Gravity and electrostatic separation of unburned coal from a selected fly ash

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Keywords

fly ash, unburned coal, gravity separation, electrostatic separation, mineral inclusions

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Abstract

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1. Introduction

The Polish energy industry is based primarily on commercial power plants. In 2021, the production volume at these facilities amounted to 154,599 GWh. The most important fuel for electricity generation in 2021 was hard coal, with a 53% share, and lignite, with a 26% share. Renewable energy sources produced 18,984 GWh, and their share increased to 11%. By-products like fly ash and furnace slag (CBP) are generated during the coal gasification and combustion process in a power plant or combined heat and power plant. CBP has been increasingly seen not as a waste but as valuable raw material, as its industrial use in cement is possible if the fly ash contains a low content of unburned coal (UC) [1−5]. The unburned coal content in CBP is an indicator of the inefficiency of the combustion process and is most often an obstacle to its economic use [6,7].

UC, which has been separated from fly ash and suitably enriched, has significant potential as a high-value product with many possible applications [2,3,8−10]. Although in some cases, unburned coal particles or chars in fly ashes could adsorb hazardous volatile elements (e.g., Hg) [11−14], the unburned coal grains, due to good sorption properties, may be used to remove B, As, Cu, Pb, Zn, Mn, Cr and Ni from wastewater, or to capture CO_2, SO_2 or NO_x from exhaust gases [2,9,11−14]. In the simplest case, the separated coal may be used for re-combustion or be a substitute for natural graphite-bearing raw materials [2,14−16]. Recovering unburned coal and using it as a substitute for natural graphite in "green energy" technologies is a new and important research direction.

In the literature, there is a series of reviews on the origin [2,17,50], the purification [3,18−25], the characteristics [7,26−31] and the use of char [2,3,32]. A number of wet and dry methods have been developed for the recovery of unburned carbon from coal fly ash, and there are various options for the beneficiation of coal fly ash to reduce the LOI value. Most of the current methods for separating unburned carbon from fly ash use sieving, gravity separation, electrostatic separation, froth flotation, and oil agglomeration [2,3,16,18−25,33,51]. The

above-mentioned methods have their advantages and disadvantages, and their possible use must take into account the properties of fly ash as well as the requirements for separation products. This applies especially to the purity level of the products and the carbon recovery. Froth flotation and oil agglomeration methods often cannot be used due to the harmful adsorption of hydrocarbons. The screening method is usually inefficient and cumbersome due to the small size of the separated grains. In such cases, gravity or electrostatic methods and their combination are available. Gravity methods can be dry or wet, and electrostatic methods – are only dry. Due to the need to dewater the separation products, dry methods are more economical.

The paper presents the research results on the possibility of separating unburned coal from fly ash using gravity methods and the electrostatic method. One two-step wet gravitational separation method and two dry gravitational separation methods were applied. It has been found that despite very fine ash grains, the quality and quantity of separation products are significantly dependent on the separation method used and the separated grains' qualitative characteristics.

2. Materials and methods

The material used for testing was fly ash from pulverized coal boilers taken from a power plant located in southern Poland. The power plant utilises a blend of energy coal, classified according to ISO 11760 [34] as subbituminous coals. The coal contained 20.7% ash, and its calorific value was 21,300 kJ/kg (dry basis). The ash sample was collected and prepared in accordance with EN 14899 [35].

The chemical and phase composition of the fly ash was determined on the basis of tests carried out using:

- X-ray fluorescence spectrometer (XRF)
- X-ray diffractometer (XRD)
- scanning electron microscope-energy dispersive X-ray analysis (SEM/EDS)

"Main chemical component and trace element content by wavelength dispersive X-ray fluorescence spectroscopy (WDXRF) using a ZSX PRIMUS II analyser (Rigaku, Tokyo, Japan) equipped with a 4 kW X-ray Rh tube; the samples were prepared by borate fusion (1 g sample: 9 g flux), the beads were obtained by melting the resulting mixture at a temperature of 1050 C." [48].

