Morphology and chemical composition of mineral matter present in fly ashes of bituminous coal and lignite

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Abstract

The changing properties of energy waste force us to look for alternative directions of their use. The aim of the research presented in this paper was to indicate the possibilities of managing fly ashes from bituminous coal and lignite based on the observation of individual particles of these ashes. Innovative research techniques were used, among others: SEM with EDS detector and Image Analysis. Microscopic observations have shown that fly ashes are a heterogeneous material composed of particles occurring in three basic morphological forms: spherical, irregular and fine-detritic. A high proportion of spherical particles was found in bituminous coal ashes (rich in SiO_2 and Al_2O_3), which gives them high pozzolanic activity. This kind of morphology and chemical composition make these ashes a potential material for zeolites synthesis. The amorphous phase of ash from lignite coal has aluminium–silicate–calcium chemical composition, which is what determines pozzolanic and hydraulic properties of these ashes. Magnetic separation of iron-rich particles may be a way to manage the researched ashes. One of the valuable components of hard coal ash are microspheres, which are characterized by high variability in chemical composition, so further utilization will require prior refining. The main component of irregular particles found in ashes is unburned coal and, to a lesser extent, a mineral substance. The separation of all useful components from the tested ashes before their further management will allow the full use of their raw material potential.

Keywords Ferrospinel · Fly ash · Mineral matter · Microspheres · Morphology

Abbreviations

AR	Aspect ratio
EDS	Energy dispersive spectroscopy
LOI	Loss on ignition
SEM	Scanning electron microscope
WB	WB Fly ash from lignite combustion
WK	Fly ash from bituminous coal combustion

Introduction

Fly ashes, created as a result of burning coal in power plants, have some specific properties, which that allow their various uses. Attempts of using these side products of burning in diverse ways have been made for years and

are ecologically beneficial as well as economically. One of the most significant recipients of fly ashes is building materials industry, where they are used as mineral additive in the production of cement, multi-component binders or concrete. (Galos, Uliasz-Bocheńczyk 2005; Giergiczny 2013; Uliasz-Bocheńczyk et al. 2015). To a slightly lesser extent, ashes are used in the production of special ceramics or thermal insulation materials (Erol et al. 2008b; Stoch 2015). Expansion of the road network and highways created greater opportunities for the use of ashes in engineering and road works (Gawlicki, Wons 2012; Swamy, Das 2012; Wong et al. 2020). Due to the high content of aluminium and silicon, fly ash can also be a valuable raw material for the synthesis of zeolites. The first attempts in this area begun already in the 1980s (Holler, Wirsching 1985). In the next years, the methods of synthesis were gradually developed and improved. (Querol et al. 1997; Ojha et al. 2004; Yao et al. 2013). The synthesis of zeolites from fly ash may be an alternative method of this waste management, which is all the more valuable as zeolites are commonly used in various fields. They are mainly used in the chemical industry. An interesting fact is that zeolites

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can be used to stiffen bitumen (Tausif et al. 2020) or to remove metals from sewage (Winczaszek 2006).

Also attempts to recover metals from fly ash, mainly iron, were made (Hycnar 1987; Michaliková et al. 1994; Ratajczak, Piestrzyński 2000). Recent research indicates that high-temperature ashes can also be a promising source of rare earth elements (Blissett et al. 2014; Jarosiński 2016; Adamczyk et al. 2018).

Due to the different fuel composition as well as the different combustion and cooling temperatures, ash grains show a high degree of morphological differentiation, phase and chemical composition (Apostolidou, Georgakopoulos 2018). Therefore, choosing the right direction for using fly ash forces the need to broaden knowledge about their physico-chemical properties. One of the things that researchers' focus on are morphological forms of ash particles. These data can be used e.g. to predict the mechanical and physical properties of a material. Retrieving all useful fly ash components can also improve the economics of their use.

