

Properties of fly ash derived from coal combustion in air and in oxygen enriched atmosphere in a pilot plant installation Oxy-Fuel CFB 0,1 MW²⁾

Właściwości popiołów lotnych pochodzących ze spalania węgla w powietrzu i w atmosferze wzbogaconej w tlen w instalacji pilotowej Oxy-Fuel CFB 0,1 MW

The progressing development of Polish energy sector, which is based mainly on fossil fuels enforces continuously improvement of technological processes. Combustion in oxygen enriched atmosphere, so called oxy-combustion, is one of the innovative ways to develop modern energy. Second important aspect is that in recent years there is placed a special emphasis on care for the environment. Activities in this direction are carried out not only for the protection of atmosphere, manifested in such techniques as carbon capture and storage (CCS), removal of sulphur and nitrogen oxides or particulate matter from exhaust gases. Environmental protection is also seen as a development of utilization of burnt fuel solid residues, which are mostly ashes and due to its properties, fly ashes especially.

Fly ash is used mostly in the construction industry as a raw material or additive in concrete, aggregates, etc. But there is much other possible application of these materials. Among others: the alumino-silicate basis for production of adsorbents [1], directly as sorbents [2] and addition supports sequestration of CO₂ [3]. Since the properties of fly ash depend on both: the combustion process and type of used fuel, their characteristics are very diversified. Effective utilization and re-use requires knowledge of ashes and appropriate referring to that properties. This fact justifies conducted research both in understanding mechanism of fly ash formulation during combustion processes, as well as the directly designation of these properties.

In recent years there could be noted progress in the development of oxy-combustion technologies, as a one of the most promising way for fossil fuels burning with simultaneous carbon dioxide separation and capture. There are many newer and newer research reports on all aspects of this technology.

Several different sources provide scientific information and describe experiences of working on oxy-combustion pilot-plant installations. Yu and co-workers [4] report on the results of comparison of two oxy-fuel cases with air-firing in 100 kW test furnace. Authors put special attention on studies of formed deposits and ashes. Small laboratory scale studies is represented inter alia by paper by Fryda et al. [5], where was showed investigation on ash deposition under oxy-combustion of two coals and their biomass blends in drop tube pulverized combustor.

Other aspect of studies is analysis of flue gas composition, with particular emphasis on polluting agents such as sulfur and nitric oxides. Ahn and co-workers [6] show analysis of SO₃ concentration in oxy-fired PC boiler in comparison with air-firing. Analysis was conducted under different parameters: range of temperatures, degree of limestone addition and two types of coals as fuel. Some papers concerns on impact of flue gas impurities produced in oxy-combustion on carbon capture and storage processes. Stanger and Wall [7] formulate several conclusion for SO_x influences for CCS technologies. There are sources of knowledge about other pollutants emissions during oxy-combustions, such as comparison of black carbon, fine particle and sodium in oxy- and air-firing in working of Morris and co-workers [8].

There is many sources, that does not focus on studies on particular aspects, but looking at all the issues of oxy-combustion as a whole. Example of such papers could be the review on actual state of art of oxy-fuel combustion prepared by G. Scheffknecht and co-workers [9] or comprehensive overview by M.B. Toftegaard et al. [10] on oxy-firing with detailing various research groups and pilot plants and semi-technical installations that they are working on. Another review, taking into account also thermodynamics, chemistry of oxy-combustion and some modeling aspects, is paper of Chen and associates [11].

¹⁾ E-mail: jbieniek@fluid.is.pcz.pl

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From a certain point of view, oxy-firing is a key technology for innovative way of CO₂ separation. Kanniche et al. [12] compare post-combustion and pre-combustion methods of carbon dioxide capture with oxy-fuel techniques. Moreover Liu and Okazaki [13] showed capacities of oxy-combustion to CO₂ separation together with limitation of SO_x and NO_x emissions.

As one of the main by-products of combustion, fly ash should be utilized in proper way. But to make this possible it is required to examine precisely its physico-chemical properties, which may be significantly various, depending on differences of fuel used, selected combustion technology, characteristic of boiler or process parameters. There are many scientific reports in the worlds' literature showing various results from studies on fly ash from different sources.

