Recycling Rare-Earth-Metal Waste Using Hydrometallurgical Methods

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Abstract—The present review describes the state of the problem of extracting rare earth metals from secondary raw materials, such as high-coercivity magnets, nickel–metal hydride batteries (NiMH), and phosphors of coatings of fluorescent lamps. Methods for waste processing with mineral acids and other solvents, the removal of associated impurities from rare-earth components, separation, and deep purification (mainly by solvent extraction) have been considered. Data on the industrial applications of hydrometallurgical processes for rare earth metals recycling have been presented.

Keywords: rare earth metals, wastes, hydrometallurgy, leaching, precipitation, solvent extraction **DOI:** 10.1134/S0040579516040266

The recycling of rare earth metals (REM)-containing wastes are presently of high interest due to the deficiency of rare earth metals on the global market and the increase in the amounts of the waste. The most widespread REM-containing wastes are FeNdB magnets (contain Nd, Pr, Dy, and Tb), nickel–metal hydride batteries (contain La, Ce), and phosphors of fluorescent lamps (contain Y, Eu, Tb, and Gd). A considerable number of studies on recycling REMcontaining wastes were carried out with application of hydrometallurgical methods for REM extraction in solutions. The diversity of hydrometallurgical methods was presented in the work [1]. These methods include leaching, selective precipitation, solvent extraction, and ion-exchange and insignificantly differ from the methods of the final stages of mineral processing. The goal of the presented work was to summarize global experience in the study and application of hydrometallurgical methods in this field. To ease the analysis, the cited works were grouped according to the principle of selecting the leaching agent at the stage of raw-material leaching.

Sulfuric Acid

REM can be extracted from pregnant solutions of sulfuric acid for leaching rare earth scraps in the form of double sulfates and oxalates. These methods were compared in [2]. It was shown that sodium sulfate enabled higher purity and extraction level to be achieved (above 82%). Hydrometallurgical methods

of recycling magnet waste includes the following stages: dissolution in 2 M sulfuric-acid solution in stirred reactors at a liquid-to-solid ratio $(L : S) = 2$ without heating, precipitation in the form of rareearth double salts, and conversion to fluorides or direct fluorination with hydrofluoric acid. The calcination of finely ground magnets at 700°C for 8 h with hydrochloric acid followed by solvent extraction at pH 2 was proposed as an alternative method. At the first stage, the extraction levels of neodymium and samarium were 97 and 94% and, at the second stage, the extraction level was 95%, respectively [3]. Usually, the aim of the considered methods for rare earth-containing scrap processing is the extraction of REM in the form of individual compounds with fairly high purity. Another approach was considered in [4]. Magnet scrap contains 59.44% $Fe₂O₃$, 31.38% Ln₂O₃, and 6.53% $Co₂O₃$ was leached with 34–36% sulfuric acid without the preliminary removal of organic binder, then incubated at 5–8°C using a thermostat to crystallize the salts. The residue was dissolved in water in order to obtain a pregnant solution that contains 80– 100 g/L iron. Then, the solution was subjected to thermal hydrolysis at a pH of 3–4 in order to precipitate ferric hydroxide, after which solid residues were filtered in order to obtain concentrated REM solution. REMs were precipitated in the form of fluorides or hydroxides depending on the aim of the further application. The lanthanide content in ferric hydroxide reached 4%, and the iron content in the mixture of lanthanides was 3.5%. It was shown that ferric hydroxide can be used as raw material to produce high-quality pigments and as a component of the lux coat of welding electrodes, which allows one to significantly increase the reliability of welded joints. Methods of the sulfuric-acid treatment of REM-containing magnets were patented by Nippon Magnetic Dressing Company [2]. It was proposed to leach REM-containing magnetic scrap by sulfuric acid solutions with concentrations of 3.0 to 6.5 M and precipitate lanthanides in the form of double sulfates. Then, impurities were removed from REM sulfates by solution and reprecipitation. It was demonstrated that the application of solutions with decreased concentrations sulfuric acid (1.5–3 M) followed by the separation of undissolved impurities could be used as an alternative method. The technology for REM-containing magnetic materials includes abrasive machining process (grinding). It was proposed to dissolve grinding wastes in sulfuric acid, precipitate REM sodium double salts, and convert these salts to hydroxides. After washing, residues were subjected to oxalate conversion, drying, and oxalate calcination in order to obtain total rare-earth oxides with 99% purity. Iron-containing and cobalt cakes were extracted from mother liquors of double salts [5].

