

# Properties of fly ash from thermal treatment of municipal sewage sludge in terms of EN 450-1

Łukasz Szarek\*, Małgorzata Wojtkowska

Warsaw University of Technology, Poland  
Faculty of Building Services, Hydro and Environmental Engineering

\*Corresponding author's e-mail: lukasz.szarek@is.pw.edu.pl

**Keywords:** fly ash from thermal treatment of municipal sewage sludge, chemical analysis, physical properties, EN 450-1, concrete additive, hardening slurry.

**Abstract:** Along with the increase in popularity of the sewage sludge thermal treatment methods in Poland resulting from the implementation of European Union law, a management problem with ash, which is produced as a result of this process, appeared. The paper analyses the chemical composition and physical properties of fly ash from thermal treatment of municipal sewage sludge in terms of its use in concrete technologies in relation to EN 450-1 *Fly ash for concrete. Definition, specifications and conformity criteria (2012)* and EN 197-1 *Cement. Composition, specifications and conformity criteria for common cements (2011)* standards. The tested material did not meet the requirements related to use of fly ash for concrete production (chemical composition, low activity index, high water demand and fineness), and as main and minor components for cement production. On the basis of the carried out research and analyses, it was found that the hardening slurry technology creates the greatest possibilities related to the management of fly ash from thermal treatment of municipal sewage sludge.

## Introduction

Along with the increase in popularity of the sewage sludge thermal treatment methods in the entire European Union, and especially in Poland, with about 1% of produced sewage sludge in 2008 to about 15% in 2014 (Falaciński et al. 2016), a management problem with fly ash, which is produced as a result of this process, appeared. The produced fly ash is characterised by specific features, which are unique among coal combustion by-products. The properties of fly ash from thermal treatment of municipal sewage sludge largely depend on the composition of sewage, treatment technology and the combustion process, similarly, as in the case of fly ash from coal combustion, in which a crucial impact on properties includes the type of minerals present in coal and the accompanying gangue (Lutze and vom Berg 2010), as well as the type of furnace, and combustion and ash removal conditions.

In the EU legislation, the sewage sludge management of the Council Directive of 21 May 1991 is in force (91/271/EEC 1991). In Poland, this directive is implemented by the National Programme of Urban Wastewater Treatment (NPUWT), and i.e. the Ordinance of the Minister of Economy (Journal of Laws of 2013, item 38), which has prevented sewage sludge disposal without its processing since 1 January 2016. In addition raw sewage sludge is a material which is rich in micro-organisms, including pathogenic bacterial species, such as: *Escherichia coli*, *Salmonella typhimurium* and *Proteus mirabilis* (Bień and Nowak 2014). This situation constitutes a field for the

use and dissemination of thermal methods in sewage sludge treatment (also co-combustion (Rajczyk et al. 2014)), and therefore, the intensity of ash production. According to the Circular Economy idea, in which the economy is treated as a closed circuit (near-zero-waste), finding the ways of waste management in order to make it a full-value product, ready for use, becomes necessary.

The fly ash produced in thermal processes can be used for concrete and cement production according to the EN 206 *Concrete standards. Specification, performance, production and conformity (2014)*, EN 450-1 *Fly ash for concrete. Definition, specifications and conformity criteria (2012)* and EN 197-1 *Cement. Composition, specifications and conformity criteria for common cements (2011)*. The European standard EN 206 (2014) defines an additive to concrete as a fine-grained component used to improve certain properties or to obtain special properties. This standard distinguishes two types of inorganic additives:

- Type I (almost neutral), they are most often pigments used to dye concrete or aggregate filler, e.g. limestone flour.
- Type II, with pozzolanic or latent hydraulic properties, the most popular additives of this type in construction which include fly ash and silica dust.

The EN 450-1 (2012) standard, with its scope, includes the chemical and physical requirements which should be met by silica fly ash (with or without the participation of co-combustion materials), used as an additive of II type in the concrete production (Table 1).

