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Article

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Energy Reports

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Elsevier

Suggested Citation: Lanzerstorfer, Christof (2018) : Pre-processing of coal combustion fly ash by classification for enrichment of rare earth elements, Energy Reports, ISSN 2352-4847, Elsevier, Amsterdam, Vol. 4, pp. 660-663, <https://doi.org/10.1016/j.egyr.2018.10.010>

This Version is available at:

<https://hdl.handle.net/10419/243548>

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Research paper

Pre-processing of coal combustion fly ash by classification for enrichment of rare earth elements

Christof Lanzerstorfer

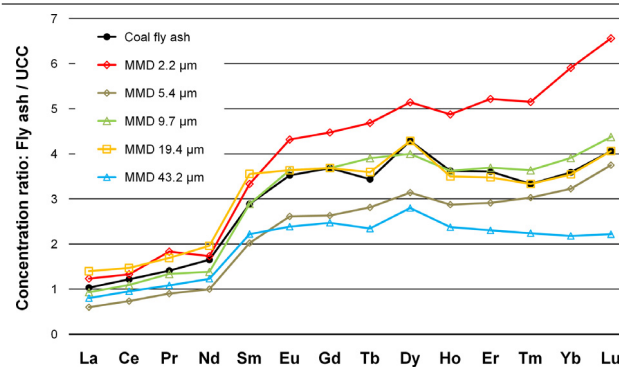
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HIGHLIGHTS

- Size dependence of various rare earth elements (REEs) was determined.
- Different distribution of light and heavy REEs was ascertained.
- Air classification might be a useful pre-treatment before REE extraction.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:

Received 25 August 2018
 Received in revised form 11 October 2018
 Accepted 18 October 2018
 Available online 30 October 2018

Keywords:

Coal combustion
 Fly ash
 Rare earth elements
 Classification
 Particle size

ABSTRACT

Fly ash from the combustion of coal shows potential as a source of valuable metals like the rare earth elements (REEs). These are present in the fly ash at low concentrations. Thus, simple dry pre-processing methods that can produce REE-enriched fractions from the fly ash could improve the feasibility of subsequent extraction processes. The performed air classification experiments with a coal combustion fly ash showed a certain size dependence of the REE concentrations. In the different size fractions the REEs were enriched or depleted. Thereby, the different groups of REEs showed differences in their behavior. In the finest size fraction all REEs were enriched by a factor of 1.05 to 1.65. In contrast, in the coarsest size fraction and in the second finest size fraction all REEs were depleted (0.63 to 0.78 and 0.58 to 0.92). In the medium size fraction the concentrations were close to those in the fly ash and in the second largest size fraction the light REEs (La, Ce, Pr, Nd and Sm) were enriched by a factor of 1.19 to 1.35, while the concentrations of the other REEs were similar as in the fly ash. Thus, air classification shows some potential as a simple dry pre-processing step for the separation of REE-enriched fractions from the bulk of a coal fly ash.

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1. Introduction

The increasing demand for rare earths elements (REEs) in a wide range of applications and the export quota reductions by China as the majority producer of REEs has increased the research interest in alternative resources for REEs as coal, coal by-products and coal combustion ash as well as expanded recycling (Haque

et al., 2014; McLellan et al., 2014; Dutta et al., 2016). Besides the valuable elements Ga, Ge and V, which can be recovered from the fly ash (Yao et al., 2015; Iyer and Scott, 2001; Font et al., 2005; Dai et al., 2018; Fang and Gesser, 1996; Dai and Finkelman, 2018; Sahoo et al., 2016), also REEs are present in fly ash from coal combustion (Blissett RS, Smalley and Rowson, 2014; Dai et al., 2014a; Franus et al., 2015; Zhang et al., 2015b; Vasconcellos and Ticianelli, 2017). In recent years, the recovery of REEs from coal fly ash has been discussed widely. However, the economic feasibility of the use of these residues in REE production is still uncertain

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Taggart et al. (2016). The utilization of coal fly ash as source for REEs shows several advantages compared to the use of REE-containing ore. First, fly ash is a readily available residue from thermal power plants, which still is partly deposited in landfills. Second, fly ash is a fine grained material which makes it ideal for processing by leaching saving the energy required for the comminution of ore.

The concentration of REEs in the fly ash depends the origin of the combusted coal. The typically range of the REE concentration of fly ash is 200 to 550 ppm (Yao et al., 2015; Iyer and Scott, 2001; Font et al., 2005; Dai et al., 2018; Fang and Gesser, 1996). However, maximum values up to 1400 ppm have been reported, too (Mardon and Hower, 2004). In contrast, the suggested cut-off grade is 1000 ppm (Dai and Finkelman, 2018). This makes recovery of the REEs from fly ash a complex task for which hydrometallurgical leaching techniques can be used (Sahoo et al., 2016). An important parameter in the leaching process is the efficiency of the recovery of the REEs (Kolker et al., 2017). The feasibility of such leaching processes might be improved by low-cost pre-processing steps in which the valuable elements are enriched.

