



Lignitic Fly Ash Byproducts from TPP Kostolac-B, Serbia And Its Usage

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Summary

In this study, the fly ash taken from the lignite thermal power plant Kostolac-B, Kostolac, Serbia, is characterized and separated from its byproducts. Fly ash is composed of unburned carbon, iron compounds typically of magnetite, pozzolanic material and cenospheres. A systematic study is conducted to establish the optimum conditions for the separation of these materials from fly ash. In this context, concentration techniques such as magnetic, gravity separations (heavy liquids separation) and flotation are put forward. Finally the lightweight fraction, from which the magnetic minerals and the unburned coal substance were removed, as well as microspheres, is an excellent material for the production of building materials – blocks and bricks, for concrete mixtures, cement additives, for fly ash pelletization process using Portland cement binder and other purposes. The main elements (fractions of valuable components) of the fly ash that determine its usability are: the quantity of unburned carbon, content of iron compounds (typically of magnetite and haematite), the pozzolanic material, microsphere (cenospheres) content, size distribution, mechanical properties of the pellets etc. The preliminary investigations and accomplished tests exhibited satisfactory results.

Keywords: lignitic fly ash, characterization, flotation, unburned coal, pelletization, building materials, cenospheres

Introduction

Thermal power plants are at present the major generators of electricity in Serbia, fueled by the lignitic low-heat coals. Lignite burning produces large amounts of fly ash, which is a reason for focusing on this waste. Fly ash (FA) is not considered a waste any more, but a technogenous raw material continuously developed for various industrial uses. The benefits from its development are smaller amounts of the ash waste and therefore less polluted environment, on one hand, and an inexpensive raw material for industries, on the other. FA is largely used in the building material industries, and in the building and road construction. Some ash constituents, seemingly unimportant, are very useful in some other sectors of economy. This study is concerned with principal characteristics of the fly ash from the thermal power plant Kostolac B with the view to its best uses.

Located some 90 km SE of Belgrade, the Drmno is the only operating open-pit mine of the Kostolac Coal Basin with the annual output is 6.5 million tons. Geological makeup of the Basin consists of crystalline schists of Paleozoic age and Tertiary (Miocene and Pliocene) and Quaternary (Pleistocene and Holocene) sedimentary deposits. An estimate of the overall Kostolac coal reserves is 388 million tons. Coal from the Drmno deposit is relatively humid (39.22% on average) and has relatively low ash content (17.70%) and elevated sulphur (average

1.18% total sulphur). The mean lower combustion heat is 10020 kJ/kg.

The TTP Kostolac A comprises two blocks, block TEK0 A1 and TEK0 A2, and the Drmno unit blocks B1 and B2, 345 MW each (producing 710500 tons of FA every year). The latter two blocks began to use a new procedure for disposal of a dense aqueous mixture of ash and slag at Čirikovac, while the former two blocks still discharge the water-abundant affluent of FA and slag into the old middle pond of Kostolac.

Samples

Fly ash samples for this study were collected from electrofilters 1 through 4 of Block 2, Drmno. The primary FA sample, a mass of ~60 kg, consisted of the materials from two collections. The FA was comparatively uniform in composition over long period of time, composed of SiO₂ (46.64%), Al₂O₃ (21.67%), Fe₂O₃ (11.44%), MgO (2.64%), SO₃ (2.27%), S* (0.88%), Na₂O (0.51%), K₂O (0.67%), with LOI 1.98% and SiO₂/AlO₂ 2.15%. The size distribution analysis of the fly ash sample was made on the standard Tyler series of sieves for wet and dry screening. Ash particles were finer than 1 mm, with the average grain diameter 0.114 mm and upper boundary size (d_{5%}) 0.570 mm. Mineral composition of the FA is based on the X-ray diffraction analysis, DTA, TGA, SEM and other analytical methods of many FA samples from different deposits [Tomanec et al., 2011].

Morphology of Fly Ash. Under an electronic microscope, the FA grains of non-magnetic fraction from Kostolac B (magnetic fraction and unburned carbon removed) are sub rounded, partly regular spheres – rounded porous grains, sponge-textured, conospheres (shells of microspheric intergrowths) and pleospheres (sphere voids filled with other microspheres).

Experimental Methods

The procedures and analyses used in this study to separate monomineral fractions of the FA from TEKO B and the methods for derivation of some products from this technogenous raw material are the following:

- Sink and float analysis in water and heavy liquid separation.