**Table 1.** Chemical and physical requirements for fly ash according to EN 450-1 (2012)

Chemical component/Physical property	Limit values for the single results	Permissible limit
	Permissible limit [% mass]	[% mass]
Chlorides (Cl)	≤ 0.10	≤ 0.10
Sulfuric anhydride as (SO <sub>3</sub> )	≤ 3.5	≤ 3.0
Free calcium oxide (CaO <sub>free</sub> )	≤ 1.6	≤ 2.5 EN 197-1 according to 450-1 for > 1.5 must meet soundness requirements
Reactive calcium oxide (CaO <sub>react</sub> )	≤ 11.0	≤ 10.0
Reactive silicon dioxide (SiO <sub>2react</sub> )	–	≥ 25
The sum of oxide content (SiO <sub>2</sub> , Al <sub>2</sub> O <sub>3</sub> , Fe <sub>2</sub> O <sub>3</sub> )	≥ 65	≥ 70
The total alkali content (Na <sub>2</sub> O <sub>eq</sub> )	≤ 5.5	≤ 5.0
Magnesium oxide (MgO)		≤ 4.0
Total phosphate (P <sub>2</sub> O <sub>5</sub> ) [mg/kg]	≤ 5.5	≤ 5.0
Total phosphate in the preliminary test [mg/kg]	–	≤ 100
Loss on ignition	cat.: A ≤ 7.0 B ≤ 9.0 C ≤ 11.0	cat.: A ≤ 5.0 B ≤ 7.0 C ≤ 9.0
Fineness*	cat. N ≤ 45.0 cat. S ≤ 13.0	cat. N ≤ 40.0 cat. S ≤ 12.0
Soundness [mm]	≤ 11	≤ 10
Activity index after 28 days of curing [%]	≥ 70	≥ 75
Initial setting time	≤ 2.25 times longer than the initial setting time of the paste made of comparative cement	≤ 2.0 times longer than the initial setting time of the paste made of comparative cement
Water demand [%]	≤ 97 (S category only)	≤ 95 (S category only)

\* – retained on 0,045 mm sieve according to EN 451-2 (1994)

Source: own elaboration based on EN 450-1 (2012)

The EN 197-1 (2011) standard defines the requirements for generally used cements and their components. The standard provisions determine the main and minor components of cement, and also impose conditions towards fly ash, including its origin, chemical composition, fineness and water demand.

The aim of the conducted research was to determine the chemical composition of fly ash from thermal treatment of municipal sewage sludge in accordance with the requirements of the EN 450-1 (2012) and EN 197-1 (2011) standards. The analysis was to determine the possibility of using fly ash in the concrete, where other coal combustion by-products have been successfully used for a long time.

## Subject of research

The subject of the research includes fly ash from thermal treatment of sewage sludge from a municipal sewage treatment plant equipped with a Sewage Sludge Thermal Treatment Station collected in multicyclon dust separator.

The fly ash production process is as follows. Dried (to 32% of dry mass by disc dryer) sewage sludge mixed with fats is injected into the fluidised bed (with a sand bed) using piston pumps. Screenings and sands travel to the bed by screw conveyor, and then are incinerated at (600÷920)°C. This temperature range allows for a reduction of the emissions of nitrogen compounds to the atmosphere. In case of the failure to achieve autothermal conditions, it is possible to supply extra fuel in the form of natural gas or biogas. Starting fuel is natural

gas. Sewage sludge stays in the combustion chamber for at least 2 seconds. Flue gas is directed to the three step Dry Flue-Gas Treatment System (1st step – multicyclon, 2nd step – bag filter, 3rd step – SCR System). By-products of combustion include: slag, ash and other by-products after the exhaust gas dry treatment.

Fly ash from three separate batches received from the Sewage Sludge Thermal Treatment Station collected in multicyclon dust separator was tested. Batch 1 was collected during the period of continuous operation of the Sewage Sludge Thermal Treatment Station at the turn of April and May 2015. Batch 2 was collected in similar conditions between September and October 2015. The composition of Batch 3 included fly ash produced by the Sewage Sludge Thermal Treatment Station in the first half of November 2015. Such a frequency of sampling was to enable the assessment of variability of the chemical and physical parameters of the concerned material. Fly ash was stored in sealed plastic containers.