There are numerous reports on physical and chemical properties of fly ash from different power plants and derived from various types of coal. A.I. Karayigit and R.A. Gayer [14] describe the XRD and elemental composition analysis of ash originated from Turkish lignite (brown coal) burned in one of the Turkish power plants. In turn Y. Nathan et al. [15] showed results of chemical and mineralogical tests of ashes from Israel power plants burning two types of coal: South African and Colombian. Rich source of information about physico-chemical properties of twenty three different fly ashes from European power plants is paper published by N. Moreno and his co-workers [16]. It seems also to be important to determine not only the composition but also a nature of individual components of fly ashes. Example is work of Ward and French [17], defining the methodology for determining the content of glass, which is important from the point of view of disposal and ash impact on the environment. Koukouzas and co-workers [18] presented mineralogical and chemical analysis of fly ash originated from lignite, bituminous coal and wood chips burned in pilot scale circulating fluidized bed boiler. Kutchko and Kim [19] showed analysis of fly ash surface by using scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS). Moreover, Matsunaga et al. [20] reported on morphology and microproperties of different particle fractions of fly ash.

Studies on elemental composition with a particular focus on heavy metal content are relevant to the utilization of fly ash and its subsequent reuse. There is many papers on this topic: Smořka-Danielowska [21] described analysis of content of Cu, Ni, Pb, Zn, Cr and Cd in fly ash from 1600 MW Rybnik Power Station; Koukouzas et al. [22] showed analysis of fly ash originated from 0,1MW circulated fluidized bed (CFB) pilot-plant with use South African coal. Studies were related to composition of fly ash including quantity and leachability of heavy metals. There is also some research on trace elements in fly ash [23]. Vincze et al. [24] described methodology for X-ray microfluorescence (XRF) analysis of metals quantity in fly ash.

Despite the innovativeness of oxy-combustion technology there is several number of literature sources reporting on the properties of ashes from combustion in oxygen enriched atmosphere. One of them is paper of Zhang et al. [25] briefly describing mechanisms of ash formation during oxy-combustion. More developed analysis was provided by Jiao and associates [26]. It concerned on impacts of flue gas recirculation and impurities such as H₂O, HCl, SO₂ on ash partitioning and its further properties. Font and co-workers [27] reports on experiences with working on 90kW oxy-fuel pilot-plant bubbling fluidized bed (BFB)

boiler. These authors studied emissions of mercury and other pollutants, but also showed some results from fly and bottom ash analysis.

Reports from studies on fly ash in oxy-combustion are still insufficient due to the fact that this is a technology still in implementing phase, coming into industry and energy production sector. For the same reason, as the oxy-combustion will be increasingly be deployed, in order to develop appropriate ways of fly ash utilization it is required to provide continuously studies on fly ashes from oxy-combustion.

Within this article we present studies on physico-chemical properties of fly ash derived from 0,1 MW_e Oxy-Fuel pilot plant furnace. This studies includes analysis of chemical composition, thermogravimetric analysis, surface properties (surface areas, pore volumes), determination of loss of ignition and analysis of infrared spectra.

EXPERIMENTAL

Materials and methods

Examined in this paper samples of fly ash originate from combustion of hard (bituminous) coal in a pilot plant Oxy-Fuel CFB circulating fluidized bed installation with electric power of 0,1 MW, which is located in the Institute of Advanced Energy Technologies at Czestochowa University of Technology. Scheme of a plant is shown in the Figure 1. The main dimensions of this object are: height of a combustion chamber – 5 m; internal diameter of combustion chamber – 0,1 m; internal diameter of the cyclone – 0,25 m. The most important sections, from the point of view of testing fly ash, are points of collecting bulk materials, in the case of fly ash are indicated in Figure 1 as FA1 and FA2.