- Wet magnetic separation (Devi s analyzer).
- Flotation concentration.
- Complete concentrate or separation product characterization; possible uses of fractions.
- Pelletization of fly ash; usable as aggregate, fill in lightweight concrete.
- Manufacture (FA mixed with FGD gypsum and CaO) of bricks for the building material industry.
- Some FA fractions (microspheres) usable for particular purposes.

The heavy mineral fraction was experimentally produced from FA, as well as the magnetic mineral fraction, which is magnetite-high and with some alloy elements can be used in the steel-making process. The sink and float analysis combined with the sieve analysis revealed hollow silicate micro-



Fig. 1. 'Water plane' method, pond A of the TPP Kostolac ash and slag landfill

Rys. 1. Metoda „Water plane”, zbiornik A składowiska popiołu i żużla z TPP Kostolac



Fig. 2. Landfill with an impervious sheet cover

Rys. 2. Składowisko pokryte warstwą nieprzepuszczalną

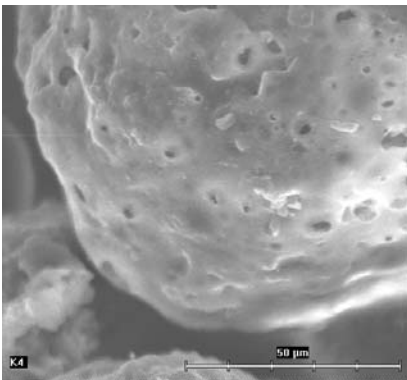


Fig. 3. Electron microscope photograph of silicate allotrimorphous, spherical microspheres

Rys. 3. Zdjęcie z mikroskopu elektronowego alotrimorficznego krzemianu, kuliste mikrosfery

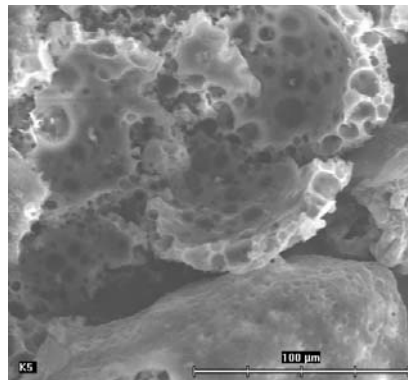


Fig. 4. Fragments of hollow silicate microspheres, with the 'shell' of porous, sponge-textured silica

Rys. 4. Fragmenty z mikrosferami rurkowymi z „muszlami” porowatymi krzemem z teksturą gąbczastą

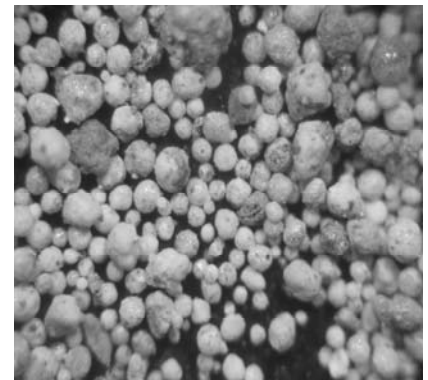


Fig. 5. Microphotograph of light fraction of the fly ash (hollow microsphere density <math>< 1.00 \text{ g/cm}^3</math>)

Rys. 5. Mikrofotografia lekkiej frakcji popiołów lotnych (gęstość mikrosfer rurkowych <math>< 1.00 \text{ g/cm}^3</math>)

spheres, which may be a commercial product. The flotation concentration tests, combined with the gravity concentration, yielded an unburned coal concentrate (semi coke) of very high heat capacity, which may be returned into the process to improve coal combustion. The FA pelletization method is separately described as a pelletization process of a single fraction from which unburned coal (coke) and iron minerals (magnetic and heavy minerals) were removed in order to use the product (pellets) as aggregate in a lightweight concrete. This FA can also be used in the building industry in combination with quicklime (CaO) and waste gypsum (the TPP flue gas desulphation gypsum - FGD), for manufacture of the building blocks and bricks (Tomanec, 2011b).

