



Association characteristic study and preliminary recovery investigation of rare earth elements from Fire Clay seam coal middlings



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ABSTRACT

Rare earth element (REE) recovery from coal and coal byproducts using physical and/or chemical methods has been the focus of recent investigations due to concerns over worldwide supply. In the current study, the association characteristics of REEs and the recovery of REEs from the organic and inorganic components were systematically studied for a Fire Clay seam coal source. The coal sample was ground in a stirred ball mill and treated by froth flotation in stages to obtain clean coal concentrates and tailing samples. The clean coal concentrate from the first stage was re-ground to liberate more micro-dispersed mineral matter followed by flotation. After six grinding and recovery stages, the cumulative grinding time was 110 min, which reduced the particle size from 90% passing 84.3 μm to 4.5 μm . Analyses conducted on the tailing samples collected from each stage revealed that the REEs have a strong association with micro-dispersed kaolinite. The REE content in the tailings material increased from around 580 ppm to 1200 ppm on an ash basis. The ratio of heavy-to-light REEs in both the clean coal concentrates and tailing samples increased, which indicated a stronger affinity between the heavy REEs and the finest mineral matter grains in the organic matrix. Based on these findings, REE concentration tests were performed using froth flotation, magnetic separation and acid leaching. A product containing nearly 7000 ppm of REEs was achieved from a feed containing 500 ppm REEs using a batch WHIMS unit. The elemental content indicated that the rare earth mineral in the concentrate was primarily monazite. Leaching the high ash content fractions generated from the sequential grinding and flotation steps using 1 M nitric acid resulted in recovering more than 80% of total REEs with the exception of scandium. A lower recovery value obtained for scandium indicated a unique association with the mineral matter relative to the other REEs.

1. Introduction

The world demand for rare earth elements (REEs) in 2011 was approximately 105,000 tonnes \pm 15% and growing at a rate between 5% and 9% due to their importance in manufacturing advanced military and renewable energy technologies as well as many commodity items used by the general public [1,2]. Coal is naturally enriched with specific REEs that have been identified as critical due to the balance between market demand and supply. The average rare earth element (REE) concentration in coal worldwide is about 69 ppm, which varies widely depending on location and coal seam. For example, REE concentrations of 62 ppm and 138 ppm were reported for US and Chinese coals, respectively [3,4]. Based on worldwide recoverable reserves of the coal and its average rare earth concentration, the amount of REEs associated with coal deposits was estimated to be more than 61 million metric tonnes, which is more than half of the traditional rare earth ore reserves and enough to meet the worldwide REE demand for several centuries [5,6]. Another attractive characteristic of the REEs in coal is the higher

concentration of the more critical, highly-valued REEs compared to commercial rare earth mineral deposits involving monazite and bastnaesite [4].

There are several well-known coal deposits with extremely high content of REEs such as the Far East coalfields in Russia (300–1000 ppm), the Fire Clay coal seam in the United States (500–4000 ppm), and the Sydney Basin in Nova Scotia, Canada (72–483 ppm) [7–10]. Various REE modes of occurrence have been reported in coal, i.e., organic associations, aluminosilicate minerals, accessory minerals, resistate minerals, authigenic minerals, etc. [7–16].

The economic potential of rare earth elements (REEs) in coal and coal byproducts such as fly ash has been the focus of recent research. The U.S. Department of Energy (DOE) selected several projects in 2015 to evaluate the recovery of REEs from coal and coal byproducts. The results from several studies have been recently published [17–26]. However, most of these studies focused solely on REE or rare earth mineral recovery without considering the association with other minerals. In the current study, a systematic study of the mineral and

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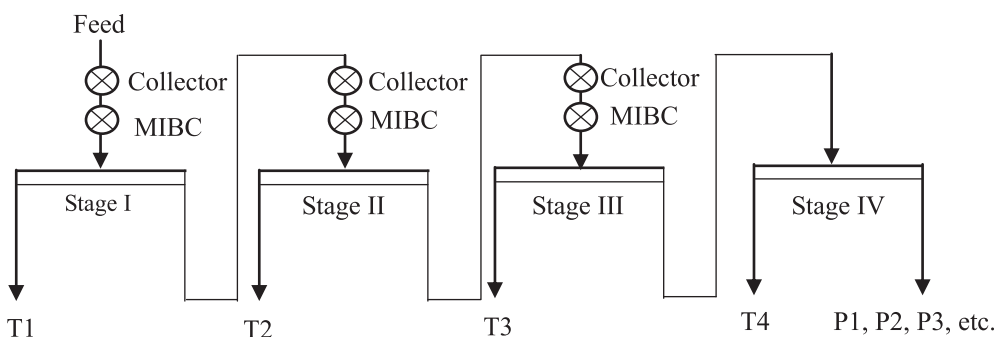


Fig. 1. Flowsheet of the coal release test (T and P mean high ash-content tailings and low ash-content clean coal, respectively).

element association characteristics and the concentration potential from the organic and inorganic components of a Fire Clay seam coal source was conducted. REE distribution within the coal source was evaluated by density fractionation and by surface properties using release analyses. The REE concentration potential was assessed by conducting tests involving froth flotation, magnetic separation, and acid leaching, which resulted in an in-depth understanding of REE recovery requirements for an REE-enriched coal source.