"Mineral composition by powder X-ray diffraction (XRD) in Bragg–Brentano geometry using a D8 DISCOVER diffractometer (Bruker, Billerica, MA, USA) with a CuKalamp, Ni filter and a LYN-XEYE_XE detector working under the following conditions: Materials 2022, 15, 3023 4 of 24voltage – 40 kV, 2theta angle step size–0.01, time–1 s by step, 2theta angle range 4–69°; sample rotation–10°/min; the composition was calculated on the basis of patterns licensed in PDF-4+ 2021 RDB ICDD (International Centre for Diffraction Data) and databases: ICSD (Inorganic Crystal Structure Database) and NIST (National Institute of Standard and Technology); the following programs were used for registration and diagnostics: DIFFRAC v.4.2 and TOPAS v.4.2. Bruker AXS; the quantitative phase composition was determined by the Rietveld method" [48].

The ash characteristics, determined using the SEM scanning electron microscope, included the determination of the morphology and size of grains and the elemental composition based on the observation of grain surface and X-ray microanalysis. The SEM/EDS analysis was performed with the help of the Hitachi SEM SU3500 variable pressure scanning electron microscope, using an X-ray spectrometer with energy dispersion of the EDD Ultra Dry from Thermo Scientific NORAN System 7. The BSE (Backscattered Electron) detector was used for analysis because of its ability to illustrate the contrast in the composition of multiphase samples.

The size distribution of ash was determined using a wet sieving method according to ISO 1953 [36]. The measure of the amount of unburned coal was a loss on ignition (LOI) [37]. The amount of unburned coal (LOI) was determined in accordance with the procedure described in EN 196–2 [38] at 900 °C.

The float and sink analysis was performed according to standard ISO 7936 [39]. Organic liquids with densities of 1.4–2.0 g/cm^3 were used for the tests for every 0.1 g/cm^3.

All analyzes were performed at the Department of Environmental Monitoring of the Central Mining Institute.

Ash samples with a grain size above 100 μm were used to study the recovery of unburned coal from ash. The distribution of the unburned coal content, in relation to the grain size in the total ash sample (Fig. 1) and the technical requirements of some separators, was the basis for this selection.

The unburned coal extraction with the wet gravitational method was carried out in two stages. In the first stage, cenospheres were separated which float on the surface of the suspension. In the second stage, the separation technique in a rising water current was used. Separation in the rising water stream was carried out at a test stand depicted in
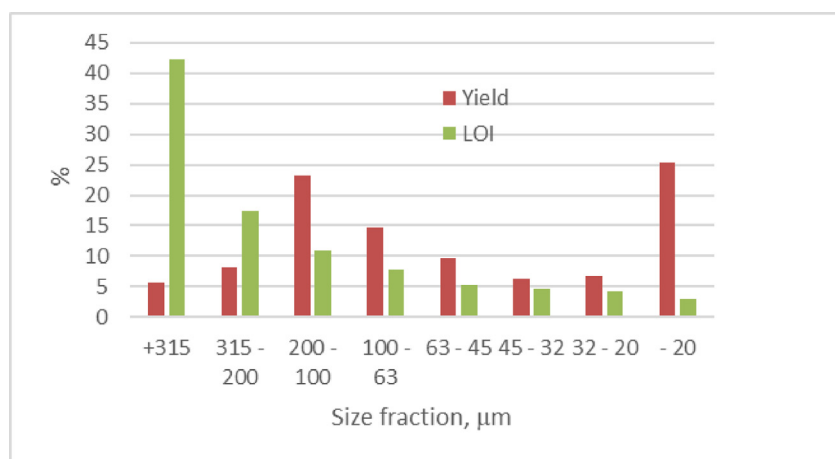
Fig. 1. The grain composition of the ash sample and unburned coal content (LOI) in the function of the size of fly ash particles.
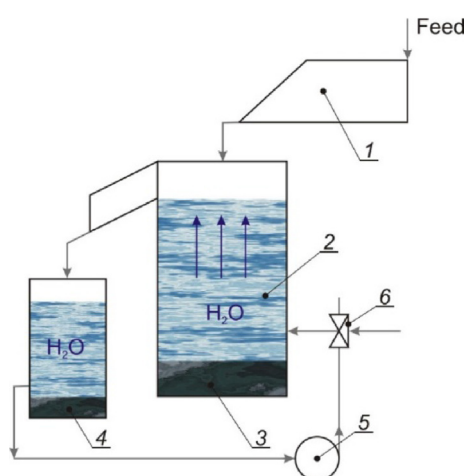