Results and Discussion

Grain size and chemical composition. After sieve analysis, the sieve size fractions were chemically analysed for burned and unburned coal and their heat capacities. The lowest ash content was found in the coarsest size fraction, $-1.168+0.833$ mm that increased to 96.10% in finer size fraction of $-0.041+0.00$ mm. The proportion of burned coal varied in the opposite direction: it was as high as 64.36% in the coarsest size fraction $-1.168+0.833$ mm (or 13.24% according to recovery) decreasing with the lesser size fraction to 3.50% (or 10.67% according to recovery). Average burned coal content in the FA is therefore 5.71%. The two coarsest size fractions had the heat capacities between 7823 kJ/kg and 16357 kJ/kg (Tab. 1).

Ore microscopy of polished sections examined magnetic fraction and identified dominantly ferromagnetic spheres, affected by hematization, ilmenite and rutile, either in mosaic arrangement – texture or individual magnetite crystallites in silicate glass. Compact ferrospheres are scarce, whilst 'shells', mainly well-rounded and smooth, are common.

Radionuclides. Gamma spectrometry of fly ash from the TPP Kostolac B, conducted in the Laboratory for Nuclear and Plasma Physics of the Nuclear Institute Vinča, Belgrade, indicated specific ash activities: for radionuclide ^{40}K 259 ± 8 Bq/kg; for radionuclide ^{232}Th 50.2 ± 2.4 Bq/kg; for radionuclide ^{226}Ra 62.8 ± 2.6 Bq/kg; and for radionuclide ^{137}Cs 1.18 ± 0.12 Bq/kg. These indicate radionuclide concentrations in FA below the allowed radioactive concentration level for the building materials; it may therefore be used in the interior and exterior components of buildings and in roads.

Light fraction of the fly ash (hollow microsphere density < 1.00 g/cm³) was analysed using the polarizing microscope and the stereobinocular magnifying glass (Fig. 5). Hollow glass beads of extremely small bulk density, low density, are excellent heat insulators; also, their large free surfaces are used to absorb large amounts of water. Note that the extraordinary qualities of this FA fraction make it also the most expensive.

Sink and float analysis of the FA of a sieve size fractions sample had the purpose of separating monomineral fractions: silicate microspheric fraction density < 1 g/cm³; unburned carbon fraction; semi-coke fraction; heavy fraction; light non-magnetic fraction; and magnetic fraction. The combined, sizing and sink and float procedure results are given in Tab. 2. Densities within the range from 1.0 to 2.76 g/cm³ form a fraction of the highest mass proportion, 93.96%. Almost without burned coal, it consists dominantly of ash, or 96.23% with respect to FA recovery. It contains Fe 2.50%, or 92.58% with respect to FA distribution in feed.

The density > 2.76 g/cm³ fraction has an elevated amount of ash, but due to the small mass proportion of 1.29%, the FA distribution in feed is 1.31% and virtually without burned coal. It contains some Fe,

Table 1. Size distribution and chemical composition of the fly ash of the TPP „Kostolac-B“

Tabela 1. Skład ziarnowy i chemiczny popiołów lotnych z TPP „Kostolac-B“

Sieve size fractions (mm)	W _t (%)	Fly ash (%)	Unburned coal (semi coke), %
-1.168+0.833	2.04	27.11	64.36
-0.833+0.589	2.72	69.44	27.79
-0.589+0.417	5.86	84.86	13.56
-0.417+0.297	9.53	94.00	5.39
-0.297+0.208	11.02	95.76	3.90
-0.208+0.147	12.18	97.07	2.62
-0.147+0.104	9.11	97.29	2.49
-0.104+0.074	18.51	97.25	2.39
-0.074+0.041	11.63	96.97	2.61
-0.041 + 0.000	17.40	96.10	3.50
Feed: Fly ash	100.00	84.76	5.71

with the FA distribution in feed being 7.42%. In addition to iron, chemical analysis identified many elements in the separated density fractions from 1.0 to 2.76g/cm³ and >2.76 g/cm³ (Tab. 3). Notable are the elevated lead, arsenic, antimony, etc. and silver and gold.

Magnetic separation. Fly ash samples were treated in Davi's analyzer for wet separation of strongly magnetic minerals. The resulting FA fractions and the distribution of total iron in the magnetic and non-magnetic fractions are given in Tab. 4. The table shows that the mean Fe content was 2.39% in the

sample with respect to 4.89% in the magnetic fraction. The distributions of Fe were 36.32% and 63.68% in the magnetic and non-magnetic fractions, respectively. The density fraction < 1 g/cm³ is without Fe, composed mainly of unburned carbon – semicoke and silicate microspheres. Chemical analysis detected many elements (Tab. 5), some notably elevated (lead, arsenic, antimony, etc.), in the magnetic and in the non-magnetic fractions. Also elevated concentrations were noted of silver (0.12 g/t in magnetic and 0.19 g/t in non-magnetic fraction) and gold (1.14 g/t and 0.45 g/t, respectively).

