

# The recovery of rare earth metals from WEEE leaching solution via liquid-liquid extraction

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## Abstract

The recovery of rare earth metals (REMs) from end-of-life products, such as Waste Electrical and Electronic Equipment (WEEE), is drawing great attention as an attractive strategy for promoting the sustainable development. The hydrometallurgical technique of solvent extraction has been reported to be one of the most interesting method to recover REMs. However, when applied to WEEE, this process is challenged by the heterogeneous composition of electronic waste, completely different from other solid matrices, and it still has much rooms of improvements.

This study investigated the extraction, stripping and recovery of REMs from a WEEE leaching solution using Versatic 10 as carrier in the organic phase and oxalic acid as stripping agent. A factorial design was carried out to evaluate the simultaneous effects of factors as the feed phase pH and the concentrations of both extractant and organic phase modifier in the extraction process. Cerium, lanthanum and yttrium were extracted at high percentages using 200 mM of Versatic 10, loaded by 100 mM of TBP in kerosene at pH 7. Moreover, 750 mM of oxalic acid successfully stripped and recovered 7.63 and 13.82 mg/kg of lanthanum and yttrium, respectively.

**Keywords:** Solvent extraction, critical metals, versatic 10, couple countered, oxalic acid, hydrometallurgy.

## 1. Introduction

Rare earth metals (REMs) are vital raw materials for developing renewable energy technologies and essential elements for modern high-tech products. Global REM demand is knocked to grow at an annual rate of 5% by 2020. This increasing demand will require a steady supply base of REMs in the long run (Tanushree *et al.*, 2016). In this view, there is an urgent need to develop efficient REM reclaiming techniques from end-of-life products or secondary sources as well as replace these critical elements with possible substitutes (Alonso *et al.*, 2012).

Waste electrical and electronic equipment (WEEE) is considered as a noticeable source of various elements, including REMs, whose recycling would have an enormous

impact on the environmental protection policies and approaches (Buchert *et al.*, 2012; Binnemans *et al.*, 2013; Innocenzi Baldé *et al.*, 2014; Baldé *et al.*, 2015; Dupont and Binnemans, 2015; Savvilotidou *et al.*, 2015; Diaz *et al.*, 2016). However, in EU countries the total amount of the recycled WEEE is still low, owing to insufficient collection rates, legal and illegal exports and poor recovery rates of end-refining processes with particular regard to some metals as REMs (Bakas *et al.*, 2014).

Numerous researchers have been trying to work out an efficient way to enhance REMs recovery from WEEE. For this purpose, hydrometallurgy, involving metals leaching into solution followed by their separation, is the favourite route for REE recovery from end-of-life products (Tunsu *et al.*, 2015). Although this process can be successfully applied for extracting different metals, the researchers are still studying the most efficient and simultaneously environmentally friendlier methods to recover these elements from WEEE.

Solvent extraction or liquid-liquid extraction (LLE) is being widely explored in various fields such as chemistry (organic, inorganic and analytical), biotechnology, biomedical technology and wastewater treatment (Muthuraman *et al.*, 2009; Singh *et al.*, 2011; Talebi *et al.*, 2012). This technique can be applied for different purposes, including the selective separation and recovery of heavy metals, separation of aromatics from hydrocarbons, antibiotics purification, protein extraction using aqueous two-phase systems, dyes and pigments removal, metallurgical purifications (Chang *et al.*, 2011).

In conventional systems for aqueous solution treatment, the recovery of metals is customarily carried out in a separate unit by elution with suitable reagents, occurring in additional cost (Kurniawan, 2006). Conversely, in liquid membrane (LM) systems the extraction and the recovery of the separated species can be combined in one stage. Hence, liquid membranes are more cost-effective and environmentally friendlier than other conventional techniques because they need lower volumes of organic reagents (Keith *et al.*, 2007; Armenta *et al.*, 2008; León and Guzmán, 2010).

The LLE is considered as the fundamental step for the LM technique and it has been investigated for REMs removal from industrial wastewaters (Harris and Tompkins, 1947; Bunus *et al.*, 1994; Preston *et al.*, 1996; Lee *et al.*, 2009; Tunsu and Retegan, 2016) and for REM ions separation in ore processing and secondary sources (Tunsu *et al.*, 2016) in order to explore the potential scale up to LM systems.

In this framework, this study aims to evaluate the application of LLE for the recovery of REMs from a WEEE leaching solution. In this regard, Versatic 10 was tested as a carrier in the organic phase. The effect on REM extraction of parameters such as pH, carrier and phase modifier concentrations was investigated. Furthermore, the effectiveness of the stripping and recovery of the targeted metals from the loaded organic phase using oxalic acid was examined.