One of the first characteristics of fly ash particles is given in the paper (Ramsden, Shibaoka 1982). Using optical and electron microscopes and research in micro-areas, the authors distinguished seven categories of particles: detrital minerals (mainly quartz), irregular-spongy particles originating from melted clay minerals, colourless glass in form of irregular particles or cenospheres, solid in various colours, depending on chemical composition, dendritic particles of iron oxides (mostly magnetite and hematite) and unburnt coal particles. On the other hand, Fisher et al. (1978) distinguished as many as eleven types of particles in ashes, differentiating them on the basis of morphology, porosity, as well as their transparency. Matsunaga et al. (2002) studied the morphology of two types of particles (solid and hollow) using a scanning electron microscope. The authors noticed that the morphology of solid particles depends on their size. Smaller particles are more spherical in their shape in contrast to the bigger particles. A similar relation was noticed for microspheres, particles filled with gases. (Strzałkowska, Stanienda-Pilecki 2018). The chemical composition of microsphere walls shows correlation with their size (Haustein, Quant 2011; Ngu et al. 2007; Strzałkowska, Adamczyk 2019).

The shape of ferrosphenes present in lignite coal ashes was close to a perfect sphere, with dendritic or skeletal structure (Sokol et al. 2002). According to the authors, those dendritic and skeletal forms of ferrosphenes are a result of their crystallization in conditions of sudden cooling. As the research conducted by Sharonovaa et al. (2015) indicates, the main phase of ferrosphenes is magnetite Fe_3O_4 (17–47 wt. %). Therefore, these particles are characterized by excellent magnetic properties which facilitate their separation from fly ash.

The main component of irregular particles found in ashes is unburnt coal and, to a lesser extent, a mineral matter

(Chengfeng et al. 2005). A numerous classifications of unburnt organic matter based on porosity, wall thickness and iso / anisotropy can be found in the literature (Lester et al. 2010; Misz 2002), therefore this work applies only to mineral matter present in ashes.

The changing properties of waste from power plants force us to look for new directions for their use. Numerous studies have shown that both the shape and particle size of the ash directly affect their further utilization. When using ashes as an additive to concrete, the spherical shape of the particles brings many benefits, while their high porosity is undesirable, as it increases the water demand of ash. Therefore, using fly ash fully in many applications can be difficult. Continuing the research goal aimed at further understanding of the morphology and chemical composition of individual fly ash particles, this paper presents the results of comparative studies of mineral matter present in bituminous and lignite coal fly ashes. The use of innovative techniques of single particle analysis will help understand the differences in their physico-chemical properties. This will allow a wider and more appropriate use of these fly ashes.

Materials and methods

To achieve the purpose of this work, two samples: fly ash from lignite combustion and fly ash from bituminous coal combustion with the code 10 01 02, have been tested (Rozporządzenie Ministra Środowiska 2014). The samples were tested for

Fig. 1 Graphical representation of the research methodology

particle size and shape using a QICPIC sensor from Sympatec GmbH. Morphology of the particles was observed using microscope for transmitted and reflected light Axioskop and Axioplan from Zeiss at magnification of 100, 200 i 500x, and high resolution scanning electron microscope (SEM) JSM 7200F from JEOL equipped with EDS Octane Elite Super from EDAX. Using EDS detector in randomly selected areas from all over the surface of the sample, microanalysis of the chemical composition of the particles was performed. Accelerating voltage of 15 kV was used in the research. Figure 1 shows graphical representation of the research methods.

Results and discussion

Granulometric composition

A complete assessment of the particle size distribution of the tested ash samples was obtained using the QICPIC/R sensor,

whose optical system enables the analysis of dynamically changing images. The signals received were used to calculate the particle size distribution, as well as to determine the frequency of appearance of selected grain classes (Fig. 2). It was experimentally found that lignite coal ash was characterized by a smaller grain size, the content of grains smaller than 45 μm was 63,65%, while in the ash from bituminous coal it was 55%.

The shape of the curves for lignite coal ash indicates a unimodal distribution of grain size with a wide maximum falling in the range of 12–65 μm , and for bituminous coal ash with a bimodal nature of the distribution, with a narrow maximum in the range of 12–15 μm and wide maximum of 90–150 μm . Bigger particle size of ash from bituminous coal can be associated with a greater proportion of unburnt coal in them (which cumulates mostly in bigger grain classes), which is indicated by higher loss on ignition for these ashes (LOI for hard coal ashes – 5.83%; for lignite coal ashes – 3.5%). This parameter reflects, among others, the content of unburnt coal in fly ash.

