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REVIEW OF METHODS FOR THE RARE EARTH METALS RECYCLING

Marinela Panayotova, Vladko Panayotov

University of Mining and Geology "St. Ivan Rilski", 1700 Sofia; marichim@mgu.bg

ABSTRACT. Rare Earth metals are materials that are essential for the development of modern industry, infrastructure and manufacturing of products used in the every-day life. They are necessary ingredients for designing and developing high-technology products, for the creation of new environment-friendly vehicles and renewable energy. Recycling is one of the main ways to secure rare metals needed for new productions. Due to underestimation of the need to own resources development and a number of technological, political, environmental and social reasons, the recycling of rare metals is not developed. Globally there is not available enough developed and accepted technology for the extraction of rare metals from waste materials. This publication is a critical review of methods for recycling of Rare Earth metals, with special attention given to the recovery from magnetic media, old batteries and catalysts.

ПРЕГЛЕД НА МЕТОДИТЕ ЗА РЕЦИКЛИРАНЕ НА РЕДКОЗЕМНИ МЕТАЛИ

Маринела Панайотова, Владко Панайотов

Минно-геоложки университет "Св. Иван Рилски", 1700 София; marichim@mgu.bg

РЕЗЮМЕ. Редкоземните метали са суровини, които са от съществено значение за развитието на съвременната промишленост, инфраструктурата и за производството на продукти, използвани в ежедневието ни. Те са необходими съставки за разработването и развитието на високотехнологични продукти, за създаването на нови, опазващи околната среда транспортни средства и ВЕИ. Рециклирането е един от основните начини за осигуряване редките метали, необходими за новите производства. Поради недооценяване на необходимостта от разработването на собствени ресурси и редица технологични, политически, екологични и социални причини, рециклирането на редки метали не е развито. В световен мащаб не съществува достатъчно разработена и общоприета технология за извличане на редки метали от отпадъци. В настоящата публикация е направен критичен преглед на методите за рециклиране на редкоземни метали, като е обърнато специално внимание на извличането им от магнитни носители, стари батерии и катализатори.

Introduction - Rare Earth elements criticality

The Rare Earth elements (REEs) include the chemical group of 15 elements called lanthanides, plus yttrium and scandium. The lanthanides are lanthanum, cerium, praseodymium, neodymium, promethium, samarium, europium, gadolinium, terbium, dysprosium, holmium, erbium, thulium, ytterbium, and lutetium. Rare Earths are moderately abundant in the Earth's crust, some of them even more abundant than copper, lead, gold, and platinum. While more abundant than many other minerals, REEs are not concentrated enough to make them easily and economically exploitable.

Modern industrial activity, the infrastructure, Renewable Energy Sources (RES) development and use, as well as products for our daily-life are impossible without the use of some strategic metals, among them – the Rare Earths play very important role. Currently, the dominant uses of Rare Earth elements are automotive catalytic converters, petroleum refining catalysts, metallurgical additives and alloys, phosphors in color television and flat panel displays (cell phones, portable DVDs, and laptops), glass polishing and ceramics: permanent magnets and rechargeable batteries for hybrid and electric vehicles, and numerous medical devices. Permanent magnets containing neodymium, gadolinium, dysprosium, and terbium are used also in numerous electrical and electronic components and new-generation generators for wind turbines. Other important applications are space-based satellites and communication systems.

World demand for Rare Earth elements is estimated at 136000 tons per year, with global production around 133600 tons in 2010. The difference is covered by previously mined and available above ground stocks. World demand is projected to rise to at least 185000 tons annually by 2015. (IMCOA, 2011). Other estimations point that by 2015 the global demand for REEs may reach 210000 tons per year (Bloomberg News, 2010). China is the main worldwide producer of REEs, however its own demand increases sharply and is expected to reach reach 130000 metric tons by 2015 and the China's production to reach only 140000 tons per year.

According to a report of Öko-Institut (Buchert et al., 2009) two main scenarios of REEs use are envisaged. The first scenario predicts an average annual growth rate of 4.5 % (left bar – Fig. 1), which is seen as a moderate scenario. The other scenario (right bar – Fig. 1) gives a growth rate of 9 %, which is seen as the upper limit. It considers the upcoming markets for manifold new applications of REEs. This scenario is supported by evaluations of specific applications with high growth rates.