## 2. Material and methods

### 2.1. Leaching feed solution

The aqueous feed solution for the LLE experiments was obtained from a leaching process involving WEEE shredding dust as solid source. The material characterization of this fraction is described in our previous studies (Marra *et al.*, 2018).

The leaching process was performed immersing 20 gr of WEEE shredding dust in a solution containing 160 mL of 2 M H<sub>2</sub>SO<sub>4</sub> and 40 mL H<sub>2</sub>O<sub>2</sub>. The solution was kept stirred at 150 rpm for 3 hours using a jar test machine. The leachate was then filtered and the resulting solution was further used for the LLE procedure. The concentration of selected REMs, namely Cerium (Ce), Lanthanum (La) and Yttrium (Y), in the leaching solution is reported in Table 1.

**Table 1.** Concentration of REMs in the WEEE leaching solution

Metal	Concentration (mg/L)
Ce	5.2 ± 0.15
La	4.4 ± 0.13
Y	5.2 ± 0.15

### 2.2. LLE experiments

The LLE procedure included an extraction step followed by a stripping phase.

Versatic acid 10 (V10, purity ≥98%) and tributyl phosphate (TBP, purity ≥99%), both supplied by SIGMA-Aldrich, were used to prepare the organic phase: the former was employed as a carrier and the latter as phase modifier. Kerosene, supplied by Honeywell, was used as diluent.

The prepared organic phase was mixed with the WEEE leaching solution at 1:1 ratio in a conical flask, which was kept shaken by a magnetic stirrer. After two hours stirring, samples were taken from the aqueous phase, and the concentration of the metals under investigation was determined. An extraction run in which the organic phase contained neither carrier nor phase modifier was also carried out as a control experiment.

A factorial design was performed to define the optimal experimental conditions regarding pH, carrier and phase

modifier concentrations for obtaining the highest metal extractions. These factors were assumed to vary between the levels reported in Table 2. A 13-run factorial design of experiments was proposed, including also central points.

pH was adjusted using HCl and NH<sub>4</sub>OH.

**Table 2.** Factors and levels of the experimental design

Factors	Levels		
	Low (-)	Medium (0)	High (+)
pH	5.0	6.0	7.0
V10 concentration (mM)	75.0	137.5	200.0
TBP concentration (mM)	50.0	100.0	150.0

The metal-loaded organic phase, obtained after the extraction process, was used for the further stripping studies. In this stage, 750 mM oxalic acid (C<sub>2</sub>H<sub>2</sub>O<sub>4</sub>) was tested as stripping agent using an organic to stripping volume ratio of 0.5.

The white precipitate resulting from 2 hours stripping was then collected and a mineral phase analysis was conducted using the X-ray diffraction (XRD, Bruker D8 advance), with the following conditions: Cu K $\alpha$  radiation, 35 keV accelerating voltage, 40 mA current, 10-80° scanning range, 0.5 s/step (0.0296°/step) scan speed. The white precipitate was, moreover, calcinated in a furnace for one hour and analysed for its metal content after acid digestion.

### 2.3. Analytical methods

The metal analyses were performed by means of the inductively coupled plasma optical emission spectroscopy (ICP-OES, Thermo iCap 6000 series).

During each extraction experiment, samples were taken from the aqueous phase, filtered, adequately diluted and analysed for the selected REM concentrations.

The extraction efficiency (E%) of the metal was, thus, calculated according to the following equation:

$$E\% = \left( \frac{[M_{aq,in}^{2+}] - [M_{aq}^{2+}]}{[M_{aq,ini}^{2+}]} \right) \times 100 \quad (1)$$

where  $[M_{aq,ini}^{2+}]$  denotes the initial concentration of the metal ion in the aqueous solution (mg/L) and  $[M_{aq}^{2+}]$  stands for the metal concentration in the aqueous phase after the extraction process (mg/L).

## 3. Results and discussion

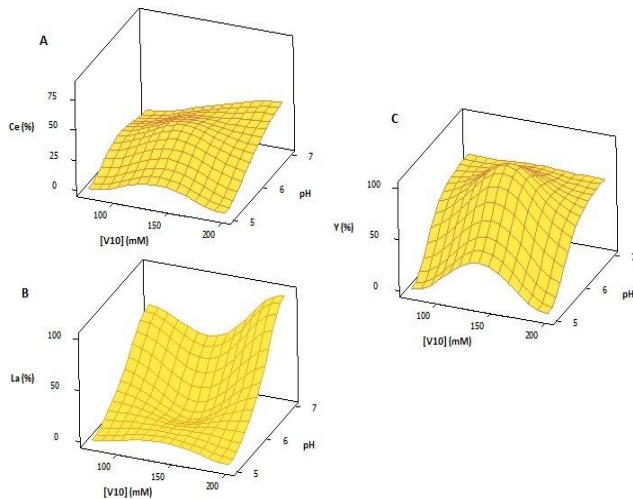
### 3.1. Extraction studies

Preliminary screening studies were conducted using only kerosene in the organic phase, without adding the carrier (V10) and the phase modifier (TBP). In these experiments, no metal ion extractions were observed, demonstrating that the solubility of the organic and the aqueous phase is a negligible factor in the extraction stage. The screening studies also revealed that the presence of TBP individually has not a significant effect on the extraction process. However, the TBP has an essential impact on the

separation of the organic-aqueous mixture as it acts reducing or eliminating emulsion phase by lowering the interfacial viscosity, tension, and elasticity of the organic-aqueous interface (Marcus, 2004; Kang *et al.*, 2006; Chagnes *et al.*, 2012).

