Recovery of critical metals from lamps and CRTs

Valentina Innocenzi

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2. Relight Srl – Milan (Italy)
3. SAT- Austrian Society for Systems Engineering and Automation- Wien – (Austria)
In this presentation the main results of the scientific works about the development of the processes for the recovery of REEs from lamps and cathode ray tubes were presented. Activities were performed in the laboratory of chemical engineering of University of L’Aquila in Italy.
Development of hydrometallurgical processes

Recycling of WEEE

Recovery of critical and precious metals from WEEE

Principal Aims

HYDROWEEE (2008-2012)

HYDROWEEE demo (2012-2016)

Realization of two industrial demo plants (stationary and mobile)

Implementation of the processes

Study the technical, economical and social feasibility of the processes

European founds (European FP7 framework)

The projects were found to support SMEs (small, medium enterprises) in research and development
HYDROWEEE projects

Recovery of metals from WEE

Printed circuit boards

Spent batteries

Liquid Crystal Display

Spent catalysts

Fluorescent lamps and cathode rays tubes (CRTs)
HYDROWEEE projects

Recycling of WEEE

Rare Earth Elements

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<tr>
<th>La</th>
<th>Ce</th>
<th>Pr</th>
<th>Nd</th>
<th>Pm</th>
<th>Sm</th>
<th>Eu</th>
<th>Gd</th>
<th>Tb</th>
<th>Dy</th>
<th>Ho</th>
<th>Er</th>
<th>Tm</th>
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Lanthanides

- Sc
- Ti
- V
- Cr
- Mn
- Fe
- Co
- Ni
- Cu
- Zn
- Ga
- Ge
- As
- Se
- Br
- Kr
- Rb
- Sr
- Y
- Zr
- Nb
- Mo
- Tc
- Ru
- Rh
- Pd
- Ag
- Cd
- In
- Sn
- Sb
- Te
- I
- Xe
- Cs
- Ba
- Lu
- Hf
- Ta
- W
- Re
- Os
- Ir
- Pt
- Au
- Hg
- Tl
- Pb
- Bi
- Po
- At
- Rn

SCANDIUM
- LANTHANUM
- CERIUM
- THULIUM
- PRASEODIMIUM
- NEODIUM
- EUROPIUM
- GADOLINIUM
- TERBIUM
- DYSPROSIUM
- HOLMIUM
- ERBIUM
- YTTERBIUM
- LUTETIUM
HYDROREEE projects

6 OF MOST IMPORTANT REEs

Lanthanum

Main application:
- Battery alloys
- Metallurgy
- Auto catalysts
- FCC
- Phosphors
- Ceramics
- Others..
HYDROWEEE projects

6 OF MOST IMPORTANT REEs

Erbium

Erbium is used to produce lasers and super fast fiber optics
HYDROREEE projects

6 OF MOST IMPORTANT REEs

Neodymium

Neodymium is used to produce magnets

Main application:
- Magnets
- Battery alloys
- Metallurgy
- Auto catalysts
- Glass additives
- Ceramics
- Others..
Europium and Yttrium

6 OF MOST IMPORTANT REEs

Europium is an important metal used to create white light
Yttrium with Europium used to create red light
Other uses: alloys and ceramics
Terbium is an important metal used to produce the green phosphors for the lamps.

Other uses: alloys and ceramics.
## ECONOMIC VALUES OF REEs

### HYDROWEEE projects

### Argus metal report

Terbium and Europium and dysprosium maintain a certain economic importance.

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### Mischmetal

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<th>5.10</th>
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### Samarium

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### Terbium

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### Yttrium

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</table>
HYDROEEE projects

Summarizing in the lamps and CRTs

Yttrium
Europium
Gadolinium
Lanthanum
Cerium
Terbium

Yttrium
35 $/kg
4.80 $/kg (oxide)

Europium
340 $/kg
90 $/kg (oxide)

Terbium
630 $/kg
410 $/kg (oxide)
HYDROWEEE projects

INITIAL MATERIALS as POWDERS were provided by RELIGTH Srl (Rho, Milan) Partner of the HydroWEEE projects

Collection/Sorting

Pretreatment (Dissambley and cutting)

Pretreatment (Crushing and sieving)

CRTs

LAMPS

- 15% Y
- 0.7% Ce
- 0.6% Eu
- 0.5% La
- 0.4% Tb
- 0.3% Gd
Details of our research - LAMPS

