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Assessment of Fly Ash from Thermal Treatment of Sewage Sludge According to the Applicable Standards

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ABSTRACT

The restrictions on carbon dioxide emissions introduced by the European Union encourage experimental work on new generation materials containing smaller amounts of clinker. Currently, silica fly ash from the combustion of hard coal is widely used in cement and concrete technology in Europe and in Poland. Their wide application is mainly determined by their chemical and phase composition, especially pozzolanic activity, their high fineness, similar to cement. The aim of the research was to assess the properties of fly ash from thermal treatment of sewage sludge in terms of use in concrete technology in relation to EN 450-1, ASTM-C618-03 and ASTM C379-65T. The obtained test results confirm that the tested material has a different physicochemical composition and does not meet the requirements related to the use of ash in the production of concrete. In addition, the research showed the possibility of producing ordinary concrete, modified with fly ash from thermal treatment of sewage sludge. The average compressive strength for concrete containing 15% of ash from Cracow was set at 48.1 MPa and 49.2 MPa after 28 and 56 days of maturation, for ash from Warsaw at 42.0 MPa and 45.1 MPa, and for ash from Łódź at 36.2 MPa and 36.2 MPa. The determined concentrations of heavy metals are below the maximum values to be met when discharging waste water into the ground or water, the leaching limits required for accepting inert waste for disposal and for substances particularly harmful to the aquatic environment. On this basis, it was found that the migration of heavy metals from concretes with ash addition to the aquatic environment is insignificant and should not be a significant problem.

Keywords: fly ash from sewage sludge, physical and chemical properties, concrete, compressive strength, heavy metals.

INTRODUCTION

In recent years, the aim of the European Union's policy has been to protect the environment and minimize the risks to human health, climate and biodiversity. With the European Green Deal, Europe is set to become the first climate neutral continent by using greener energy sources and technologies in industry. According to the document of the European Environment Agency - EEA-EIONET strategy for 2021-2030 - one of the five main goals to be achieved at this time is to support the implementation of the circular economy plan adopted by the European Commission (European Environment Agency, 2021). In the construction sector, this concerns the acquisition of electricity and the proper management of the production of building materials. According to published data, this sector is responsible for 39% of CO_2 emissionsand 36% of energy consumption, of which 11% is related to the production of building materials (International Energy Agency, 2019).

Taking into account these data, it becomes reasonable to search for innovative building materials with the lowest possible carbon footprint and to modify the already used materials so that they contribute to the improvement of the natural environment. Human activity, to a greater or lesser extent, affects the natural environment around us. The growing public awareness of the need to protect the natural environment concerns various areas of life. Taking into account the changes in environmental regulations, the energy sector was faced with challenges aimed at meeting the growing emission requirements. From January 1, 2016, the emission of nitrogen oxides, sulfur and dust was limited (Dz.U. 2015 poz. 1277). An important issue for the construction industry is that concrete - as a construction material, should become a more environmentally friendly composite. In the composition of the concrete mix, its two basic components, cement and aggregate, lead to anthropopressive effects at the stage of their acquisition and production There are already areas in Poland and in the world where obtaining good quality materials for the production of cement is a problem. In addition, the world economy each year for the production of concrete requires more and more cement (responsible for 6-8% of the total anthropogenic emission), for which no comparable substitute has been found to date (International Statistics Yearbook 2020). In Poland and in European countries, ashes from hard coal combustion are used in the technology of concrete and cement production. Their wide application is mainly determined by their chemical and phase composition, especially pozzolanic activity, their high fineness, similar to cement (Deja &Antosiak, 2012, Giergiczny, 2013, Gupta, 2007). Use of siliceous fly ash for the production of concreteit is possible after meeting the requirements of EN 450-1: 2012(EN 450-1: 2012).

Table 1. Requirements for physical and chemical properties of fly ash according to EN 450-1

Marked component	Limit values for a single result according to EN 450 -1: 2012
Properties chemical	
Loss on ignition	7% category A ; 9% category B.
SiO_2	-
Fe_2O_3	-
Al_2O_3	-
Total 2-4	In total, min. 65%
CaO	-
MgO	max. 4.0%
TiO_2	-
SO_3	max. 3.5%
On_2O	max. 5%
K_2O	-
P_2O_5	100 mg / kg
Cl^-	≤0.10%
Properties physical	
Shallowness	Cat. N <40%, cat. S <12%
Activity index after 28 and 90 days	75% 85%
Water lust for cat. S.	<95%
Density volumetric [kg/m^3]	Maximum difference of ± 200 kg/m^3 in relation to the value declared by the manufacturer, determined in accordance with EN 196-6

