Petrographic and SEM/EDS Characterization of Bottom Ash Fractions Obtained Using Magnetic Separation Equipment

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Abstract: The combustion of coal generates coal combustion products (CCPs) such as boiler slag, fly ash and bottom ash that may be used in construction, manufacturing, environmental remediation, and other industries. However, these CCPs are may be in part or globally landfilled due to environmental and economic factors.
The bottom ash landfilled at Ceplea Valley (Gorj county, Romania) is a matter of serious environmental concern, and its utilization may be based on the fractionation of this material since its global utilization was not possible due to the Romanian regulation. Therefore, this study aims to demonstrate that the sequential utilization of magnetic separation equipment may provide bottom ash fractions with different types and volumes volume of Fe-bearing morphotypes.
For this purpose, one composite sample representative of the landfill was successively fractionated using a ferrite hand magnet, an Nd hand magnet, and a Sterns separator at 10500 Gauss and 18000 Gauss. The global sample and the magnetic fractions were then petrographically characterized via reflected light microscopy using oil immersion objectives and via scanning electron microscopy with X-ray microanalysis (SEM/EDS). As a result of the utilization of these equipment's the petrographic results show that the Fe-rich morphotypes were almost all collected in the first step of the magnetic separation, partially baked clay is more concentrated in the intermediated steps of the process, whereas the char and the quartz concentration was strongly increased in the remaining sample. The SEM/EDS results show that after the first magnetic separation process the aluminosilicate glass and partially baked clay morphotypes only contain residual amounts of Fe.
Therefore, a magnetic separation process via ferrite magnet only is enough to remove the majority of the iron-rich particles from the bottom ash landfilled at Ceplea Valley. However, a further separation step with a Nd magnet or a magnetic separator at 10-11 MGauss is needed to remove the particles with minor amounts of Fe.

Key-Words: petrography; ferrospheres; calcispheres; magnesiaspheres.

1 Introduction

Recycling coal combustion products is essential to overcome the challenge of residues landfilling and using resources from primary deposits, i.e., sustainably, with preservation.

Bottom ash is not widely used in Romania as fly ash. Therefore, the landfilling of this material continues to increase despite the possibility of its utilization in the construction industry. At Ceplea Valley (Gorj county, Romania) the bottom ash is being deposited for nearly four decades that causes a major environmental impact on the region due to dust during the dry season. Several studies, however, were already made in an attempt to find ways to recycle the bottom ash [1-6].

However, in these studies only global bottom ash samples were used whereas its fractionation was not considered. Therefore, the aim of the present study is to show that the fractionation of the bottom ash landfilled at Ceplea Valley via magnetic separation can provide a fraction rich in Fe-bearing particles, and fractions with high-char volumes and poor in Fe-bearing materials.

For this purpose, the bottom ash was separated using a sequence of magnetic equipment such as ferrite magnet, Nd magnet and a Stearns separator to obtain fractions with different types of Fe-bearing particles and to search for the most efficient magnet. Finally, the characterization of the samples was made using a combination of oil immersion reflected light microscopy and scanning electron microscopy with energy dispersive spectroscopy (SEM/EDS). The combination of the two techniques increased the value of their information since it is possible to quantify the volume percent of the morphotypes present in the materials, and also to assess semi quantitatively their chemical composition and morphology.

The paper structure includes the methodology section (sampling, description of the magnetic separation sequence and characteristics of the analytical techniques), a section where the petrography and the SEM/EDS results are shown, and, the conclusions.

2 Methodology

2.1 Sample

The bottom ash sample studied is a composite sample of 25 drill samples collected at the Turceni landfill in the Ceplea Valley (Romania; Fig. 1) where bottom ash from pulverized fuel conditions (1400 – 1600 °C) is being deposited for decades. The composite sample was obtained after splitting each of the 25 samples using a “Retsch” Rotary Sample Divider PT 100, and 25 splits (one per sample) were hand-mixed.