2. Materials and methods

2.1. Materials

A representative middling coal (1.50 × 1.70 S.G.) was collected from the secondary dense-medium vessel of an eastern Kentucky coal preparation plant. Middlings material was selected for the test program due to: 1) elevated total and heavy REE contents on a dry, whole mass basis, 2) presence of coal that can be liberated and recovered for added value and 3) preliminary findings indicating that REE recovery by leaching was uniquely favorable relative to other fractions of the coal source. The Fire Clay coal is a high volatile A bituminous coal with an ash content of 29.07% and a total REE content of 270 ppm on a dry, whole mass basis or 930 ppm on a dry, ash basis. The middlings material was crushed from its original top particle size of 50 mm–1 mm using a laboratory jaw crusher and hammer mill. The crushed material was wet-screened into different particle size fractions (i.e., $-1 + 0.5$ mm, $-0.5 + 0.3$ mm, $-0.3 + 0.15$ mm, and -0.15 mm). Some of the original middlings were directly crushed to a particle size finer than 0.15 mm for the rare earth release and the coal release tests.

A lithium metatungstate (LMT) solution, which had a specific gravity of 2.95, was purchased from LMT LIQUID, LLC and used as the heavy liquid for the density fractionation tests. Fuel oil No. 2 and sodium oleate were utilized as collectors for coal and rare earth minerals, respectively. Solution pH values were regulated using hydrochloric acid and sodium hydroxide solutions. Concentrated nitric acid was used for acid leaching tests. All of the above chemicals were purchased from Fisher Scientific.

2.2. Density fractionation tests

The results from density fractionation tests provided the distribution of weight, ash content and REE content by density in the aforementioned particle size fractions. Heavy liquid solutions of desired specific gravities, i.e., 1.40, 1.60, 1.80, 2.0, 2.20, and 2.40, were prepared by mixing a certain amount of LMT liquid with tap water. Solid samples were mixed with the heavy liquid solutions (800 ml) using a stirring rod in a 1 liter beaker, after which, the slurries were settled for a period of time until clear float and sink layers were observed. The float materials were removed using a strainer. The float and sink samples were filtered and rinsed three times using hot water to remove residual heavy liquid solutions. Since the analysis is not effective for ultrafine size fractions, density fractionation tests were only performed on the $-1 + 0.5$ mm,

$-0.5 + 0.3$ mm and $-0.3 + 0.15$ mm size fractions.

2.3. Flotation tests

Separations for particles finer than 0.15 mm were evaluated using the froth flotation process, which achieves a separation based on surface hydrophobicity differences using froth flotation. Selective flotation can be achieved by selecting appropriate collectors that preferentially adsorb onto the targeted minerals. In the current study, froth flotation tests were conducted using a bench-top Denver flotation machine. Representative samples were mixed with a certain amount of water to obtain a solid concentration of 10% by weight. The slurries were initially stirred in the cells to achieve sufficient dispersion, after which, a collector and frother were added sequentially and the slurry conditioned for 5 min and 1 min, respectively. After conditioning, flotation was conducted for a given period of time using an appropriate air flowrate and agitation speed.

2.3.1. Coal release tests

Coal release tests were conducted to study the fractionation of the REEs with respect to ash content for the -0.15 mm size fraction and for the product of a 60 min attrition mill grind. The mill was a stirred ball mill containing 37% 5-mm and 63% 3-mm diameter stainless steel grinding media. The overall flotation test included four stages of cleaning and separation as labelled in Fig. 1. The first three stages were conducted according to previously stated flotation procedures except that fuel oil No. 2 and MIBC were added in batches. Flotation continued in each of the first three stages until all of the floatable material was recovered, which took about 2 h for each stage of flotation release test. The repeated stages resulted in the elimination of hydraulically entrainable material. For Stage IV, no collectors or frothers were added during the process of collecting several froth products by incrementally increasing the rotation speed and air flowrate.

2.3.2. Rare earth release tests

The results presented in this publication show that the total REE content and concentration of heavy and highly-valued REEs are the highest in the fraction of the coal containing the lowest amount of ash-bearing mineral matter. Possible explanations include an organic association and/or significantly elevated REE concentrations in the finely dispersed mineral matter within the coal. As such, a test program was conducted to determine if REEs are more concentrated in the micro-dispersed mineral matter. The tests involved liberating and separating the mineral matter in a sequential manner as shown in Fig. 2.

A representative sample of the middlings was first crushed to a particle size finer than 0.15 mm, which was below the upper particle size limit of the attrition mill. Next, the crushed sample was mixed with water to achieve a 28.5% solids concentration by weight and ground in an attrition mill for 5 min. The ground sample was diluted to 10% and subjected to froth flotation tests using the procedure detailed in Section 2.3. The froth concentrate was re-floated twice to reduce hydraulic entrainment and achieve maximum separation efficiency between the

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