Hydrometallurgical processes developed within HydroWEEE projects

In the further pages the details for the development of the hydrometallurgical process for the recycling of lamps will be presented.
**HYDROWEEE projects**

**Details of our research - LAMPS**

**WHY TO RECOVER ALL RARE EARTHS FROM LAMPS?**

Economic value of the fluorescent powders

---

**RED PHOSPHORS**

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<tr>
<th>Element</th>
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<th>kg/ton powder</th>
<th>€/ton powders</th>
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<td>35</td>
<td>150</td>
<td>5250</td>
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<td>Eu</td>
<td>0.6</td>
<td>340</td>
<td>6</td>
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**OTHER PHOSPHORS**

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<td>Tb</td>
<td>0.4</td>
<td>630</td>
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**PRICES OF METALS !!**

Yttrium has the greatest influence on the total price.
**HYDROEEE projects**

**Details of our research - LAMPS**

**WHY TO RECYCLE THE ALL RARE EARTHS FROM LAMPS?**

**Economic value of the fluorescent powders**

### RED PHOSPHORS

<table>
<thead>
<tr>
<th>Oxide</th>
<th>€/kg</th>
<th>kg/ton powder</th>
<th>€/ton powders</th>
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### OTHER PHOSPHORS

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<th>€/kg</th>
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<th>€/ton powders</th>
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<td>Ce</td>
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<td>Gd</td>
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<tr>
<td>Tb</td>
<td>365</td>
<td>4.7</td>
<td>1716</td>
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**7200 → 1370 €**

**10000 → 3180 €**

**PRICES OF OXIDES !!**

![Pie chart showing the distribution of rare earths in fluorescent powders]
Details of our research - LAMPs

Hydrometallurgical processes developed within HydroWEEE projects

- **Situation 2015**

  - Total recovery of yttrium and europium was 93%.

  - Purity of the final oxide was on average 97% with 91% of $Y_2O_3$, 4% of $Eu_2O_3$, 0.5% $Tb_2O_3$, 0.11% $Ce_2O_3$, 1% $Gd_2O_3$ with traces of lanthanum oxide.
The research activities were focused on the recovery of red phosphors (Y and Eu).

HYDROWEEE projects

Situation 2015

The processes were tested on mobile and stationary plants realized within HYDROWEEE projects
HYDROWEEE projects

Details of our research - LAMPS

Hydrometallurgical processes developed within HydroWEEE projects

Considering the high economic value of terbium the further activities were concentrated on the dissolution of the other phosphors containing terbium.

The low percentage of recovery for rare earths was mainly due to high acid resistance of green phosphors that contain terbium, lanthanum and cerium.

To leach green phosphors:
- strong leaching conditions or
- a pre-treatment at high temperature to transform the phosphates in oxides that can be leached with weak leaching conditions.

In our research activities the second approach was chosen to improve the leaching of terbium.
HYDROWEEE projects

Details of our research - LAMPS

Hydrometallurgical processes developed within HydroWEEE projects

The transformation of phosphates in oxides by thermal treatment

XRD analysis were showed in the following figures

\[ \text{REPO}_4 + \text{AM} \rightarrow \text{RE}_2\text{O}_3 + \text{AP} + \text{H}_2\text{O} \]

\( \text{RE} = \text{rare earths (La, Ce, Tb)} \)

\( \text{AM} = \text{alkaline material} \)
**HYDROWEEE projects**

**Details of our research - LAMPS**

Hydrometallurgical processes developed within HydroWEEE projects

**Leaching process**

Several acids were tested
Sulfuric acid was chosen to perform leaching

The leaching reaction

\[
\text{RE}_2\text{O}_3 + 3 \text{H}_2\text{SO}_4 \rightarrow \text{RE}_2(\text{SO}_4)_3 + 3 \text{H}_2\text{O}
\]

Investigated factors
- Alkaline concentration
- Acid concentration
- Powder concentration
- Reaction time
- Temperature

Fluorescent lamps powders

Sulfuric acid

Water
Hydrometallurgical processes developed within HydroWEEE projects

**Details of our research - LAMPS**

Precipitation process after leaching

\[ 2\text{RE}(\text{SO}_4)_3 + 3\text{H}_2\text{C}_2\text{O}_4 \rightarrow \text{RE}_2(\text{C}_2\text{O}_4)_3 + 6\text{H}_2\text{SO}_4 \]

- Oxalic acid
- Dissolution
- Filtration
- Recovery of REEs by precipitation
- Filtration
- Calcination
HYDROWEEE projects

Details of our research - LAMPS

Hydrometallurgical processes developed within HydroWEEE projects

Precipitation process after leaching

Grade of the oxides (98.62%): 82.22% $\text{Y}_2\text{O}_3$, 8.38% $\text{Eu}_2\text{O}_3$, 2.43% $\text{Ce}_2\text{O}_3$, 2.29% $\text{Gd}_2\text{O}_3$, 1.77% $\text{La}_2\text{O}_3$ and 1.52% $\text{Tb}_2\text{O}_3$.

