Acid Leaching of Rare Earth Elements from Coal Ash Obtained from a Mongolian Surface Mine

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ABSTRACT

Rare earth elements (REEs) are crucial for various renewable and clean technologies, increasing the importance of their recovery from secondary sources. This study determines if REE content and recovery from coal samples, and their ash, could be obtained from a Mongolian coal mine deposit in sufficient quantities for reuse. 9 different coal samples were examined and the highest REE concentrations were found in samples 3, 7, and 4. Upon burning, REEs in the coal ash samples significantly increased in all three samples. Direct acid leaching further improved REE recovery (S3); moreover, when alkali pre-treatment and 1M hydrochloric acid leaching were used, REE recovery was increased even further (sample 7). These findings characterize coal ash at this mining site, and indicate that it could serve as a viable secondary source of REEs, using optimized leaching methods to enhance the effectiveness, for potential industrial applications in Mongolia.

Keywords: Rare Earth Elements, coal ash, acid leaching, alkaline pre-treatment, REEs from secondary sources

INTRODUCTION

Rare Earth Elements (REEs), distinguished by their unique electronic, magnetic, and optical properties, are comprised of 17 metallic elements, which include the lanthanides, scandium, and yttrium. Over the last three decades, REEs and their alloys have become crucial for various technological applications, leading to unprecedented demand. Applications include electronics, energy solutions (such as rechargeable batteries and solar panels), automotive technologies, and healthcare (e.g., MRI agents). REEs are also essential for the production of advanced materials such as superconductors, glass additives, and fluorescent materials, making REEs indispensable for modern technology [1].

The growing importance of renewable energy technologies is another major driver of the increasing demand for REEs, particularly in the wind power and solar energy industries. REEs such as neodymium (Nd), praseodymium (Pr), and dysprosium (Dy) are crucial for wind turbine production, while elements like indium, selenium, and tellurium is essential for solar panel manufacturing. To address supply challenges for the long term, the recycling of REE-containing products must be explored as a sustainable solution [2]. Globally, the production, consumption, and processing of REEs is uneven. The most abundant supply of REEs is currently located in China; the concentration of supply has raised concerns around the world, prompting extensive research into the criticality of these elements [3]. In 2022, for example, global extraction of REEs (subsequently processed as rare earth oxides) amounted to 300,000 tons, with China accounting for 70%, followed by the U.S. (14%) and Australia (4%) [4]. Due to the heavy reliance on China for the supply of REEs, researchers are now shifting their focus to secondary sources. These sources include the recovery of REEs from recycled materials, such as electronic waste, automotive catalysts, industrial by-products, and coal fly ash (CFA) [5].

Researchers have estimated that CFA contains an average of ~69 parts per million (ppm) of REEs, with some studies reporting concentrations as high as 500 ppm, depending on the geological origin of the raw coal [6]. Studies on bituminous and lignite coals show that REEs are more concentrated in CFA compared to the parent coal: 72 ppm in bituminous coal and 469 ppm in its ash, and 69 ppm in lignite and 378 ppm in its ash [7]. Studies have identified several key REE-hosting minerals, including monazite (a phosphate mineral with the complex formula [Ce-, La-, Nd-, Th- PO_4]), xenotime (a byproduct of monazite), zircon ([$ZrSiO_4$], which is commonly found in: acidic rocks, CFA, allanite, apatite, alunite supergroup minerals), and water-bearing rare earth Y phosphates (like rhabdophane and churchite). Rarely, a few oxides, carbonates, and fluoro-carbonates also contain REEs [8].

Recent studies have demonstrated that efficient acid leaching techniques play a critical role in enhancing REE recovery. Wang et al.[9] examined REE extraction from coal gangue, achieving approximately 80% recovery using mild acid leaching, emphasizing the importance of optimizing leaching conditions to reduce impurity dissolution. Similarly, van Wyk et al. [10] analyzed the leaching behavior of discard coal and CFA, revealing that temperature strongly influences REE leaching efficiency, while acid concentration plays a lesser role. These findings highlight the necessity of understanding the relationship between coal composition and leaching efficiency to maximize REE recovery.