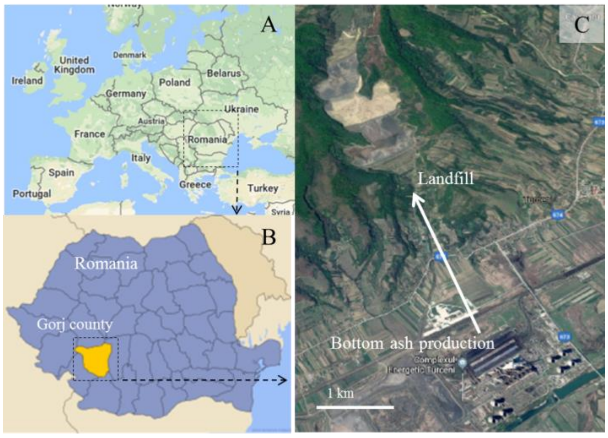

Fig. 1 A) Europe map; B) Romania map with Gorj county location; C) pathway of the bottom ash production to the landfill at Ceplea Valley.

2.2 Magnetic separation

Approximately 50 g of bulk bottom ash sample was magnetically separated using a ferrite hand magnet, a Nd hand magnet, and a Stearns Magnetic separator. These equipments were used in sequence and separated the bulk bottom ash samples in five fractions as shown in Figure 2. Finally, all samples were dried at 50 °C, and each fraction weighed.

2.3 Petrography

The bottom ash samples were prepared as polished blocks for petrographic analysis at the Geoscience Department (University of Porto) according to standard procedures [7], and analyzed using a Leica DM4000 microscope equipped with a Discus-Fossil system using ×20 and ×50 oil immersion objectives in non-polarized and polarized reflect white light. In some petrographic images it was used polarized reflected light with a retarder plate of 1ʎ.

Point count method was used to quantify the ash morphotypes following the fly ash classification proposed by Hower [8], and Valentim et al. [9]. The particles were counted (500 points for each sample) only when the cross-wire was superimposed on a particle, i.e., ignoring voids.

2.4 Scanning electron Microscopy and X-ray microanalysis - SEM/EDS

The samples were analysed by SEM/EDS at the Materials Centre of the University of Porto, Portugal (CEMUP) using an FEI Quanta 400 FEG-ESEM/EDAX Genesis X4M instrument. The SEM was operated at 15 kV in high-vacuum mode, manual aperture, and 4.5 beam spot size. To improve the analysis quality under high-vacuum conditions all samples were previously covered by sputtering a thin Au film coating. The samples were fixed to the mounting block by double sided adhesive carbon tape. The main phases observed in the micrographs at ×200 magnification under backscattered electron detection mode (BSE) were analyzed by X-ray microanalysis (EDS).

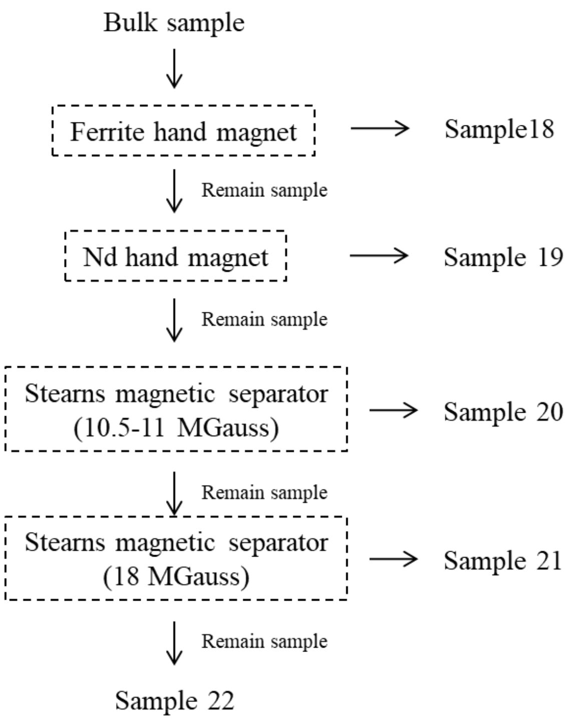

Fig. 2 Magnetic separation scheme.