Fig. 2 Particle size distribution for the examined fly ashes, code 10 01 02 – cumulative Q and density q **a** from lignite coal **b** from bituminous coal

Morphology of particles

The QICPIC / R measuring sensor was also used to determine the AR (Aspect Ratio) of the tested ash particles. The shape of cumulative curves (Fig. 3) illustrating the content of grains with a specific AR (Aspect Ratio) in the studied ash indicates that hard coal ash particles are more spherical. 13.27% of the particles of this ash have an AR value higher than 0.9 while for lignite coal it is only 5.54%. AR equal to one usually represents maximal symmetry, such as a circle or sphere.

Because ash particles can have very different shapes (not only spherical) which is a result of, among others, differences in the process of combustion as well as the type of coal burnt. Therefore, microscopic examinations of transformed mineral matter were conducted to determine their morphology.

Microscopic observations of both types of fly ashes allowed to distinguish the following morphological forms of transformed mineral matter (Fig. 4):

- solid spherical,
- irregular solid,
- irregular porous,
- fine-detrital.

In hard coal ashes, the transformed inorganic matter is mainly present in the form of spherical particles. These

Fig. 4 Distinguished morphological forms of particles in examined ashes

particles are created as a result of combustion of mineral matter from coal, its melting and then solidification (Fig. 5). High surface tension leads to creation of perfectly spherical forms.

Fig. 3 Cumulative curves picturing the content of grains in the examines ashes with a certain aspect ratio WB – lignite coal ash, WK – bituminous coal ash

Fig. 5 Spherical particles in hard coal ash. Microscopic analysis in reflected light, magnification 500x, 1 N

This form is extremely rare in lignite coal ashes. The range of sizes of these particles is very wide, it varies from a few to several dozen micrometres. Most of the particles are massive and their colour is dependent on chemical composition (Fig. 6).

In bituminous coal ashes, these spherical particles are mostly made of SiO_2 and Al_2O_3 with addition of sodium and potassium (Fig. 7a). Sodium oxide and potassium oxide are especially present in particles of ash whose size is below $45\ \mu\text{m}$, which is probably due to their lower melting temperature and condensation on the smallest ash particles. High content of SiO_2 and Al_2O_3 in glass phase conditions pozzolanic reaction. In result of this reaction, hydrated silicates and calcium aluminosilicates, which increase in the strength

of the construction material. However, pozzolanic activity depends not only on the chemical composition, but also on the shape and size of the ash particles. Small glass spheres improve the fluidity and workability of fresh concrete (Bastian 1980).

Research shows that amorphous aluminosilicate can also be useful in zeolites synthesis from ash, even more so, that its chemical composition closely resembles that of rocks from which natural zeolites crystallize (Czarna et al. 2016). The decisive factor influencing the formation of various types of zeolites is undoubtedly presence of silicon and aluminium oxides. The EDS analysis shows that the ratio of both of these elements in the majority of tested coal ash particles is within the range of 1–2. This is the optimal range for the formation of Na-A zeolite (Tanaka et al. 2006).

In lignite coal ashes, spherical particles contain not only SiO_2 and Al_2O_3 , but also calcium oxide CaO (Fig. 7b). The presence of calcium ions in aluminosilicate glass affects its lower stability, thanks to which it dissolves and reacts faster than the glass in silica ashes. Hydraulic activity of these ashes grows in proportion to the increase in their calcium oxide content. This is evidenced by research presented in the work (Enders 1996). According to the author glassy spheres are the most important CaO bearing mineral phase in calcium fly ashes. Since the content of CaO in the glass phase of the tested ash exceeded 25%, it can be considered highly reactive (Giergiczny 2006).

Predomination of aerodynamic and hydrostatic forces promotes formation of particles characterized by the appearance of hollows of various sizes and shapes.