Given this background, the objective of this research is to analyze REES in samples of Mongolian coal and coal ash, focusing on its: mineralogy; constitutive components; distribution; and its efficient recovery. Specifically, this study examines the recovery rate of REEs using direct and indirect leaching methods, at varying acid concentrations. Compared to other studies on this topic, the current study is aimed at characterizing the REEs at a geochemically and mineralogically unique Mongolian coal deposit. This focus contributes to the understanding of REEs and their distribution, which could be important for Mongolian mining coal companies to make reuse of their coal ash.

MATERIALS & METHODS

Materials

Samples were taken from a Mongolian coal deposit, corresponding to 9 different coal seams. Analytical grade hydrochloric acid was used in the leaching of coal ash, and analytical grade NaOH was used for pretreatment. Deionized water purified by Milli-Q Element A10 (resistivity=18.2 $M\Omega$) was used in all of the experiments.

Sample Preparation and Handling

The following treatments were conducted on each of the 9 coal samples collected from the mine site. First, a sample was divided using a splitter, then ground to a powder. The powder was then separated by size, in a Retsch AS 200 basic B Sieve Shaker. A 1 g sample of the <212 μm sieved fraction was used to determine moisture; another 1 g sample of this fraction was used to determine the amount of ash produced when burned. Finally, an approximate 10 g sample of the sieved fraction was sent to KhanLab LLC, Mongolia to determine the constitutive elements and concentration of REEs.

Based on KhanLab results, the ash was prepared from the <212 μm sieved fraction that were shown to have exhibited the highest concentration of REEs (Samples 3, 4, 7). Ash from coal sample 3 was sent to the Erdenet Mining Research Institute to determine microstructure and mineralogy of the coal ash fraction sample.

Moisture Content

The sample of the 212 μm fraction was heated at 105°C for 1.5 hours in a drying oven (Shanghai Jingke Scientific Instrument, Model-JK-DO-9240A). After cooling to room temperature, the sample was weighed again to determine the moisture content. The values from three trials were averaged, for each sample, to determine reported moisture content.

Ash Content

The sample of the 212 μm fraction was placed in a preheated muffle furnace (KarpatDental Germany, Model – KM1 MIHM-VOGT) at 850 °C for 2 hours. The ash was then cooled to room temperature weighed again. The ash content was determined by calculating the weight difference, and the average ash content was obtained from three trials for each sample.

REE Content and Main Constituents

At KhanLab, each 212 μm fraction from the 9 coal samples was chemically treated with a mixture of four acids (hydrochloric, nitric, perchloric and hydrofluoric acids), to solubilized them. The type of element, and its quantity, was determined by inductive coupled plasma-optical emission spectroscopy (ICP-OES), and the intensity of the excited atomic light emission in a high-temperature plasma environment was recorded and quantified. In addition, all coal fraction samples were sent to KhanLab to determine the main constituents of each sample.

After being sent the data on which samples contained the highest amount of REES, coal ash fractions from each of these samples and leaching residues were sent to the Baotou Research Institute for Rare Earth Elements (BRIREE); REE concentration was determined by ICP-OES.

Morphological and Mineralogical Analyses

At Erdenet Mining Research Institute, the mineral identification and composition of the sieved fraction from Sample 3, which has the highest REE levels, were determined using the TESCAN Integrated Mineral Analyzer (TIMA), and SEM at magnifications from 100x to 23,000x. Only the highest REE-containing sample was analyzed due to time and material costs.

