

**THE POSSIBILITY TO USE MODIFIED FLIGHT ASH
AS A NEUTRALIZER IN THE ACID SOILS
RECLAMATION PROCESSES**

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Abstract

Using the alkaline fly ash after combustion of lignite as the acid soils neutralizer is a technique known for decades. Due to many disadvantages of the direct fly ash application it is sought to modify this material prior to its use. The process of fly ash modification in the magnetic activator involved breaking up fly ash to small grain sizes in order to obtain a material with a very large specific surface and modified properties. The purpose of the research was to compare the properties of unmodified fly ash with those of ash modified in the magnetic activator in terms of its usefulness in the neutralization of acidic soils. Unmodified fly ash was classified as a medium-grained calciferous material. The basic components of ash were silicates (33.28% of SiO₂) and calcium compounds (31.26% of CaO). It has a low heavy metal content falling within a range characteristic of coal ash and meeting soil quality standard requirements. As a result of activation, the following changes were obtained in the properties of modified ash compared with unmodified ash: sand fraction content – reduced to 0.40, silt fraction content – increased by 1.40, silt fraction content – increased by 1.68, content of the sum of the dust and silt fractions – increased by 1.49, specific surface – increased by 1.65, fineness – reduced by 0.48. Modification of fly ash in the magnetic activator was

found to have improved the physical properties of ash as acidic soil neutralizer, and its chemical properties make such an application possible.

Keywords: fly ash, magnetic activator, reclamation, soil neutralization

1. INTRODUCTION

Scientists and practitioners present diverse opinions on fly ash, ranging from it being considered an environmental problem [1] to being perceived as a valuable material for construction, soil reclamation, and even agriculture [2-5]. The effects of ash application to the soil referred to in the literature relate first and foremost to raising their reaction, but also to raising C content and sorption properties – BS and CEC [6], as well as improving water capacity [3,6]. Tejasvi and Kumar described the impact of fly ash admixed to sandy soil on the improvement of its grain size distribution, water retention capacity, increased porosity, increased pH, and presence of the majority of analyzed minerals [5]. Moreover, Veranis et al. observed the presence of unburnt residual carbon in lignite fly ash in the amount of 0,54-1,69% [4]. This is a significant finding, both as regards its agricultural value and sorption capacity of these materials. While analyzing the impact of ash on soil properties, one has also reported undesirable effects such as increase in solubility of sodium, proper conductivity, contamination with heavy metals, as well as induced deficiency of assimilated phosphorus, iron, and zinc and sometimes copper, manganese, and boron [7,8]. As regards forest ecosystems, sudden increase in reaction may also interfere symbiotic relationships with certain groups of soil microorganisms and may bring about changes in soil microbiology [9], which must be taken into account when planning ash applications in relation to the form of the final development. As regards the reclamation of post-mining land, the references are available on the methods for preventing oxidation of pyrites through sealing soil with energy ashes [10,11]. On the other hand, the application of ash containing heavy metals to highly acidic soil environment can result in their increased solubility with lowered pH below 6.0 [12]. However, there are findings available that support the lack of negative reaction of crops to fertilizing soil with fly ash [13]. The best properties and suitability for the neutralization of acidic soils are demonstrated by fine-grained fly ash with a large specific surface. One of the ways to improve those properties of fly ash is by modifying it in a magnetic activator [14]. Owing to vibrations of ferromagnetic elements in the activator, the fly ash grain crushing process takes place, which results in their large specific surface and hence improved reactive properties [15,16].

A large percentage of soils in Poland have a low pH. About 38% of the surface area of the country at the end of 2016 was covered with soils with pH of less than 5.5, of which 12% are below 4.5 (Fig. 1). Soil liming needs in Poland were estimated for 49% of the total area under cultivation [17]. Fly ash with a high pH, usually above 9 (even reaching over 12 pH – [18]), can be used for the deacidification of soils on reclaimed or agricultural land. The use of ash for soil fertilization has been the subject of a number of studies. In terms of properties, wood ash lends itself best for the purpose, whereas hard coal ash is considered to be the worst. Unlike P, K, Ca, Mg and Na, undesirable components, such as certain heavy metals, are poorly soluble owing to a high pH value of the material, and thus their availability to vegetation is low. Waste generated in the combustion of lignite usually ranks in between, and its composition and properties may differ considerably between deposits. It should be noted, however, that excessively high doses of ash may lead to excessive enrichment of soil with salts easily soluble in water. This results in soil salinity [6].