Since the fly ash is separated from the off-gas usually by an electrostatic precipitator (ESP) which consists of several electric fields in series, some fractionation of the fly ash happens already during the separation. Thereby, the particle size of the separated dust decreases from the first field to the last field of the ESP (Ahn and Lee, 2006). However, in a study by Mardon and Hower (2004) only very little difference in the REE concentration of fly ash from the different fields of the ESP of a coal combustion power plant was found. A similar observation was made by Liu et al. (2017).

Querol et al. (1995) studied the composition of very fine size fractions of coal fly ash ($< 10 \mu\text{m}$). They used a cascade impactor method to separate such fine size fractions from the fly ash. Slightly higher concentrations of REEs were found in the finest size fraction $< 2.5 \mu\text{m}$. However, the increase of the REE concentration (excluding Eu, Gd and Sm which were not analyzed) was only approximately 5%. In the size fraction $2.5\text{--}10 \mu\text{m}$ the concentration of the REEs was even lower than in the total fly ash. In contrast, Blissett RS, Smalley and Rowson (2014) noticed a distinct increase of the concentrations of the rare earth elements with decreasing particle size. They separated coal fly ash into six size fractions by sieving. The finest size fraction produced was $< 38 \mu\text{m}$. The REE concentration in the finest fraction was approximately 18% higher than in the original fly ash. In a similar study by Zhang et al. (2015a) the results were likewise. The concentration of the REEs in the finest sieve fraction of $< 25 \mu\text{m}$ was somewhat increased. In another study by Dai et al. (2014b) an even higher enrichment of REEs in the finest sieve fraction of $< 25 \mu\text{m}$ was observed. There, the REE concentration was increased by approximately 90%. Thus, literature is somewhat inconsistent about the enrichment of REEs in the fine size fractions of coal combustion fly ash.

Pre-processing steps for the production of a REE-enriched fraction from coal fly ash has been proposed by various authors. Lin et al. (2017) investigated the enrichment of REEs from coal by-products by physical separation processes. They investigated classification by sieving, magnetic separation and density separation and found that the highest enrichment of REEs can be achieved by density separation. The particle size dependence of the REE concentration of the two fly ash samples investigated was inconsistent. Generally, the concentration of REEs was somewhat lower in the coarser size fractions compared to the fine size fractions ($< 38 \mu\text{m}$). Thus, a combination of density separation, magnetic separation and classification for the separation of a REE-rich fraction from the fly ash was recommended (Lin et al., 2017). However, such a process would be a wet process and therefore hardly fit into the dry off-gas de-dusting system of a power plant combusting coal. Blissett RS, Smalley and Rowson (2014) suggested a process

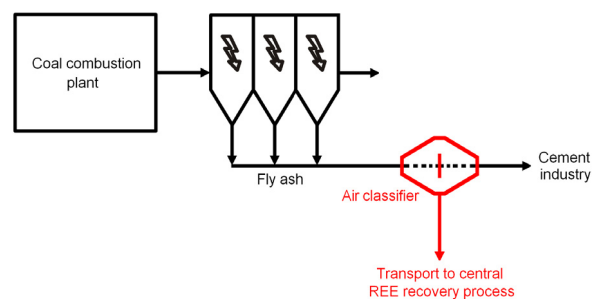


Fig. 1. Process flow diagram of the fly ash system (additional air classifier in red).

for pre-processing of fly ash consisting of a flotation step, a magnetic separation stage and classification by hydrocyclones. Also this process would be a wet process which is quite complex and difficult to integrate into dry off-gas de-dusting systems of power plants. Such processes would rather require a separate process unit.

In contrast to the suggested process combinations for the production of REE-enriched fractions from fly ash, air classification would be an entirely dry process which is relatively simple on an industrial scale, even with a cut size in the range of few micrometers. Such a process could be integrated directly into a power plant de-dusting system. There, the fly ash discharged from the ESP would be separated into two or more REE-enriched and REE-depleted size fractions (Fig. 1). Such on-site fractionation would increase the REE concentration in the fly ash fraction transported to a central hydrometallurgical processing plant for recovery of REEs and, maybe, other valuable metals.

In a recent study (Lanzerstorfer, 2018) the size dependence of various metals in fly ash from coal combustion was investigated applying air classification. The concentration of Ga, Ge and V showed a significant increase from the coarsest to the finest size fraction. In the finest size fraction with a mass median diameter of $2.2 \mu\text{m}$, Ga, Ge and V were enriched by a factor of 2.2 to 2.4 compared to the whole fly ash. The aim of this study was to extend this investigation to REEs.