These hollow spherical particles are microspheres (Fig. 8). As in solid spherical particles, the largest mass share in this morphological form is SiO_2 silica and Al_2O_3

Fig. 6 Glass of different colour. Fly ash from **a** bituminous coal **b** lignite coal. Microscopic analysis in transmitted light, 1 N

Fig. 7 EDS spectrum of spherical ash particles from **a** bituminous coal **b** lignite coal

alumina, which ensure high mechanical strength (Żyrkowski et al. 2016). Microspheres from lignite coal also contain calcium oxide CaO and magnesium oxide MgO. High variability of Fe_2O_3 content indicates differences in magnetic properties of hollow spherical particles. Other components, such as SO_3 , K_2O , Na_2O and TiO_2 appear in small amounts, what confirms the results of previous research (Vassiley et al. 2004; Ngu et al. 2007; Strzałkowska 2017). Low stability of chemical composition of microspheres is noticeable even within a single particle (Fig. 8 and Table 1).

Microspheres are one of the most valuable products obtained from ashes. An important quality feature of these particles is their low bulk density, so they can be used to produce a wide range of building materials, including construction materials, a light filler for the production of synthetic materials or lightweight concrete. So far, the only method of obtaining these particles in a large-scale is flotation method, which uses the difference of density (Kiani, Galvin 2017).

A much smaller content of microspheres in lignite coal ashes results from the fact that molten phase, which is created from lignite coal in high temperature, has alkaline character, due to which it has low viscosity. Gases trapped

Fig. 8 Electronic image of microspheres from bituminous coal fly ash. Points 1–8 where microanalysis of the chemical composition was performed (Table 1)

inside the microspheres can easily diffuse through their walls and as a result they break apart. A unique form of microspheres is plerospheres, big particles filled with

Table 1 Results of chemical composition microanalysis in selected areas of the microspher (Fig. 8)

Spot 1	Spot 2	Spot 3	Spot 4	Spot 5	Spot 6	Spot 7	Spot 8						
Chemical component	mass %	Chemical component	mass %	Chemical component	mass %	Chemical component	mass %						
SiO ₂	54.24	SiO ₂	73.47	SiO ₂	53.85	SiO ₂	76.50	SiO ₂	44.43	SiO ₂	56.84	SiO ₂	60.85
Al ₂ O ₃	34.60	Al ₂ O ₃	19.26	Al ₂ O ₃	38.65	Al ₂ O ₃	16.36	Al ₂ O ₃	31.81	Al ₂ O ₃	16.36	Al ₂ O ₃	48.59
CaO	3.45	CaO	2.01	CaO	2.62	CaO	1.42	CaO	4.00	CaO	1.42	CaO	2.07
MgO	2.11	MgO	1.23	MgO	1.54	MgO	1.44	MgO	1.98	MgO	1.44	MgO	1.80
Na ₂ O	1.58	Na ₂ O	1.57	Na ₂ O	0.88	Na ₂ O	2.26	Na ₂ O	1.25	Na ₂ O	2.26	Na ₂ O	0.88
K ₂ O	1.12	K ₂ O	1.5	K ₂ O	0.82	K ₂ O	1.19	K ₂ O	0.99	K ₂ O	1.19	K ₂ O	0.48
P ₂ O ₅	0	P ₂ O ₅	0	P ₂ O ₅	0	P ₂ O ₅	0	P ₂ O ₅	0	P ₂ O ₅	0	P ₂ O ₅	0
Fe ₂ O ₃	1.38	Fe ₂ O ₃	0.22	Fe ₂ O ₃	1.35	Fe ₂ O ₃	0.28	Fe ₂ O ₃	1.96	Fe ₂ O ₃	0.28	Fe ₂ O ₃	0.25
SO ₃	0	SO ₃	0	SO ₃	0	SO ₃	0	SO ₃	0	SO ₃	0	SO ₃	0
TiO ₂	1.51	TiO ₂	0.75	TiO ₂	0.33	TiO ₂	0.56	TiO ₂	1.92	TiO ₂	0.56	TiO ₂	1.50
MnO	0	MnO	0	MnO	0	MnO	0	MnO	0	MnO	0	MnO	0

Fig. 9 Plerosphere. Fly ash from bituminous coal. Microscopic analysis in reflected light, magnification 500x, 1 N

smaller spherical particles (Fig. 9). In order to release these smaller particles, the ash should be grind, thus increasing the chemical reaction surface.