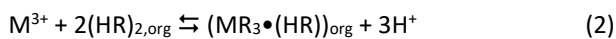
The surface responses of the extraction efficiencies of Ce, La and Y are plotted in Figure 1.

Results revealed that the organic phase, consisting of kerosene, V10 and TBP was highly efficient to extract up to the 100% of La and Y (Figure 1b and c, respectively) whereas for Ce the highest extraction percentage remained around 50% (Figure 1a).



**Figure 1.** Surface plot for cerium (a), lanthanum (b) and yttrium extraction (c)

During the extraction step, a complexation mechanism occurs between REM ions and V10 which can be described as below:



where *org* subscript denotes the organic phase,  $M^{3+}$  represents the trivalent REM whereas HR is the carrier. As V10 is an acidic carrier in forms of dimers in aliphatic solvents, it is aggregated as dimers or larger oligomers in non-polar organic solvents (Mason *et al.*, 1987).

The surface plot for Ce extraction versus pH and V10 (Figure 1a) shows that the highest efficiency took place when both variables were set in the corresponding middle level. This extraction behaviour is similar for Y, while for La the obtained surface plot displays a different pattern.

The typical extraction behaviour of the REMs lies in the increase in extraction efficiency with increase in the pH, as broadly observed. The obtained results are in agreement with those reported in literature, in which the significant effect of pH on the extraction efficiency is contributed to the metal ions extraction as described in Eq. 2 (Feng *et al.*, 2014).

After each run, the aqueous phase pH was measured, and the results showed a decrease in pH as soon as the extraction process took place (Table 3). Indeed, the uptake of metal ions decreases the pH of the aqueous feed phase (Agrawal *et al.*, 2012; Babakhani *et al.*, 2014; Noori *et al.*,

2014; Devi, 2015) as a result of an increase of  $[H^+]$  ions in the aqueous feed phase, which is a side effect of the metal-carrier complexation in the feed-organic interface (Eq. 2).

**Table 3.** pH variation ( $\Delta$ pH) in the aqueous phase for each run of the design of experiments

Run	pH	V10 (mM)	TBP (mM)	$\Delta$ pH
1	5.0	200.0	50.0	0.35
2	6.0	137.5	100.0	0.46
3	7.0	200.0	150.0	0.54
4	5.0	75.0	150.0	0.31
5	7.0	75.0	50.0	0.47
6	5.0	75.0	150.0	0.29
7	7.0	75.0	50.0	0.50
8	5.0	200.0	50.0	0.29
9	5.0	75.0	150.0	0.31
10	7.0	200.0	150.0	0.52
11	7.0	75.0	50.0	0.41
12	5.0	200.0	50.0	0.23
13	7.0	200.0	150.0	0.57

The extraction efficiency is affected by the solution pH as well as by the carrier concentration. This condition can be explained by applying the equilibrium constant ( $K_{eq}$ ) to Eq.2:

$$K_{eq} = [MR_3-RH_3]_{org}[H^+]^3/[M^{3+}] [(HR)]^2 \quad (3)$$

The distribution ratio (D) relating to the aqueous/organic system is:

$$D = [M^{n+}]_{org}/[M^{n+}]_{aq} \quad (4)$$

where  $[M^{n+}]$  represents the concentration of metal ion and the subscripts *org* and *aq* denote the organic and the aqueous phase, respectively.

Considering the metal concentration in the organic phase equal to metal-carrier complex concentration:

$$[M^{n+}]_{org} = [MR_3-RH_3] \quad (5)$$

Substituting Eq. 3 and Eq. 4 into Eq. 5 and rearranging, the distribution ratio results equal to:

$$D = K_{eq} \cdot [(HR)_2]^2/[H^+]^3 \quad (6)$$

Taking logarithm of Eq. 6 it gives:

$$\log D = \log K_{eq} + 2 \log [(HR)_2] - 3 \log [H^+] \quad (7)$$

Since  $-\log [H^+] = \text{pH}$ , Eq. 8 can be written as:

$$\log D = \log K_{eq} + 2 \log [(HR)_2] + 3 \text{pH} \quad (8)$$

Eq. 8 explains how the metal extraction, in terms of distribution ratio, is a function of both carrier concentration and aqueous feed phase pH.