3 Results

The morphotypes found in the composite bottom ash sample and in the respective magnetically separated fractions are particles of partially baked clay, aluminosilicate glassy spheres, char (irregular, angular and rounded in shape), ferrospheres, oxidised Fe, (Ca, Mg, Fe)-spheres (calcispheres, magnesiacalcispheres, and magnesiaspheres; other Ca particles, and quartz (Fig. 3) [9].

Partially baked clay and aluminosilicate glassy spheres are found in major amounts (>10 Vol. %) in all samples, while (Ca, Mg, Fe)-spheres are found in major amounts only in the MS2 fraction, and char and quartz in the MS5 fraction. The other morphotypes, and (Ca, Mg, Fe)-spheres (except in sample MS), char and quartz (except sample in MS5) occur in minor amounts (<10 Vol.%) or are residual (Fig. 4).

The morphotypes bearing high concentrations of Fe, which includes ferrospheres, oxidized Fe, (Ca, Mg, Fe)-spheres, and some aluminosilicate glassy spheres were mainly removed in the first magnetic separation stage using the Fe hand magnet (Fig. 5).

Therefore, sample MS1 shows the highest volumes % of aluminosilicate glass and of (Ca, Mg, Fe)-spheres, and the lowest volumes % of partially baked clay, char and quartz (Fig. 4).

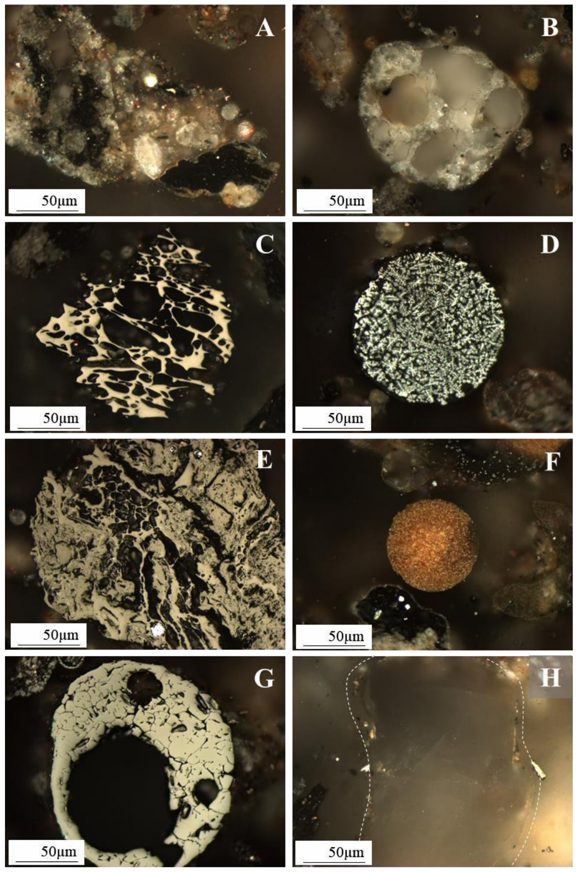

Fig. 3 Bottom ash morphotypes (reflected white light, + polars; ×500) A) partially baked clay (sample MS1; + polars); B) glass (sample MS3); C) char (sample MS5); D) ferrosphere (MS1); E) oxidized Fe (sample MS2); F) calcisphere (sample MS1; G) magnesiasphere (sample MS1); H) quartz (dashed lines limiting; sample MS5).

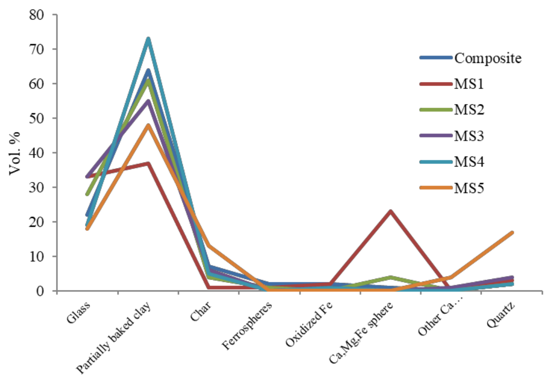

Fig. 4 Petrographic results: distribution of the different morphotypes in the magnetic and non-magnetic fractions.