Due to the development of new generation concretes, the production of concrete mixtures with additives has increased over the last several decades. The EN 206 + A2: 2021-08 standard "Concrete - requirements, properties, production and compliance" defines an additive to concrete as "*fine-grained inorganic material with pozzolanic or latent hydraulic properties, which can be added to concrete to improve certain properties or the achievement of special properties*" (EN 206 + A2: 2021-08).Added in an amount greater than 5% by weight of cement. The standard distinguishes between:

- type I additives - these are almost inert additives, including pigments and mineral fillers, primarily stone flour - quartz, lime.
- type II additives - these are additives with pozzolanic or latent hydraulic properties, which include mainly fly ash (silica and lime), silica dust and blast furnace slag.

Typical silica ash and silica dust are pozzolanic additives, while limestone ash and ground blast furnace slag are additives with latent hydraulic properties. These substances contain a certain amount of amorphous, fine-grained silica.

The requirements for the chemical and physical properties of the ash used for concrete are specified in EN 450-1 and the technical rules for production control are given in EN 450-2 - Table 1 (EN 450-1: 2012,EN 450-2:2006)

So far, there is no European Standard on limestone ash as an additive to concrete, while in the USA the requirements are included in ASTM C618-03 - Table 2 (ASTM C618-03).

An important aspect of environmental protection is the proper management of waste generated in sewage treatment plants, the number of which is growing every year. Along with the growing

Table 2. Requirements for limestone fly ash according to ASTM C618-03

Composition chemical - acceptable content [%]	
CaO total	≥ 10.0
Sum of $SiO_2 + Al_2O_3 + Fe_2O_3$	≥ 50.0
SO_3	≤ 5.0
Moisture	≤ 3.0
Loss on ignition	≤ 6.0

costs of waste storage, specialized, environmentally friendly forms of disposal are sought, which would contribute to reducing the costs of storage, and thus lowering the prices of manufactured products. One of the forms of waste disposal is their reuse. In the case of post-production waste, it is possible in the production processes of building materials. Municipal wastewater is primarily a mixture of domestic and industrial wastewater fed with rainwater. The quality and quantity of generated wastewater depends on the living conditions of the population, the amount of water used, the technical condition and the type of sewage system. They are subject to changes in the annual, monthly, weekly and daily cycle.

The regulations in force in Poland treat the problem of storing sewage sludge in landfills more and more severely, introducing a ban on the storage of waste with a calorific value above 6 MJ/kg of dry matter (Dz.U. 2015 poz. 1277) developed on the basis of the directive(WE/2003/33).Additionally, taking into account the ban on the storage of sludge from January 1, 2016, their management is a technical problem, economic and ecological due to sanitary risk, hydration and high weight (Dz.U. 2015 poz. 1277, WE/2003/33).According to the Municipal Waste Act (Dz.U. 2013, poz.21), sewage sludge may be used if it is stabilized according to the purpose and method of its application. The generated sewage sludge was then deposited in landfills or released to the environment after its initial stabilization. Stabilization is carried out in order to eliminate the risk to human health and the environment and to reduce the susceptibility of sewage sludge to rotting by chemical, biological or thermal treatment (Sadecka et al., 2011, Bień et al., 2011, Środa et al., 2012).

Wastewater treatment takes place in several stages:

1. Mechanical cleaning - 1st degree of cleaning. This process consists in removing solid contaminants (dragged and floating), sand, fats and easily settling suspensions from sewage. The sewage flowing into the treatment plant mixes in the hermetized collecting chamber, and then flows through grates, sand traps, preliminary settling tanks to the intermediate pump.
2. Biological treatment - II stage of treatment. After leaving the primary settling tanks, the sewage goes to 10 biological technological lines. In this process, there is a continuous growth of microorganisms. Thickened excess and initial sludge are sent to fermentation chambers, where biological decomposition of the organic part of the sludge takes place under anaerobic conditions (stabilization of sewage sludge) (www.mpwik.com.pl).