Several studies were conducted on the hydrometallurgical processing of nickel–metal hydride batteries. In these batteries, the negative electrode is made of nickel lanthanum (which can be replaced with mischmetal) alloy, which contains small amounts of manganese, cobalt, aluminum, and other metals, whereas a positive electrode consists of nickel oxyhydroxide. In [6], both electrodes were treated with 3 M sulfuric acid at 95°C. Due to low solubility about 94.8% of REM sulfates were separated from other metals. Then, iron, zinc, and manganese, which contain 5.2% REM, were separated from nickel and cobalt by solvent extraction using 20% P204 (Di-(2-ethylhexyl)phosphoric acid). After zinc and manganese removal, REMs were stripped from organic phase with 2M HCl. REM sulfates were converted to hydroxides by treatment with sodium hydroxide, and cake was dissolved using hydrochloric acid. Cobalt was separated from nickel by solvent extraction with 20% Cyanex 272 ((bis(2,4,4 trimethylpentyl)dithiophosphinic acid), whereas pure cobalt and nickel sulfates were obtained via the evaporation of extract and raffinate, respectively. Nickel sulfate was used to produce a spheroidal powder of hydroxide, which can be used to produce batteries. The total extraction of REM, Co, and Ni exceeded 98%. A similar approach was applied by Japanese researchers to process electrodes from nickel– metal hydride batteries [7]. Leaching with 2M sulfuric acid at 95°C allowed one to obtain a pregnant leaching solution with a pH of 0.95 that contains 10.6 g/L Ni, 0.85 g/L Co, 1.70 g/L Fe, 0.36 g/L Zn, 0.21 g/L Al, 0.54 g/L Mn, 1.73 g/L La, 0.10 g/L Ce, 0.33 g/L Pr, 1.10 g/L Nd, and 0.032 g/L Sm. REMs were extracted with 25% DEHPA in kerosene, followed by oxalate precipitation. The oxalates were calcinated to obtain concentrate that contains up to 99.8% of REMs. The total extraction of REMs was 93.6%. Cobalt and nickel

were separated by solvent extraction in 20% Cyanex 272 in kerosene and extracted in the form of oxalates with purity of 99.6 and 99.8%, respectively. The total extraction of the metals exceeded 96%. The possibility of extracting nickel, cobalt, and rare earth metals from nickel–metal hydride batteries that contain 50% Ni, 5.1–5.5% Co; 15.3–29.0% REM; 2.8% Cd, and 2.2– 10.9% K was considered in [8]. It was demonstrated that the leaching of nickel and cobalt with sulfuric acid was effective and depended slightly on temperature and hydrogen peroxide concentration. The REM mixture was precipitated by the addition of sodium hydroxide. Then, cadmium, cobalt, and nickel were selectively extracted by DEHPA and Cyanex-272. Several patents on processing these wastes were compared in [2]. Several approaches were developed in order to improve sulfuric acid extraction, including preliminary dry-magnetic separation to extract the magnetic fraction before sulfuric acid leaching; leaching of the negative electrode of nickel– metal hydride batteries, followed by a mixture of H2SO4–H2O2, followed by REM precipitation (it was revealed that the presence of wastes of nickel cadmium batteries did not affect the process); and the application of sulfidation to precipitate nonferrous metals from pregnant leaching solutions.

Both acid and alkaline leaching was used to extract valuable components from spent batteries [9]. The most promising results (98% REM extraction) were achieved during leaching with sulfuric acid, followed by selective precipitation at pH 1.2 using sodium hydroxide. Then, pregnant solution was neutralized (pH was increased to 7) to precipitate 100% of dissolved iron and 70% of zinc and obtain a solution with a high content of nickel and cobalt. According to the authors, the results demonstrated in the work were comparable with those of solvent extraction, but the developed method was environmentally friendly, easier than solvent extraction, and did not require complex equipment.

Processing optical industry wastes is not presently of high interest, probably due to the low value of REMs contained in polirit, which mainly consists of cerium compounds. However some studies have been conducted in this field [10]. The number of the works on treating spent catalyzers and sorbents is low due to the low prices and availability of lanthanum and cerium. However, the increase in the prices of rare earth metals and the deficient on global market in the last decade will surely lead to growing interest in this field.