By increasing the magnetic influx density using the Nd hand magnet, and after the Stearns separator at 10500 and 18000 Gauss particles with less Fe are also collected (Fig. 6, and Fig. 7A-7D), and for that reason samples MS2, MS3 and MS4 show a decreasing volume % of aluminosilicate glass, and an increasing volume of char (Fig. 4). Finally, the remaining material (sample MS5) in composed by a major volume of quartz, and the lowest volumes % of aluminosilicate glass and partially baked clay, which are composed of materials with residual Fe (Fig. 4; Fig. 7E-7H).

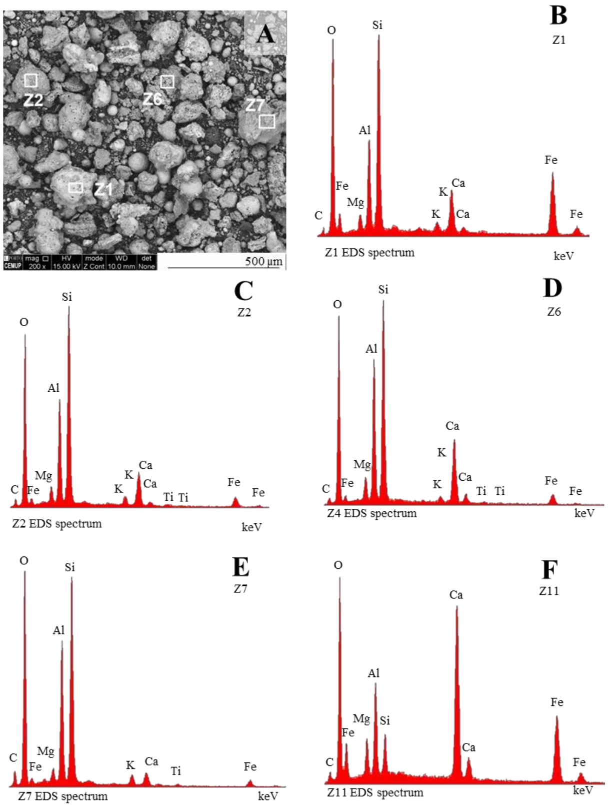

Fig. 5 SEM/EDS analysis of the bottom ash separated using a ferrite hand magnet (Sample MS1; backscattered electron mode).

4 Conclusions

The composite sample of the bottom ash landfilled at Ceplea Valley is composed by more than 80 vol.% of aluminosilicate glass and partially baked clay, and, less than 10 vol.% of char and Fe-bearing particles. However, the Fe-bearing particles are composed of ferrospheres and a diversity of other particles with residual to minor amounts of Fe.

The first step of the magnetic separation of bottom ash deposited at Ceplea Valley (Gorj county, Romania), i.e., the separation via an ferrite magnet was enough to separate ferrospheres, Fe oxide relics, calcispheres, magnesiacalcispheres, and magnesiaspheres. However, the separation of particles with residual Fe was only effective via the Nd magnet or the Stearns magnetic separator at 10-11 MGauss. The magnetic separation above this value only separates a residual amount of sample and the remaining sample is mainly composed of char and SiO_2 particles.