Spherical particles with high reflectance are spinels (identified as magnetite with a diverse structure, most commonly dendritic-skeletal, joined by aluminosilicate glass (Fig. 10) or in a solid form (Fig. 11 a). While melted, silica bonds spinel grains, which take spherical shapes.

The amount of oxygen in the boiler determines the degree of oxidation of iron in coal. As a result of the oxidation of some iron in magnetite, martite is formed, which is a mineral type of hematite occurring in the form of pseudomorphosis from magnetite (Fig. 11b). Only particles with increased iron content were found to contain manganese oxides, which may be the result of the crystal-chemical relationship of these elements and their ability to replace each other. Already before in the literature, a strong correlation between Fe₂O₃ and Mn was noticed (Singh et al. 2011).

The separation of iron compounds from fly ash can be one of ways to use them. On the one hand, this will improve the properties of ash (e.g. for the dissociation of alumina from it), on the other hand, the separated magnetite may find further use, e.g. for the preparation of heavy liquids used in coal enrichment. These particles can be successfully isolated by magnetic separation (Vassilev et al. 2004).

In addition to spherical forms, the occurrence of irregular solid amorphous particles, usually larger than spherical particles, is observed in ashes (Fig. 4). Their shape may be the result of collisions of solid spherical particles during combustion or result from their rapid cooling. Their chemical composition is therefore similar to this of spherical solid particles.

Irregular porous particles are, among others, unburned aggregates of clay minerals (Fig. 12). The EDS analysis of these particles shows that they show high variability in

Fig. 10 Electron image of magnetite with dendritic – skeletal structure in bituminous coal ashes with EDS spectrum

Fig. 11 Ferrospheres **a** magnetite with massive microstructure **b** hematite, visible red internal reflexes. Fly ash from lignite coal. Microscopic analysis in reflected light, magnification 500x, 1 N

Fig. 12 Electron image of irregular porous particles together with the EDS spectrum **a** lignite coal ash, **b** bituminous coal ash

Fig. 13 Quartz. Fly ash from bituminous coal. Microscopic analysis in transmitted light, magnification 200x, XN

chemical composition. The most significant changes were observed in the case of SiO_2 and CaO content. The effect of high porosity of these particles is increased water demand of the ash.

The crystalline components of silica ashes are, among others mullite (which is a product of decay of kaolinite) and quartz (Fig. 13) also present in lignite coal ashes. According to previous studies (Kapuściński, Strzałkowska 2007), the detritic quartz grains are of high purity (content of SiO_2 is about 98%) (Fig. 14) while the secondary silica phase formed as a result of the mullitization process is associated with the presence of an aluminosilicate substance. Quartz in fly ash has similar applications as natural mineral aggregates, whereas mullite (which appears in the temperature of around 1000°C) gives refractory properties to ash, often used in ceramics and construction.

Conclusion

The presented above chemical and morphological characteristics of two types of fly ashes from bituminous coal and lignite coal, allowed the formulation of the following conclusions.

- The researched ashes are heterogeneous material, built of particles with different physical, mineralogical and chemical properties.
- Ash from bituminous coal characterizes with bigger particles than lignite coal ash, which is related to the greater proportion of unburned organic matter in it. This is evidenced by both microscopic observations and higher loss on ignition for these ashes. Removing larger particles (above $100\ \mu\text{m}$) will also remove a huge part of unburned coal. The most suitable method for separation of unburned coal from these ashes seems to be flotation, which uses differences in the wettability of the particles. Unburned coal particles have hydrophobic properties and will be easily floatable after adding optimal amounts of flotation reagents.
- Mineral matter in the studied ashes occurs in three basic morphological forms: spherical, irregular and fine-detritic, differing in porosity. Considering the usefulness of these ashes as building materials, ash from bituminous coal characterises with high pozzolanic activity, because of high content of spherical particles (rich in SiO_2 and Al_2O_3). For the same reason, these ashes can also be a valuable material for zeolites synthesis.
- Spherical glass phase from lignite coal has aluminium–silicate–calcium chemical composition, which is what determines pozzolanic and hydraulic properties of these ashes. Presence of calcium ions in aluminosilicate glass will surely influence its stability. Low density, spheri-