Different options are available, which complement each other, to cope with the problem: supporting / encouraging greater exploration for REE worldwide aimed at identifying

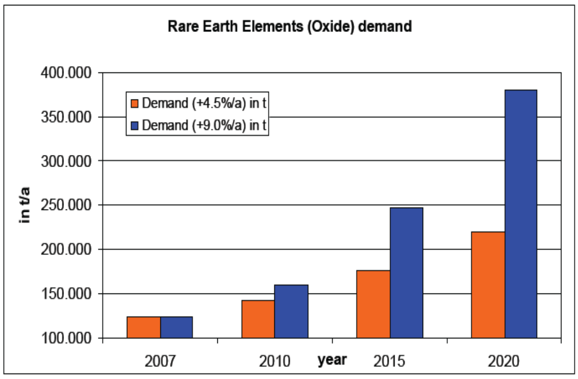

Fig. 1. Rare earths oxides demand scenarios based on calculations from the Öko-Institut

economically exploitable REEs deposits and / or REE production as a byproduct; investment in R&D that would address the three areas: greater efficiencies in materials use; substitutes or alternatives for Rare Earths; and recycling of Rare Earth elements. The present paper is devoted to the latter option.

Current state of REEs recycling

A successful recycling is very important regarding increase of resource efficiency, avoidance of possible scarcities and reduction of the overall environmental impacts. Furthermore, the positive contribution of the recycling sector to employment should be taken into account. Concerning the recycling of metals two major fields should be differentiated: pre-consumer recycling, which means the recycling of production scrap of manufacturing processes ('new scrap') and the post-consumer recycling. Pre-consumer recycling is the easier task compared to post-consumer recycling. It displays the following general advantages:

- Relatively high concentrations of the distinguished metal in the new scrap;
- Well-known and definite source of waste generation;
- Continuous new scrap formation;
- Relatively high volumes of new scrap formation.

Post-consumer recycling is a much more difficult task because:

- Low metal concentrations in waste flows: dissipative applications;
- The critical metal is a minor composition in a complex material matrix (many other metals, plastics etc.);
- Regarding consumer applications like automotives or electrical and electronic equipment (EEE), metal concentration in a single unit is very low and the final end-use often takes place in emerging or developing countries without sufficient take-back and collection systems for secondary materials.
- Some applications of critical metals are so new, that relevant mass flows of post-consumer materials will reach the waste management sector not until in some years.

In addition, other recycling restrictions can be pointed out:

- The recycling processes for the REEs are quite complex and extensive if re-use is not possible and a physical and chemical treatment is necessary;
- Most of the recycling procedures are energy-intensive;
- Physical / chemical limitations for recycling - Rare Earths hold a disposition to slag (as oxides) in smelter plants;
- Lack of suitable recycling technologies and/or recycling infrastructures;
- Lack of price incentives for recycling.

Recycling Process Technologies

Rare Earth elements are used in high strength magnets, in high power density batteries, lighting and catalysts. These potential waste streams will be considered separately as they present different challenges and opportunities.

Magnets

Rare Earth magnets are fragile and fracture very easily. It is estimated that between 20-30 % of the Rare Earth magnet is scrapped during manufacturing because of breakages or waste cuttings such as swarf and fines (Akai, 2008). There are three categories of waste materials derived from Rare Earth magnets production:

- Material left in furnaces after vacuum melting, atomising or rapid quenching;
- Rejected finished magnets;
- Residues from grinding operations used to fabricate magnets.

All these scrap materials are designated as hazardous wastes. Recovery or recycling methods of Rare Earth magnet scrap can be classified into four generic processes (Schüler et al, 2011):

- Recover the Rare Earth scrap in its unoxidised state by re-melting the scrap. The re-melting process generally suffers from low yields from almost all scrap sources, although it is considered that recovery from melted residues by this method may be economical (Kara et al., 2010).
- Recover the Rare Earth scrap in its oxide state. The recovery of Rare Earths in their oxide state is not fully economically proven. It is considered to be the most appropriate method of handling the materials. However it assumes the commercial value of REEs, in their waste form, to be near to zero.
- Recover material in a form suitable for recombination into another magnet. The recovery of the magnetic material in a form that is suitable for preparing a new magnet requires that the waste is not chemically processed and remains as a high quality metal alloy.
- Selective direct extraction of neodymium and dysprosium.