Stabilized sludge is sent to the Thermal Sewage Sludge Treatment Station (STUOŚ). The main product of the sewage treatment plant, in addition to technological waste, i.e. sand, fats and screenings, is sewage sludge. STUOŚ uses the most efficient fluidized bed combustion technology in the world. Thanks to it, not only sewage sludge can be thermally neutralized, but also sand, grease and screenings. Before the sludge goes to fluidized bed furnaces, it is dried in disc dryers to 32% of dry weight. Dried and dried sludge and fats into the fluidized bed are introduced by means of piston pumps. On the other hand, crushed screenings and sand are introduced over the sand bed by means of screw conveyors. In the fluidized bed furnace there is a chamber into which air is introduced at a temperature of 570-650 °C. This air keeps the sand layer, heated to 750 °C, in constant motion. In such a deposit, sewage sludge and technological waste are burned. Despite the fact that sewage sludge contains a significant amount of organic substances, it can be burned without the addition of auxiliary fuel, when it is dehydrated to 35 - 50% DS. The combustible substance of the sludge has a calorific value similar to that of brown coal. The parts that will not burn are burnt in special chambers at the temperature of 850 °C. The product of the thermal neutralization process is fly ash (Bień et al., 2011, Środa et al., 2012, www.mpwik.com.pl). In addition, as a result of cleaning exhaust gases from acid gaseous pollutants (S_x,HCl, HF, NO_x) and heavy metals, hazardous waste is generated with the code 190107 - solid waste from the treatment of flue gases or with the code 190106 - sludges and other hydrated wastes from gas cleaning off-road (www.mpwik.com.pl).

The decisive influence the properties of ashes are influenced by the composition of municipal wastewater, the method of its treatment and combustion, as well as the type of mineral substances present in the coal and the accompanying waste rock, the type of boiler and technological combustion conditions, the method of fuel preparation, methods of ash capture, discharge and storage (Grabowski &Oleszkiewicz, 1998, Kępys et al., 2013, Lutze &Vom Berg, 2010, Sear, 2001). Additionally, raw sewage sludge contains microorganisms and bacteria (Bień et al., 2011). Potential benefits of using fly ash in building materials include the destruction of pathogens during the burnout process, the immobilization of heavy metals in the burned-out matrix, the oxidation of organic substances and increased resistance to low temperatures.

The experimental works on fly ashes from thermal treatment of sewage sludge presented in the literature focus on the assessment of the possibility of using them for composite materials such as: ceramics (Suzuki et al., 1997, Park et al., 2003, Merino et al., 2007), burnt tiles, bricks (Lin et al., 2016, Lin et al., 2017, Lynn et al., 2016), mortars, pastes and concretes (Piasta & Lukowska, 2016, Vouk et al., 2017, Chakraborty et al., 2017, Rutkowska et al., 2020). The introduction of a certain amount of ashes from sewage sludge as a partial replacement for cement allows for obtaining comparable parameters in relation to concrete made on the basis of siliceous and calcareous fly ashes (Yusur et al., 2012, Rutkowska et al., 2016, Wichowski et al., 2017).The use of ashes from sewage sludge is related to the restrictions contained in the applicable regulations (Dz.U. 2016 poz. 108). implementing Directive EU/2010/75, with a different chemical and physical composition, with the content of pollutants – heavy metals (UE/75/2010).

An important rule is that there is no typical composition and quality of the delivered to municipal wastewater treatment plants, and thus there is no typical, constant composition of waste - fly ash generated during thermal treatment of sludge. So far, no guidelines and subject standards have been developed regarding the use of ashes from sewage sludge incineration in the technology of concrete and cement production as an additive.

The main purpose of the research was to analyze and evaluate the physicochemical composition of fly ash from thermal treatment of sewage sludge in accordance to EN 450-1:2012, ASTM-C618-03 and ASTM C379-65T, and thus to determine their variability (EN 450-1:2012, ASTM-C618-03 and ASTM C379-65T). In addition, the analysis was aimed at determining the effect of fly ash properties on the compressive strength of concrete produced with its participation, where other by-products from coal combustion are used. As part of the pilot studies, the impact of this additive on the natural environment was also determined – the leaching values of selected heavy metals from ashes and from ordinary concretes containing fly ashes from thermal treatment of sewage sludge were determined. It is essential not only during the operation of a given building structure, but also in the post-exploitation period, when the materials from the demolished building are to be reused or stored.