Experimental Methods

The leaching processes for REEs from primary and secondary sources require the use of strong acids such as H_2SO_4, HCl, and HNO_3. The efficiency of REE extraction and the optimization of the leaching process depend on variables such as acid molarity, leaching temperature, run time, solid/liquid ratio, and stirring speed [8]. Additionally, alkali pretreatment, which helps break down aluminosilicate phases, has been studied to enhance REE extraction. In this context, [11] investigated the effects of various roasting additives such as Na_2O_2, $NaOH$, CaO, Na_2CO_3, $CaSO_4$, and $(NH_4)_2SO_4$ at 450 ℃, followed by acid leaching. Their findings showed that pre-treatment with alkali solutions, such as sodium hydroxide ($NaOH$), was the most effective additive, enabling the recovery of over 90% of REEs, thus complementing the acid leaching process by further optimizing the extraction efficiency.

Direct acid leaching: In order to obtain the highest concentration possible of REEs, leaching experiments were conducted on the coal ash sample 3 that contained the highest levels of REE. Leaching was performed using 1M, 3M, 6M, and 9M concentrations of hydrochloric acid, maintaining the solid-to-liquid ratio at 1:10. The process was carried out at a temperature of 80°C with a stirring speed of 300 rpm for 2 hours using a Heidolph Magnetic Stirrer (MR HEI-TEC $\emptyset 145$ (EU). At this point, all residues were sent to an outside laboratory so that the resulting REE concentration could be determined.

Indirect acid leaching (Alkali treatment which is then followed by acid leaching): Alkali treatment involved subjecting a high REE-containing sample to 12M NaOH, with a solid-to-liquid ratio of 1:10, at 150 °C for 2 hours, with a stirring speed of 450 rpm. Next, the sample was cooled in a water bath for 1 hour at room temperature, then filtered with ionized water using a 25-μm filter paper. To eliminate possible contamination by the sample during subsequent acid leaching analysis, the sample was rinsed 10 times more with ionized water. The remaining solid residue was then treated with HCl in a 1:10 ratio, at 80 °C for 2 hours with stirring at 300 rpm to possibly obtain even higher concentrations of REEs in the residue.

RESULTS AND DISCUSSION

Moisture and Ash Content

As shown in Figure 1, the total moisture content of all samples was relatively low, ranging from 0.8% to 2.1%. This is due to the prolonged exposure of the sample to air after sampling. The ash content in each sample varied from 8.6% to 28.4%, and the color of the ash differed depending on the chemical composition, as observed by other researchers [12].

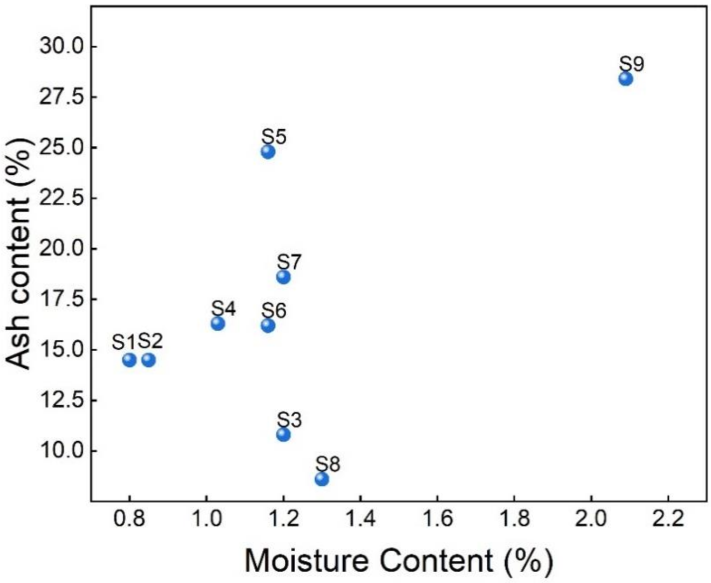

Figure 1. The total moisture and ash content for 9 coal samples (S1-S9)

Initial characterizations (moisture and ash content) and material preparation (uniform coal fraction turned to ash) were completed before sending all samples to KhanLab for analysis of constituent elements and REE content.