Table 2. The combined, classification and sink and float procedure results (Size distribution and chemical composition of the fly ash, heavy liquid separation)

Tabela 2. Analiza wzbogalności i klasyfikacji (skład ziarnowy i chemiczny popiołów lotnych, wzbogacanie w cieczy ciężkiej)

Products	Size fractions, [mm]	W _t , [%]	Fly ash, [%]	Distribution of FA in < 1,0 g/cm ³ , [%]	Distribution of FA in feed, [%]	Unburned carbon W _t , [%]	Distribution of burned coal, D _c , [%]	Lower heat capacity [kJ/kg]	Fe, [%]	Distribution of Fe, [%]
Light fraction < 1,0 g/cm³	+ 1.000	1.17	10.84	5.28	0.13	79.37	44.72	22 071	0.00	0.00
	- 1.000+0.500	1.72	35.56	25.46	0.63	56.38	46.70	15 589	0.00	0.00
	- 0.500+0.400	0.42	64.52	11.28	0.28	32.82	6.64	7900	0.00	0.00
	- 0.400+0.315	0.36	91.54	13.72	0.34	7.18	1.24	0	0.00	0.00
	-0.315 +0.000	1.08	98.43	44.26	1.08	1.36	0.70	0	0.00	0.00
	Feed:	4.75	50.57	100.00	2.47	43.72	100.00			0.00
1.0 – 2.76 g/ cm³		93.96	100.00		96.23	0.00	0.00		2.50	92.58
>2.76 g/cm³		1.29	100.00		1.31	0.00	0.00		13.57	7.42
Feed: Fly ash		100.00	97.64		100.00	2.08			2.54	100.00

Table 3. Chemical composition of fly ash (heavy liquid separation)

Tabela 3. Skład chemiczny popiołu lotnego (wzbogacanie w cieczy ciężkiej)

Products	Pb, %	Sb, %	As, %	S, %	Bi, %	Ni, %	Cd, %	Zn, %	Mg, %	Ca, %	Mn, %	Cu, %	Ag, g/t	Au, g/t
1.0 – 2.76 g/ cm ³	0.0033	0.21	0.01	0.43	0.0043	0.0040	0.0001	0.0055	0.074	1.32	0.51	0.0054	1.74	0.11
> 2.76 g/ cm ³	0.010	2.09	<0.01	1.07	0.0014	0.022	0.0004	0.015	1.19	6.36	0.33	0.010	1.67	0.33

Table 4. Wet magnetic separation (Devi 's analyzer) of fly ash

Tabela 4. Separacja magnetyczna na mokro popiołu lotnego (analyzer Devi)

Products	W _t , %	Fe, %	Distribution of Fe, %
Magnetic fraction (M. F.)	17.75	4.89	36.32
Non-magnetic fraction (N. M. F.)	76.08	2.00	63.68
Light fraction (< 1 g/cm ³)	6.17	0.00	0.00
Feed: Fly ash	100.00	2.39	100.00

Table 5. Chemical composition of fly ash (electromagnetic separation)

Tabela 5. Skład chemiczny popiołu lotnego (separacja elektromagnetyczna)

Products	Pb, %	Sb, %	As, %	S, %	Bi, %	Ni, %	Cd, %	Zn, %	Mg, %	Ca, %	Mn, %	Cu, %	Ag, g/t	Au, g/t
M. F.	0.020	0.013	0.020	0.94	0.0043	0.0091	0.0001	0.0063	0.074	1.82	0.12	0.0077	1.14	0.12
N. M. F.	0.020	0.0083	0.010	0.94	0.0024	0.0044	0.0006	0.011	0.63	1.27	0.032	0.0071	0.45	0.19

Flotation concentration of unburned carbon from FA. Additionally to the sink and float analysis, the flotation tests were expected to separate unburned coal and any possible organic compound by using different collector and frothing agents. For repulping, one kilogram of FA and water were used to the level adequate for operation of a Denver type flotation cell volume 2.8 l. The pulp feed pH was 8.7 that in ten minutes rose to 11.17. After five-minute settling in the calm pulp and separation of the floating light fraction and the sinking heavy fraction, the light

fraction was mechanically removed. The five-minute conditioning began with the addition (100 g/t of ash) of the frothing agent F 521, made by CYTEC. During the ten-minute main flotation, 400 g/t more F 521 was added, and the product was cleaned for five minutes. The cleaning flotation outflow was an middling product and the frothy product was the concentrate. The test results are summarized in Tab. 6. The products were chemically analyzed for the amounts of ash and unburned carbon and for its heat capacity (Tab. 6).