RESEARCH MATERIAL AND METHODOLOGY

In order to understand and evaluate the physico-chemical properties, tests were carried out on fly ash from thermal treatment of sewage sludge. Separate batches of material were collected for the study during the continuous operation of the sludge from three wastewater treatment plants: Warsaw, Cracow and Łódź, in three periods. The batches were collected in April/May 2018, 2019 and 2022. Such frequency of sampling made it possible to assess the variability of the properties of a given material. Fly ash was stored in closed plastic containers. Fly ash tests were carried out in the Laboratory of the Faculty of Civil Engineering and Architecture of the Lublin University of Technology according to the methodology

Table 3. Collective indicators with reference to standards

Research	Method tests / standard
Properties chemical	
Cl chlorides -	PN - EN 196-2: 2013-11
Free oxide carbon (CaO)	PN - EN 451-1: 2012
Reactive oxide carbon (CaO)	PN - EN197-1: 2012
Reactive oxide silicon (SiO_2)	PN - EN197-1: 2012
Anhydride acid sulfuric acid (SO_3)	PN - EN 196-2: 2013-11
Sum of oxides SiO_2, Al_2O_3, Fe_2O_3	PN - EN 196-2: 2013-11
Oxide of aluminum Al_2O_3	PN - EN 196-2: 2013-11
Oxide iron Fe_2O_3	PN - EN 196-2: 2013-11
Whole contents alkali (Na_2O)	PN - EN 196-2: 2013-11
Oxide magnesia (MgO)	PN - EN 196-2: 2013-11
Phosphate (P_2O_5)	PN - EN 450-1: 2012
Loss on ignition	PN - EN 196-2: 2013-11
Properties physical	
Shallowness	PN - EN 451-2: 20017-06
Indicator activity	PN - EN 451-1: 2012
Density grains	PN - EN 196-6: 2011
Stability volume	PN-EN 196-3: 2016-12
Time beginning bindings	PN-EN 196-3: 2016-12
Water lust	PN-EN 450-1: 2012
Surface right by Blaine	PN-EN 196-6; 2011

presented in previous studies (Rutkowka et al., 2018, 2020). Table 3 presents a collective list of indicators with a reference to the standards used.

In order to make an effect of fly ash on the compressive strength, plain concrete of class C25/30 with a consistency of K3 was prepared in accordance with EN 206 + A1: 2016-12 (currently 206 + A2: 2021-08).The fine aggregate was selected by the sieve analysis method, while the coarse aggregate was selected by the iteration method. The individual components were designed by the method of three equations according to Bukowski (Jamroży, 2015). For the preparation of concrete samples, natural aggregate with a grain size of 0.125 - 16 mm, CEM I 32.5 Portland cement and additives - fly ash from thermal combustion from wastewater treatment plants in Warsaw, Cracow and Łódź were used. The following concrete samples were prepared:

- reference concrete without any additives - CON,
- concrete with the addition of fly ash from thermal treatment of municipal sewage sludge - FA - W (Warsaw), FA - K (Cracow), FA - L (Łódź).

In individual samples with the addition, cement was replaced with fly ash from sewage sludge in the amount of 5%, 10%, 15%. The concrete mix recipe is presented in Table 4.

Compressive strength tests were carried out after two maturing periods in accordance with the guidelines contained in EN 12390-3: 2011 in a hydraulic machine H011 Matest (Italy)(EN 12390-3: 2011).

For all concrete samples with the addition of fly ash (FA15%), leaching tests were carried out.After the compressive strength test, concrete samples were ground to fractions below 4 mm. The obtained granulate was dried to a constant weight, and then leaching tests were carried out according to the EN 12457-2 standard(EN 12457-2:2006). “The tests were carried out at the temperature of 20 °C in triplicate. Using the method of atomic emission spectrophotometry with inductively excited plasma on the ICP-AES Thermo spectrometer Scientific iCAAP 6500 (USA), the content of heavy metals (Cd, Cr, Cu, Ni, Pb, Zn, As, Sb, Se, Ba, Hg and Mo) was determined. Additionally, the pH was checked using the CyberScan pH -510 pH meter from Eutech Instruments with the GPX-105s pH head, the IJ44C type pH electrode, and the conductivity was checked using the CyberScan Con-510 conductometer from Eutech Instruments. Color was determined using a Hach DR 4000 spectrophotometer, turbidity - Turbidimeter 2100N IS turbidity meter, salinity and TDS content - Hach Sension 156 ionometer with combined TDS probe. The determination of the content of sulphates, chlorides, hardness and alkalinity was performed by the titration method. Quick COD analyzer by LAR with a combustion temperature of 1200 °C (Wichowski et al., 2017, Rutkowska et al., 2018).

RESULTS AND DISCUSSION

Fly ash from the thermal treatment of sewage sludge

The results of the research on the chemical composition of individual batches of fly ash from sewage sludge (Fig. 1). The results for the limit values for single results were compared with the requirements (Table 1) of EN 450-1: 2012(EN 450-1: 2012).

