



Leaching kinetics study of neodymium from the scrap magnet using acetic acid



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ABSTRACT

Leaching kinetics of neodymium was investigated from Nd–Fe–B scrap magnet using green solvent as CH₃COOH. The Characterization study of the scrap magnet was performed by XRD and SEM–EDAX to ascertain the phase as Nd₂Fe₁₄B (31.05% Nd, 65.15% Fe and 0.66% B). Influence of the factors such as CH₃COOH concentrations (0.05–0.8 M), agitation speed (200–1000 rpm), particle size (45–150 μm), temperature (308–353 K) and S/L ratio (1–5% (W/V)) affecting on leaching of Nd was investigated to obtain an optimum condition. Maximum yield (99.99%) of Nd along with definite extraction of Fe was resulted at the condition: Agitation speed: 800 rpm, S/L: 1% (W/V), temp. 353 K, 0.4 M CH₃COOH and particle size: 106–150 μm). The key factors such as CH₃COOH concentration and temperature appears to be critical on effective dissolution of both Nd and Fe from the scrap magnet phase. Leaching kinetic results showed best fit with the shrinking-sphere model ($1 - (1 - X)^{1/3} = k_{ap}t$) ensuring the overall leaching process is governed by surface chemical control mechanism. The activation energy determined from the experimental study was of +17.13 kJ/mol further more supports the proposed chemical control leaching process. Thermodynamics variables like ΔG^0 , ΔH^0 values determined from the Arrhenius plot at varied temperature indicating the feasibility of the leaching reaction of endothermic type. Linear dependence of the plot of $\log k_{ap}$ on $\log 1/d_0$ and a first order dependence of k_{ap} with CH₃COOH at 0.4 M was derived from the above proposed leaching model.

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1. Introduction

There is a growing demand of the high-tech rare earth metals such as Nd, Pr and Dy in day by day life owing to their numerous usages in various electronic and mechanical devices like computers, audio-visual components, magnetic separators, cranking motor of automobiles, military and aerospace system [1–5]. To meet the global demand of neodymium (Nd) metal a number of investigations for extraction of above metals being proposed by researchers [1–10] from various primary and secondary sources. At present scenario, the neodymium bearing minerals are limited and therefore, secondary sources appear to be the potential candidates claiming the above metals up to a considerable amount. Waste printed circuit board (PCBs), spent/scrap magnet, battery waste and magnet waste are the vital source of secondary containing significant amount of rare earth metals [7–13]. Nevertheless, these secondary sources are being produced from various industries and/or as a solid waste generated from domestic utilities and subsequently they are dumped to soil level. This leads to contaminate

the soil, water as well as air of earth due to liberation of toxic gases and hazardous metals which substantially causing the environmental issues [14,15]. It is therefore urged to develop a promising process to recycle the solid waste by recovering the valuable metals vis-à-vis yielding zero waste to the environment. Off the above secondary source, scrap magnet (Nd–Fe–B) was found as one of the important source of Nd while bearing major content of Nd (~30%) and Fe (~64%) along with trace amount of B (~0.5%). As reported by the investigators [16,17], at about 25% of sintered Nd–Fe–B alloy materials are wasted as useless scraps in manufacturing processes during sintering, cutting/polishing, magnetization. The sintered magnet scraps are mainly of two types (i) Bulk-type scrap, reused as a part of raw alloy materials for producing the Nd–Fe–B sintered magnets where oxygen content of such cracked magnets is usually small (<0.5 wt%) and (ii) Fine powder scrap which is difficult to reuse directly as magnet components, since it is highly oxidized for the strong oxygen affinity of rare earth. Hence recycling of second type is highly necessitated because of above reasons. In addition, Nd–Fe–B composite type of magnet has huge application in electronic and other devices in consequence of have number of advantages such as high value of energy product, high resistant to the demagnetization at room temperature and cost effective

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[10,12,18–21]. Therefore, to lower the production costs, to reduce waste and in order to prevent the environmental pollution, recovery of Nd from scrap magnet through hydrometallurgical method can be of suitable approach [13,15,22–27]. The proposed leaching study for extraction of Nd from scrap magnet from using green solvent can be an alternate solution for the recycling of the above solid waste while yielding sizable quantity of pure neodymium/neodymium compound without making any environmental impact.

Various pyro- and hydro- metallurgical approaches for recovery of Nd from scrap magnet have been reported by many researchers [28–31]. The processes such as carbonylation [6,7], chlorination using NH_4Cl [17], glass slag method [28], molten magnesium metal technique [30], chemical vapour transport [31] and leaching of Nd using mineral acid [9–12] were investigated for substantial recovery of Nd. However, due to number of disadvantages in pyro processing method especially energy intensive, less selectivity, hydrometallurgical leaching method appears to be most promising for extraction of rare earth metal such as Nd. Up to now, a number of acidic, alkaline and ammoniacal reagents are being employed for leaching of Nd from scrap magnet and overall yielding of Nd are also impressive. Although, the use of mineral acids (sulphuric acid, nitric acid and (or) hydrochloric acid), ammonia, NaOH are more common while developing the hydrometallurgical leaching process, at the same time these reagents do produce hazardous/toxic gases to environment during leaching. Moreover, subsequent acid or alkali based leach solution generated after leaching is not being recycled in general and thereby loss of the above reagents is encountered [9–13]. Therefore, having a number of advantages like low energy consumption, low gas emissions, low waste generations and recyclability nature, adaptations of green organic reagents in hydrometallurgical leaching processes are being well practiced for leaching of rare earth metals [11].