Fig. 2. Test stand diagram for separation in a rising water stream 1 — feeder, 2 — separator, 3 — sediment (bottom product), 4 — overflow (top product), 5 — pump, 6 — three-vay valve [49].

Fig. 2. "The water flow intensity was selected experimentally, so as to obtain the most optimal separation possible. The water flow intensity was within the range of 1—3 dm^3/min" [49].

Two different gravitational separation techniques were also applied using the dry method. One of them was the traditional fluid bed method, and the other was a fluid bed method with vibrations and a classifier.

The separation fluid bed method was carried out in a laboratory fluid bed separator, which was a properly instrumented quartz tube with a diameter of 100 mm and a height of 500 mm (Fig. 3). Quartz sand with a grain size of 300—385 μm was used as a fluid-forming agent. The separation ash was gradually dosed in portions to ensure the required pressure drops over the deposit. After the
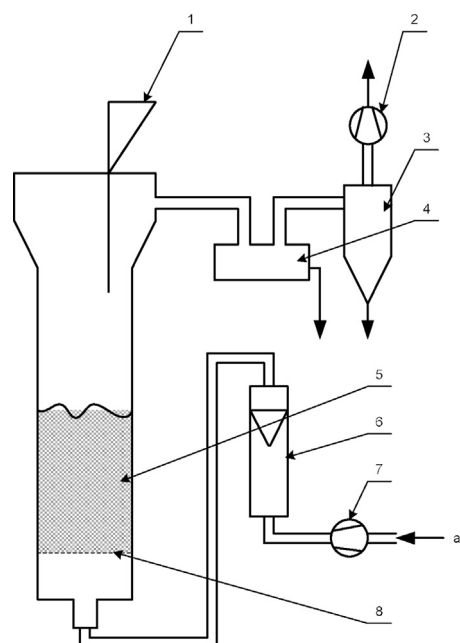
Fig. 3. Scheme of the fluid bed separator: 1 — ash dispenser, 2 — exhaust fan, 3 — cyclone, 4 — settlement chamber, 5 — fluidized bed, 6 — flow meter, 7 — air fan, 8 — distributor.

end of the ash dosing and setting the pressure over the deposit at a stable level, the air flow velocity was reduced in order to arrange the grains in characteristic layers. In the upper layer of the deposit, unburned coal grains formed, and in the lower layer, ash grains and a layer of quartz sand grains formed.

The fluid bed separation method with vibrations was carried out in a device consisting of a separator with a flat nozzle bottom and a classifier with vertical airflow. This is a combination of the classification method with vertical airflow and elutriation.

Furthermore, vibrations were used to loosen the grains at the fluid deposit forming stage.

Electrostatic enrichment tests were carried out using the Boxmag-Rapid Limited device from England. The main variable was the voltage between the electrodes regulated within 10−25 kV. Increasing the voltage between the electrodes to above 25 kV caused a spark and prevented separation.

The separation products of individual tests were dried (in the case of wet methods), the losses on ignition (LOI) were determined as a measure of unburned coal content, and a mass balance was prepared. The recovery of unburned coal in the separation process products was calculated from the dependence:

$$\varepsilon = \frac{\beta}{\alpha}\gamma \qquad (1)$$

where: ε − unburned coal recovery,

    β − loss on ignition value in the product,
    α − loss on ignition value in the enrichment feed,
    γ − product weight yield.