2. Material and methods

The coal combustion fly ash investigated originated from a 420 MW_{th} power plant using Polish hard coal as fuel. For de-dusting of the flue gas a three-field ESP is installed. A fly ash sample of approximately 5 dm³ was collected at the fly ash discharge of the ESP. In the laboratory, the fly ash was dried. Subsequently, the sample volume was reduced for the air classification tests using a Haver&Boecker HAVER RT sample divider.

Classification of the fly ash sample was performed using a laboratory air classifier 100 MZR from Hosokawa Alpine. In the sequential classification procedure the finest size fraction SF1 was separated from the bulk in the first step. The remaining coarse fraction was used as feed material in the second classification step. Thereby, the classifier wheel was operated at a reduced speed. In this classification step the material was split into the fine fraction SF2 and a new coarse fraction. This procedure was repeated twice. Details about the fly ash and the separation into size fractions by air classification have been published previously (Lanzerstorfer, 2018).

The determination of the concentration of REEs was performed by the testing laboratory of the Environment Agency Austria (Umweltbundesamt). Prior to chemical analysis the samples were digested according to EN 13656 using a mixture of hydrochloric acid, nitric acid and hydrofluoric acid. The concentration of the REE elements (La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb and

Table 1
Chemical composition of the fly ash size fractions; concentrations in mg/kg based on dry weight.

	SF1	SF2	SF3	SF4	SF5	Total fly ash	Recovery rate
Mass fraction	7.3%	13.5%	20.3%	35.3%	23.6%	100%	–
MMD	2.2 μm	5.4 μm	9.7 μm	19.4 μm	43.2 μm	17.6 μm	–
La	37	18	28	42	24	31	101%
Ce	85	47	70	94	61	78	95%
Pr	13	6.4	9.5	12	7.7	10	98%
Nd	45	26	36	51	32	43	92%
Sm	15	9.1	13	16	10	13	100%
Eu	3.8	2.3	3.2	3.2	2.1	3.1	92%
Gd	17	10	14	14	9.4	14	90%
Tb	3	1.8	2.5	2.3	1.5	2.2	97%
Dy	18	11	14	15	9.8	15	88%
Ho	3.9	2.3	2.9	2.8	1.9	2.9	90%
Er	12	6.7	8.5	8	5.3	8.3	91%
Tm	1.7	1	1.2	1.1	0.74	1.1	97%
Yb	13	7.1	8.6	7.8	4.8	7.9	95%
Lu	2.1	1.2	1.4	1.3	0.71	1.3	94%
REE	270	150	213	271	171	231	95%
LREE	195	107	157	215	135	175	96%
HREE	75	43	56	56	36	56	91%
HREE/LREE	0.38	0.41	0.36	0.26	0.27	0.32	–

Lu) was determined according to EN ISO 17294-2 by inductively coupled plasma mass spectrometry (ICP-MS). The total carbon content of the size fractions SF1 to SF5 was 21.9%, 17.4%, 10.2%, 5.4% and 2.5% (Lanzerstorfer, 2018). The concentrations of the REEs are reported on a whole sample basis.

3. Results and discussion

Table 1 summarizes the REE concentrations in the original fly ash and in the size fractions produced. Additionally, the mass

fractions and the mass median diameters (MMD) are shown (Lanzerstorfer, 2018). The calculated recovery rates were close to 100% for most REEs. Only for Dy it was less than 90%.

Generally, the REE concentration of the investigated fly ash was on the lower end of the reported range. In SF1 and SF4 the REE concentration was higher compared to the original fly ash sample while in the other size fractions (SF2, SF3 and SF5) the concentration was lower. Distinct differences were observed in the distribution of the group of the light rare earth elements (LREEs) consisting of La, Ce, Pr, Nd and Sm and the group of heavy rare earth elements (HREEs) consisting of Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb and Lu. The ratio of HREE to LREE was in the range of 0.36 to 0.41 for the fine size fractions SF1 to SF3. For the coarser size fractions SF4 and SF5 the ratio dropped to 0.26–0.27.

Fig. 2 shows the concentration of the REEs in the fly ash and the various size fractions normalized with the concentration of the REEs in the Upper Continental Crust (UCC) (Taylor and McLennan, 1995).