On the basis of the obtained results, it was found that the variability of the free CaO content in the fly ash was small (lot: FA-W- 0.09% by weight, FA-K- 0.08% by weight, FA-L- 0.12% by weight). All samples met the requirements ofEN 450-1: 2012 - the limit value for a single result is - 1.6%. The variability of the total calcium oxide content can be considered high (lot: FA-W- 18.64% by weight, FA-K- 11.90% by weight, FA-L- 22.02% by weight). The requirements for reactive calcium oxide (limit value for a single result

Table 4. Concrete mix proportions by weight

	Mass of concrete ingredients (kg/m ^{3})			
	Water	Aggregate	Cement	Fly ash
Concrete without additives	186.23	1838.10	372.46	-
Concrete with 5% ash	186.23	1838.10	353.84	18.62
Concrete with 10% ash	186.23	1838.10	335.22	37.24
Concrete with 15% ash	186.23	1838.10	316.59	55.87

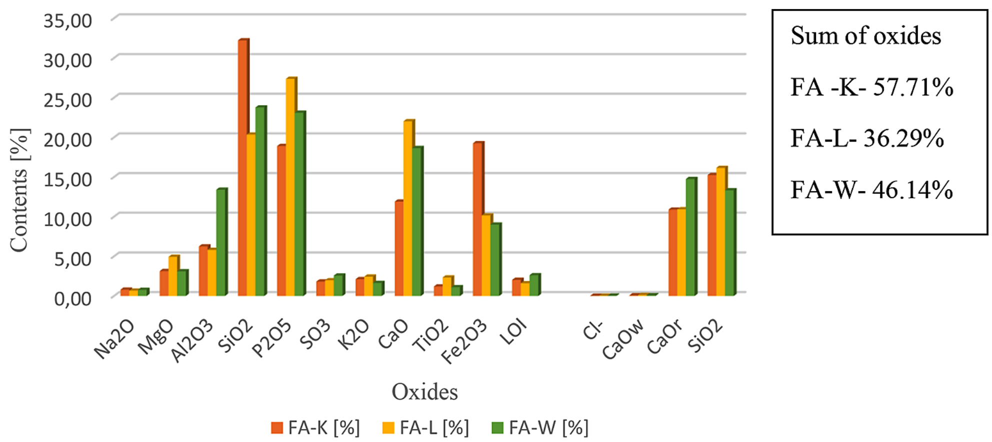

Figure 1. Chemical composition of fly ash from sewage sludge

lower than 11% by weight), influencing the formation of the CSH phase, were met for ash from Cracow (10.89% by weight) and Łódź (10.92% by weight). The ash from Warsaw exceeded the permissible value by 3.74% by weight. A higher content of reactive calcium oxide affects the hydraulic properties of the ash. None of the tested sewage sludge ash batches (batch: FA-W- 13.32% by weight, FA-K- 15.24% by weight, FA-L- 16.14% by weight) meets the requirements of the standard regarding the amount of reactive silicon dioxide (lower limit is 25% by weight). The results indicate a variability of 88%. The total SiO_2 content was respectively for: FA-W- 23.76% by mass, FA-K- 32.21% by mass, FA-L- 20.34% by mass, which gives us a variability of 58%.

Taking into account the fact that the basic oxides of cement clinker are: CaO, SiO_2, Al_2O_3, Fe_2O_3, the requirements related to the sum of the three oxides (SiO_2, Al_2O_3, Fe_2O_3) have not been met for any of the parties. The tested samples of ashes from thermal treatment of sewage sludge were characterized by a spread of results at the level of 59% (FA-W- 46.14% by weight, FA-K- 57.71% by weight, FA-L- 36.37% by weight). Taking into account ASTM-C618-03(ASTM-C618-03), the ash from the sewage treatment plant in Cracow meets the requirements of the sum of oxides ($SiO_{2,}$ Al_2O_3, $Fe_2O_3 \geq 50\%$). Due to the low SO_3/Al_2O_3 ratio((FA-W- 0.19%, FA-K- 0.29%, FA-L- 0.34% by mass)) addition in the form of fly ash from thermal sludge treatment due to the formation of ettringite, it will not adversely affect the swelling of concrete (Rajczyk, 2012).