Fig. 14 EDS spectrum of quartz. Fly ash from bituminous coal combustion

cal shape and glassy character of microspheres present in ash qualify this raw material for many applications, among others as a material improving rheological properties or in the production of lightweight concrete. The EDS analysis of individual microsphere particles shows that they are characterized by high variability in chemical composition. Before further use of this raw material, it is necessary to obtain a more homogeneous material.

- One of the methods of usage of these ashes can be separation of iron-rich particles from them. The product obtained in the process of magnetic separation, after further processing and separation of impurities, can be used as ore for the production of iron.
- After separating all useful components from researched ashes, the refined waste can be used in the following industries: ceramics, refractory, construction, etc.

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References

- Adamczyk Z, Komorek J, Lewandowska M (2018) The high temperature ashes (HTA) from bituminous coal combustion as a potential resource of rare earth elements. *Gospodarka Surowcami Mineralnymi - Miner Resour Manag* 34(3):135–150. <https://doi.org/10.24425/122576>
- Apostolidou Ch, Georgakopoulos A (2018) Morphology, mineralogy and chemistry of fly ash from the Ptolemais Power Stations, Northern Greece, and its potential as partial portland cement substitute. In: *Proceedings of the 14th International Symposium of Continuous Surface Mining, ISCSM 2018*
- Bastian S (1980) *Betony konstrukcyjne z popiołem lotnym*. Arkady, Warszawa (in Polish)
- Blissett R, Smalley N, Rowson N (2014) An investigation into six coal fly ashes from the United Kingdom and Poland to evaluate rare earth element content. *Fuel* 119:236–323. <https://doi.org/10.1016/j.fuel.2013.11.053>
- Chengfeng Z, Qiang Y, Jiuming S (2005) Characteristics of particulate matter from emissions of four typical coal-fired power plants in China. *Fuel Process Technol* 86:757–768. <https://doi.org/10.1016/j.fuproc.2004.08.006>
- Czarna D, Wdowin M, Franus W, Liu H, Panek R, Snape CE, Stevens LA, Sun CG (2016) Application of Fly Ash Derived Zeolites as CO₂ Sorbents. IGSMiE PAN Publishing, Kraków
- Enders M (1996) The CaO distribution to mineral phases in a high calcium fly ash from Eastern Germany. *Cem Concr Res* 26(2):243–251. [https://doi.org/10.1016/0008-8846\(95\)00211-1](https://doi.org/10.1016/0008-8846(95)00211-1)
- Erol M, Küçükbayrak S, Ersoy-Meriçboyu A (2008) Comparison of the properties of glass, glass-ceramic and ceramic materials produced from coal fly ash. *J Hazard Mater* 153:418–425. <https://doi.org/10.1016/j.jhazmat.2007.08.071>
- Fisher GL, Prentice BA, Silberman D, Ondov JM, Biermann AH, Ragaini RC, McFarland AR (1978) Physical and morphological studies of size-classified coal fly ash. *Environ Sci Technol* 12(4):447–451. <https://doi.org/10.1021/es60140a008>
- Galos K, Uliasz-Bocheńczyk A (2005) Źródła i użytkowanie popiołów lotnych ze spalania węgla w Polsce. *Gospodarka Surowcami Mineralnymi - Miner Resour Manag* 21(1):23–42 ((in Polish))
- Gawlicki M, Wons W (2012) Popioły lotne z kotłów fluidalnych jako składniki popiołowo-cementowych spoiw drogowych. *Prace Instytutu Ceramiki i Materiałów Budowlanych*, 8 (in Polish)
- Giergiczny Z (2013) *Popiół lotny w składzie cementu i betonu*. Wydawnictwo Politechniki Śląskiej, Gliwice (in Polish)
- Giergiczny Z (2006) *Rola popiołów lotnych wapniowych i krzemionkowych w kształtowaniu właściwości współczesnych spoiw budowlanych i tworzyw cementowych*. Wydawnictwo Politechniki Krakowskiej (in Polish)
- Haustein E, Quant B (2011) The characteristics of selected properties of the cenospheres-fraction of fly ash-by-product of coal combustion. *Gospodarka Surowcami Mineralnymi-Miner Resour Manag* 27(3):95–111
- Holler H, Wirsching U (1985) Zeolite formation from fly ash. *Forsch Miner* 63:21–43
- Hycnar J (1987) *Metody wydzielania koncentratów metali z popiołów elektrowniowych*. Fizykochemiczne Problemy Mineralurgii 19:243–257 ((in Polish))
- Jarosiński A (2016) *Możliwości pozyskiwania metali ziem rzadkich w Polsce*. Zeszyty Naukowe Instytutu Gospodarki Surowcami Mineralnymi i Energią Polskiej Akademii Nauk 92:75–88 ((in Polish))