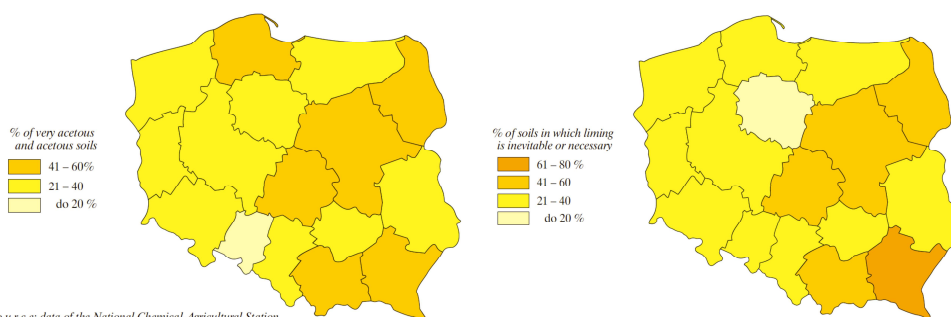
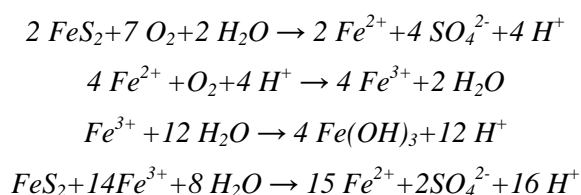


Fig. 1. Evaluation of pH of agricultural land in Poland, 2013-2016 (on right) and the necessity of liming agricultural land soils in Poland, 2013-2016 (on left)

Opencast mining of lignite and different mineral aggregates is planned in western Poland and eastern Germany. After the mining operations are discontinued, land reclamation must be carried out in order to provide the desired functionality to the site. As regards the former lignite mining sites in western Poland and eastern Germany, the main issues are a low pH value of soils, their infertility, lack of pedogenic carbon, an unfavourable structure resulting in an increased susceptibility to erosion [19]. In the latter part of the 20th century, such soils were considered as being useful only for the revegetation of dumps following their drainage, or as toxic materials unsuitable for reclamation. Over time, however, they started to be viewed differently and be

reclaimed with the use of specifically selected reclamation methods [20]. In the sand overburden deposited on spoil heaps in the western Polish lignite-bearing region, pyrite content may reach $59.9 \text{ mol} \cdot \text{m}^{-3}$, which is three times more than in the majority of overburden materials [21,22]. As a result of oxidation of those sulfides, a multistage transformation process takes place that leads to the formation of sulfuric acid which strongly acidifies the soil [23]:



A consequence of high acidity of the overburden soil is the inability of it being directly overgrown with vegetation owing to phytotoxicity. Also the consequences related to the easy transmission of mineral components are important, which, on one hand, leads to the depletion of what is already a deficient environment [35-38], and, on the other hand, poses the risk of pollution of surface waters and groundwater with various ions, including heavy metal ions [24,25]. The basic measure is to ensure the appropriate preparation of subsoil for the introduction of vegetation. Soil neutralization is an important measure [26]. Agricultural lime is expensive in Poland, given the high doses used for reclamation of the soil types in question (ranging from several dozens to more than $100 \text{ Mg} \cdot \text{ha}^{-1}$). For this reason, tests and analyses are conducted concerning the use of ash for the reclamation of such soils [35-38].

In 2016 in Poland 84 mln Mg of hard coal and 61 mln Mg of lignite were used. Consequently, annual generation of fly ash from coal amounted to 3.3 million Mg. At the end of 2016 a total accumulation in landfills accounted for 26 million Mg of fly ash from coal [27,28]. The use of combustion waste for reclamation purposes, including land levelling, is acceptable and often used. In 2010, in the USA a total of 37 million Mg of coal combustion products were used, including fly ash 22,220 Mg in agriculture and 785,552 Mg for reclamation purposes – modification and stabilization of soils [29]. At the same time, it was recommended to gradually increase the use of fly ash and other coal combustion products in individual sectors. Similar trends were also described by Veranis et al. as regards other countries of the world [4].