## Research methodology

The studies of the chemical composition and physical properties of fly ash were carried out in the laboratories of the Faculty of Building Services, Hydro and Environmental Engineering of the Warsaw University of Technology. For the purposes of indication, samples of fly ash with a weight dependent on the test procedure were used. Table 2 presents a collective summary of indicators, along with a reference to

**Table 2.** Indicators of the analysed samples

Examination	Test method / standard
Chlorides (Cl <sup>-</sup> )	EN 196-2 (2005)
Sulfuric anhydride as (SO <sub>3</sub> )	EN 196-2 (2005)
Free calcium oxide (CaO <sub>free</sub> )	EN 451-1 (2004)
Reactive calcium oxide (CaO <sub>react</sub> )	EN 197-1 (2011)
Total calcium oxide (CaO)	EN 196-2 (2005) – using EDTA
Reactive silicon dioxide (SiO <sub>2react</sub> )	EN 197-1 (2011)
The residue insoluble in hydrochloric acid and potassium hydroxide	EN 196-2 (2005)
Total silicon dioxide (SiO <sub>2</sub> )	EN 196-2 (2005)
Aluminium oxide (Al <sub>2</sub> O <sub>3</sub> )	EN 196-2 (2005) – using EDTA
Iron oxide (Fe <sub>2</sub> O <sub>3</sub> )	EN 196-2 (2005) – using EDTA
The sum of oxide content (SiO <sub>2</sub> , Al <sub>2</sub> O <sub>3</sub> , Fe <sub>2</sub> O <sub>3</sub> )	EN 196-2 (2005)
The total alkali content (Na <sub>2</sub> O <sub>eq</sub> )	Flame emission spectroscopy FES ISO 9964 (1993)
Magnesium oxide (MgO)	EN 196-2 (2005) – using EDTA
Total phosphate (P <sub>2</sub> O <sub>5</sub> )	EN 450-1 (2012)
Carbon dioxide (CO <sub>2</sub> )	EN 196-2 (2005) – alternative method
Loss on ignition	EN 196-2 (2005)
Fineness	EN 451-2 (1994)
Activity index	EN 450-1 (2012)
Soundness	EN 196-3 (2011)
Particle density	EN 1097-7 (2008)
Initial setting time	EN 196-3 (2011)
Water demand	EN 450-1 (2012)

Source: own elaboration

the standards/methods according to which the analyses were carried out. The analyses were conducted separately for three fly ash batches.

Fly ash decomposition with sodium peroxide was conducted. The indication of the iron oxide, magnesium oxide, aluminium oxide and calcium oxide content made by a versenate method with the use of EDTA as a titrant reagent was possible thanks to the manganese oxide content lower than the limit one EN 450-1 (2012). To evaluate reactive calcium oxide (CaO<sub>react</sub>) content the total calcium oxide content was reduced by the corresponding fraction to calcium carbonate (CaCO<sub>3</sub>), based on the measured carbon dioxide (CO<sub>2</sub>) content, and the fraction corresponding to calcium sulfate (CaSO<sub>4</sub>), based on the sulfate (SO<sub>3</sub>) content after substrate of SO<sub>3</sub> taken up by alkalis.

The quantity of reactive silicon dioxide (SiO<sub>2react</sub>) is determined by subtracting from the total silicon dioxide content that fraction contained in the residue insoluble in hydrochloric acid and potassium hydroxide, both on a dry basis.

Pastes that are necessary for marking the initial setting time and soundness were made after mixing the fly ash, cement CEM I 42.5R and tap water at (20 ± 2)°C in accordance with EN 196-3 (2008) and EN 450-1 (2012). In order to carry out the strength and water demand tests, mortars were prepared with use of fly ash, cement CEM I 42.5R and tap water at (20 ± 2)°C in accordance with EN 450-1 (2012), to which standard sand complying with EN 196-1 (2005) was added. Cement used in experiment was CEM I 42.5R Lafarge Specjal. Initial setting time for paste based on the cement was 200 min. Compressive

strength for mortar based on the cement was 55.7 MPa after 28 days of curing.

## Research results

In Table 3, the results of indication of chemical and physical properties for three different batches of fly ash from thermal treatment of municipal sewage sludge are demonstrated.

### Analysis of the research results in the light of requirements of the EN 450-1 standard (2012)

The research results (Table 3) are referred to the requirements of the EN 450-1 (2012) standard (Table 1). The analysis of the results included the limit values for a single result, as well as the permissible value treated as a characteristic value for a given parameter.

#### Chemical properties

In the fly ash samples, the content of chlorides (Cl<sup>-</sup>) was indicated. Chlorides in concrete affect the form of a matrix of hydrated silicates of calcium (C–S–H phase) and reduction of pH, which, in turn, may cause corrosion of reinforcement. In each batch, the share of chlorides is similar (on average 0.03% mass), by almost an order of magnitude lower than the limit value (0.1% mass), both in relation to a single result and a characteristic value.