- Kapuściński T, Strzałkowska E (2007) Wykorzystanie mikroskopy elektronowej do badań odpadów paleniskowych. *Zeszyty Naukowe Politechniki Śląskiej, seria Górnictwo* z 280:57–72 ((in Polish))
- Kiani A, Galvin KP (2017) Detailed characterisation and separation of fly ash fed to the inverted reflux classifier. *Fuel Process Technol* 155:114–123. <https://doi.org/10.1016/j.fuproc.2016.04.028>
- Lester E, Alvarez D, Borrego AG, Valentim B, Flores D, Clift DA, Rosenberg P, Kwiecińska B, Barranco R, Petersen HI, Mastalerz M, Milenkova KS, Panaitescu C, Marques MM, Thompson A, Watts D, Hanson S, Predeanu G, Misz M, Wu T (2010) The procedure used to develop a coal char classification – commission III combustion working Group of the international committee for coal and organic petrology. *Int J Coal Geol* 81:333–342. <https://doi.org/10.1016/j.coal.2009.10.015>
- Matsunaga T, Kim JK, Hardcastle S, Rohatgi PK (2002) Crystallinity and selected properties of fly ash particles. *Mater Sci Eng A*. [https://doi.org/10.1016/S0921-5093\(01\)01466-6](https://doi.org/10.1016/S0921-5093(01)01466-6)
- Michalíková F, Búgel M, Zelenák F (1994) Przeróbka popiołów z węgla kamiennego metodą flotacji i separacji na mokro w słabym polu magnetycznym. XII Międzynarodowy Kongres Przeróbki Węgla, Kraków 23–27 maja, Kraków: 227–237 (in Polish)
- Misz M (2002) Comparison of chars in slag and fly ash as formed in pf boilers from Będzin Power Station (Poland). *Fuel* 81(10):1351–1358. [https://doi.org/10.1016/S0016-2361\(02\)00022-4](https://doi.org/10.1016/S0016-2361(02)00022-4)
- Ngu L, Wu H, Zhang D (2007) Characterization of ash cenospheres in fly ash from Australian power station. *Energy Fuels* 21(6):3437–3445. <https://doi.org/10.1021/ef700340k>
- Ojha K, Pradhan NC, Samanta AN (2004) Zeolite from fly ash: synthesis and characterization. *Bull Mater Sci* 27(6):555–564. <https://doi.org/10.1007/BF02707285>
- Querol X, Alastuey A, Lopez-Soler A, Plana F (1997) A fast method of recycling fly ash: microwave assisted zeolite synthesis. *Environ Sci Technol* 31:2527–2533. <https://doi.org/10.1021/es960937t>
- Ramsden AR, Shibaoka M (1982) Characterization and analysis of individual fly-ash particles from coal – fired power stations by a combustion of optical microscopy, electron microscopy and quantitative electron microprobe analysis (1967). *Atmos Environ*. [https://doi.org/10.1016/0004-6981\(82\)90290-6](https://doi.org/10.1016/0004-6981(82)90290-6)
- Ratajczak T, Piestrzyński A (2000) Wzbogacanie magnetyczne popiołów lotnych jako metoda odzyskania i możliwości identyfikacji niektórych ich składników. *Zeszyty Naukowe: Górnictwo* 245, Kraków: 143–157 (in Polish)
- Rozporządzenie Ministra Środowiska z dnia 9 grudnia (2014) w sprawie katalogu odpadów, poz. 1923 (in Polish)
- Sharonova OM, Fedorchak MA, Zhizhaev AM, Mazurova EV, Anshits AG (2015) Composition of individual ferrospheres of different morphological types. *Inorg Mater* 51(11):1143–1150
- Singh S, Ram LS, Masto RE, Santosh K, Verma SK (2011) A comparative evaluation of minerals and trace elements in the ashes from lignite, coal refuse, and biomass fired power plants. *Int J Coal Geol* 87:112–120. <https://doi.org/10.1016/j.coal.2011.05.006>
- Sokol EF, Kalugin VM, Nigmatulina EN, Volkova NI, Frenkel AE, Maksimova NV (2002) Ferrospheres from fly ashes of chelyabinsk coals: chemical composition, morphology and formation conditions. *Fuel* 81:867–876. [https://doi.org/10.1016/S0016-2361\(02\)00005-4](https://doi.org/10.1016/S0016-2361(02)00005-4)
- Stoch A (2015) Fly ash from coal combustion – characterization. PhD thesis, Tecnico Lisboa
- Strzałkowska E, Adamczyk Z (2019) Influence of chemical composition of fly-ash cenospheres on their grains size. *Int J Environ Sci Technol* 17(2):809–818. <https://doi.org/10.1007/s13762-019-02512-2>
- Strzałkowska E, Stanienda-Pilecki K (2018) Morphology, size and density of cenospheres from Polish and foreign power plants. *Przegląd Górniczy* 74(12):44–53 ((in Polish))
- Strzałkowska E (2017) Selected properties of cenospheres from fly ashes. *Przegląd Górniczy* 73(11):37–43 ((in Polish))
- Swamy AK, Das A (2012) Possible use of some waste materials in road construction. *The Masterbuilder* 10:44–48
- Tanaka H, Eguchi H, Fujimoto S, Hino R (2006) Two-step process for synthesis of a single phase Na–A zeolite from coal fly ash by