Nitric Acid

The authors of patent [11] set the task to develop a method that allows one to produce high-purity REM from magnetic scrap, regardless of its composition. The scraps that contain $0.1-60\%$, $0.2-2\%$ Fe, up to 36% Co, up to 20% of total REMs, 3–90% Nd (of total REMs), $0.1-30\%$ Sm (of total REMs), $1-12\%$, 0.2–7% Si, and up to 15% water-soluble organic matter were subjected to thermal treatment at temperature of 80–700°C followed by leaching with nitric acid (0.4–3.0 M). This treatment enabled moisture to be removed, organic impurities to be burned out, and iron to be partially converted to an insoluble form, as well as release of nitrogen oxides to be decreased and the release of hydrogen to be avoided during treatment with nitric acid. Total REMs were extracted from obtained pregnant solutions in organic phase. To separate neodymium from total REMs, other REMs were extracted from this solution with tributyl phosphate (TBP). The obtained neodymium nitrate solution contained less than 0.05% of other REM. The value of neodymium extraction was 95%. Other REMs were extracted as the samarium concentrate, which contained 60–70% samarium, obtained after stripping REM of organic solvent. The liquor obtained during the stripping of organic phase with nitric acid or part of the loaded aqueous phase that contains REM was used as the aqueous phase for neodymium separation. The method for the selective dissolution of REM was developed in [12]. It has been proposed to leach nickel-REM alloy with diluted nitric acid at a temperature no higher than 50°C and at a pH of 5 or higher and separate nickel by filtration.

Hydrochloric Acid

It was shown that leaching with hydrochloric acid allowed one to extract 98% of nickel and 99% of cobalt from the scrap of spent batteries [13]. REM may be precipitated using either oxalic acid or hydrogen fluoride depending on the proposed application. It was noted that the cost-effective processing of the pregnant solution after REM extraction posed a problem. A new approach for separating the iron-group metals from REMs was developed in [14]. It has been proposed to separate metals via solvent extraction with ionic liquid instead of the usual organic reagents. The scrap of neodymium iron (NdFe) and samarium– cobalt (SmCo) magnets were leached with hydrochloric acid. Iron and cobalt were extracted from pregnant leaching solutions with trihexyl(tetradecyl)phosphonium chloride. The separation factors of Nd–Fe and Sm–Co with 8.5 and 9 M HCl were 5.0×10^6 and 8.0×10^5 , respectively. Cobalt and iron were stripped from the ionic liquid phase with water and ethylenediaminetetraacetic acid (EDTA), respectively. Unfortunately, distribution ratios of chromium, nickel, aluminum, calcium and magnesium were very low under the test conditions. Leaching scrap from nickel–metal hydride (NiMH) batteries with 3–4 M HCl at 95°C for 3 h enabled the extraction of 99% of REM [15]. After leaching REMs, iron, aluminum, manganese, and zinc were separated from nickel and cobalt by solvent extraction with 25% DEHPA in kerosene. Then REMs were stripped with 2 M HCl and precipitated by oxalic acid. The purity of the obtained REMs and the value of extraction were 99 and 98%, respectively. Nickel and cobalt contained in the raffinate were separated by solvent extraction with trioctylamine and precipitated by oxalic acid.