Similar problems with soil reaction have been noted in areas connected with sulfur exploitation. Very strong acid conditions are making the soil unfertile and phytotoxic [30-32]. The one and only solution is neutralization using high doses of lime. Unfortunately, lime fertilizer is a very expensive material, which should

first be used on non-degraded arable fields. This sought to look for other neutralizers, effective, environmentally safe but much cheaper than fertilizer lime. Many authors describe the use of various waste materials as soil neutralizers, like: dusts, ashes and sediments, including flotation lime sludge [19,20,30-32].

The purpose of the study is to present the results of tests of the properties of unmodified fly ash and ash modified in the magnetic activator in terms of neutralization of acidic soils. A research thesis was made that the modification of fly ash resulting from the combustion of lignite in a magnetic activator causes an effect in the form of a change in its properties beneficial from the point of view of the use for neutralization of acidic soils.

2. MATERIALS AND METHODS

The research was elaborated using the fly ash produced during the combustion of lignite from the Konin Basin. Test samples were taken from a spoil heap of the Pątnów Power Plant. The laboratory tests involved the determination of:

- LOI (loss on ignition) and sulfate content (as SO_3), chlorides Cl^- , free and reactive calcium oxide CaO , silicon dioxide SiO_2 (including reactive SiO_2), aluminium(III) oxide Al_2O_3 , iron(III) oxide Fe_2O_3 , magnesium oxide MgO , sodium oxide Na_2O and potassium oxide K_2O ,
- heavy metal content: cadmium Cd , cobalt Co , chromium Cr (including hexavalent chromium Cr^{6+}), copper Cu , manganese Mn , nickel Ni , lead Pb , thallium Tl and zinc Zn ,
- radioactivity,
- grading,
- pulverizing degree,
- specific surface,
- pH,
- EC (electric conductivity).

The elementary chemical content was determined by the ICP-OES method using the Thermo iCAP 6500 Duo ICP plasma spectrometer with AS CRM-010 as reference substance, while LOI was determined by igniting a weighed portion of material (for a minimum of 2 hours), which involved the measurement of sample mass before and after igniting in a muffle furnace at 975°C ($\pm 25^\circ\text{C}$) until its mass ceased to change.

Radiation was tested using a comparative method of gamma-ray spectrum analysis with the use of a natural radioactive contamination analyzer. Concentrations of naturally radioactive elements were determined: potassium K^{40} , radium Ra^{226} and thorium Th^{228} in balance with radium Ra^{228} as well as the

activity coefficients f_1 and f_2 . The activity coefficient f_1 – defining the content of natural radioactive isotopes – was determined by the formula:

$$f_1 = \frac{S_K}{3000 \text{ Bq/kg}} + \frac{S_{Ra}}{300 \text{ Bq/kg}} + \frac{S_{Th}}{200 \text{ Bq/kg}}$$

and the activity coefficient f_2 – defining the content of radium Ra^{226} – was determined by the formula:

$$f_2 = S_{Ra}$$

where the symbols S_K , S_{Ra} , S_{Th} mean concentration values of potassium K^{40} , radium Ra^{226} and thorium Th^{228} .

Ash grain size distribution was determined by the laser diffraction method, i.e. with a light beam passing through a suspension on the edge of mineral particles contained in it. The Fritsch Analysette 22 laser particle sizer was used for the purpose, while the specific surface was determined by the Blaine method.

Ash pulverizing degree was determined as 0.045 mm sieve residue in the wet ash sieving process.

The pH value of the ash and water mixture (ash to water ratio of 1:2.5) was measured potentiometrically on the basis of the measurement of the electromotive force EMF of a cell consisting of a reference electrode and an indicator electrode using the Radelkis OP-208/1 pH-meter, whereas electrical conductivity was measured in saturated paste using the conductometric method by means of the Elmetron CX-701 conductometer.