**Table 3.** The results of the research of chemical and physical properties of fly ash from thermal treatment of municipal sewage sludge

Chemical component/Physical property	Batch 1	Batch 2	Batch 3	Average
	[% mass]			
Chlorides (Cl)	0.04	0.03	0.03	0.03
Sulfuric anhydride as (SO <sub>3</sub> )	2.78	2.62	2.54	2.65
Free calcium oxide (CaO <sub>free</sub> )	0.12	0.26	0.65	0.34
Reactive calcium oxide (CaO <sub>react</sub> )	10.49	12.13	13.53	12.05
Total calcium oxide (CaO)	13.20	14.80	16.30	14.80
Reactive silicon dioxide (SiO <sub>2react</sub> )	9.10	15.50	19.95	14.85
Total silicon dioxide (SiO <sub>2</sub> )	36.40	41.40	39.0	38.90
The sum of oxide content (SiO <sub>2</sub> , Al <sub>2</sub> O <sub>3</sub> , Fe <sub>2</sub> O <sub>3</sub> )	60.18	64.90	63.24	62.77
The total alkali content (Na <sub>2</sub> O <sub>eq</sub> )	4.20	4.42	5.40	4.67
Magnesium oxide (MgO)	4.15	4.06	3.13	3.78
Total phosphate (P <sub>2</sub> O <sub>5</sub> ) [mg/kg]	5.50	6.18	6.18	5.95
Loss on ignition (Category)	2.09 (A)	2.09 (A)	2.49 (A)	2.22 (A)
Fineness	62.50	58.20	47.00	55.90
Soundness [mm]	0.1	0.6	1.1	0.6
Compressive strength [MPa]	30.1	37.0	34.0	33.7
Activity index after 28 days of curing (w/b* = 0,5) [%]	54.0	66.5	61.1	60.5
Initial setting time (w/b*)	2.03 (0.38)	2.05 (0.38)	2.00 (0.40)	2.03 (0.39)
Water demand [%]	129	131	132	131
Particle density [Mg/m <sup>3</sup> ]	2264	2293	2358	2305

\* – water to binder ratio

Source: own elaboration

Likewise, in the case of chlorides, the content of sulfuric anhydrides (indicated as SO<sub>3</sub>) in considered fly ash is similar (on average 2.65% mass). All the samples fulfilled the EN 450-1 standard (2012), both in relation to a single result (the upper limit value of 3.5% mass) and a characteristic value (the upper limit value of 3.0% mass) related to fly ash for concrete. The content of SO<sub>3</sub> above 3% of cement may result in sulphate corrosion (Eglinton 1987), however (Garcés et al. 2008) demonstrates that sulphates present in fly ash from thermal treatment of municipal sewage sludge are not reactive towards cement.

Another indicator that is analysed in samples was the content of free calcium oxide (CaO). The variability of the content of free CaO in fly ash (Batch 1 – 0.12% mass; Batch 2 – 0.26% mass; Batch 3 – 0.65% mass) can be considered small. All the samples fulfilled the requirements of the standard (the upper limit value for a single result – 1.6% mass, for a characteristic value – 2.5% mass). Due to the low free calcium oxide content in fly ash (on average 0.34% mass), it was not necessary to indicate the volume constancy. The average content of total calcium oxide was 14.80% mass. The variability of the content of total CaO in fly ash (Batch 1 – 13.2% mass; Batch 2 – 14.8% mass; Batch 3 – 16.3% mass) can be considered small.

The requirements of the reactive calcium oxide content (the upper limit value for a single result of 11% mass), which can form the C–S–H phase, were fulfilled only by fly ash from Batch 1 (10.49% mass), and the characteristic value

(≤ 10% mass) was exceeded only by 5% (in relation to the permissible value). Other fly ash did not meet the requirements. The average value of reactive calcium oxide for the material (12.05% mass) also exceeded the permissible value. The increased reactive calcium oxide content may show the fly ash hydraulic properties.

None of the tested batches of fly ash (Batch 1 – 9.10% mass; Batch 2 – 15.5% mass; Batch 3 – 19.95% mass) meets the requirements of the standard related to the reactive silicon dioxide content (the lower limit content of 25% mass), which is involved in the pozzolanic reaction in concrete. The results are characterised by the variability of up to 100%. The average content of total silicon oxide was 38.90% mass. The variability of the content of total SiO<sub>2</sub> in fly ash (Batch 1 – 36.4% mass; Batch 2 – 41.4% mass; Batch 3 – 39.0% mass) can be considered small.