The main factor influencing the volume changes (swelling) of concrete is too high a content of free calcium and magnesium oxides. Fly ash from Warsaw and Cracow met the standard requirements for the content of magnesium oxide (permissible content up to 4.0% by weight), ash from Łódź slightly (0.93%) exceeded the permissible value. The low content of magnesium oxide, which is released in free form slower than CaO, will probably not cause a change in volume when maturing concrete produced on the basis of the tested fly ash (Pachowski, 2002). The total alkali content (limit value for a single result max. 5%) was met for all ash samples. The tested ashes were characterized by a low dispersion of the results (FA-W- 0.77% by weight, FA-K- 0.80% by weight, FA-L- 0.68% by weight).

The indicator analyzed in each sample was also the content of chlorides Cl^- and sulfuric anhydride $SO_{3.}$ In each batch of ash from sediment, the chloride content is similar at the level of 0.03 and 0.04% by weight and is an order of magnitude less than the limit values for a single result. When considering the SO_3 content, the following results were observed for: FA-W- 2.57% by mass, FA-K- 1.82% by mass, FA-L- 1.98% by mass, which indicated a variation of 41%. All samples met the requirements of EN 450-1 and ASTM-C618-03(EN 450-1: 2012, ASTM-C618-03). According to the literature on the subject, the presence of chlorides and sulphates has a negative effect on the properties of concrete, causing its corrosion. Of all the ions, chloride ions penetrate deep into the cement matrix the fastest. Chloride aggression leads to a decrease in the pH of the concrete and the formation of expansive compounds that may cause concrete cracking and corrosion of the reinforcement. The chloride content in concrete also affects the form of the matrix of hydrated calcium silicates (CSH phases) (Szarek&Wojtowska, 2018).

The content of SO_3 above 3% by weight may cause sulphate corrosion of concrete (Eglinton, 1987). However, studies carried out by Garcés have shown that sulphates contained in fly ash from thermal treatment of sewage sludge do not react with cement (Garcésa et al., 2008).

The loss on ignition, expressing the content of unburned carbon in the sample of fly ash FA, was clearly lower than that of silica ash. This is due to the combustion technology in a fluidized bed furnace and a higher combustion temperature exceeding 850 °C. The ash from sewage sludge obtained losses on ignition respectively: FA-W- 0.5% by weight, FA-K- 0.7% by weight, FA-L- 0.5%. According to the standard, these values correspond to category A (Table 1) and meet the requirements specified in Table 2. The low variability of the obtained results indicates the stability of the municipal sewage sludge incineration technology in furnaces. A characteristic feature of ashes from thermal treatment of sewage sludge is the high content of phosphates, exceeding the limit values for single results (less than 5.5% by weight) three and five times. The amount of phosphates was as follows: FA-W- 23.09% by weight, FA-K- 18.91% by weight, FA-L- 27.37% by weight.

It is assumed that such a high content of them in the ash delays the hydration of the cement and thus the slow increase in the compressive strength of concretes produced with their participation (Małolepszy & Tkaczewska, 2006, De Noirfontaine et al., 2009, Targo et al., 2014). According to De Noirfontainewhen using this type of ash for cement production, too much phosphorus deteriorates the quality of the clinker, breaking down alite (C_3S) into belite (C_2S) and lime (CaO) (De Noirfontaine et al., 2009).The particle size distribution of the tested fly ash is monomodal, with a maximum of 65 µm(FA-K), 125 µm(FA-L) and 85 µm(FA-W) - Figure 2. Grains with a diameter from 2 to 250 µm constitute over 91% of the volume in all batches of the tested ash. The dominant grain fractions in this range are 20-50 µm (20.18% FA-L, 27.25 FA-K and 25.01% FA-W%), 50-100 µm (25.88% FA-L, 29.39% FA-K and 28.12% FA-W) and 100-250 µm (34.79% FA-L, 22.89% FA-K and 27.75% FA-W) - Figure 3.The specific ash density was respectively: FA-W- 2.530 kg/dm^3, FA-K- 2.780 kg/dm^3, FA-L-2.620 kg/dm^3.