The fly ash studied was modified in the magnetic activator shown in Fig. 2. The purpose of fly ash modification in the magnetic activator was to obtain as fine-grained fractions as possible within the shortest possible time, in order to obtain a final product with a much greater specific surface and better properties than the input material. The basic element of the activator is a rotating magnetic field inductor with a tube situated on its axis, forming a work chamber with 20% of its space filled with ferromagnetic grinding media. Grinding media rotating in the space filled with the ground material produce impacts that allow the ground material to be crushed into smaller parts, down to ultra-fine ones.

The modification of fly ash was performed with assumed technological specifications of the magnetic activator, such as: ash grinding time – 60 seconds; number of ferromagnetic grinding elements used – approx. 6000, which represented ca. 20% of the work chamber tube capacity.

The testing of the modified lignite fly ash included the determination of grading, fineness and specific surface.

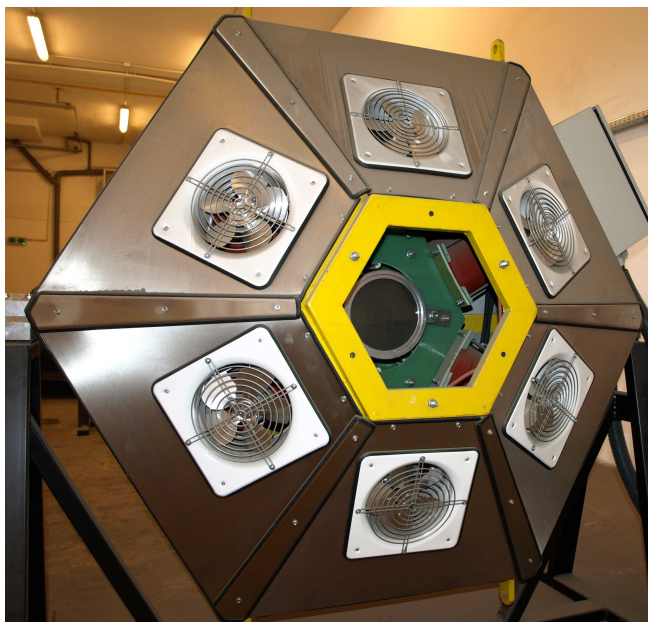


Fig. 2. Magnetic activator used in the experiment

3. RESULTS AND DISCUSSION

The basic chemical composition of ash was found to be as follows:

- silicon oxides (SiO_2), representing 33.28% of ash by weight (including reactive SiO_2 – 9.25%),
- aluminium oxides (Al_2O_3), representing 3.55% of ash by weight,
- calcium oxides (CaO), representing 31.26% of ash by weight (including free CaO – 8.71%),
- sulfates (as SO_3), representing 11.5% of ash by weight,
- iron oxides (Fe_2O_3) – 7.64%,
- magnesium oxides (MgO) – 6.86%.

Based on the results of analysis of the chemical composition, the material was classified as calcic fly ash (class C according to ASTM C618). For this class a typical phenomenon is a presence of the free lime.

The ash was characterized by the absence of chlorides and trace quantities of oxides Na_2O (0.24%), K_2O (0.28%) and a low loss on ignition (0.11%). Heavy metal content in the tested fly ash is shown in Table 1. pH value of the ash and water mixture ranged at 12.5÷13.2. Electrical conductivity in saturated paste extracts was 4.89÷6.76 $\text{mS}\cdot\text{cm}^{-1}$.

The ash tested was also characterized by high leachability of certain components, which is testified by the formation of an alkaline water extract. The electrical conductivity levels obtained are indicative of its suitability for use as a safe neutralizer of acidic soils. Ugurlu also noted this property in the context of the potential environmental impact of fly ash [33]. He concluded it was possible to reduce the electrical conductivity of dust leachate from 10.3 to 5.4 mS·cm⁻¹ and from 7.4 to 3.6 mS·cm⁻¹ by washing it 3 times with deionized water at 23°C.