In Table 2 the enrichment or depletion of the different REEs in the various size fractions expressed by the concentration ratio $C_{SFx}/C_{Fly\ ash}$ is summarized. The calculated ratios ranged from 0.55 to 1.65. Thus, the enrichment and depletion of the REEs in the size fractions of the fly ash is significantly less than which was found for V, Ga and Ge (Table 3). In contrast to the consistent decrease of the concentration ratio with increasing particle size established for V, Lanzerstorfer (2018) the behavior of the REEs is different. Generally, all REEs were enriched in the finest size fraction SF1, although the enrichment was quite small for Nd and Ce. The highest enrichment was found for the HREEs (1.20–1.65). The total REE concentration was increased by 17%, which is considerably higher as reported by Vasconcellos and Ticianelli (2017) for a fine size fraction of similar size.

In SF2 all REEs were depleted. Thereby, the depletion was highest for the LREEs and lower for the HREEs. In SF3 the concentrations were quite close to the concentration in the original fly

Table 2
Enrichment of the various REEs in the different size fractions.

	SF1	SF2	SF3	SF4	SF5
La	1.19	0.58	0.90	1.35	0.77
Ce	1.09	0.60	0.90	1.21	0.78
Pr	1.30	0.64	0.95	1.20	0.77
Nd	1.05	0.60	0.84	1.19	0.74
Sm	1.15	0.70	1.00	1.23	0.77
Eu	1.23	0.74	1.03	1.03	0.68
Gd	1.21	0.71	1.00	1.00	0.67
Tb	1.36	0.82	1.14	1.05	0.68
Dy	1.20	0.73	0.93	1.00	0.65
Ho	1.34	0.79	1.00	0.97	0.66
Er	1.45	0.81	1.02	0.96	0.64
Tm	1.55	0.91	1.09	1.00	0.67
Yb	1.65	0.90	1.09	0.99	0.61
Lu	1.62	0.92	1.08	1.00	0.55
REE	1.17	0.65	0.92	1.17	0.74
LREE	1.11	0.61	0.89	1.23	0.77
HREE	1.34	0.78	1.01	0.99	0.65
Ranges of enrichment					
> 1.4		1.2-1.4		1.1-1.2	

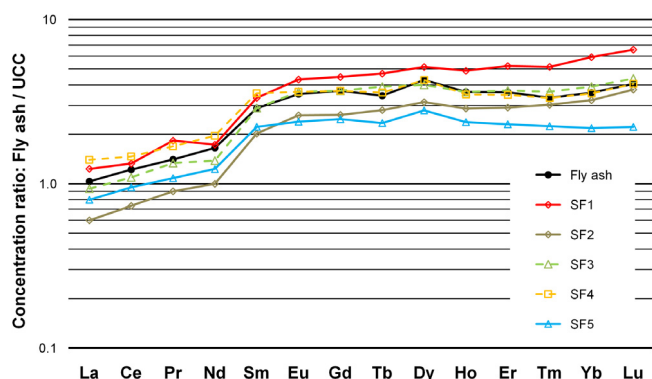


Fig. 2. Normalized concentrations of the REEs.

Table 3

Enrichment of V, Ga and Ge in the different size fractions (calculated from data published in Lanzerstorfer, 2018).

	SF1	SF2	SF3	SF4	SF5
V	2.44	1.53	0.97	0.77	0.45
Ga	2.20	1.73	1.22	0.85	0.39
Ge	2.23	1.92	1.31	0.62	0.15

ash. The LREEs were slightly depleted, while the concentrations of the HREEs were somewhat increased or unchanged. Exceptions were found for Dy, which was depleted and for Tb, which was enriched. In SF4 the LREEs were noticeably enriched (1.19–1.35). The concentrations of the HREEs were close to those in the fly ash. In the coarsest size fraction SF5 all REEs were depleted. However, for the LREEs the depletion was less compared to the depletion in SF2. For the HREEs the depletion in SF5 was most pronounced.

Besides enrichment, extractability of the REEs from the material obtained by fly ash pre-treatment is a key issue (Kolker et al., 2017). Thus, the leachability of the REEs in the different size fractions would have to be investigated in the future.

4. Conclusions

The concentration of REEs in coal combustion fly ash is size dependent. In contrast to the consistent size dependence of the concentration of, for example, V, Ga and Ge, for REEs the behavior is more complex.

In the finest size fraction SF1 all REEs were enriched, while in the coarsest size fraction SF5 and in SF2 all REEs were depleted. However, the degree of enrichment and depletion differed for the various REEs. The trends were similar for REEs of the same group.

Thus, air classification shows the potential for being used as a simple pre-processing step for the separation of REE-enriched fractions from the bulk of a coal fly ash. For the recovery of the HREEs, the finest size fraction would be appropriate, while for production of the LREEs, SF4 would be preferable. However, further studies using fly ash with higher REE concentrations would be required. Additionally, the leachability of the REEs in the different size fractions has to be investigated.

Acknowledgment

REE analysis by the Environment Agency Austria is gratefully acknowledged.

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