (a), 2 M LiCl (b) and 3.5 M LiCl (c) as a function of the indium concentration. EG (1), 5 gL⁻¹ indium (2), 10 gL⁻¹ indium (3), 20 gL⁻¹ indium (4), 40 gL⁻¹ indium (5), 60 gL⁻¹ indium (6). λ_{laser} = 532.0 nm.



Fig. S5 Raman spectra of solutions containing 5 gL⁻¹ indium (a), 10 gL⁻¹ indium (b), 20 gL⁻¹ indium (c) and 40 gL⁻¹ indium (d) as a function of the LiCl concentration. EG (1), 0 M LiCl (2), 1 M LiCl (3), 2 M LiCl (4), 3.5 M LiCl (5). λ_{laser} = 532.0 nm.



Fig. S6 Infrared spectra of solutions containing 0 M LiCl (a), 1 M LiCl (b), 2 M LiCl (c) and 3.5 M LiCl (d) as a function of the indium concentration. EG (1), 5 gL^{-1} indium (2), 10 gL^{-1} indium (3), 20 gL^{-1} indium (4), 40 gL^{-1} indium (5), 60 gL^{-1} indium (6).



Fig. S7 Infrared spectra of solutions containing 5 gL^{-1} indium (a), 10 gL^{-1} indium (b) and 20 gL^{-1} indium (c) as a function of the LiCl concentration. EG (1 0 M LiCl (2), 1 M LiCl (3), 2 M LiCl (4), 3.5 M LiCl (5).



Fig. S8 Infrared spectra of solutions containing 40 gL⁻¹ indium (a) and 60 gL⁻¹ indium (b) as a function of the LiCl concentration. EG (1), 0 M LiCl (2), 1 M LiCl (3), 2 M LiCl (4), 3.5 M LiCl (5).



Fig. S9¹¹⁵In NMR spectra of the LP phase 5 vol% of Cyphos IL 101 in toluene (a) and the LP phase 5 vol% of Aliquat 336 in toluene (b) obtained after extraction with 5 g L^{-1} indium(III) and varying LiCl concentration: 0 M LiCl (1), 1 M LiCl (2), 2 M LiCl (3), 3.5 M LiCl (4).



Fig. S10 Raman spectra of the less polar phase: 5 vol% Cyphos IL 101 in toluene (a) and 5 vol% Aliquat 336 in toluene (b) obtained before (1) and after extraction (2) with 5 g L^{-1} indium(III) and 3.5 M LiCl in EG.



Fig. S11 Effect of the LiCl concentration on the mutual solubility for EG+(2.56 vol% Cyphos IL 101 in toluene) system (\blacksquare) and the EG+(2.56 vol% Cyphos IL 101 in *p*-cymene) system (\blacktriangle): (a) solubility of EG in the toluene/p-cymene phase; (b) solubility of toluene/p-cymene in the EG phase; (c) solubility of Cyphos IL 101 in the EG phase.

	McCabe–Thiele diagram extraction	McCabe–Thiele diagram scrubbing
MP:LP phase ratio	%E _{Zn}	%S _{Zn}
1:11	98	20
1:7	91	33
1:5	80	42
1:3	47	55
1:2	27	68
1:1	9	80
2:1	4	87
3:1	2	92
5:1	1	97
7:1	0.8	98
11:1	0.4	98

Table S1 % E_{zn} and % S_{zn} at different MP:LP phase ratio. Conditions: room temperature, 600–1000 rpm, 3 h, [Cyphos IL 101] = 2.56 vol%, [LiCI] = 3.5 M, [1-decanol] = 1 vol%. Initial metal concentrations extraction: [In(III)] = 5 g L⁻¹, [Zn(II)] = 5 g L⁻¹. Metal concentration scrubbing solution: [In(III)] = 5 g L⁻¹.



Fig.S12 Percentage extraction (%E) of indium(III) (\blacksquare) and zinc(II) (\blacktriangle) as a function of the reaction time for the EG+(2.56 vol% Cyphos IL 101 in *p*-cymene) system. Conditions: volume MP:LP = 1:2, room temperature, 600 rpm, [LiCl] = 3.5 M, [1-decanol] = 1 vol%. Initial metal concentrations: [In(III)] = 5 g L⁻¹, [Zn(II)] = 5 g L⁻¹.



Fig. S13 Percentage extraction of indium(III) (%E_{in}) (\blacksquare) and percentage scrubbing of zinc(II) (%S_{zn}) (\blacktriangle) as a function of the reaction time for the loaded MP phase. Conditions: volume MP:LP = 1:1, room temperature, 600 rpm, [LiCI] = 3.5 M. Metal concentration scrubbing solution: [In(III)] = 5 g L⁻¹.



Fig. S14 Concentration profile of In(III) (black) and Zn(II) (red) in the extraction stages of the mixersettler units. Conditions: volume MP:LP = 1:2, room temperature, 1000 rpm. Initial metal concentrations: $[In(III)]_{MP} = 5 \text{ g L}^{-1}$, $[Zn(II)]_{MP} = 5 \text{ g L}^{-1}$ in EG. LP phase: 2.56 vol% Cyphos IL 101, 1 vol% 1-decanol in *p*-cymene.



Fig. S15 Concentration profile of In(III) (black) and Zn(II) (red) in the scrub stages of the mixer-settler units. Conditions: volume MP:LP = 1:2, room temperature, 1000 rpm. Initial scrub feed concentration: $[In(III)]_{MP} = 5 \text{ g L}^{-1}$ in EG. Loaded LP phase: 2.85 g L⁻¹ indium(III), 0.55 g L⁻¹ zinc (III), 2.56 vol% Cyphos IL 101, 1 vol% 1-decanol in *p*-cymene.