The requirement related to a sum of the content of SiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, and Al<sub>2</sub>O<sub>3</sub> oxides (the lower limit value for a single result of 65% mass, 70% mass for the characteristic value), which in addition to CaO, are main components of cement, were not met by any of three batches of fly ash (Batch 1 – 60.18% mass; Batch 2 – 64.90% mass; Batch 3 – 63.24% mass). The tested samples are characterised by low dispersion of results.

The requirements related to the total alkali content are important because of the possibility of their reaction with opal silica in aggregates, which can lead to swelling and cracking of concrete (Hewlett 2003). The ranges determined in the standard (the upper limit value for a single result of 5.5% mass, 5% mass

for the characteristic content) were not met for the characteristic value only by fly ash from Batch 3 (5.40% mass). Taking into account the average value of the parameter for three batches considered together (4.67% mass), the requirement should be regarded as met. Fly ash from all batches is characterised by the similar content of alkali (Batch 1 – 4.20% mass; Batch 2 – 4.42% mass; Batch 3 – 5.40% mass).

Only fly ash from Batch 3 (3.13% mass) met the standard requirements related to the magnesium oxide content (permissible content  $\leq 4.0\%$  mass). The remaining samples slightly exceeded the permissible value (Batch 1 by 4%; Batch 2 by 1.5% in relation to the threshold). However, taking into account the average value of the magnesium oxide for three batches considered together (3.78% mass), the requirement should be regarded as met. The magnesium oxide content (along with free calcium oxide) in the cement components is important due to the possibility of the concrete swelling.

In the fly ash samples, an analysis of the phosphate content was carried out. Too high phosphorus content deteriorates the clinker quality, decomposing alite ( $C_3S$ ). Furthermore, it has a negative impact on the cement hydration process delay (De Noirfontaine et al. 2009). The requirements for this parameter ( $\leq 5.0\%$  mass) were met by all samples. Moreover, each of the three batches of fly ash (Batch 1 – 5.50 mg/kg; Batch 2 – 6.18 mg/kg; Batch 3 – 6.18 mg/kg) met the requirements related to preliminary tests ( $\leq 100$  mg/kg).

Each of the tested fly ash batches is characterised by a low loss of ignition (Batch 1 – 2.09% mass; Batch 2 – 2.09% mass; Batch 3 – 2.49% mass), equivalent to A category (Table 1). It gives hope to the possibility of cooperation between fly ash and airing admixtures. The low variability of results proves a stable combustion process of municipal sewage sludge.

According to (Lin and Lin 2005), the chemical composition of fly ash from thermal treatment of municipal sewage sludge does not negatively affect the hydration process of cements (especially after 90 days of curing), in which fly ash constitutes up to 20% of the binder. However, it should be taken into account that the material, which was considered in the paper, was different in terms of the silicon dioxide content from the one analysed in tests (Lin and Lin 2005), and the binder mixture was specially prepared (incinerated together in the furnace, rapidly cooled, then milled in the ball mill). In addition, a significant impact of changes in the content of individual oxides on the content of minerals in the clinker, and consequently in cement, is commonly known (Neville 2000). The results suggest the need for further research, in order to ensure the quality of the tested material.

### Physical properties

The requirements on fineness (cat. N  $\leq 40.0\%$  mass cat. S  $\leq 12.0\%$  mass) according to the EN 450-1 standard (2012) were not met by any of the tested samples of fly ash (Batch 1 – 62.50% mass; Batch 2 – 58.20% mass; Batch 3 – 47.00% mass) even for N category (Table 1). This implies a negative impact of the fly ash additive on the concrete mixture workability.

The free calcium oxide content indicates that fly ash from thermal treatment of municipal sewage sludge should not negatively affect the concrete swelling, however, due to the exceeded magnesium oxide contents for Batches 1 and 2, the indication of the volume constancy of fly ash pastes was

made in the paper. All the pastes (Batch 1 – 0.1 mm; Batch 2 – 0.6 mm; Batch 3 – 1.1 mm – values correlate with a content of free CaO) met the requirements of the EN 450-1 (2012) ( $\leq 10$  mm) standard. The composition of tested fly ash (mainly free lime, magnesium oxide, sulfuric anhydrides (Neville 2000)) does not negatively affect the paste swelling.