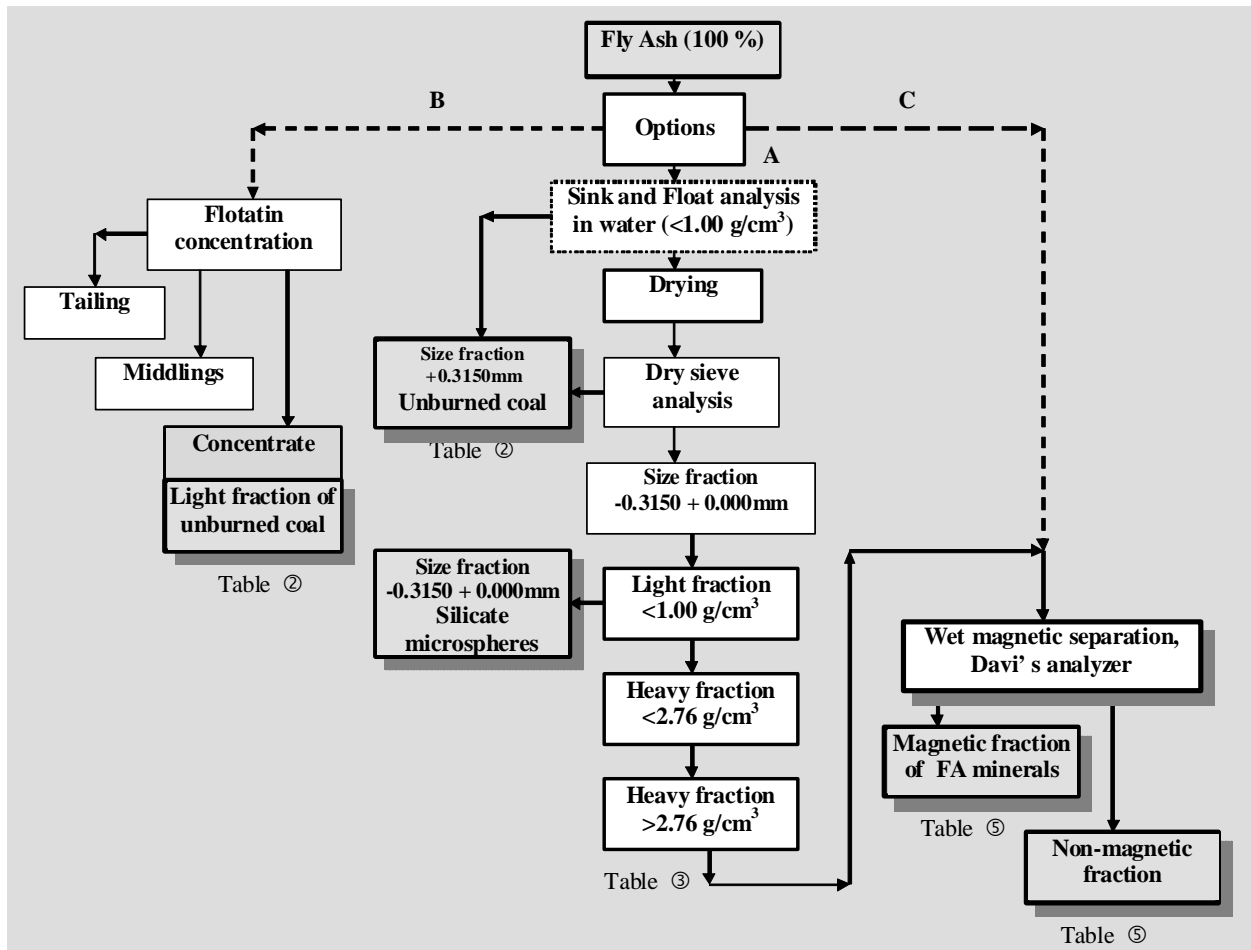


Fig. 6. Optimum flow sheet for separation of fly ash byproducts with options A, B and C