Main Constituents of the Coal Ash Fraction

In general, the chemical composition of coal ash is derived from inorganic minerals such as quartz, feldspars, clays and metal oxides, minerals closely related to the numerous rock types found in the Earth's upper crust [13]. Coal fractions were prepared for each of the samples provided by the mining company, and sent to KhanLab for chemical analysis. Results showed varying concentrations of the following major elements, depending on the coal sample: silicon (39-64.9%), aluminum (11.8-31.9%), iron (1.39-12.8%), calcium (0.64-11.6%), and sulfur (0.25-2.1%). These variations indicate that the coal fractions from each coal sample of the deposit exhibits distinct compositional characteristics, as summarized in Table 1.

Table 1. Main constituents of all coal sample fractions by ICP-OES, wt%

	S1	**S2**	**S3**	**S4**	**S5**	**S6**	**S7**	**S8**	**S9**
Zr	0.04	0.04	0.03	0.04	0.05	0.04	0.05	0.04	0.04
Sr	0.09	0.36	0.27	0.42	0.08	0.39	0.07	0.33	1.05
Pb	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0
Zn	0.03	0.02	0.05	0.01	0.02	0.01	0.03	0.08	0.02
Cu	0.03	0.04	0.04	0.03	0.03	0.03	0.04	0.12	0.01
Ni	0.04	0.05	0.03	0.02	0.03	0.03	0.04	0.02	-
Fe	12.8	1.92	9.28	3.37	1.39	4.86	4.71	8.39	5.6
Mn	0.15	0.03	0.17	-	0.03	0.1	0.2	0.15	-

Ti	0.81	1.43	1.19	1.5	0.88	1.28	1.4	1.66	0.68
Ca	6.93	5.06	6.94	11.26	2.56	5.56	5.29	6.89	0.64
K	1.3	0.8	0.9	0.8	0.6	1.7	0.9	1.4	3.1
Al	18.3	22	31.4	21.5	11.8	22.2	21.5	31.9	22.2
P	1.4	1.8	0.4	0.7	0.2	0.2	<LOD	1.3	1
Si	39.1	54.1	42.3	43.2	64.9	40.9	55.7	43.1	40.8
Cl	0.03	<LOD	0.02	0.02	<LOD	<LOD	0.01	0.02	-
S	0.7	0.9	1.4	2.1	1.3	2	1	1.4	0.3
Mg	-	3.2	5.4	6.8	2.2	2.5	3.3	3	-

Concentration of REEs

Table 2 below, presents the total REE content from each coal sample received from the mine site, as determined by KhanLab. Among the coal samples analyzed, S3 showed the highest total REE concentration at 311.36 g/t, followed by S7 with 201.06 g/t and S4 with 151.17 g/t. In contrast, the lowest REE concentration was observed in S1, at 60.3 g/t, with S8 and 6 also having relatively low values of 65.02 g/t and 75.07 g/t, respectively as shown in Table 2.

Table 2. Total REE concentration for each coal fraction

Coal Sample	La	Ce	Yb	Y	Eu	Sm	Dy	Nd	Er	Lu	Tm	Tb	Ho	Gd	Pr	Total REE (ppm)
S1	10.9	16	<1	8.65	<1	<3	<1	22.6	<1	<1	<2	<2	<5	2.1	<1	60.3
S2	15.9	22.7	<1	9.92	<1	5.6	<1	53.6	<1	<1	<2	<2	<5	8.1	4.17	119.96
S3	37.4	85.5	1.5	11.8	<1	<3	1.2	169	<1	<1	<2	<2	<5	5.1	<1	311.36
S4	10.1	14	1.4	8.89	<1	3.4	<1	101	<1	<1	<2	<2	<5	8.6	4.35	151.17
S5	13.6	31.3	<1	5.79	<1	5.5	<1	25	<1	<1	2.4	<2	<5	2.8	5.38	91.65
S6	14.7	24.3	<1	9.19	<1	<3	<1	10.4	<1	<1	<2	<2	<5	4.6	11.9	75.07
S7	30.4	57.5	<1	8.78	<1	3.7	<1	98.2	<1	<1	<2	<2	<5	2.5	<1	201.06
S8	12.5	22.4	<1	2.71	<1	<3	<1	27.5	<1	<1	<2	<2	<5	<2	<1	65.02
S9	18.4	35.4	<1	11.9	<1	4.6	1.1	9.7	<1	<1	<2	<2	<5	6.2	<1	87.18