Other impurities were 0.62% phosphor oxide, 0.48% calcium oxide and 0.28% silica.

The total recoveries are 70% Y, 100% Eu, 60% Tb, 40% Ce, La and Gd.
Only the red phosphor of color television tubes contains a rare earth, i.e. europium. The first red phosphor, YVO4:Eu3+, was replaced by Y2O2S:Eu3+ due to the increased brightness of the latter phosphor.

The blue and green phosphors in CRTs are based on zinc sulfide: the green phosphor is ZnS:Cu while the blue phosphor is ZnS:Ag
## HYDROWEEE projects

### Details of our research - CRTs

**WHY TO RECOVER RARE EARTHS (Y and Eu) FROM CRTs?**

**Economic value of the fluorescent powders**

<table>
<thead>
<tr>
<th>Element</th>
<th>%</th>
<th>€/kg</th>
<th>kg/ton powder</th>
<th>€/ton powders</th>
</tr>
</thead>
<tbody>
<tr>
<td>Y</td>
<td>14</td>
<td>35</td>
<td>140</td>
<td>4900</td>
</tr>
<tr>
<td>Eu</td>
<td>0.9</td>
<td>340</td>
<td>6</td>
<td>3060</td>
</tr>
</tbody>
</table>

**Prices of Metals!!**

<table>
<thead>
<tr>
<th>Element</th>
<th>€/kg</th>
<th>kg/ton powder</th>
<th>€/ton powders</th>
</tr>
</thead>
<tbody>
<tr>
<td>Y</td>
<td>4.3</td>
<td>1177</td>
<td>761</td>
</tr>
<tr>
<td>Eu</td>
<td>80</td>
<td>10</td>
<td>800</td>
</tr>
</tbody>
</table>

**Prices of Oxides!!**
HYDROEEE projects

Details of our research - CRTs

Hydrometallurgical processes developed within HydroWEEE projects

- Total recovery of yttrium and europium was 90%
- Purity of the final oxide was on average 96%

New activities are focused on the valorization of the residual cake after leaching rich in zinc
Details of our research - CRTs

Hydrometallurgical processes developed within HydroWEEE projects

Hydrogen peroxide

Dissolution
ZnS + 3H₂O₂ → ZnO + 3H₂O
ZnO + 3H₂SO₄ → ZnSO₄ + H₂O

Filtration

Recovery of Zn by precipitation
ZnSO₄ + H₂C₂O₄ → ZnC₂O₄ + H₂SO₄

95% Zn leaching

Oxalic acid

Filtration

95% Zn oxalate
0.08% Y
0.006% Eu
0.08% Al
0.19% S
HYDROWEEE projects

Details of our research - CRTs

Hydrometallurgical processes developed within HydroWEEE projects
HYDROWEEE projects

Details of our research

Hydrometallurgical processes developed within HydroWEEE projects

Preliminary economic aspects

2.5 - 3 €/kg production cost
HYDROWEEE projects

Details of our research

Preliminary economic aspects

Hydrometallurgical processes developed within HydroWEEE projects

The economic value is most influenced by Eu, Tb and Y.

- Final oxides from lamps
  - Eu: 43%
  - Tb: 23%
  - Y: 32%

- Final oxides from CRTs
  - Eu: 51%
  - Y: 49%

The economic value is most influenced in the same way by Y and Eu.
Details of our research

**HYDROWEEE projects**

**FINAL DISCUSSIONS**

The developed processes will be tested on HYDROWEEE industrial demo plants.

Other developments are necessary to implement the processes, increase the economic feasibility and reduce the market fluctuations of REE prices.

Anyway the new researches performed within HydroWEEE projects represent an important growth on the fields of treatment of WEEE.
Thanks to all partners of HYDROWEE projects

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University of Ancona (Italy)

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Relight Srl (Italy)

Pupin Institute (Serbia)

SET-trade (Serbia)

Greentronics (Romania)
Thanks
For the attention

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