



Review

Recovery of indium and yttrium from Flat Panel Display waste using solvent extraction

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ABSTRACT

Oxides of indium and yttrium are two of the key components in Flat Panel Displays (FPDs). In recent years the need to recycle these metal oxides from waste FPDs has been growing. In this work a process to recycle indium and yttrium based on acid leaching and solvent extraction was proposed and studied. Solid waste was leached by acid at S/L ratio = 0.1 g/ml, HCl was found to be more effective than HNO₃, possibly due to the formation of soluble metal chloride complexes. The extraction of indium using Cyanex 923 and yttrium using DEHPA from chloride media studied using lab-scale mixer-settlers at a flow rate of 3 ml/min. Leachate of real FPD waste was used as aqueous feed. Good separation between indium, yttrium and other impurities such as iron, copper and aluminum could be achieved by extraction from 1 M HCl with 0.25 M Cyanex 923 diluted in kerosene, followed by stripping with 1 M HNO₃ and further purification with 0.2 M DEHPA diluted in kerosene.

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Contents

1. Introduction	118
2. Experimental	118
2.1. Acid leaching	118
2.2. Batch solvent extraction	118
2.3. Mixer-settler tests	118
2.4. Sample analysis	118
3. Results and discussion	119
3.1. Leaching experiments	119
3.1.1. Composition of soluble metals in the FPD waste	119
3.1.2. Acid leaching behavior of HCl and HNO ₃	119
3.1.3. Optimal leaching acid and conditions	120
3.2. Solvent extraction process	120
3.2.1. Batch extraction experiments	120
3.2.2. Mixer-settler tests	122
3.3. Third phase and crud formation	123
3.4. Solvent extraction process	124
4. Conclusion	124
Acknowledgements	124
References	124

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1. Introduction

During the last decade, the global shipment of Flat Panel Displays (FPDs), especially Liquid Crystal Displays (LCDs) has surpassed Cathode Ray Tube (CRT) displays and has become the most popular type of displays on the market [1]. A typical LCD contains approximately 0.7 g indium per m² in the form of Indium-Tin Oxide (ITO) [2]. In addition, small amounts of Rare Earth Elements (REEs) can be found in the backlight units and loudspeakers. These metals are considered to be critical raw materials by the European Commission due to their economic importance, availability and supply risk [3]. Therefore in recent years the recovery of indium and REEs from secondary sources such as production waste and End-of-Life (EoL) products has been gaining interest. Previous studies have shown that many different methods can be used to recover indium from waste LCDs, such as chloride vaporization [4], supercritical CO₂ extraction [5] and solvent extraction. In addition, ionic liquids have also been tested for separation of REEs [6]. In this study solvent extraction was chosen due to its simplicity in terms of material, equipment and process conditions required, and its efficiency in metal separation. Numerous previous studies have shown that indium can be recovered from aqueous solutions using methods such as solvent extraction, and extensive studies on organic molecules suitable for indium extraction have been carried out [7]. Many different extractants, especially organophosphate compounds such as Tributyl Phosphate (TBP) [8], (Cyanex 272) [9], bis-2,2-ethylhexyl phosphate (DEHPA) [10,11], 2-ethylhexyl phosphonic mono-2-ethylhexyl ester [12] and Cyanex 923 (a mixture of four trialkyl phosphates) [13] have been tested. Results of these earlier studies showed that apart from Cyanex 272, other extractants were all able to selectively extract indium from many other elements such as transition metals from either acidic chloride or sulfate media. With respect to the extraction of REEs from aqueous solutions, earlier studies have shown that Cyanex 923 can be an effective extractant for the extraction of REEs from e.g. phosphor powders of EoL fluorescent lamps and electrode materials of Nickel Metal Hydride (NiMH) batteries [14,15]. In addition, there have been a number of previous studies on the recovery of indium from leachate of LCD waste [12,2,16]. However, since the FPD waste material studied in the present work contain both indium and yttrium (>10 ppm), modification of the previously designed method for indium recycling [2] was necessary. In this work, a recycling method based on acid leaching followed by solvent extraction was explored and presented. In the acid leaching studies, composition of soluble metals in the waste and optimal leaching conditions were determined. Based on a previous study [17], Cyanex 923 and DEHPA were used as the extractants in the design of a laboratory-scale solvent extraction process for the separation and recovery of indium and yttrium from the acidic leachate containing different metal ions. Furthermore, a problem encountered in the solvent extraction process regarding crud formation was discussed and a few possible solutions were proposed.