3. Results and discussion

The results of the mineral analysis of ash compositions are presented in Table 1.

The dominant ash component is the amorphous − (Am) phase. The content during this phase was 75.46%. The second quantitatively mineral component is (Mu) mullite −with a content of 13.90%. The last significant quantitative component is (Q) quartz, whose content is 9.04%. All three phases (Am, Mu and Q) account for over 90% of the mineral composition of ash, which indicates such: anhydrite, hematite, magnetite, maghemite and periclase.

The results of the chemical analysis are presented in Table 2.

The dominant components of the ash are SiO_2 and Al_2O_3 (Table 2). The share of SiO_2 is 51.68%, while Al_2O_3 is 22.19%. Together, these two components account for over 70% of the total share. An important ash element is unburned coal (UC), whose content is 8.93%. The chemical components which are present in amounts ranging from approximately 1%−6% are: Fe_2O_3 (6.16%), CaO (3.36%), K_2O (2.7%), MgO (2.56%), Na_2O (1.05%) and TiO_2 (0.99). The average content of other remaining oxides (Mn_3O_4, P_2O_5 and SO_3) does not exceed 0.50%. It should be noted that the above-mentioned mineralogical and chemical characteristics are typical for fly ashes from subbituminous coals. They are produced in coal power plants [2,31,33,40−42].

The yield in each ash fraction and the respective content of UC is shown in Fig. 1. The content of UC for the whole sample was 9,4%. A typical picture of the ash grains smaller than 20 μm in size, magnified 2000 × under the electron microscope, is shown in Fig. 4.

Visible numerous light gry balls are microspheres. In the background of the microspheres are visible irregular dark coal grains. The point analysis spots 1 and 2 in Fig. 4 of the chemical composition results in their main ingredient being carbon. Its content, calculated as oxide, oscillates in the range of 88−92%. The main components of the cenospheres are silicon and aluminium compounds [43−45]. For example, in spot 3 of Fig. 4, the Al content in terms of Al_2O_3 reaches 33.8%, and the content of Si in terms of SiO_2 is 25.3%. The carbon content in cenospheres is low − below 20%, calculated in CO equivalent (spot 3). Grey grains (spots 4 and 5) are probably mineral due to its low carbon content (less than 8%) and a very high content of Ca (about 46.4%), Sio_2 (about 53.2), and P (about 38.1%). In the grain classes of 20−32 μm, 32−45 μm and 45−63 μm, the losses of UC amount to 5% (Fig. 1). From the 100−63 μm class, the content of UC increases, reaching more than 40% in the thickest class. This shows that the content of UC in the ash increases with the increasing grain size. The grain composition of the ash and the increase in the content of unburned carbon, along with the increase in ash graining, are consistent with the data given in the literature [2,33,40−42].

Table 1. Mineral composition of fly ash.

Mineral composition	Q	Mu	Ah	He	Mgt	Mgh	Pe	Am	Sum
Weight yield, %	9.04	13.90	0.20	0.10	0.10	0.60	0.60	75.46	100.00

Table 2. The main chemical components of fly ash.