The requirements of the EN 450-1 (2012) standard related to an activity index after 28 days of curing (the lower limit value for a single result of 70% of the compressive strength of samples from comparative mortar, 75% for the characteristic value) were not met by any of mortars made on the basis of fly ash from thermal treatment of municipal sewage sludge (Batch 1 – 54.0%; Batch 2 – 66.5%; Batch 3 – 61.1%). The compressed fly ash samples obtained much lower strength values (Batch 1 – 30.1 MPa; Batch 2 – 37.0 MPa; Batch 3 – 34.0 MPa) than reference samples made only of comparative cement CEM I 42,5R (55.7 MPa). Despite the reactive calcium oxide content above 10%, the tests showed a low hydraulic and pozzolanic potential of ash.

Among the tested ash pastes, only the one made on the basis of fly ash from Batch 3 (2.0 times longer than the initial setting time of the paste made of comparative cement) met the standard requirement related to the initial setting time for characteristic values ( $\leq 2,0$ ). The requirements for a single result ( $\leq 2.25$ ) were met by all kinds of fly ash (Batch 1 – 2.03; Batch 2 – 2.05). Longer binding time may positively affect the concrete workability.

Although the requirements on water demand are used only for fly ash of S category (Table 1), the obtained results of pastes made on the basis of fly ash from thermal treatment of municipal sewage sludge were compared in order to identify the material. All the considered mortars (Batch 1 – 129%; Batch 2 – 131%; Batch 3 – 132%) showed similar values of this parameter and did not meet the requirements ( $\leq 95\%$ ) included in EN 450-1 (2012). Water demand demonstrates the high impact of fly ash on the mortar consistency which contributes to the concrete workability. The largest water demand of mortar on the basis of fly ash from Batch 3 correlates with fineness and the amount of water required to obtain the standard consistency when testing the setting time (the highest w/b ratio among three batches).

The EN 450-1 (2012) standard does not pose requirements to the fly ash density, however, it imposes certain requirements on manufacturers of fly ash (density should not differ more than 200 kg/m<sup>3</sup> from its declared value). All three batches of fly ash oscillate around the mean value (2305 Mg/m<sup>3</sup>) in the range of about 2%. These are the values similar to the density of fly ash found in the literature, e.g. (Blanco et al. 2005, Berndt 2009).

According to the definition itself, fly ash from thermal treatment of municipal sewage sludge does not fulfil the assumptions of the EN 450-1 standard (2012). A similar situation occurs in the case of fly ash from lignite coal combustion (too large content of free calcium oxide (Ilic et al. 2003) and reactive calcium oxide (Giergiczny et al. 2014)), however, it is widely used as an additive in various types of binders and concrete mixtures under technical approvals (Feuerborn 2011). The provisions of the EN 197-1 standard (2011) disqualify fly ash from thermal treatment of municipal sewage sludge as main and minor components of cement due to the origin (furnace not fired with coal dust), and also because of fineness and high water demand (Table 3).

## Conclusion

The discussed research results allow to make the following conclusions:

- 1) The variability of the chemical composition and physical properties of fly ash from thermal treatment of sewage sludge is assessed as insignificant. In the future, further reduction of the composition variability of fly ash will be possible as a result of using the methods of its treatment and if ash is not treated as waste no longer and starts to be considered as a full-value combustion by-product. Such an effect in the case of calcareous fly ash from Bełchatów Power Plant (Poland), which thanks to treatment or use and the related control of properties, reached the stability of physical and chemical parameters (Baran and Drożdż 2013).
- 2) Fly ash from thermal treatment of sewage sludge does not meet the requirements of the EN 450-1 (2012) standard, not only by definition, but also due to physical and chemical properties (exceeded thresholds of the chemical composition, low activity and high fineness and water demand). This prevents from using the material as an additive of II type for concrete. In order to employ such ash in concrete technology, it will be necessary to obtain the European technical approvals.
- 3) According to EN 450-1 (2012) there is a possibility to achieve fly ash from the co-combustion of sewage sludge with coal dust in appropriate conditions. However it cannot be done within waste water treatment plant, where fluidized bed is considered as BAT (Best Available Technology).
- 4) Due to its properties (high water demand and fineness) and its production method, fly ash from the thermal treatment of municipal sewage sludge cannot be used as main and minor component in cement production. It is possible to use it as an additive, which will reduce the consumption of natural resources.
- 5) Due to the chemical composition of fly ash from thermal treatment of municipal sewage sludge, further research on the phase structure of the material and on hardened composites produced on its basis is needed.
- 6) The hardening slurry technology (which does not include guidelines in the form of the European standards) is not so demanding in respect to fly ash as the concrete technology. Despite this, fly ash is successfully used in hardening slurry technology (Klędyński 2004, Falaciński 2011). The conditions that are posed by it mainly refer to the slurry composition, which defines a specific application. This results from the way of performance and the nature of operation in the liquid form and after hardening. The relatively large water demand of tested fly ash does not adversely affect the properties of slurries by reason of the volume majority of water. The influence of water demand on the frost resistance is negligible. It results from the fact that slurries are not frost resistant, and the structures made of them (mostly in the ground) are protected from frost (Klędyński and Rafalski 2009). Taking into account the chemical composition and physical properties of fly ash from the thermal treatment of municipal sewage sludge, and especially its high water