Transport of magnetic materials is restricted: their fields can interfere with aircraft instruments, so they are deemed hazardous materials. (Any magnetic field greater than 0.00525 Gauss at 15 feet is prohibited from air transport.) Therefore, the recycling of Rare Earth magnets will require both regional and centralised collection and processing facilities.

Additional issues that must be solved are the low yields, contamination, expensive re-processing (e.g. of Rare Earth oxides) and chemistry adjustment needs (Schüler et al, 2011).

Recycling of post-consumer magnets, which has the greatest potential in terms of material amounts, is however more complex. Except for the costly recycling process and difficulty of product collection and dismantling, there are technical issues connected to contamination (due to platings, glues, plastics etc.), highly variable magnet compositions (whose mixing may destroy the desired material properties) and magnet corrosion, especially of NdFeB, requiring additional refining processes to remove oxides and hydroxides (Goodier, 2005). Recent progress in research shows that high recovery rates of Rare Earths as well as automatic dismantling processes are possible (Schüler et al, 2011).

Several ways of recycling Rare Earths have been described in the literature, and the methods include the use of molten salts, hydrometallurgical processes, extraction with liquid metals, melt spinning, the formation of slags, and re-sintering.

Molten Salts

The Rare Earth metals are chlorinated and dissolved in sodium chloride (NaCl) and potassium chloride (KCl) melts (Yamamura, 2003). The electro-winning of lanthanum, cerium, neodymium, samarium and dysprosium from these melts has been studied and it was concluded that the low current efficiency of neodymium and dysprosium electro-winning is caused by the solubility of those metals in the melt.

Binary chloride mixtures of Rare Earths were separated by a reduction-vacuum distillation process, giving greater selectivity than solvent extraction methods (Uda, 2003). Magnet sludge was chlorinated by iron chloride ($FeCl_2$) to remove neodymium and dysprosium as their chlorides. Recovered chlorides are converted to their oxides by reaction with water and these oxides can be used directly as the raw material in the conventional oxide electrolysis process.

Laboratory tests were carried out in Japan with temperatures of around 1000 °C (Shirayama and Okabe 2009) to extract selectively Nd and Dy directly from magnet scrap by using molten magnesium chloride as the selective extracting agent.

Hydrometallurgical Processes

Hydrochloric acid was used to dissolve 98% nickel, 100% cobalt and 99% Rare Earth metals (Ellis et. al, 1994). Rare Earth elements can be precipitated from aqueous solutions by the addition of oxalic acid or hydrogen fluoride to form the oxide or fluoride. As Rare Earth elements are produced by the calciothermic reduction of the fluoride, it is advantageous to precipitate it in this form. However, although high quality material can be produced by aqueous processing, removal of other dissolved species can cause problems.

Tang et al. (2009) compared two methods employing Na_2SO_4 double-salt precipitation and oxalate secondary precipitation which achieve a recovery rate of Nd_2O_3 of more than 82 %.

Zhang et al (2010) researched a separation method based on the electrical reduction by using P507 extraction. Test results showed that this newly electrical reduction technology may result in a recovery rate of 96.1 % of Rare Earth from neodymium magnet scraps und save about 650 Euro per ton of Rare Earth recycled, compared to the traditional separation methods in terms of material consumption and costs.

Treatment with Liquid Metals

Neodymium is selectively extracted from magnet scrap with liquid magnesium, leaving the iron behind. The magnesium is then distilled away from the melt (Okabe, 2003; Takeda, 2006). Neodymium is selectively leached from magnet scraps using molten silver (Takeda, 2004). The neodymium is selectively removed from the silver by oxidation to form neodymium oxide (Nd_2O_3).

Melt Spinning

Isotropic NdFeB magnetic powder was recycled directly from nickel-coated waste sintered magnets by melt spinning. The resultant magnets had good magnetic properties (Itoh, 2004).

Glass Slag Method

Boric acid is used to extract the Rare Earth elements as RE-BO_3 from Rare Earth-iron alloys.