As shown below, in Figure 2, all coal samples exhibited a broadly similar distribution of light (LREE) and medium (MREE) atomic weight, with light REE being clearly dominant.

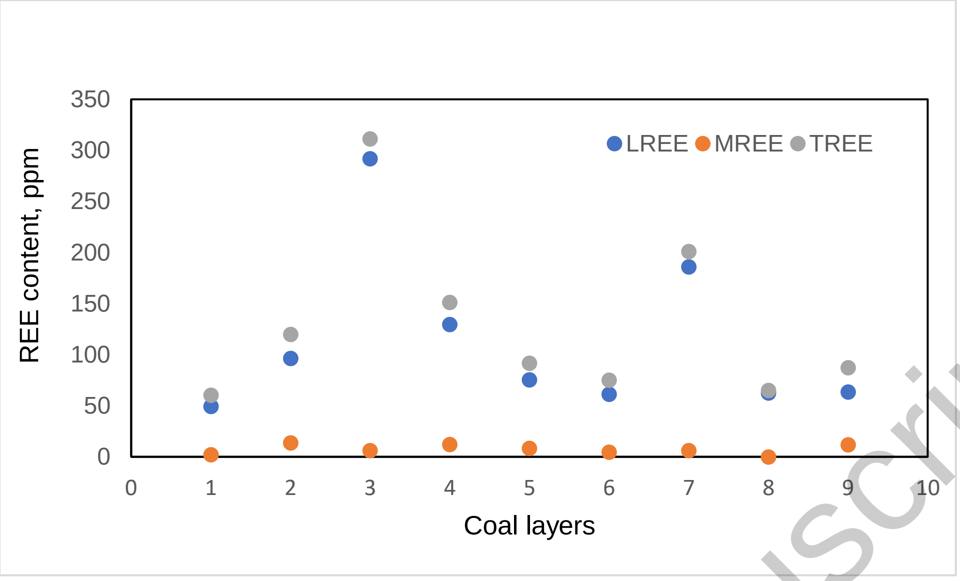

Figure 2. REE content in coal fractions from different samples LREE = Light, MREE = Medium, TREE=Total

Notably, La, Ce and Nd are the most widespread elements present. Based on these results, the coal ash fractions containing the highest concentration of REEs (S3) were sent to Erdenet Mining Research Institute to conduct the mineralogical and morphological analyses.

Morphological and Mineralogical Analysis

The morphology of the sieved fraction of coal ash from Sample 3, examined by Erdenet Mining Research Institute, is shown in Figure 3A. Figure 3C is an individual grain of coal ash from Sample 3, for comparative purposes. Although not a main focus of this research, the difference in morphology between coal ash and coal fly ash can be seen in Figures 3A and 3B, respectively. The morphology of coal ash in Figure 3A shows more angular and porous morphology, whereas the coal fly ash image in Figure 3B, which is from a previous experiment (unpublished data), predominantly exhibits smooth, spherical shapes. These spherical shapes are characteristic of complete combustion of coal, which has been associated with higher REE concentrations [14].