Leaching of the rare earth metals with various organic and inorganic acids have been investigated by the researchers [10–12]. Lyman et al. have developed a process flow sheet which includes the recovery of valuable rare-earth materials by leaching of scrap magnet using H_2SO_4 solution followed by precipitation of recyclable rare-earth salts [20]. Leaching kinetic study of neodymium from Nd–Fe–B scrap magnet was carried out using H_2SO_4 acid & almost all (~99.99%) quantity of Nd was recovered to the aqueous phase [10]. A comparative study of leaching of rare earth metals (La, Nd & Lu) from bauxite residue (red mud) was examined by Borra and his co-workers. In which they used different mineral acid (Nitric, hydrochloric, sulphuric and methano sulphonic acid) and organic acid (citric and acetic acid) for leaching process. Neodymium (Nd) and dysprosium (Dy) was quantitatively recovered from magnetic waste sludge generated from the Nd–Fe–B (-Dy) manufacturing process using H_2SO_4 solution [11]. Recovery of Nd from Nd–Fe–B scrap magnet by selective chlorination process using NH_4Cl was studied by Itoh et al. [7]. The above literature survey indicated about effective recovery of neodymium and other rare earth metals by hydrometallurgical leaching methods using mineral acid as well as their mixtures. Though, leaching of scrap magnet by mineral acid as reported in the above literatures showed the significance on effective recovery of rare earth metals, there is still the issue on regeneration, liberation of toxic gases during leaching and inconvenience on handling high conc. acid in industrial scale operation as has been discussed in previous section. Therefore, the adaptation of green organic solvent as the lixiviant on hydrometallurgical leaching process could be an environmentally friendly in contrast to the use of hazardous, concentrated inorganic or mineral acid and alkali reagents [11,22]. Moreover, Leaching of metal using green solvents (acetic acid, citric acid, and oxalic acid) in hydrometallurgical process [22–25] has several advantages including ease degradable property under

aerobic and anaerobic conditions, less emission of hazardous gases, possible regeneration property ascertaining the loss of reagents. Off these organic acids, acetic acid found to be effective on efficient leaching of valuable as well as rare earth metals from several sources [31,32]. But the investigation pertaining to the leaching of Nd or other rare earth metals using organic acid especially acetic acid is scanty. Therefore, the dissolution of Nd from waste magnet using acetic acid was attempted in this investigation.

The leaching of Nd from Nd–Fe–B scrap magnet was investigated using dilute glacial acetic acid. The process optimization and kinetics study on the dissolution of Nd from Nd–Fe–B scrap magnet in the function of stirring speed, acid concentration, and temperature & particle size was examined and established. Typical samples are characterized by SEM/ XRD and reported.

2. Experimental

2.1. Materials and reagents

The scrap permanent magnet sample was procured and used as it is without any further treatment. For leaching study, magnet sample was grinded to a sizable fine fraction by the pulveriser. The particle were sieved to obtain a desired size fraction and in the present study and the particle size range of 106–150 μm was kept fixed in most of the leaching study unless otherwise specified. The chemical assay of the spent magnet was ascertained by XRD and phase analysis was carried out by XRD (Model PAN ANALYTICAL PW 1830). However, to ensure the absolute content of the said metals the quantitative analysis of the each of the element (Nd, B and Fe) was performed after digesting the original scrap magnet using hydrochloric acid solution. The neodymium (Nd), iron (Fe) and boron (B) bearing aqueous samples were analysed by MP-AES (Agilent 4200).

2.2. Leaching procedure and conditions

The leaching experiment was carried out in a three necked round bottom flask which was placed on a heating mantle (set at desired temperature). The solution was kept under agitation using an externally connected agitator. When the lixiviant attained the desired leaching temperature the scrap magnet powder sample of the appropriate particle size range was charged into the flask containing 100 ml of the acetic acid solution. The effect of changing of one of the five parameters: (i) agitation speed, (ii) acid strength, (iii) temperature and (iv) pulp density and (v) particle size range was investigated. During leaching experiments one of the parameters was changed while other parameters were maintained constant. The effect of stirring speed was tested at 200–1000 rpm. The average particle size of the spent catalyst was 106–150 μm in most of the experiments, excepting effect of particle size study where particle size range was of 46–75 μm to 151–180 μm . The acid concentration was of 0.4 M CH_3COOH , except when the effect of acid strength was studied at the concentration range of 0.05–0.8 M. The pulp density was maintained low at 0.05–5% solids (w/v). In order to ascertain reproducibility, some of the leaching experiments in present studies were repeated and the experimental error was found to be of $\pm 2\%$.

3. Results and discussion

3.1. Characterization of scrap magnet

The powder scrap magnet sample was characterized by XRD, SEM and further confirmed by wet chemical analysis using MP-AES. From Fig. 1 SEM–EDAX results indicated the elemental profile

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