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Preparation of Sm_2O_3 and Co_3O_4 from SmCo magnet swarf by hydrometallurgical processing in chloride media

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Abstract

The recycling of rare earth elements (REE) from end-of-life REE based products is an environment friendly proposition. Waste Sm-Co based permanent magnet generated during machining is a good source for both Sm and Co. In the present study a simpler process of acid leaching at 80 ºC followed by solvent extraction, oxalate precipitation and calcination is described for producing pure Sm_2O_3 and Co_3O_4. With either 10 vol% H_2SO_4 or 15 vol% HCl at 80 ºC more than *95%* Sm and Co are leached in 1 h. Extraction of Sm from sulphate leach liquor with TBP or Aliquat 336 was poor. Although extraction with TOPS-99 is quantitative but Sm from sulphate leach liquor precipitated as $Sm_2(SO_4)_3 \cdot 8H_2O$. The chloride leach liquor at an initial pH of 2.5 and with 1.2 mol/L TOPS-99 shows requirement of 4-stages at A:O=3:2. Stripping with oxalic acid precipitates Sm-oxalate which is calcined at 800 ºC to produce Sm_2O_3. Co_3O_4 is recovered from the raffinate through oxalate precipitation followed by calcination at 450 ºC.

Keywords: SmCo magnet; Samarium; Cobalt; TOPS-99; Solvent extraction; Precipitation stripping

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1 Introduction

The use of rare earth elements (REEs) is increasing day by day due to its diverse applications in several sectors such as energy, environment, automobiles, aerospace, and defence. This increasing demand of REEs increases excavation of primary REE ores and environmental pollution due to excavation. On the other hand, the recycling of REEs from end-of-life REE based products is environment friendly. The permanent magnets, NiMH battery, phosphor powder and FCC catalyst are main sectors for recycling of REEs[1–3]. Due to constraint of waste management and collection system, the recycling rate of the rare earths is very low. In a critical review[4] on recycling of rare earths it is shown that less than 1% REE had been recycled by 2011.

The SmCo rare earth magnets have high coercivity, excellent thermal stability and good corrosion resistance. These magnets are available in two forms: one is $SmCo_5$ and the other one is Sm_2Co_{17}. The magnets are machined to have desired shape and size for particular application and during this machining process some magnets are broken and damaged. Samarium is an important and expensive rare earth element and should be recycled for valuable products. Stanton[5] investigated the sulfation-roasting and leaching process for recycling of Sm-Co magnet swarf and determined the effects of acid addition, roasting temperature and time, pulp density, gas composition, flow rate, etc. on samarium recovery. These magnets mainly contain Sm and Co and these elements can be recovered by hydrometallurgical route using leaching, solvent extraction and precipitation process.

In general, literature reveals that samarium extraction can be carried out with various extractants such as Cyanex 301 (bis(2,4,4-trimethylpentyl)dithiophosphinic acid), D2EHPA (di 2-ethyl hexyl phosphoric acid), Cyanex 923 (a mixture of four trialkyl-phosphine oxides including: $R_3P(O)$, $R_2R'P(O)$, $RR'_2P(O)$ and $R'_3P(O)$ Where R = $[CH_3(CH_2)_7]$-normal octyl, R'= $[CH_3(CH_2)_7]$-normal hexyl), Ionquest-801 (2-ethylhexyl 2-ethylhexyl phosphate), PC88A (2-ethylhexyl)phosphonic acid mono-2-ethylhexyl ester) and high molecular weight amine. Sm (III) from aqueous nitrate solution was extracted using Cyanex 301 alone and in combination with D2EHPA[6,7]. Similarly the extractant Cyanex 923 in different diluents such as chloroform, carbon tetrachloride, n-octane, cyclohexane, 1,2-dichloroethane, benzene, toluene, xylene and nitrobenzene was used for the extraction of Pr(III) and Sm(III) from nitrate medium[8]. The extracted species was found to be $(M(NO_3)_3 \cdot 2CY923)$ where CY923 refers to Cyanex 923. With a micro pilot unit of mixer-settler, the separation of samarium and gadolinium was achieved from a chloride leach liquor of monazite containing both heavy and medium RE [9]. The extractant used was Ionquest-801. Other authors performed the separation of Sm from chloride medium with PC88A and partially saponified PC88A[10]. The extraction reaction of Sm with PC88A was determined and the distribution coefficient of Sm with saponified PC88A was predicted with help of chemical model. El-Hefny et al.[11] studied the extraction of Sm using sodium salt of Cyanex 272 (bis(2,4,4-trimethylpentyl)phosphinic acid). The effect of different parameters on extraction equilibrium was investigated and they observed that samarium extraction increased with increase of pH. The kinetic study was carried out with stirred Lewis Cell and the reaction mechanism was found to be controlled by chemical reaction in the bulk phase. Samarium extraction was also carried out by high molecular weight amines[12].