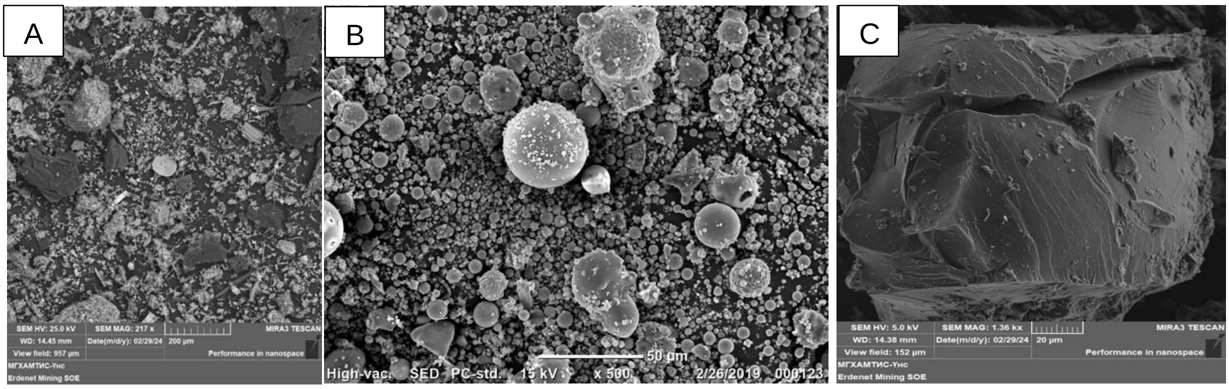

Figure 3. (A) SEM image of Sample 3 coal ash, at magnification 217x, 2024; (B) coal fly ash magnification 500x, 2019; and (C) a single grain of Sample 3 coal ash, at magnification 1360x, 2024

Mineralogical studies on CFA have identified mineral crystals, unburned carbon, and non-crystalline aluminosilicate glasses. These analyses have revealed four common glass compositions: (a) Al-Si aluminosilicate glasses, (b) aluminosilicate glasses enriched with calcium (Ca), iron (Fe), or both, (c) iron oxide minerals, and (d) quartz. Consistent with ~~this~~ previously published work [15] the mineralogical and morphological data in the current study confirmed that the Sample 3 test by Erdenet Mining Research Institute consisted mainly of amorphous and cryptocrystalline aluminosilicates (Figure 4a and 4b).

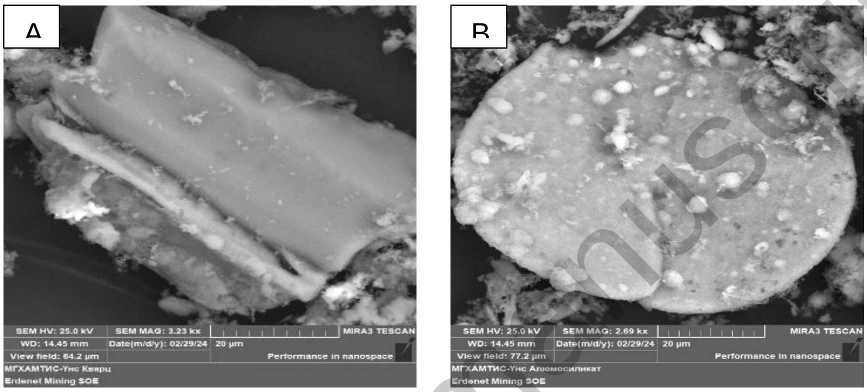

Figure 4. (A) The quartz magnification 3230x; and (B) aluminosilicate magnification 2690x.

Further, the TESCAN TIMA results, shown in Figure 5, reveal that the primary gangue minerals from Sample 3 are quartz (26.7%) and kaolinite (23.4%), and that apatite (a main REE-bearing mineral) is also present (3.38%).