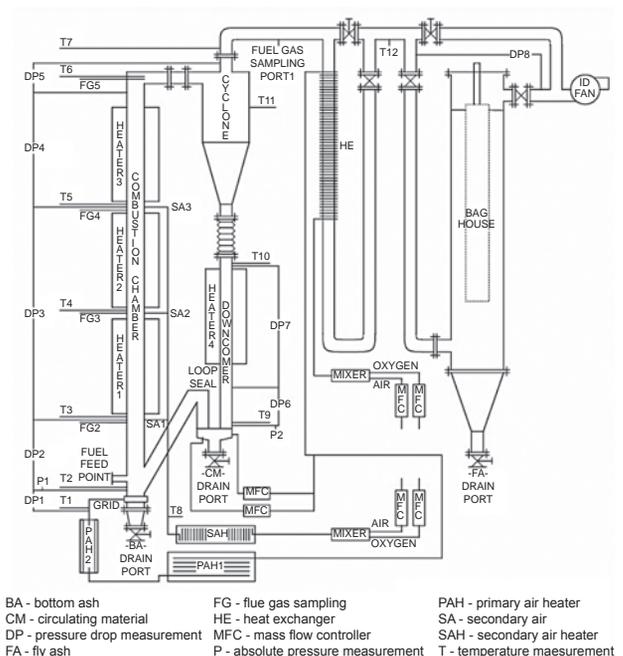


Fig. 1. Diagram of the pilot plant Oxy-Fuel CFB installation with power of 0,1 MWe

During experiments was used hard (bituminous) coal, which the most important parameters are determined. Average shares of particular elements in analysis state and transient moisture content are presented in Table 1. Transient moisture, which value ranges from 6,6 % to 10,0 %.

The schedule of combustion processes measurements, from which samples of ash have been obtained, is shown in Table 2. Names of ash samples are analogous to symbols of measurements (1a, 2a and 3a). Sample 1a is reference sample for combustion in air atmosphere and the other two are derived from the combustion in oxygen and carbon dioxide mixture.

Analysis of carbon, hydrogen, sulphur and nitrogen content were carried out using Leco TruSpec analyzer equipped with infrared sensor and thermal conductivity detection system. Analysis of metal contents (Al, Si, Ca, Fe, Mg, P, K, Mn, Na, Ti) were performed on X-ray fluorescence spectrometer (XRF) PW 4025/00 MiniPal. There were also performed studies of ash surface area (BET and Langmuir specific surface areas, micropore volumes and average pore diameters) using Micrometrics' ASAP 2000 analyzer. Thermogravimetric decompositions studies was performed using TGA/SDTA 851e Mettler Toledo analyzer. Decompositions were carried out under air atmosphere and in temperature range of 251000°C. Series of spectrographic analysis were made on FTIR Nicolet iS10 infrared spectrometer with Fourier transform. Additionally there were determined loss of ignition values according to Polish Standard PN-77/G-04528 "Determination of the chemical composition of ash".

Results and discussion

First phase of chemical composition analysis was determination of carbon, hydrogen, sulphur and nitrogen content, the results of which are shown in Table 3. Decreasing content of unburned carbon – unburned part of organic matter in fact – of fly ash samples, indicate on better coal combustion conditions in CO₂/O₂ atmosphere in relation to combustion in air. Although for the samples of bottom ash there could be observed the opposite trend. For better burnout of organic matter in the case of oxy-combustion indicates also the hydrogen content both in samples of fly and bottom ash. Analysis of sulphur content shows greater tendency of deposition of sulphur in the bottom ash during the oxy-combustion. Lower values of nitrogen content in the samples from the combustion in oxygen leads to the conclusion that CO₂/O₂ atmosphere promotes conversion of fuel nitrogen to nitrogen oxides. However, there is need to note that in the nitrogen-free

atmosphere much easier follows the reaction of NO_x to N₂ reduction by heated to a high temperature coke particles, which consequently results in much lower emissions of nitrogen oxides in the oxy-combustion case [28]. However, this trend of lower nitrogen content in bottom ash is not observed.

Table 3

Contents of basic elements: carbon, hydrogen, sulphur and nitrogen in fly ash (FA) and bottom ash (BA)

Sample of ash Component		1a		2a		3a	
		FA	BA	FA	BA	FA	BA
Carbon	% _{mas}	19,33	0,432	15,50	1,393	7,43	2,350
Hydrogen	% _{mas}	0,164	0,005	0,022	~0	0,011	~0
Sulphur	% _{mas}	1,249	0,031	1,122	0,126	1,589	0,223
Nitrogen	% _{mas}	0,915	0,104	0,371	0,106	0,300	0,101

Values of loss of ignition were shown in Table 4. Comparing contents of organic elements (unburned organic matter) with loss of ignition, it could be observed that, despite its lower value in sample 2a than in 1a, loss of ignition have comparable values. For the better illustrate this dependence values of loss of ignition and carbon content was compared in Figure 2.