The processing of traditional fluorescent energysaving lamps, light-emitting diodes, cathode ray tubes, and monitors posed a specific problem due to the specificity of their applications and chemical composition. Fluorescent lamps consume 75% less energy and last up to ten times longer than regular incandescent lamps with equal light outputs, which, along with measures of state regulation, cause high growth rates in their production. At the same time, processing spent fluorescent lamps is a pressing issue, as they contain both nontoxic (expensive and deficient oxides and phosphates of yttrium, europium, terbium, gadolinium) and toxic (mercury, the natural reserves of which are limited) components. The mercury content in the lamps has been constantly declining of late; it partially decreases the problem of utilization, but does not allow it to be solved completely. After grinding and separating plastic, metal, and glass components, mercury should be thoroughly removed from REM phosphates and oxides. Standard 40W fluorescent lamps contain 4–6 g of phosphors, which transform ultraviolet radiation into visible light and contain up to 27.9% of REM. Contents of thorium, cerium, europium, cerium, and lanthanum in phosphors in the form of oxides, phosphates and halophosphates are 23.2, 2.4, 1.8, 0.2 and 0.3%, respectively, and vary depending on the manufacturer. In addition to terbium, the composition of green phosphors usually includes gadolinium. Thus, REM extraction from phosphors of fluorescent lamps cannot be an easy task due to the complexity of their chemical composition [16]. The preliminary stages of processing fluorescent lamps, such as separating metal parts, grinding glass, demercurization, and washing phosphors are not various. In [17], phosphorous residue was dried at 110°C and autoclaved at 125°C for 4 h using a mixture of sulfuric ant nitric acid to dissolve REMs. The value of extractions of yttrium and europium were 96.4 and 92.8%, respectively. Then, calcium was precipitated and REMs were converted into thiocyanates. REMs were extracted from thiocyanate solution using trimethylphenylammonium (TMPA). Solvent extraction enabled the recovery of 98.8 and 96.5% of yttrium and europium, respectively. Both rare-earth components were extracted using 1 M nitrate solution that contains TBP. Then, yttrium was stripped using ethanol (the separation factor was 9.4). A wide range of extragents, including 2-thenoyltrifluoroaeetone (TTA) in heptane, ÐÑ-88À in kerosene can be used to recover REM from phosphors. It has been shown that the extraction efficiency decreases in the series Cyanex $923 >$ Aliquat 336-P204 = Aliquat 336-P507 > P350 > TBP. Also, ion-exchange resins can be effectively used to recover REMs from pregnant solutions for leaching phosphors from fluorescent lamps. It was shown that yttrium can be effectively separated from other REMs by elution with some organic acids. It was demonstrated that nitric, hydrochloric, and sulfuric acid and ammonia can be used to dissolve a mixture of phosphors. Effects of pH, temperature, stirring intensity, and the S : L ratio on extraction were studied. Furthermore, solvent extraction, ionic exchange, and precipitation methods were tested for REM recovery. Gravitation and flotation were used for the preliminary separation of calcium halophosphates. However, despite the efforts of researchers, a simple and efficient process suitable for industrial application has not yet been developed [16]. Patents owned by R. S. Otto (e.g., [18]) can demonstrate current state of technology of REM extraction from spent fluorescent lamps. The developed technology includes six stages, i.e., the mechanical separation of the coarse fraction of grinding, the separation of halophosphates using cold hydrochloric acid, the leaching of easily soluble components (yttrium and europium oxides) using hot hydrochloric acid, the leaching of poorly soluble components (rare earth phosphates) using hot sulfuric acid, the grinding of insoluble residue that contains rare earths and its treatment with acid or alkali, and final operations (precipitation of the rare-earth components in a form suitable for further use). Examples of technological approaches to this issue that used in Japan are presented in the book [19]. One of the proposed technologies includes the dissolution of phosphors from fluorescent lamps in 1.5 M sulfuric acid (pulp density of 30 kg/m3) at 70° C for 1 h. This enabled the extraction of solely yttrium and europium (92 and 98%, respectively), which are contained in phosphors in the form of oxides. Lanthanum, cerium, and terbium, which are contained in the form of phosphates, were almost unaffected and remain in solid residue. Further operations are carried out to purify yttrium and europium. The proposed approach did not allow one to extract valuable terbium. Pilot trials allowed one to treat 10 kg of phosphorous slurry and obtain rare-earth components of 98.2% purity. The total extraction of yttrium and europium were 65 and 67%, respectively. Then, precipitation with oxalic acid was replaced by solvent extraction for separating and purifying yttrium and europium. At the first stage of extraction, yttrium was extracted at pH 1.5. Then, europium was extracted at pH 2.0. Since Al, Si, P, Mn, Sr, Mg, and Na are not extracted at these pH values, yttrium and europium oxides were obtained with purities of 99.7 and 90%. To achieve the higher extraction of rare-earth components from all components of phosphors, including phosphates, leaching with concentrated sulfuric acid and mechanical and chemical treatment were used. Another promising approach is fusion with alkalis. The approach includes the following stages: grinding the glass component of fluorescent lamps; sieving; processing the fine fraction with hydrochloric acid for separating alkaline earth and heavy metals; filtering and fusing dried solid residue, which contains a rare-earth component with an alkali; dissolving the residue in hydrochloric acid; additionally purifying the pregnant solution; and separating Y, Eu, Tb, La, and Ce by liquid extraction in multistage mixer-settlers. In [20], it was attempted to simplify the complex technology of REM extraction from phosphors. At the first stage, phosphors were activated using alkaline reagent at a temperature above 300°C. Then, the non-rare-earth component was leached at an elevated temperature, and solid residue was dissolved with nitric acid to recovery REMs, which were extracted from nitric acid solution using common methods. In addition, the acid pretreatment of phosphors could be carried out prior to activation in order to dissolve yttrium oxide. Unfortunately, alkaline treatment led to the partial dissolution of a fine fraction of ground glass, which resulted in the pollution of pregnant solutions with silicic acid and problems during filtration.