demand, the most appropriate direction of the material management includes its use in hardening slurries. In this sector of construction binders, there are no such strict limits related to the w/b ratio as in the concrete technology. Work connected with this way of using tested fly ash is already in progress (Falaciński et al. 2016, Falaciński and Szarek 2016).

## Acknowledgement

Funding: This work was supported by the Dean of the Faculty of Building Services, Hydro and Environmental Engineering of the Warsaw University of Technology [grant number 504/02102 titled *Fly ash from thermal treatment of sewage sludge and the PN-EN 450-1:2012 standard*].

## References

- EN 1097-7:2008. (2008). Tests for mechanical and physical properties of aggregates. Determination of the particle density of filler. Pycnometer method.
- EN 196-1:2005. (2005). Methods of testing cement. Determination of strength.
- EN 196-2:2005. (2005). Methods of testing cement. Chemical analysis of cement.
- EN 196-3+A1:2008. (2008). Methods of testing cement. Determination of setting times and soundness.
- EN 197-1:2011. (2011). Cement. Composition, specifications and conformity criteria for common cements.
- EN 206:2014. (2014). Concrete. Specification, performance, production and conformity.
- EN 450-1:2012. (2012). Fly ash for concrete. Definition, specifications and conformity criteria.
- EN 451-1:2003. (2004). Method of testing fly ash. Determination of free calcium oxide content.
- EN 451-2:1994. (1994). Method of testing fly ash. Determination of fineness by wet sieving.
- ISO 9964-3:1993. (1993). Water quality – Determination of sodium and potassium – Part 3: Determination of sodium and potassium by flame emission spectrometry.
- 91/271/EEC. (1991). Council Directive 91/271/EEC of 21 May 1991 Concerning urban waste-water treatment.
- Journal of Laws of 2013 item 38 Regulation of the Minister of Economy of 8th January 2013 On the criteria and procedures for acceptance of waste for landfilling of waste that type of. (in Polish)
- Baran, T. & Drożdż, W. (2013). Evaluation of properties of domestic calcareous fly ash and its processing methods, *Roads and Bridges – Drogi i Mosty*, 12, 1, pp. 5–15.
- Berndt, M.L. (2009). Properties of sustainable concrete containing fly ash, slag and recycled concrete aggregate, *Construction and building materials*, 23, 7, pp. 2606–2613. Doi: 10.1016/j.conbuildmat.2009.02.011
- Bień, J. & Nowak, D. (2014). Biological composition of sewage sludge in the aspect of threats to the natural environment, *Archives of Environmental Protection*, 40(4), pp. 79–86.
- Blanco, F. Garcia, M.P. & Ayala, J. (2005). Variation in fly ash properties with milling and acid leaching, *Fuel*, 84, 1, pp. 89–96. Doi: 10.1016/j.fuel.2004.05.010
- De Noirfontaine, M.N. Tusseau-Nenez, S. Signes-Frehel, M. Gasecki, G. & Girod-Labianca, C. (2009). Effect of phosphorus impurity on tricalcium silicate T1: from synthesis to structural characterization, *Journal of the American Ceramic Society*, 92, 10, pp. 2337–2344. Doi:10.1111/j.1551-2916.2009.03092.x
- Eglinton, M.S. (1987). *Concrete and its chemical behaviour*, Thomas Telford Ltd, London 1987.