Electroslag Remelting

Relatively large scrap magnetic material can be melted either as a consumable anode or by addition to a molten bath. A reactive flux, ($CaCl_2$/CaF_2/RE-F_3) is used to remove carbon, nitrogen, oxygen and metallic impurities such as lithium, sodium and aluminium. This method cannot be used for swarf or fine materials (Ellis,1994).

Milling and re-sintering

Zakotnik et al (2009) recycled neodymium magnets from disk drives successfully by milling and re-sintering with the addition of 1 % new neodymium in a technical scale. Kawasaki et al (2003) developed a similar recycling process for sintered neodymium magnets by adding Nd-rich alloy powders to the ground magnet scrap powder before re-sintering.

Post-consumer recycling

Research is ongoing in Japan into the post-consumer recycling of Rare Earths from motors / generators (permanent magnets). Pyro-metallurgical and hydrometallurgical approaches are described which focus on the recovery of REEs as metals. (Takeda 2009, Koyama 2009).

Hitachi announced that it has developed a machine for the dismantling of neodymium magnets from hard disks and compressors (Clenfield and Shiraki 2010). The machine has a capacity of 100 magnets per hour, eight times faster than manual labour. The developed dismantling process shall commence operation in 2013.

Batteries

Well-established methods are available for the recycling lead, nickel-cadmium (NiCd), nickel hydride and mercury batteries. For some batteries, such as newer nickel-hydride and lithium systems, recycling is still in the early stages and not designed for the recovery of their Rare Earth contents. The focus of the recycling of NiMH cells has been on the recovery of the nickel, chromium and iron fractions. The Rare Earths and other metals contained in the hydride alloy were not separated and ended up in the slag. The negative electrode in Ni-MH is made of nickel hydride alloy that can include REEs, such as lanthanum, cerium, neodymium and praseodymium. Mischmetal with naturally occurring Rare Earth combinations is the major source for the electrode alloy. The amount of Rare

Earths used in N-iMH cells varies between manufacturers. For example, Motorola uses only 5-15 % Rare Earths in its negative electrodes. However, the scrap from nickel hydride alloy cells typically contains about 33 % Rare Earths metal, 60 % transition metals and 7 % others, such as manganese and aluminium.

The most active European organisation that recycles batteries is Umicore, which recycles both Ni-MH and Li-ion cells. In November 2009, Umicore announced it was to invest in a new facility in Belgium to recover Rare Earths from batteries, but process details are not yet available.

It may be stated that Japan is leading the efforts in Rare Earth metals recycling. There the uptake of hybrid and electric vehicles (HEV) is high and the shortage on Rare Earth metals could have a major impact not only in the field of electric cars but also in the electronics industry. There is also a requirement to restrict the environmental impact of waste magnets.

A hydrometallurgical process has been developed for the recovery of cobalt, nickel and Rare Earth metals from the electrode materials of spent Ni-MH batteries (Kuzuya, 2003). Mischmetal nickel-cobalt intermetallic compound was separated from the electrode materials mixture by sedimentation (56 % nickel, 13.4 % cerium, 10.6 % lanthanum and 7.9 % cobalt). By use of sulphuric acid Rare Earths are dissolved, followed by precipitation at pH 1.2 using sodium hydroxide (NaOH). At pH 5 - 7 iron, zinc and manganese were precipitated. Nickel and cobalt can be obtained by electro-winning from the remaining solution (Bertuol et. al, 2009).

In Japan Toyota recycles their HEV batteries by removing them from the vehicles and returning them to Panasonic EV Energy Co Ltd, where they are disassembled into their various higher value components, namely resins and plastics, metals and precious metals. The precious metals, including cobalt, nickel and the Rare Earths, are processed by a battery recycling company and the recovered metals are used as raw material for stainless steel.

Japanese automobile manufacturer Honda announced that it has teamed up with Japan Metals & Chemicals Co., Ltd. to create a technology to recover Rare Earth metals such as lanthanum and cerium from spent Ni-MH batteries used for HEVs, and to refine the recovered metals for re-use in new batteries. The electrodes are first treated with a multi-element refinery process, then the separated reduction of the Rare Earths takes place (JOGMEC 2010). The successful stabilisation of the extraction process at the plant of Japan Metals & Chemicals Co., Ltd. made possible the extraction of approximately 80 % of Rare Earth metals contained in used nickel-metal hydride batteries, with purity as high as that of newly mined and refined metals (Honda.com).