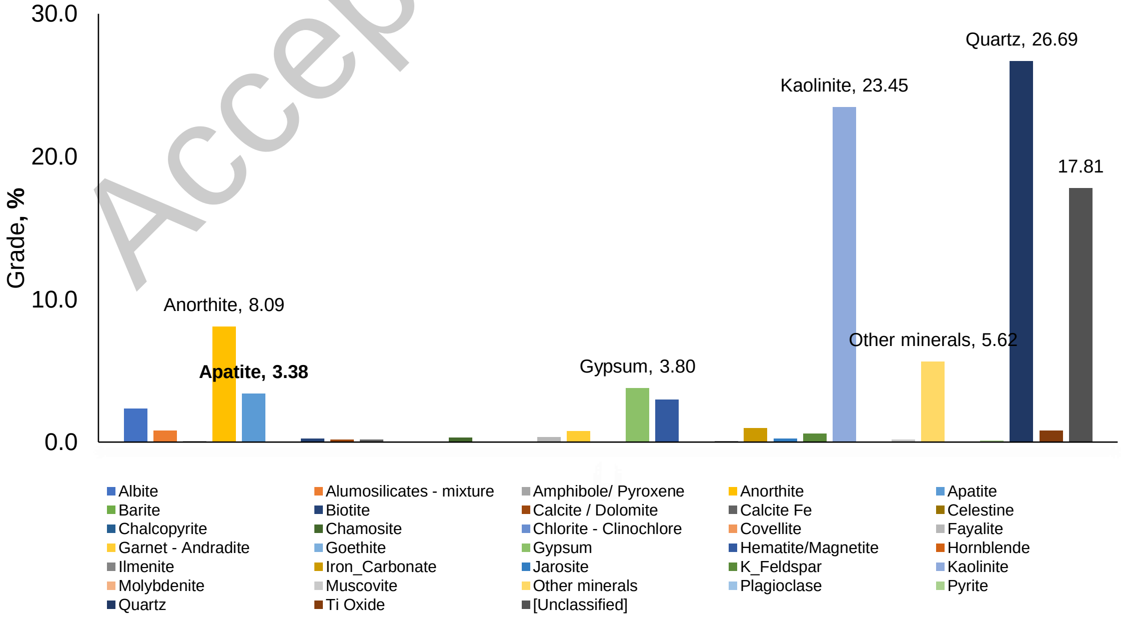

Figure 5. The mineral composition of coal ash of Sample 3

Direct Acid Leaching of REEs from the Coal Ash Fraction

The Baotou Research Institute for Rare Earth Elements (BRIREE) in China was chosen for direct and indirect acid leaching experiments due to their expertise in this area, as well as timing and capacity constraints with partner laboratories. Coal ash fraction from Sample 3 were treated as described in the Methods Section and sent to BRIREE to determine REE concentration in the coal ash and in the resulting solid residue. The REE recovery was calculated using the following equation:

$$REE\ recovery\ in\ the\ solid\ residue = \frac{M_1\ C_2}{M_2 C_1} \times 100\%$$

where M_1 is the mass solid residue (mg), C_2 is the REEs concentration in the solid residue (ppm), C_1 is the REEs concentration in the coal ash sample (ppm), and M_2 is the mass of coal ash sample (mg). The effect of hydrochloric acid concentration on leaching of REEs was examined, and the findings show that REE recovery decreases with increasing concentration of acid (Table 3). A decline in REE recovery is primarily attributed to the formation of silica gel which acts as a sorbent matrix, potentially adsorbing REE ions, obstructing their diffusion, thereby reducing their leaching efficiency [9], [16].

Table 3. Direct acid leaching of REEs for S3 coal ash on samples analyzed by BRIREE

Initial weight of coal ash (g)	Hydrochloric Acid Concentration (M)	Final weight of solid residue (g)	Concentration of REEs (ppm) in 10 g coal ash	Concentration of REEs in solid residue (ppm)	Recovery rate in the solid residue, (%)	REE recovery (%)
10	1	8.87	1340	840	55.60	44.40
10	3	8.9	1340	860	57.12	42.88
10	6	8.7	1340	1100	71.42	28.58
10	9	8.67	1340	1360	87.99	12.01

Since the objective in the present study was to systematically investigate the specific effect of acid concentration on REE recovery, while minimizing the contradictory effect of other variables, parameters such as a solid to liquid (S/L) ratio, leaching temperature, and contact time were held constant, based on values optimized by previous studies [11].