Table 1. Heavy metal content in fly ash from the Pątnów Power Plant

No	Metal	Content [mg·kg ⁻¹]
1	Cadmium (Cd)	< 0.60
2	Cobalt (Co)	< 24.0
3	Chromium (Cr)	< 2.0
4	Copper (Cu)	10.0
5	Manganese (Mn)	3670
6	Nickel (Ni)	41.0
7	Lead (Pb)	< 70.0
8	Thallium (Tl)	< 49.0
9	Zinc (Zn)	8.0
10	Hexavalent chromium (Cr ⁶⁺)	< 2.0

An analysis of the results shows that based on soil standards and the average content of those elements in the lithosphere and in surface soil horizons in Poland [34], the following concentrations of heavy metals occur in the fly ash tested:

- falling within a range characteristic of coal ash,
- lower than or close to the average content in the lithosphere: Cr, Ni, Zn, Co, Pb, Tl,
- exceeding the average content in the lithosphere: Mn, Cu, Cd,
- falling within the content range occurring in surface soil horizons in Poland: Cd, Co, Ni, Cu, Cr, Zn, Pb, Tl,
- exceeding the content range occurring in surface soil horizons in Poland: Mn.

The results of grading tests of unmodified ash are shown in Table 2. The analyses of grain size distribution showed the content of each fraction in unmodified ash as follows:

- sand – 45.1%,
- dust – 37.0%,
- silt – 17.9%,
- dust + silt – 54.9%.

Table 2. Grain size composition of unmodified fly ash from the Pątnów Power Plant

Grain size distribution d [mm]	Content of each grain or particle fraction f [%]	Sum of fractions with a diameter of less than d [%]
2.0	0.0	100.0
1.0	0.0	100.0
0.5	0.1	99.9
0.25	3.3	96.6
0.125	14.8	81.8
0.1	6.0	75.8
0.09	2.8	73.0
0.063	8.3	64.7
0.05	9.8	54.9
0.04	8.9	46.0
0.03	8.5	37.5
0.02	8.0	29.5
0.01	7.0	22.5
0.002	4.6	17.9
<0.002	17.9	-

The other properties of fly ash that determine its potential use in reclamation are fineness and specific surface. The above properties are shown in Table 3.

Table 3. Results of fineness and specific surface tests of unmodified fly ash from the Pątnów Power Plant

Property	Test result
Pulverizing degree [%]	47.60
Specific surface, Blaine [$\text{cm}^2 \cdot \text{g}^{-1}$]	2150

An analysis of physical properties of fly ash from the Pątnów Power Plant shows that it does not meet fineness requirements for Category N (fineness should not exceed 40%). In terms of grading and Blaine specific surface, it falls within the medium-grained ash category, containing 40÷75% of particles smaller than 0.075 mm and having a specific surface of 1500÷3000 $\text{cm}^2 \cdot \text{g}^{-1}$. The results of grading tests of modified ash are shown in Table 4.

Table 4. Grain size composition of modified fly ash from the Pątnów Power Plant

Grain or particle diameter d [mm]	Content of each grain or particle fraction f [%]	Sum of fractions with a diameter of less than d [%]
2.0	0.0	100.0
1.0	0.0	100.0
0.5	0.0	100.0
0.25	0.0	100.0
0.125	5.0	95.0
0.1	2.9	92.1
0.09	0.9	91.2
0.063	3.2	88.0
0.05	6.2	81.8
0.04	5.1	76.7
0.03	8.0	68.7
0.02	11.7	57.0
0.01	10.0	47.0
0.002	17.0	30.0
<0.002	30.0	-

The analyses of grain size distribution showed the content of each fraction in modified ash as follows:

- sand – 18.2%,
- silt – 51.8%,
- clay – 30.0%,
- silt + clay – 81.8%.

The results of fineness and specific surface tests of modified fly ash from the Pątnów Power Plant are shown in Table 5.

Table 5. Results of fineness and specific surface tests of modified fly ash from the Pątnów Power Plant

Property	Test result
Pulverizing degree [%]	22.80
Specific surface, Blaine [$\text{cm}^2 \cdot \text{g}^{-1}$]	3540

As a result of activation, the following changes were obtained in the properties of modified ash compared with unmodified ash:

- sand fraction content – reduced to 0.40,

- silt fraction content – increased by 1.40,
- clay fraction content – increased by 1.68,
- content of the sum of silt and clay fractions – increased by 1.49,
- specific surface – increased by 1.65,
- pulverizing degree – increased by 0.48.

The basic components of the ash tested were silicates and calcium compounds (Fig. 3).