- dialysis. *Fuel* 85(10–11):1329–1334. <https://doi.org/10.1016/j.fuel.2005.12.022>
- Tausif M, Zaidi SBA, Ahmad N, Jameel MS (2020) Influence of natural zeolite and paraffin wax on adhesion strength between bitumen and aggregate. *Civil Eng J* 6(4):733–742
- Uliasz-Bocheńczyk A, Mazurkiewicz M, Mokrzycki E (2015) Fly ash from energy production - a waste, byproduct and raw material. *Gospodarka Surowcami Mineralnymi - Miner Resour Manag* 31(4):139–150. <https://doi.org/10.1515/gospo-2015-0042>
- Vassilev SV, Menendez R, Borrego AG, Diaz-Somoano M, Martinez-Tarazona MR (2004) Phase-mineral and chemical composition of coal fly ashes as a basis for their multicomponent utilization 3. Characterization of magnetic and char concentrates. *Fuel* 83(11–12):1563–1583. <https://doi.org/10.1016/j.fuel.2004.01.010>
- Winczaszek BA (2006) Analiza możliwości zastosowania zeolitów syntetycznych z popiołów lotnych w procesach oczyszczania ścieków. PhD thesis, Wrocław (in Polish). <https://civilejournal.org/index.php/cej/article/view/2021>
- Wong JKH, Kok ST, Wong SY (2020) Cementitious, pozzolanic and filler materials For DSM binders. *Civil Eng J* 6(2):402–417
- Yao ZT, Ye Y, Xia MS (2013) Synthesis and characterization of lithium zeolites with ABW type from coal fly ash. *Environ Prog Sustain* 32:790–796. <https://doi.org/10.1002/ep.11689>
- Żyrkowski M, Neto RC, Santos LF, Witkowski K (2016) Characterization of fly-ash cenospheres from coal-fired power plant unit. *Fuel* 174:49–53. <https://doi.org/10.1016/j.fuel.2016.01.061>