Indirect Leaching: Alkali Pretreatment Followed by Acid Leaching of the Sample 7

In order to try and obtain the highest rate of recovery of REEs, alkali pretreatment was used on a coal ash containing one of the highest levels of REEs (as determined in Table 4), followed by direct leaching. Sample 7 coal ash was used for this set of determinations as the Sample 3 material needed to be kept in reserve. Thus, coal ash fraction samples from Sample 7 were treated as described in the Methods Section, and sent to BRIREE to determine REE concentration in the coal ash and in the resulting solid residue (Table 4).

To determine the optimal conditions for indirect leaching, preliminary experiments were conducted on coal ash samples, varying the alkali concentration, S/L ratio, temperature, and contact time (unpublished results).

To investigate the effect of alkali treatment on REE recovery, a comparison was made between direct and alkali-treated samples. In the case of direct leaching, the REE content in the solid reside was 840 ppm (Table 3). However, after alkali treatment, the REE content in the solid residue decreased to 650 ppm. An initial sample of 15g of coal ash, containing a total REE concentration of 1250 ppm, was subjected to an alkali pretreatment step, resulting in a reduced solid mass to 12.51g. Subsequent acid leaching further reduced the solid mass to 9g. The alkali treatment helped dissolve problematic mineral phases, leading to an improvement in REE recovery, which increased to 62.6 %.

In summary, across the coal samples (S1-9), the moisture content of each sample was relatively low, ranging from 0.8 – 2.1%. The deposit was heterogenous, with an ash content ranging from a minimum of 8.6 % to a maximum of 28 %. Combustion of sieved coal significantly enriched the total REE concentration in the ash. Further, the REE content in the Mongolian coal deposit is mainly composed of lanthanum, cerium, and neodymium, suggesting that light REEs are more common. TESCAN TIMA analysis identified apatite, a well-known REE-bearing mineral, as comprising 3.38 % of the total mineral composition. The highest REE recovery rate, 44.4%, was achieved through direct acid leaching using 1M hydrochloric acid. Additionally, a sequential process involving alkali treatment followed by hydrochloric acid leaching of 1M, enhanced the total REE recovery rate to 62.6%. Therefore, the pre-alkali treatment and acid leaching method is recommended for extracting REEs from the coal ash of this Mongolian coal deposit.

CONCLUSION

This research set out to characterize REEs from a Mongolian mining company's coal ash, and to determine REE recovery under experimental conditions, using previously published methods (10, 11) that suggested this possibility. These preliminary findings were significant for several reasons: first, the presence of apatite (an REE bearing-mineral) was found to be present in the coal ash; second, REEs were enriched in the coal ash, and further enriched using experimental leaching methods; and third, these data are consistent with previously published data on CFA, suggesting that the coal ash from this mine could be further enhanced should its CFA be produced. However, further research is necessary to optimize the processing of REE enrichment techniques, to increase efficiency and cost-effectiveness.

AUTHOR CONTRIBUTIONS

Battsengel Baatar, Bayardulam Jamiyansuren and Baasandorj Myagmarsuren conceptualized and designed the research methodology. Bayardulam Jamiyansuren, Baasandorj Myagmarsuren and Narangua Khongorzul conducted the experiments, collected data and interpreted the experimental results. Baasandorj Myagmarsuren and Bayardulam drafted the manuscript, while Battsengel Baatar revised it. All authors approved the final version of the manuscript.

CONFLICT OF INTEREST

The authors declare no conflict of interest

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