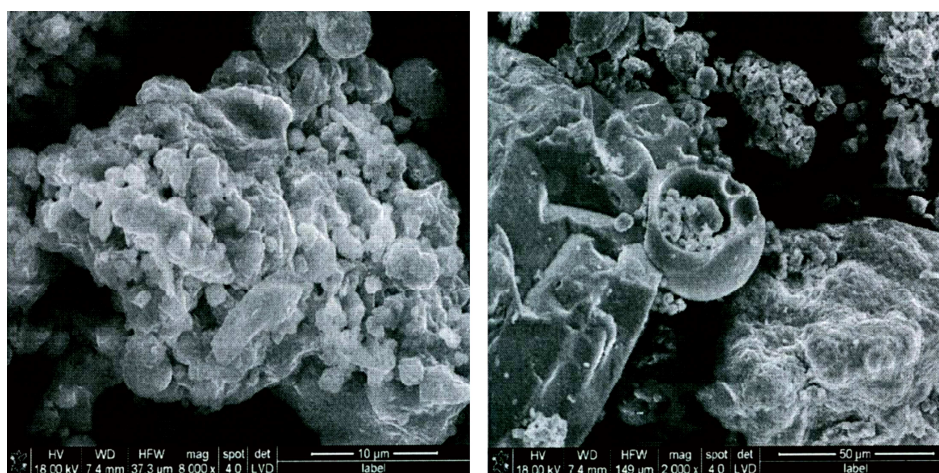


Fig. 3. Electron microscope images of the fly ash from Pątnów Power Plant, generated after the lignite combustion

Heavy metals occur in low concentrations falling within a range characteristic of coal ash and meeting soil or earth quality standard requirements. Comparable results were published by who concluded that only V, Cu, Zn and Cd were found to be active in ash, while more than 80% of Cr, Co, Ni, Sn and Pb were permanently bound [1,4]. Ugurlu demonstrated the possibility of significant quantities of Ca, Na, K and Mg being washed out at room temperature [33]. He also noted, to a smaller extent, the mobility of Fe, Pb and Mn. Gitari et al. pointed to the mobility of the analyzed components from washed fly ash in the sequence: B > Cr > Zn > Mo > Ni > Cu > Co > Se > V > Ba > Cd [18]. A proposition of a different nature was proved by Stouraiti et al. by providing evidence of the reduced availability to organisms of Pb, Zn and Cd present in contaminated soil in an industrial area near Monteverchio, Italy, caused by the addition of lignite fly ash [2]. With the addition of 5% of ash, they reached the reduction of the available form of Pb by as much as 10%, Zn by 5% and Cd by 13%. With higher additions, the reduction was progressing, though at a low

pace for additions above 10%. It can be demonstrated that in the case of the tested lignite ash the risk of heavy metal being released into the soil environment of reclaimed land is low. The analyzed ash was characterized by a low loss on ignition (< 5%) and high bonding properties owing to a high content of free calcium oxide (> 7%). This property should result in the ability of ash to stimulate the formation of soil aggregates, especially with regard to light and medium soils. This fact is indicated e.g. by the research of Yunusa et al. [8].

A radiation analysis showed that the ash tested was not a hazardous material. This is confirmed by radioactivity levels determined by the gamma-ray spectrum analysis. The following concentrations of radioactive elements were found:

Potassium: $S_K = 57.51 \pm 33.29 \text{ Bq/kg}$

Radium: $S_{Ra} = 40.87 \pm 8.56 \text{ Bq/kg}$

Thor: $S_{Th} = 19.58 \pm 3.87 \text{ Bq/kg}$

The respective values of activity levels determined on the basis of the above concentrations are as follows:

$$f_1 = \frac{S_K}{3000 \text{ Bq/kg}} + \frac{S_{Ra}}{300 \text{ Bq/kg}} + \frac{S_{Th}}{200 \text{ Bq/kg}} = 0.25 \text{ Bq/kg}$$

$$f_2 = S_{Ra} = 40.87 \text{ Bq/kg},$$

whereas: $\Delta f_1 = 0.03 \text{ Bq/kg}$, $\Delta f_2 = 8.56 \text{ Bq/kg}$,

Based on the tests performed, it was found that the fly ash from the Pątnów Power Plant met the requirements for the content of:

- natural radioactive isotopes for raw materials and building materials used in buildings intended for occupation by people or livestock, as the activity factor f_1 did not exceed the value of 1.2, and $f_2 < 240 \text{ Bq/kg}$,
- natural radioactive isotopes for industrial waste used in above-ground civil structures erected in developed areas or in areas intended for development under the local area development plan, and for the levelling of such areas, as $f_1 < 2.4$, a $f_2 < 480 \text{ Bq/kg}$,
- natural radioactive isotopes for industrial waste used in the above-ground parts of civil structures not mentioned in point 2, and for the levelling of areas not mentioned in point 2, as $f_1 < 4.2$, a $f_2 < 1200 \text{ Bq/kg}$,
- natural radioactive isotopes for industrial waste used in the above-ground parts of the civil structures mentioned in point 3 and in underground structures, including railway and road tunnels, except industrial waste in underground workings, as $f_1 < 8.4$, a $f_2 < 2400 \text{ Bq/kg}$.

4. CONCLUSIONS

Unmodified fly ash was classified as medium-grained calciferous material. The basic components of ash were silicates and calcium compounds. Heavy metals occurred in low concentrations in the ash, falling within a range characteristic of coal ash and meeting soil quality standard requirements.

The fly ash under examination can be subject to the modification process in the magnetic activator. The tests showed that the ash modification process resulted in significant changes in its grading, specific surface, and pulverizing degree. Owing to vibrations of ferromagnetic elements in the activator, the fly ash grain crushing process takes place, which results in the fragmentation of grains and an increase in their specific surface.

Modification of fly ash in the magnetic activator improved the physical properties (grading, specific surface, and pulverizing degree) of ash as acidic soil neutralizer, and its chemical properties make such an application possible. The changes have a favourable effect on the properties of ash as acidic soil neutralizer. This results from finer grain size of modified fly ash, its greater specific surface, and lower fineness. Based on the results of those tests and the tests of chemical properties, it can be concluded that the ash can be potentially used for the neutralization of acidic soils.

5. ACKNOWLEDGMENTS

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MOŻLIWOŚĆ ZASTOSOWANIA PYŁU LOTNEGO
JAKO NEUTRALIZATORA
W PROCESACH REKULTYWACJI GLEB KWAŚNYCH

Abstract

Wykorzystanie alkalicznego popiołu lotnego po spalaniu węgla brunatnego jako neutralizatora gleb kwaśnych to technika znana od dziesięcioleci. Ze względu na wiele wad bezpośredniego stosowania popiołu lotnego dąży się do modyfikacji tego materiału przed jego użyciem. Proces modyfikacji popiołów lotnych w aktywatorze magnetycznym polegał na rozbiciu popiołów lotnych na małe ziarna w celu uzyskania

materiału o bardzo dużej powierzchni właściwej i zmodyfikowanych właściwościach. Celem badań było porównanie właściwości niezmodyfikowanego popiołu lotnego z właściwościami modyfikowanego popiołu w aktywatorze magnetycznym pod względem jego przydatności do neutralizacji kwaśnych gleb. Niezmodyfikowany popiół lotny został sklasyfikowany jako średnio ziarnisty materiał wapienny. Podstawowymi składnikami popiołu były krzemiany (33,28% SiO₂) i związki wapnia (31,26% CaO). Odznacza się niską zawartością metali ciężkich, mieszczącą się w zakresie charakterystycznym dla popiołu węglowego spełniając wymagania normy jakości gleby. W wyniku aktywacji uzyskano następujące zmiany we właściwościach zmodyfikowanego popiołu w porównaniu z popiołem niemodyfikowanym: zawartość frakcji piaskowej – zmniejszona do 0,40, zawartość frakcji pylistej – zwiększona o 1,40, zawartość frakcji pylastej – zwiększona o 1,68, zawartość sumy frakcji pyłu i mułu – zwiększona o 1,49, powierzchnia właściwa – zwiększona o 1,65, rozdrobnienie – zmniejszona o 0,48. Stwierdzono, że modyfikacja popiołu lotnego w aktywatorze magnetycznym poprawiła właściwości fizyczne popiołu jako środka neutralizującego glebę, a jego właściwości chemiczne umożliwiają takie zastosowanie.

Słowa kluczowe: pył lotny, aktywator magnetyczny, rekultywacja, neutralizacja gleb

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