Table 4

Values of loss of ignition of fly ash samples

Sample	1a	2a	3a
Loss of ignition, %	20,42	20,38	11,02

Comparison: carbon content - loss of ignition

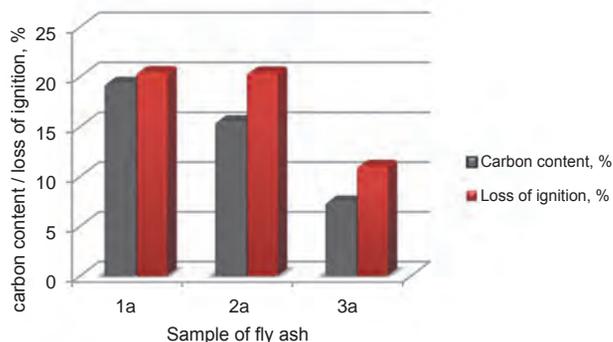


Fig. 2. Comparison of carbon content in fly ash samples with their loss of ignition values

Table 1

Physicochemical composition of coal used in tests

Component	Carbon C ^a	Sulphur S ^a	Hydrogen H ^a	Nitrogen N ^a	Ash A ^a	Volatile matter V ^a	Hygroscopic moisture W ^a	Transient moisture W ^r – W ^a
Content	77,79%	1,67%	5,70%	1,23%	8,50%	38,11%	5,90%	6,6-10,0%

Table 2

Measurement parameters of the combustion processes, from which samples of fly ash was collected

Measure label (symbol of fly ash)	Atmosphere	Oxygen content in gas inlet, [%vol.]	Graduation: primary gas / secondary gas	Oxygen excess coefficient	Temperature in combustion chamber
1a	Air - N ₂ /CO ₂	21	70 / 30	1,15	850
2a	O ₂ /CO ₂	21	70 / 30	1,15	850
3a	O ₂ /CO ₂	25	70 / 30	1,15	850

Contents of metallic elements was shown in Table 5. Silica, aluminum, calcium, iron, magnesium, phosphorus and potassium were presented as calculated in oxide form (due to applied methodology of XRF analyzer measurement). All samples of fly ash characterize similar contents of aluminum and silica. Highest content of rest of metals shown sample 3a. In most cases it could be explained by significantly lower residue of unburned carbon, but not for considerably higher amounts of iron, phosphorus, manganese and sodium. In case of potassium and titanium this trend was not observed. Comparing contents of metals in samples 1a and 2a no significant differences were noted.

Table 5

Contents of metal elements in fly ash (FA) and bottom ash (BA)

Sample		1a		2a		3a	
		FA	BA	FA	BA	FA	BA
Al ₂ O ₃	% _{mas}	19,36	13,26	20,82	18,88	18,65	9,44
SiO ₂	% _{mas}	33,43	63,37	34,53	53,33	31,6	58,48
SiO ₂ /Al ₂ O ₃ ratio		1,73	4,78	1,66	2,82	1,69	6,19
CaO	% _{mas}	4,09	3,19	3,98	3,56	6,84	4,78
Fe ₂ O ₃	% _{mas}	12,31	1,73	11,66	5,72	23,41	3,31
MgO	% _{mas}	1,5	2,18	1,69	1,51	1,82	1,79
P ₂ O ₅	% _{mas}	0,66	0,32	0,38	0,4	2,7	5,00
K ₂ O	% _{mas}	1,88	1,03	1,86	1,35	1,48	0,79
Mn	% _{mas}	0,0237	0,0269	0,0312	0,0491	0,0518	0,0381
Na	% _{mas}	0,376	0,488	0,255	0,354	0,455	0,442
Ti	% _{mas}	0,70	0,40	0,73	0,61	0,62	0,37

Results of surface analysis were shown in table 6. Lowest values of specific surfaces and micropore volume were observed for sample 3a, due to its lower value of carbonaceous residue, which has strongly developed microporous structure. Interesting conclusion brings comparison of samples 1a and 2a. Despite lower content of organic matter in the ash 2a both the BET and Langmuir specific surfaces and the micropore volume is greater than in the ash 1a. Simultaneously the average pore diameter of the 2a sample is higher than in the sample 1a.