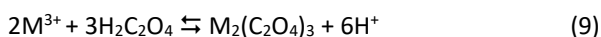
Based on the extraction studies, pH, carrier and phase modifier concentrations were set to 7, 200 mM and 100 mM, respectively, for the further stripping studies.

### 3.2. Stripping studies

The chemical characterization of the white precipitate resulting from the stripping studies revealed that oxalic acid was capable of 100% stripping of the two targeted REMs, lanthanum and yttrium, from the organic phase whereas in case of cerium, no stripping efficiency was

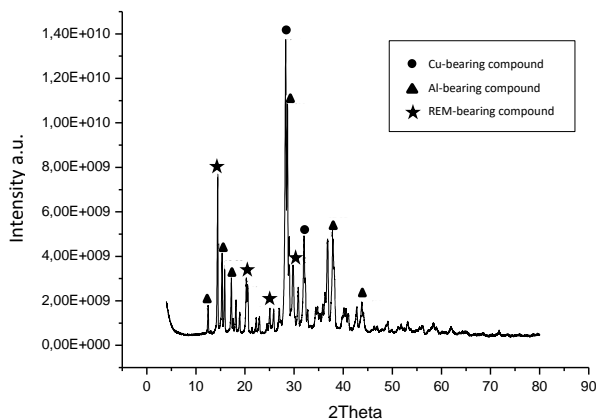
observed. At the end of the process, around 7.63 and 13.82 mg/Kg of lanthanum and yttrium, respectively, were recovered.

Stripping process, which consists of the decomplexation of metal-carrier compounds and the subsequent complexation between the released metal ions and the stripping agent, follows the Eq. 2 in left to right direction. The use of oxalic acid causes the occurrence of stripping and precipitation simultaneously as it can act as both stripping and precipitation agent. Indeed, the oxalic acid dissociates in the aqueous phase in hydrogen ions and oxalate anions: the hydrogen ions provide the stripping of the rare earth ions present in the organic phase and the oxalate anions their precipitation in form of white powders (Konishi *et al.*, 1998). Complexation between REM ions and oxalic acid ( $H_2C_2O_4$ ) can be presented as follow:



where  $M^{3+}$  represents the trivalent REM (Chi and Xu, 1999).

Figure 2 shows the XRD pattern of the white precipitate.



**Figure 2.** XRD pattern for the white precipitate

The mineral phase analysis confirmed the existence of rare earth compounds, mainly in the form of rare earth oxalates and oxides, although rare earth silicates and carbonates were found as well.

The occurrence of rare earth silicates is related to the presence of silicon dioxide in the WEEE dust source (Marra *et al.*, 2018). The rare earth carbonate existence is due to the reaction of the oxalic acid with Ca and Mg ions in solution to form oxalates. Reported electro-kinetic measurements indicated that  $Ca^{2+}$  and  $Mg^{2+}$  cations actively reduce the negative charge at the pyrochlore surface hindering cationic collector adsorption (Rao *et al.*, 1988; Chelgani *et al.*, 2012 a, b; Xia *et al.*, 2015). The higher concentration of oxalic acid might be a useful remedy to decrease the amount of rare earth-carbonate compounds. However, according to Chelgani *et al.* (2017), the acid addition could promote an increase in the rate of carbonate dissolution increasing the production of Mg and Ca in the solution.

However, both chemical characterization and mineral phase analysis pointed out that, apart from REMs, other metals ions including base metals and heavy metals (such as aluminium, cadmium, nickel, iron, copper and zinc) were co-stripped along with the REMs. The co-stripping of the metals ions is due to the simultaneously presence in WEEE shredding dust of a wide range of different metals, including base metals, in relative high concentrations (Marra *et al.*, 2018). In this regard, a prevalence of alumina compounds was found (Figure 2), due to the high concentrations of aluminium detected in the WEEE shredding dust (Marra *et al.*, 2018) as well as the strong chemical interaction existing between alumina and rare earth oxides (Bernal *et al.*, 2005).

#### 4. Conclusions

The solvent extraction of REMs from WEEE leaching solution using Versatic 10 as a carrier and TBP as phase modifier in kerosene was investigated. High efficiency in REM ion extraction was found. The results of a two-level factorial design revealed that high percentage of cerium, yttrium and lanthanum could be extracted at pH 7, [V10] 200 mM and [TBP] 100 mM. For the recovery of the leachate REMs from the organic phase, 750 mM of oxalic acid showed 100% of the REM recovery, except for cerium. XRD analysis revealed that the recovered REMs mainly occurred in forms of REM oxalates, but silicate and carbonate rare earth compounds were even found, as well as other heavy metal-bearing compounds.

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