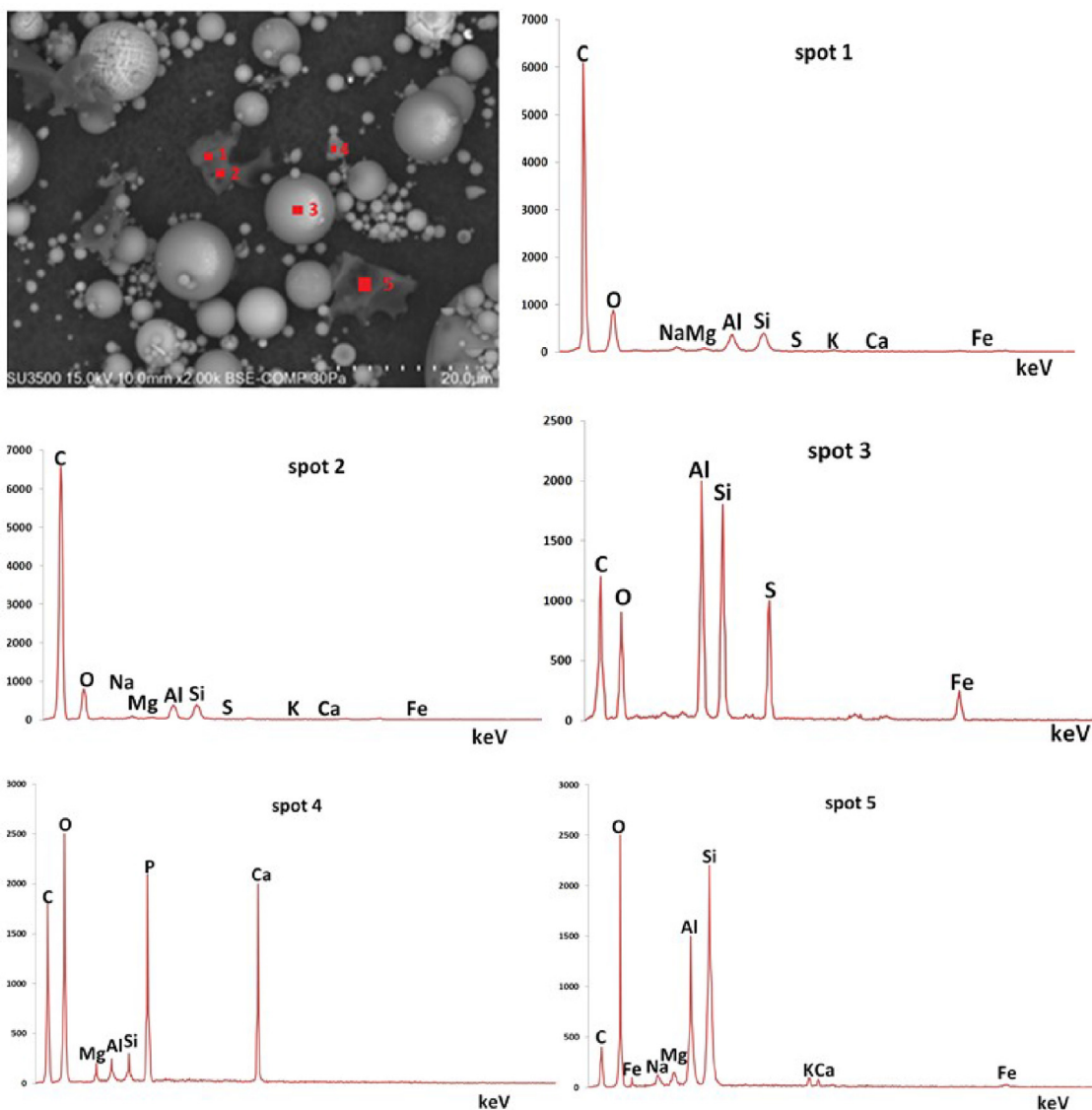| Chemical components	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	Mn_3O_4	MgO	CaO	Na_2O	K_2O	P_2O_5	SO_3	LOI	Sum
Weight yield, %	51.68	0.99	22.19	6.16	0.11	2.56	3.36	1.05	2.70	0.17	0.10	8.93	100.00

Fig. 4. The SEM-BSE image of ash grains of the class below 20 μm in 2000 × magnification and SEM-EDX spectra of the selected different grains.

Table 3. Float and sink analysis.

