Selective neodymium recovery from model permanent magnets using cost-effective organic acid systems

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Experimental section

1. Materials and methods

1.1. Materials

Neodymium (III) oxide (Nd_2O_3, 99.9% Merck) and iron (III) oxide (Fe_2O_3, >96% Sigma Aldrich) were used to mimic spent magnets components in the dissolution experiments. The chemicals used in this study were guanidine hydrochloride (GUC, >99% Merck), as a hydrogen bond acceptor (HBA) and DL-lactic acid (90%, Sigma Aldrich), and acetic acid (100%, Univar) as hydrogen bond donors (HBDs).

1.2. Dissolution experiments

The leaching efficiency and selectivity of Nd_2O_3 and Fe_2O_3 by two DES-like solvents; GUC: lactic acid and GUC: acetic acid were investigated. To investigate the influence of the separate

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components of these mixtures – HBA or HBD – with water mixtures, water was used in the same ratio as the component it replaced. Thus, GUC-water, lactic acid-water, and acetic acid-water were prepared with molar ratios of HBA:water, and HBD:water of 1:6, and 6:1, respectively. All the solvents were prepared by mixing the components for 3 h at 50 °C with 500 rpm.

To understand the dissolution mechanism, the experiments were performed in different steps: (i) separate leaching of Nd_2O_3 and Fe_2O_3 in the above-mentioned mixtures (ii) leaching of Nd_2O_3 and Fe_2O_3 mixtures with the mass fraction ratio of 1:1 (iii) leaching of Nd_2O_3 and Fe_2O_3 mixtures at around the same mass fraction ratio as REE magnet composition (1:7 wt.%) delivered by AMF magnetics.

A 10 mg solid sample was placed in a glass vial and 1.5 mL of solvent was added. The glass vial was immersed in thermal beads to ensure a uniform temperature distribution in the samples. The reaction mixture was then heated at 50 °C and stirred at 500 rpm for 24 h. Then, the solution obtained from the leaching experiments was filtered using a syringe filter (PTFE Filter, pore size: 0.22 μm, diameter: 30 mm, Membrane Solutions, LLC) and the filtrate was analysed using Inductively coupled plasma mass spectrometry (ICP-MS). The leaching efficiency (L %) was calculated as follows:

$$L = \frac{w_{M^{n+}}(l)}{w_{M^{n+}}(i)} \times 100 \text{ \%} \qquad (1)$$

where $w_{M^{n+}}(i)$ is initial mass concentration of metal in solvent before leaching, $w_{M^{n+}}(l)$ is the calculated mass concentration of metal in solvent after leaching.

A selectivity factor (SF) was calculated to determine the extent of preferential dissolution of one element over another (A and B respectively) in a mixture, according to the following equation from the literature[28]:

$$SF_{A/B} = \frac{\dfrac{L_A}{1-L_A}}{\dfrac{L_B}{1-L_B}} \qquad (2)$$

where L_A and L_B are the leaching efficiencies of elements A and B, respectively.

1.3. Analytical and characterization methods

Inductively coupled plasma mass spectrometry (ICP-MS, PerkinElmer NexION 350X) was employed to determine the concentration of metals in the solution (leachate) after leaching experiments. ICP samples were prepared by diluting the leachate with 2 % (v/v) nitric acid.

X-ray Powder Diffraction (XRD) characterization was performed between 20 and 100° on a Panalytical X'pert Powder powder diffractometer fitted with a 10 mm slit. XRD measurements were performed to analyse the phase composition of Fe_2O_3 before and after leaching. Fe_2O_3 before leaching was directly used for the XRD measurement. Fe_2O_3 after leaching was filtered from the leachate using a syringe filter (PTFE Filter, pore size: 0.22 μm, diameter: 30 mm, Membrane Solutions, LLC) and the residue analysed using XRD.