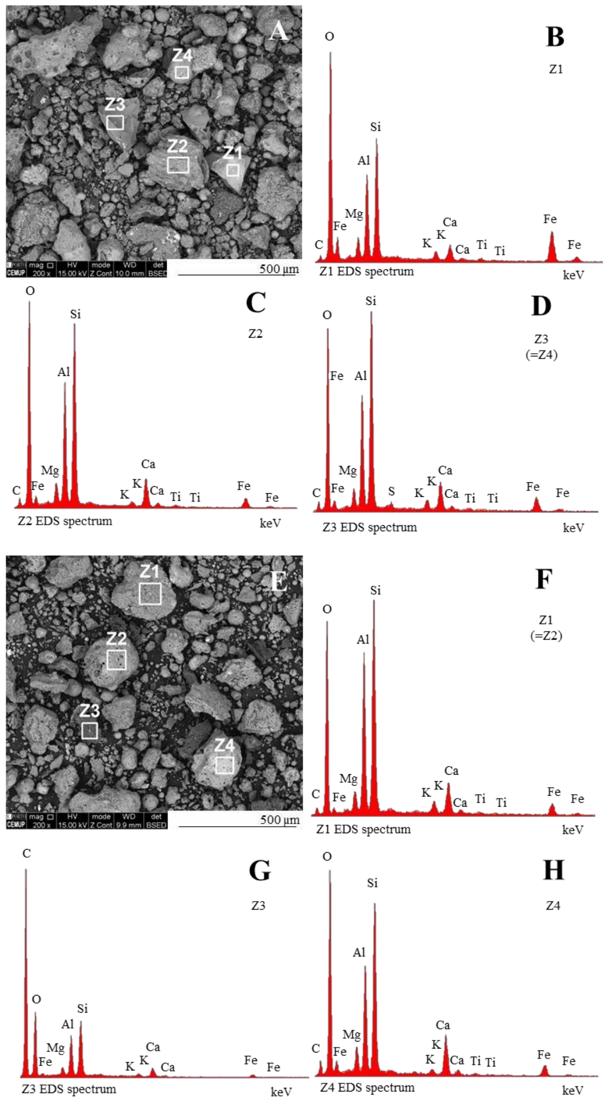

Fig. 6 SEM/EDS analysis of the bottom ash (backscattered electron mode): A to D) fraction separated using a Nd hand magnet (Sample MS2); E to H) fraction separated using Stearns magnetic separator at 10500 Gauss (Sample MS3).

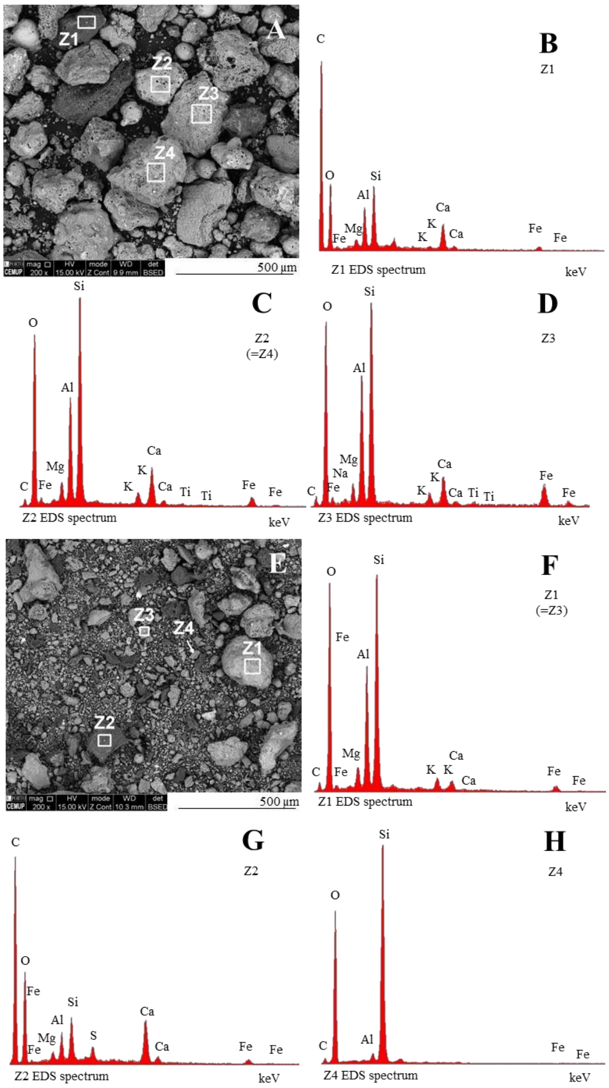

Fig. 7 SEM/EDS analysis of the bottom ash (backscattered electron mode): A to D) fraction separated using Stearns magnetic separator at 18000 Gauss (Sample MS4); E to H) non-magnetic fraction (Sample MS5).

Acknowledgments

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