Fraction, g/cm^3	Yield, %	Sum of the yield, %	LOI in fraction, %	Cumulatively LOI, %
−1.4	17.2	17.2	2.69	2.69
1.4−1.5	9.4	26.6	18.44	8.25
1.5−1.6	6.9	33.5	16.15	9.87
1.6−1.7	4.9	38.4	15.11	10.54
1.7−1.8	7.2	45.6	30.06	13.64
1.8−1.9	16.7	62.3	39.05	20.45
1.9−2.0	7.4	69.7	31.75	21.65
+2.0	30.3	100	3.57	16.16
Sum	100		16.16	

The float and sink analysis of the grain class +100 μm ash are presented in Table 3.

The float and sink analysis indicated that the two extreme fractions, i.e. the lightest (−1.4 g/cm^3) and the heaviest (+2.0 g/cm^3), together constituted approximately 47.5%. The lightest fraction was virtually pure cenosphere, found floating on the water's surface. The unburned coal content in the lightest and heaviest fractions was 2.69% and 3.57%, respectively. The other fractions' yield is 4.9−16.7%,

and the contents of unburned coal are in the range of 15.11−39.05%. These characteristics indicate that the emission of unburned coal using gravity methods is very difficult because the undesirable fractions in the concentrate are the two extreme fractions, i.e. −1.4 and + 2.0 g/cm^3. These characteristics require two-stage gravity enrichment, using the wet method. In the first stage, the lightest density fraction, i.e. cenospheres floating on the surface of the suspension, should be separated. In the second stage, grains with a density above 2.0 g/cm^3 should be separated.

This is also confirmed by the results of calculations of unburned coal in concentrate and wastes (Table 3). Separating the lightest fraction will

increase the UC to 18.96%. On the other hand, separating only the heaviest fraction will result in the UC in the remaining concentrate equaling 21.65%. For the aforementioned reasons, unburned coal removal by wet gravity was carried out in two stages. The results of the selected unburned coal exuding tests using specific methods are presented in Table 4.

The content of unburned coal in concentrates separated by individual methods varies considerably in the range of 30.30−62.00%. The product with the highest coal content is a concentrate from fluidized separation with vibration, and the smallest concentrate is from two-stage wet separation. The second parameter characterizing the separated coal concentrates is their yield. This fluctuates widely between 2.4 and 17.8%. The largest yield of coal concentrate was obtained by electrostatic separation and the smallest by the fluid bed separation method. Using the above parameters and the coal content in the distribution feed, the coal recovery in the individual separation methods was calculated. The highest recovery, equal to 50.5%, was obtained by the electrostatic method and the smallest, 8.3%, by the fluid bed method. In the other two distribution methods, coal recovery is similar and amounts to 24.2% in the wet method and 21.5% in the fluid

Table 4. Characteristics of unburned coal obtained in individual separation methods.