Worcester Polytechnic Institute (WPI), Colorado School of Mines (CSM), and the Catholic University of Leuven in Belgium have established a collaborative research Center for Materials Resource Recovery and Recyclability (CR3) for developing technologies for treating different wastes, including REM-containing waste [21]. The goal of one of the projects was to develop fluorescent lamps that recycle technologies for REM extraction. The developed technology included several stages. First, lamp sockets and metallic parts were separated from the glass. Then, glass covered by phosphors was ground and sieved. REMs were concentrated in a fraction of less than 10 μm. The REM content in this fraction and the initial material was 19.8 and 5.8%, respectively. The rate of REM extraction was 94%. The obtained ground material was treated with 4M sulfuric acid at 90°C for 4 h with stirring (600 rpm); the pulp density was 50 g/L and the total REM extraction reached 80% (85% Eu, Y > 95%, La, Ce, 10–30% Tb). It was revealed that YEO phosphor, which contains oxides of Y and Eu, was completely dissolved in 2 M acid at 90°C, but was insoluble at an ambient temperature. The solubility of LAP phosphor, which contains La, Ce, and Tb phosphates, increased in concentrated acid solutions.

The same hydrometallurgical methods and mechanical operations were used to extract rare-earth components from the spent cathode ray tubes and monitors [17, 22]. Several poorly accessible works on recycling scrap fluorescent energy-saving lamps and monitors, REM extraction from automobile catalytic converters, catalysts used in oil refinery, and red mud of the aluminum industry were cited in a report by the Oeko-Institute [1]. Recent data on the development of REM extraction from different types of wastes were presented in works [23, 24], as well as in the proceedings of the 1st Conference on European Rare-Earth Resources. For example, it was reported that, in 2012, plants in La Rochelle and Saint Fons owned by the Solvay Group began the deep processing of phosphors from utilized fluorescent lamps using solvent extraction for REM separation. Terbium production reaches several tons per year [25].

Spent compact and linear fluorescent lamps are the most suitable and promising raw material for the hydrometallurgical processing of REM-containing wastes in the Russian Federation. These lamps contain europium, terbium, and yttrium, which are the most expensive and deficient REM. Furthermore, these rare-earth components are demanded by the market. Currently, the collection of the lamps and primary processing (grinding and demercurization) are carried out in Russia. In Moscow, Moscow Oblast, and St. Petersburg, more than 10 million lamps are processed annually. The development of REM extraction from these wastes will allow one to extract up to 500 kg of yttrium and several dozen kilograms of europium and terbium. It can almost completely cover domestic demand.

CONCLUSIONS

The application of waste for REMs and the production of their compounds is not alternative to recovering rare-earth components from mineral resources and large-scale man-made raw materials (phosphogypsum and red mud), as well as from middlings (such as phosphoric acid). The attractiveness of the considered REM-containing wastes for recycling is due to their high REM content, the absence of radioactivity, which is characteristic of all types of REM-containing mineral raw materials), environmental safety, low costs required for production development, and by high demand. Not all natural mineral resources and large-scale manmade waste possess the above-listed advantages.

The estimation of hydrometallurgical approaches for processing different REM-containing wastes allows one to note the following advantages: low costs for developing compact plants, the possibility of using all kinds of raw materials, application of the same approaches as in mineral processing, and the possibility of obtaining very pure oxides suitable for any application. Common disadvantages of the considered hydrometallurgical technologies include the large number of required operations, the application of the large number of reagents, and the need to recycle large volumes of waste solutions contaminated by organic and inorganic compounds.

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