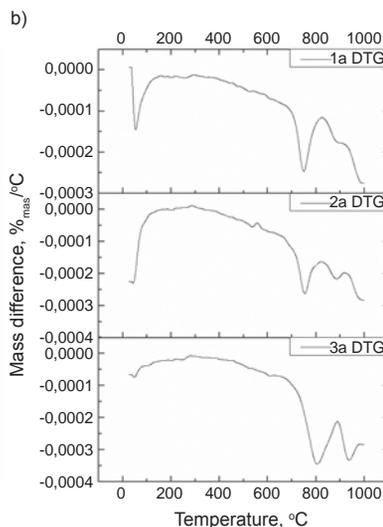
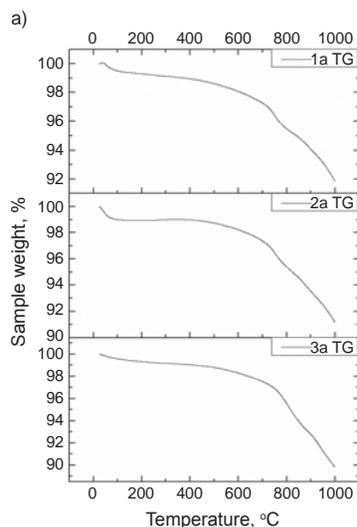


Fig. 4. Diagrams of thermal decomposition – TG (on the left) and differential thermal analysis – DTG (on the right) of fly ash samples

All these observations allow to conclude that oxy-combustion creates fly ash of more complex mesoporous structure than combustion in air. For better interpretation this dependence was illustrated in Figure 3.

Table 6

Results of analysis of surface area and porosity of fly ash

Parameter	BET specific surface area	Langmuir specific surface area	Micropore volume	Average pore diameter
Sample	m ² /g	m ² /g	cm ³ /g	Å
1a	53,0170	67,1504	0,010352	28,6886
2a	56,7012	71,9213	0,010680	34,2201
3a	18,8282	23,9701	0,002254	39,7728

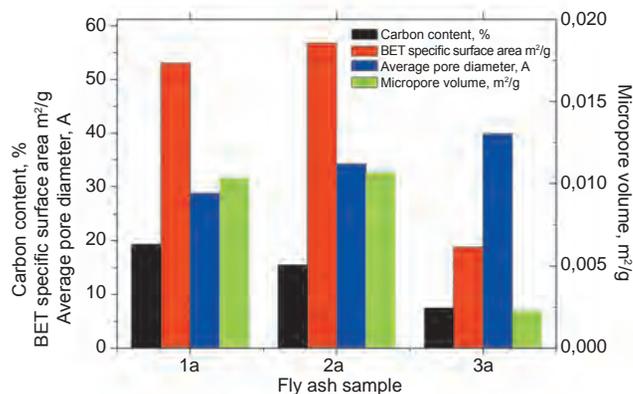


Fig. 3. Comparison of surface properties and carbon content of fly ash samples

Results of thermogravimetric analysis were illustrated in Figure 4. First observed weight loss for all samples were related to evaporation of adsorbed water molecules – it has lowest value for sample 3a, due to lower content of hygroscopic carbon. Continuously weight loss in the temperature range of 200 – 700 °C, was connected to degassing of volatiles and burnout of organic

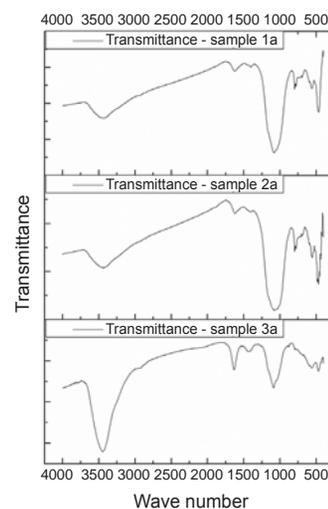


Fig. 5. FTIR spectra of fly ash samples

matter – as might be expected, this loss is lower for sample 3a too. For all three samples there appear two peaks: in the range of 750-800°C and around 900°C. They are related to loss of water associated with the hydrated calcium oxide and decomposition of calcium carbonate (according to equation: $\text{CaCO}_3 \rightarrow \text{CaO} + \text{CO}_2$) [29].

Diagrams of FTIR spectra of studied samples of fly ash was shown in Figure 5. Observed for all samples band of wave number 3450 cm^{-1} is related to hydroxyl groups, and this peak is clearest for sample 3a. In all ashes are peaks indicating the presence of quartz, these include peaks with numbers 1100 cm^{-1} and 792 cm^{-1} corresponding to a basic unit – tetrahedrons TO_4 (T = Si, Al). In addition, the peak of number 1100 cm^{-1} indicates asymmetric stress Si – O – Si and the peak of 792 cm^{-1} is linked to symmetric stress of Si – O – Si. Another visible for all samples band of number around 460 cm^{-1} is responsible for surface groups Si – O. Similarly, for all ash samples, could be seen small peak at wave number around 1636 cm^{-1} indicating adsorbed or associated water molecules. Vibrations in the vicinity of the wave number 694 cm^{-1} and peaks at 516 cm^{-1} also indicate the presence of silica formation: kaolinite, quartz and mullite.