Rys. 6. Optymalny schemat wzbogacania produktów pośrednich z popiołu lotnego (opcje A, B, C)

Table 6. Account of carbon flotation concentration (with respect to burned carbon)

Tabela 6. Ilość koncentratów flotacyjnych węgla (z uwzględnieniem niedopału węgla)

Products:	W _t , [%]	Unburned carbon, [%]	Distribution of burned carbon D _c , [%]	Fly ash, [%]	Distribution of FA, [%]	Lower heat capacity, [kJ/kg]	Distribution of lower heat capacity, [%]
Light fraction (< 1 g/cm ³)	2.75	38.65	19.32	57.66	1.69	13483	92.05
Concentrate	0.41	29.72	2.22	67.57	0.30	7465	7.60
Middlings	2.14	11.81	4.60	87.02	1.98	660	0.35
Tailing	94.70	4.29	73.86	95.15	96.03	0.00	0.00
Feed:	100	5.50	100.00	93.83	100.00	402.80	100.00

The resulting concentrate (light fraction and flotation concentrate) with weight of 3.16% has the highest amount of unburned carbon, as it consists dominantly of semicoke (37.49%), or according to recovery 21.54%. Middlings product with weight of 2.14% contain unburned carbon 11.81% or with 4.60% of recovery. Waste, which is the highest constituent of even 94.70%, has the lowest amount of unburned carbon (only 4.29%), but due to the large weight percentage distribution of unburned carbon is 73.86% (Table 6).

The concentrate 3.16 % by mass has the highest value of the lower heat capacity 12702.18 kJ/kg, in respect to 99.65% carbon distribution in feed. This is because the concentrate consists essentially of unburned carbon – a semicoke. The purpose of the experiment was to test and demonstrate the elimination of unburned carbon from the fly ash for use of the remaining FA in building and road construction, in the process of pelletization, etc (Bircevic, 2011a).

Pelletization process agglomerates wet fine-powdered particles on a rotating disc with the addition of a binding agent during or after the process. The resulting pellets must be adequately strong to withstand transportation and storage. Pelletization products prevailing over the world are concentrates of magnetite, hematite, limonite, siderite, some clays, coal and fly ash, produced in Belgium, China, Columbia, Germany, Italy, Japan, Holland, Great Britain, Russia and USA (Gorkhan, 2000). Pellets of the FA are used as aggregate for lightweight concrete, filler in drainage channels, for soil stabilization and made up ground, stabilization of upper waste landfill layers, and the like. Machines and devices. The equipment used for aggregation was a laboratory pelletization disc unit, Eirich TR.04 (made by Maschinenfabrik Gustav Eirich) power 0.5 kW, 40 cm in diameter.

Physical and mechanical properties of FA pellets must be of standard quality in chemical composition, mechanical properties, physical properties, etc. Impact strength of pellets was tested for ten representative grains-from each size fractions. Each test recorded the pellet diameter and the number of drops to the breaking instant. The test data were statistically processed and interpreted. The pellets used in the tests had diameters from 5.90 mm to 7.39 mm and the number of drops exceeded 150, which suggests their high impact strength. Compressive strength of pellets was tested in modified Penetrometer LC 2, Soiltest Inc., Evanston, Illinois, USA. The pellet was loaded by a piston with increasing force to the point of pellet fracturing and breaking. The mean equivalent diameter and mean resistance to pressure were calculated for each size fractions tested. Compression test was performed on a sample of ten

pellets (from each size fractions). The binding agent used was cement type Titan PC 35 M. The test intervals were 7, 14 and 21 days. Compressive strengths of the pellets varied from 2.952 MPa to 9.264 MPa, or average 6.322 MPa.