Figures 4–6 show scanning electron micrographs of fly ash from sewage sludge incineration. It can be noticed that in the fly ash samples irregular grains of variable size predominated

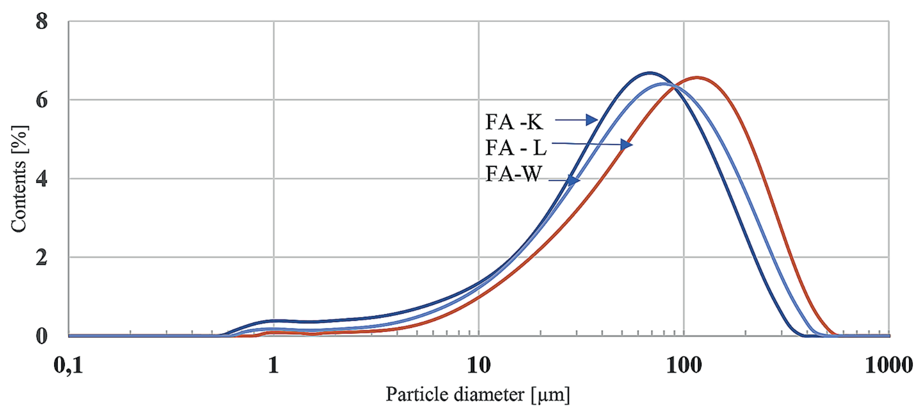

Figure 2. Particle size distribution curve in the tested fly ash

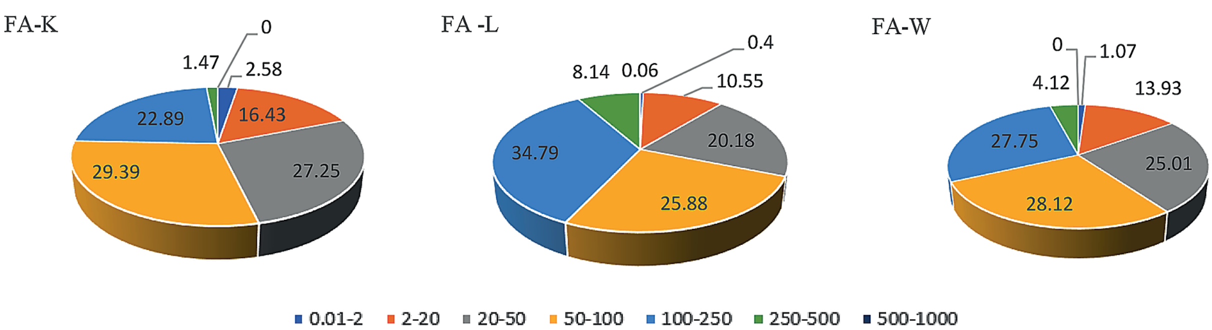

Figure 3. Volumetric distribution of individual particle fractions in the investigated ashes

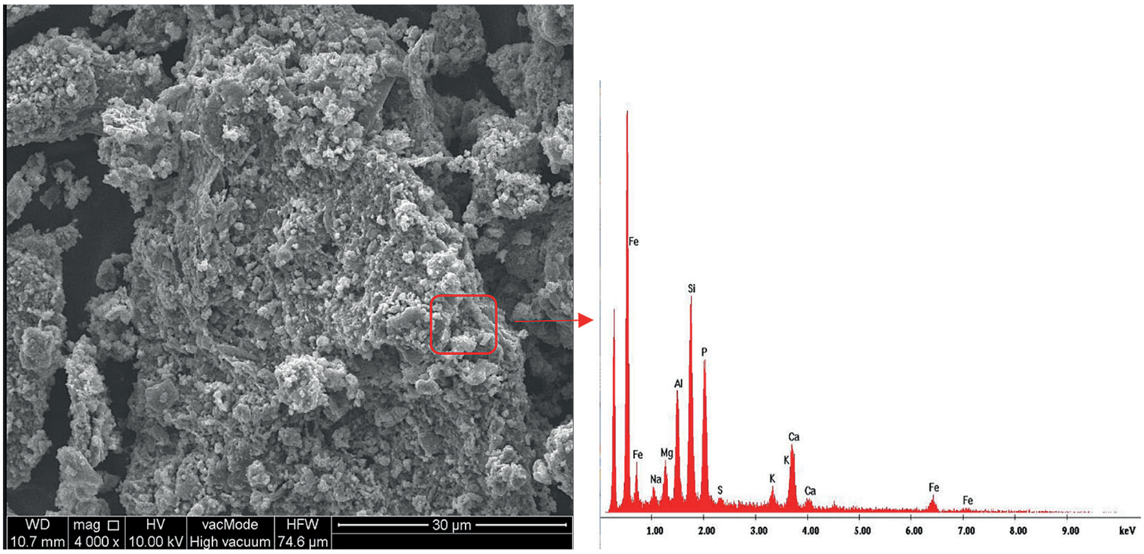

Figure 4. SEM photos of the tested ash from Cracow together with the EDS analysis