Researchers from Freiberg - Germany developed a hydro-metallurgical process to recover Rare Earth metal from the slag of the pyro-metallurgical treatment of used Ni-MH batteries (Heegn 2009).

The recovery of REEs from Ni-MH batteries is examined by Luidold (2010). The work attempts to extract the REEs out of slags from smelting operations by means of hydrometallurgical methods. It is also aimed at the previous separation of the REEs from the other materials.

The Chinese researchers Wu and Zhang (2010) studied the recovery of Ni, Co and Rare Earths (lanthanum, cerium, neodymium and praseodymium) from used Ni-MH batteries by leaching with sulphuric acid. The tests showed recovery rates of 95 %.

Lighting and luminescence

There are some research activities and new patents in the field of post-consumer recycling:

- Recycling of yttrium and europium from discharge lamps and fluorescent lamps (OSRAM 2009, Wojtalewicz-Kasprzak 2007);
- Guarde et al (2010) reported on the recycling of fluorescent lamps and tubes and the output of a distilled powder fraction which contains up to 10 % Rare Earths. Currently this fraction is disposed;
- Research activities are being undertaken which focus on yttrium and europium recovery not only from lamps but also from TV tubes and computer monitors (Rabah 2008, Resende and Morais 2010);
- A scientific overview of conceivable recovery methods for the recycling of rare earths fluorescent powder containing yttrium, europium, lanthanum and cerium is provided by the Chinese publication (Mei et al, 2007).

Catalysts

The recycling of REE from spent catalysts (industrial as well as automotive catalysts) is not common due to relative low prices of REEs in the past. It is an open question whether a recovery of the REEs (mostly lanthanum) from Fluid catalytic cracking (FCC) catalysts could be interesting from an economic point of view in the next years. This will mainly depend on the price development of lanthanum. From a technical point of view the large global mass flow of FCC catalysts – 600000 t per year (Hykawy 2010) – with about 2 % REEs content means an interesting REEs potential for recycling from this specific application. It should be mentioned that Öko-Institut and Umicore (Hagelüken et al., 2005), after in-depth investigations regarding the recycling flows of platinum group metals, came to the clear conclusion that the usual business-to-business relationships (e.g. between the catalysts suppliers and the oil refineries) are a very good pre-condition for very high recovery rates of the platinum group metals (almost 100 % collection rate of the spent catalysts). Nevertheless, the development of a technically feasible and economically acceptable solution for the recycling of REEs from FCC catalysts is a task for the future.

The recycling activities of the automotive catalysts focus worldwide on the recovery of the valuable platinum group metals (Hagelüken et al., 2005). Therefore the recovery of the REEs content (mainly cerium) from these catalysts has not yet become a focus. Currently the REEs moves into the slags from smelter processes due to the high affinity of the REEs to oxygen. It remains an open question whether a recovery of REEs from spent automotive catalysts could feature in the future.

Conclusions

As a conclusion it can be stated that there are potentially a number of recycling processes but none of them is developed commercially due to draw-backs on yields and cost.

Further intensive research and work is needed to build-up a recycling scheme and to develop the most appropriate recycling technologies.

Taken the current situation and upcoming developments into account, three different spheres of activities can be pointed out to promote the recycling of critical metals in the future and thus to contribute to ensuring the supply base for future sustainable technologies:

- The enlargement of recycling capacities including efficient collection system;
- The development and realization of new recycling technologies;
- The accelerated improvement of international recycling infrastructures.

The efforts should be supported by policy measures:

- Improved legal frameworks for the distinction of used goods and scraps, designated for the export in non-EU countries to raise the collection rate;
- Establishment of Best Practice Guidelines for the entire recycling chain;
- Campaigns and initiatives to draw attention of the public to the importance of recycling of special metals;
- Creation of conditions for technology transfer and international cooperation.

We have not to forget even for an instant, that recycling provides a number of advantages:

- The secondary Rare Earths potential of Europe will arise;
- Europe will be less dependent on foreign material supply;
- Know-how on Rare Earth processing will be built up;
- No radioactive wastes arising in secondary Rare Earth processing;
- Additional environmental benefits are created - concerning air emissions, groundwater protection, acidification, eutrophication and climate protection.

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