2. Experimental

2.1. Acid leaching

The solid waste sample was in the form of powders (<1 mm) and was one of the fractions obtained from a FPD waste shredding line. All leaching experiments were performed in 120 ml plastic beakers. The rate of stirring was approximately 700 rpm with 6 × 20 mm stirring magnets. Aqua regia was used to determine the total metal composition in the solid waste. For other studies the initial concentration of HCl or HNO₃ was 1 M, samples were taken between 1 h and 4 days after leaching started, the solid to

liquid ratio was 0.1 g/ml. A simple test on the effect of increased temperature on leaching efficiency was performed by leaching the samples at 22 and 80 °C. The acid solutions used for leaching were diluted from concentrated HNO₃ (65%, Sigma Aldrich) or HCl (>37%, Sigma Aldrich) with de-ionized water. It was calculated from the difference in pH of acid before and after leaching that acidity after leaching was reduced by approximately 15% from 1 M. Due to this reduction in acidity had little effect on metal extraction, the leachate was used as aqueous feed in subsequent solvent extraction processes.

2.2. Batch solvent extraction

Batch extractions were performed using the following procedure: 1.5 ml of each organic and aqueous phase was added to a 3.5 ml glass vial (O/A = 1). The extractions were performed at room temperature (21 ± 1 °C). For batch tests, an extraction kinetics study was performed and it was found that metal extraction using Cyanex 923 reached equilibrium after approximately 5 min of mechanical shaking (IKA VIBRAX VXR basic) at 1500 rpm. A contact time of 5 min was therefore used. The organic diluent studied was kerosene (Solvent 70, Statoil), and the extractants were Cyanex 923 (93%, Cytec) and DEHPA (97%, Sigma Aldrich). For the construction of a McCabe-Thiele diagram (calculation of number of ideal stages), the volume ratio of the organic and aqueous phases (O/A) varied from 1:5 to 5:1.

2.3. Mixer-settler tests

Mixer-settler experiments were performed using laboratory scale mixer-settlers in counter-current arrangement as described in an earlier work [18]. The mixer-settlers were made of PVDF, for both indium and yttrium extraction experiments, the volumes of the mixing and settling chambers were 100 ml and 500 ml, respectively. The pump rate was set to approximately 2 ml/min for both organic and aqueous flows. The rate of stirring was set to approximately 1000 rpm, and samples of both phases were taken after 600 ml (2 stages) or 900 ml (3 stages) of solution had been pumped through the system, in order to ensure that equilibrium was reached. Leachate of the solid waste was used as aqueous feed (HCl with an initial concentration of 1 M). The organic feed was 0.25 M Cyanex 923 diluted in kerosene, with 1% toluene.

The separation and recovery process of indium and yttrium from acidic leachate was divided into four major steps: (1) separation of indium and yttrium with Cyanex 923. (2) back-extraction of indium from loaded organic phase to another aqueous phase. (3) Extraction of yttrium from the aqueous phase in step (1) with DEHPA. (4) back-extraction of yttrium from the loaded organic phase from step 3.

2.4. Sample analysis

Metal concentrations in the acidic leachate and the aqueous phase of solvent extraction experiments were determined using Inductively Coupled Plasma with Optical Emission Spectrometer (Thermo iCAP-6000). Aqueous concentrations of metals were quantified by calibration with 1000 ppm external standards (Sigma-Aldrich). The detection limit of the instrument for the metals of interest is in the range of parts per billion (<0.1 µM), which is low enough for the quantification to be reliable. More precise values of distribution ratios below 0.01 were not presented due to measured values being near the detection limit of the instrument.

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