Analysis was also made of grain distribution of fly ash. The results are shown in the graph in Figure 4. While the particle size distribution of samples of ash from the combustion of oxygen is similar, the Gauss distribution, when it compares them to the distribution of ash from the combustion of the air we note the following differences. Firstly, the diameter of the ash particles of sample 1a is strongly shifted towards the coarse grain. This indicates O_2/CO_2 atmosphere to promote the formation of a finer grain ash. Second, the ash 1a seeing more coarse particles with diameters between 200 and 500 microns.

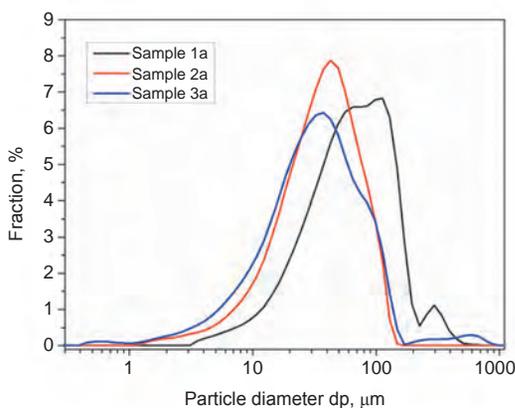


Fig. 6. Particle size distribution of fly ash samples

Conclusion

A series of comparative analyzes of ash from the combustion in oxygen-enriched atmosphere to the ash from the combustion in air were conducted. Chemical analysis revealed some differences. Analysis of unburned organic residue shows the efficiency of the combustion process and on the basis of the results conclude that the oxy-combustion can be more efficient than the conventional combustion of fuel. XRF analysis of the chemical composition showed a number of differences in the contents of

individual components, and the results of this analysis should be taken into account when selecting the particular route management of fly ash. Analysis of physical properties: TGA - DTG, surface area, loss of ignition, showed differences between the ashes, but they result mainly from the chemical composition, in particular the content of unburned carbon and they are not particularly surprising, especially after a careful analysis of the chemical composition. FTIR analysis indicates the similarity of aluminosilicate construction. An important difference between the samples of fly ash is particle size distribution. This analysis shows that in oxy-combustion process, we have to deal with the ashes of finer grain than the combustion of oxygen. All these results are only a prelude to more extensive physico-chemical properties of fly ash, which will be necessary to develop efficient processes of ash utilization.

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Karol Witkowski¹⁾, Sławomir Grądziel²⁾

Wpływ współspalania biomasy na stopień efektywności cieplnej rur RHI dla kotła OP-380

Influence of biomass co-combustion on the coefficient of an OP-380 boiler heating surfaces thermal efficiency

W kotle energetycznym w wyniku spalania paliw generowane są duże ilości popiołu oraz żużla. Ze względu na wysokie temperatury w rejonie komory paleniskowej oraz grodzi należy się tam spodziewać odkładania się żużla czy też osadów popio-

łowych. Na intensywność ich odkładania wpływa głównie rodzaj i jakość spalane go paliwa oraz budowa kotła i warunki przepływu w nim panujące. Osady w rejonie komory i grodzi ulegają stopieniu lub spiekaniu, w rejonie międzyciągu są one sypkie z tendencją do spiekania, drugi ciąg to w dużej mierze osady sypkie. Osadzony popiół na powierzchniach ogrzewalnych przegrzewaczy wpływa negatywnie na strumień ciepła przepływający od spalin do rur, zwiększa opory przepływu, co przyczynia się do wzrostu zużycia energii przez wentylator spalin. Wyżej wymienione czynniki

¹⁾ Mgr inż. Karol Witkowski, EDF Polska S.A., Pion Badań i Rozwoju; e-mail: Karol.Witkowski@edf.pl

²⁾ Dr hab. inż. Sławomir Grądziel, Politechnika Krakowska, Instytut Maszyn i Urządzeń Energetycznych; e-mail: gradziel@mech.pk.edu.pl