- Falaciński, P. (2011). Leak tightness of hardening slurries with fluidal fly ashes in chemically aggressive environments, *Archives of Environmental Protection*, 37, 1, pp. 115–134.
- Falaciński, P. Hejko, W. & Jakubowicz, M. (2016) Ash from thermal treatment of municipal sewage sludge as additive to hardening slurries, *Materiały Budowlane*, 523, 3, pp. 46–49. (in Polish) Doi: 10.15199/33.2016.03.14
- Falaciński, P. & Szarek, Ł. (2016). Possible applications of hardening slurries with fly ash from thermal treatment of municipal sewage sludge in environmental protection structures, *Archives of Hydro-Engineering and Environmental Mechanics*, 63, 1, pp. 47–61. Doi: 10.1515/heem-2016-0004
- Feuerborn, H.J. (2011). Coal combustion products in Europe – An update on production and utilization, standardisation and regulation, In: *World Coal Ash Conference*, Denver, pp. 9–12.
- Garcés, P., Pérez Carrión, M. García-Alcocelb, E. Payác, J. Monzó, J. & Borrachero, M.V. (2008). Mechanical and physical properties of cement blended with sewage sludge ash, *Waste Management*, 28, 12, pp. 2495–2502. Doi: 10.1016/j.wasman.2008.02.019
- Giergiczny, Z., Dziuk, D. Puzak, T. & Batog, M. (2014). Practical use of calcareous fly ash in portland-composite cement CEM II/B-M (V-W) 32,5R industrial production, *International Journal of Research in Engineering and Technology*, Special Issue 13(03), pp. 195–202. DOI: 10.15623/ijret.2014.0325031
- Hewlett, P. (2003). *Lea's chemistry of cement and concrete*, Butterworth-Heinemann, 4th edition 2003.
- Ilic, M., Cheeseman, C. Sollars, C. & Knight, J. (2003). Mineralogy and microstructure of sintered lignite coal fly ash, *Fuel*, 82, 3, pp. 331–336. Doi: 10.1016/S0016-2361(02)00272-7
- Kledyński, Z. (2004). Influence of fly ashes on hardening slurries resistance to sulphate attack, *Archives of Hydro-Engineering and Environmental Mechanics*, 51, 2, pp. 119–133.
- Kledyński, Z. & Rafalski, L. (2009). *Hardening slurries*, Committee of Civil and Water Engineering Polish Academy of Science, Warsaw 2009. (in Polish)
- Lin, K.L. & Lin, C.Y. (2005). Hydration characteristics of waste sludge ash utilized as raw cement material, *Cement Concrete Research*, 35, 10, pp. 1999–2007.
- Lutze, D. & vom Berg, W. (2010). *Handbook on fly ash in concrete*, Bau+Technik GmbH, Essen 2010.
- Neville, A.M. (2000). *Properties of concrete*, Pearson Education Limited, Harlow 2000.
- Rajczyk, R. Bień, J. Palka, H. Pogodziński, A. & Smorąg, H. (2014). Co-combustion of municipal sewage sludge and hard coal on fluidized bed boiler WF-6, *Archives of Environmental Protection*, 40(3), pp. 101–113.

## Właściwości popiołów lotnych z termicznego przekształcania komunalnych osadów ściekowych w świetle wymagań normy EN 450-1

**Streszczenie:** W pracy przeanalizowano skład chemiczny i właściwości fizyczne popiołu lotnego z termicznego przekształcania komunalnych osadów ściekowych w zakresie stosowania go w technologii betonu w odniesieniu do wymagań zawartych w normie EN 450-1 Popiół lotny do betonu (2012) oraz EN 197-1 Cement (2011). Przedmiot badań stanowił popiół lotny z termicznego przekształcania komunalnych osadów ściekowych, pochodzący z miejskiej oczyszczalni ścieków, wyposażonej w Stację Termicznej Utylizacji Osadów Ściekowych. Przeprowadzono badania popiołu pod względem składu chemicznego oraz właściwości fizycznych zgodnie z wytycznymi normy EN 450-1 (2012) i EN 197-1 (2011). Badany materiał nie spełnił wymagań stawianych głównym i drugorzędny składnikom cementu oraz popiołom lotnym do produkcji betonu (skład chemiczny, niski indeks aktywności, wysoka wodożądność i mialkość). Na podstawie przeprowadzonych badań i analiz stwierdzono, iż technologia zawieszin twardniejących stwarza największe możliwości wykorzystania popiołu lotnego z termicznego przekształcania komunalnych osadów ściekowych.