Building material pilot tests. Given the large amounts of FA daily generated and deposited as waste, and the sizable amounts of waste gypsum (Flue Gas Desulphuration gypsum), there is the need and the available source for combining these technogenous raw materials, with addition of lime, in pilot tests. FA is a basic raw material to which were added waste gypsum (FGD gypsum semihydrate $\text{CaSO}_4 \times \frac{1}{2} \text{H}_2\text{O}$) and quicklime (CaO) to produce building blocks and bricks. The test ratio of fly ash and waste gypsum was 1:1, with the quicklime proportion between 3% and 14% of the total sample mass. Test samples were homogenized, placed in a mould and tested for compressive strength after 28 days. The result was satisfactory. The sample with previously removed magnetic fraction, unburned carbon, and heavy minerals (heavy FA fraction) attained the highest compressive strength. The optimum amount of lime was about 6%. Compressive strength of the prepared samples varied from 5 MPa to 10 MPa, which exceeds the published data for similar samples (without pressure, without firing, a 'cold process') of about 3.5 MPa. With only 5% lime added, the compressive strength can be higher than 10 MPa. The pilot test of this study substantiated the hypothesis that some FA fractions can give products of satisfactory quality for the building industry.

Conclusion

Laboratory tests of this study suggest the following:

1. Separation of monomineral fractions from FA of the TPP Kostolac B was pilot tested and the obtained fractions analyzed for chemical and mineral compositions. The FA contained unburned carbon (semicoke) 5.77% that decreased with the grain fining, as the coarsest size fractions contained 64.36% and the finest only 3.50%. The light fraction of grain size +0.315 mm had the heat capacity 13483 kJ/kg.
2. Concentrate from the flotation separation had the heat capacity 7465 kJ/kg; mixed with the light fraction ($< 1.00 \text{ g/cm}^3$) could give a product of FA 3.16% by mass and 12702.18 kJ/kg heat capacity. This product has the smallest amount of ash or the greatest amount of unburned coal, and semicoke particles 37.49%. Middlings with weight of 2.14% contained unburned carbon 11.81% and ash 87.02%. The weight percentage of waste was 94.70.

3. Heavy fraction ($> 2.76 \text{ g/cm}^3$) of the FA with 1.29% of microscopic ferrospheres consisted mainly of magnetite, hematite, ilmenite and partly rutile.
4. Sieve undersizes fraction -0.315 mm , the light fraction ($< 1.00 \text{ g/cm}^3$), mainly of silicate microspheres, had the ash content of 98.43%.
5. Light FA fraction, a constituent of only 4.75%, consisted of unburned carbon (semi coke) and silicate microspheres.
6. Radionuclides in the ash test samples from the current production are below the contamination level for building materials.
7. Fly ash can be palletized mixed with cement (10% or 35%). The compressive strength of pellets, for 30% cement binder, was between 2.952 MPa and 9.264 MPa and the impact strength higher than 150 drops per pellet (Tomanec, 2011c).
8. Fly ash from the Thermo Power Plant Kostolac B has constituents suitable for use in the building industry for production of bricks and blocks or for pellets as light-concrete aggregate. Some fractions of fly ash, specifically magnetic fraction or light fraction of silicate microspheres also may be a commercial product, and thus contribute to the complex beneficiation of this technogenous raw material, therefore also to the environmental protection.

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Półprodukty z popiołów lotnych z węgla brunatnego z TPP Kostolac B, Serbia i ich wykorzystanie

W artykule przedstawiono charakterystykę oraz wyniki badań nad wzbogacaniem popiołów lotnych ze spalania węgla w Ciepłowni Kostolac –B w miejscowości Kosolac w Serbii. Popioły lotne składają się z niedopału węgla, składników żelazo nośnych (najczęściej magnetyt, materiałów pucolanowych i cenosfer. Celem badań było określenie optymalnych warunków separacji popiołów. Badania przeprowadzono wykorzystując metody wzbogacania magnetycznego, grawitacyjnego (wzbogacanie w cieczy ciężkiej) i flotacji. W końcowym etapie wzbogacania uzyskano frakcję lekką, pozbawioną części magnetycznej i niedopału oraz cenosfer, która może być wykorzystana na do produkcji materiałów budowlanych – bloczków, cegieł, betonu, dodatków do cementu, jako dodatek do procesu peletyzacji z wykorzystaniem cementu portlandzkiego i podobnych zastosowań. Najważniejsze czynniki, które decydują o jakości popiołu są ilość niedopału, zawartość minerałów żelaza (magnetyt, hematyt), zawartość pucolanów i mikrosfer (ceno sfery), skład ziarnowy, właściwości mechaniczne peletów itd. Wstępne badania i testy dały zadowalające wyniki.

Słowa kluczowe: popiół lotny z węgla brunatnego, charakteryzacja, flotacja, niedopał, peletyzacja, materiały budowlane, cenosfery