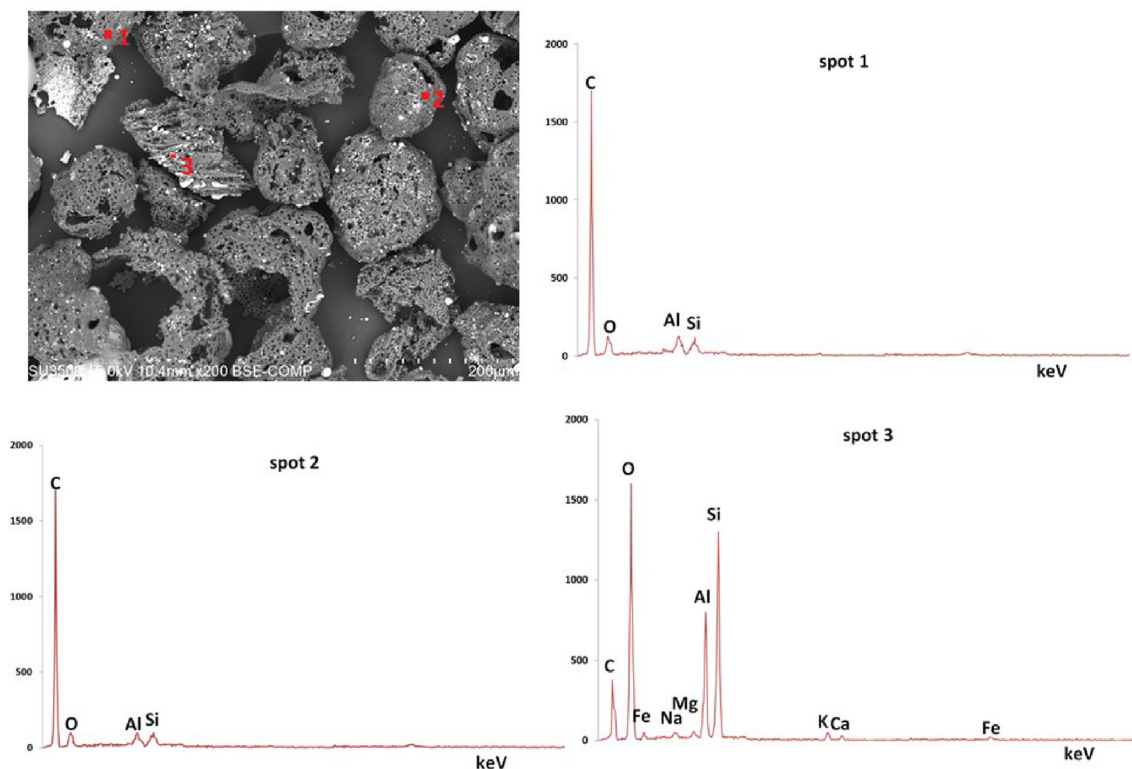| Separation methods	Value of loss ignition of the product	Mass yield of the product	Carbon recovery
	β, %	γ, %	ε, %
Separation in a rising water stream	30.30	12.9	24.2
Fluid bed separation	55.95	2.4	8.3
Fluid bed separation with vibration	62.00	5.6	21.5
Electrostatic separation	45.90	17.8	50.5

Fig. 5. The SEM-BSE image of seeds of one of the concentrates and SEM-EDX spectra of selected seeds.

bed separation with vibration. Similar conclusions were also published in the paper [37].

Figure 5 presents an image from an electron microscope of one of the concentrates, including the point analyses of the chemical composition of selected grains. For the other concentrates, the grain views were very similar. The vast majority of grains have an irregular shape, as well as grains of an elongated shape and grains in the shape of plates or stripes. The very large porosity of dark coalaceous grains, with bright shiny areas that constitute mineral inclusions, is noteworthy. The content of elemental coal in spots 1 and 2, designated as coal monoxide, is over 92%. The elemental coal content in bright shiny areas is much lower and amounts to around 10% (spot 3). This is a typical picture of the coal grain with mineral inclusions.

As shown in Fig. 5, the dimensions of the mineral inclusions are smaller than 20 μm. In the tested case, increasing UC in the separation products is difficult without prior grain grinding. The crushing can release coal from the mineral inclusions. Similar observations come from the work [46,47]. However, crushing the grains will reduce their dimensions, making it more difficult to separate them.

4. Conclusions

The possibility of separating unburned coal from selected fly ash was evaluated in this paper. Recovering unburned coal with one wet method and three dry methods was proposed. From fly ash with a grain size above 100 μm and unburned coal content of 16.16%, it was possible to obtain a concentrate containing 30.3—62% of unburned coal. The highest amount of unburned coal in the concentrate, equal to 62%, was obtained using a fluid bed method with separation. The largest yield of concentrate, equal to 17.8%, was obtained using the electrostatic method. Further cleaning of the concentrate is very difficult without crushing the grains in order to release the carbon grains from mineral inclusions.

Ethical statement

The authors state that the research was conducted according to ethical standards.

Funding body

Conflict of interest

The authors declare no conflict of interest.

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