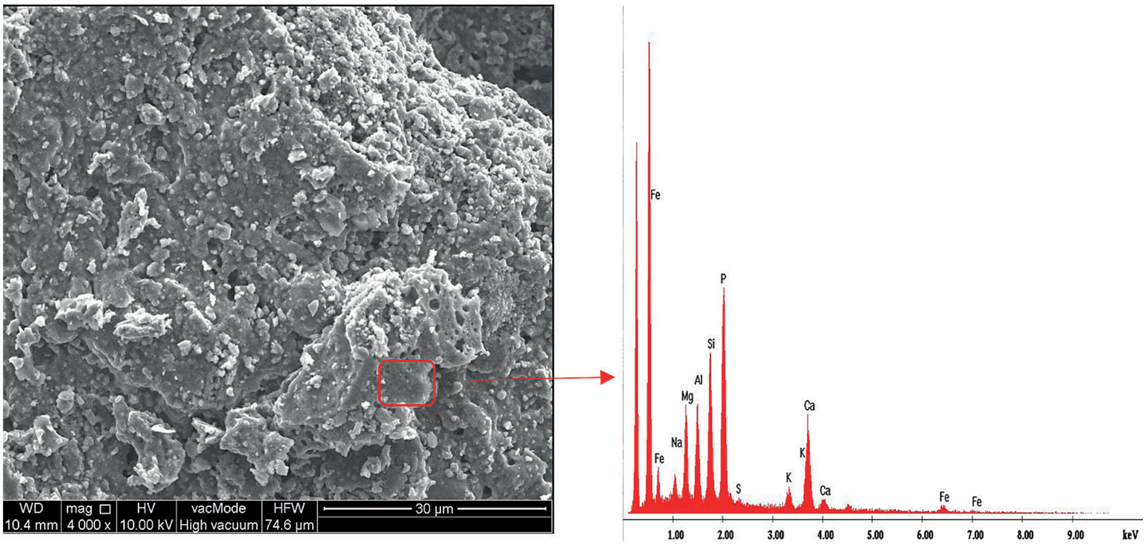

Figure 5. SEM photos of the tested ash from Łódź together with the EDS analysis

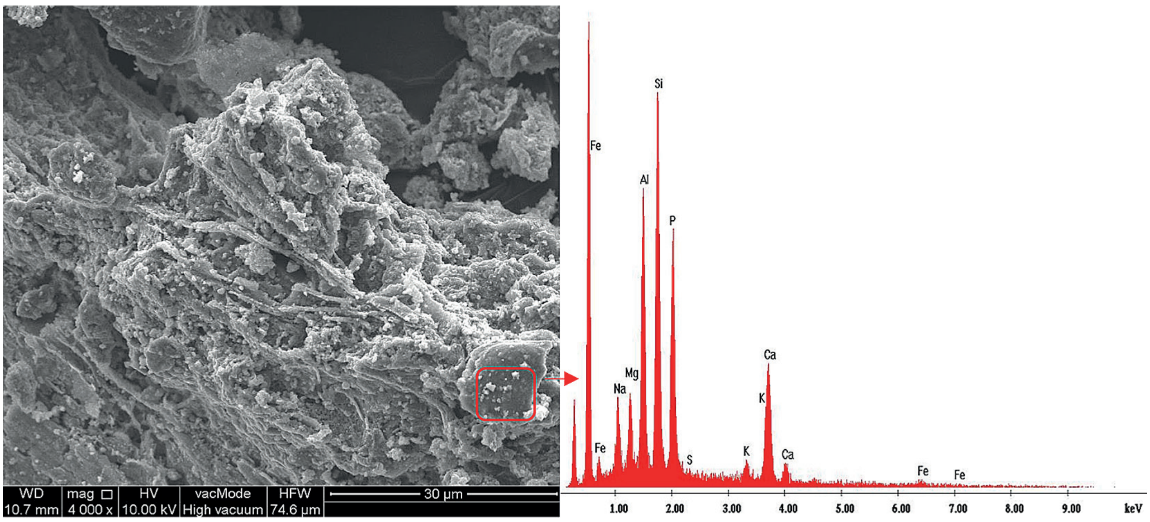

Figure 6. SEM photos of the tested ash from Warsaw together with the EDS analysis

with a strongly developed surface showing high porosity of the material with a loose and rough structure, which leads to greater water absorption (Lynn et al., 2016). Spherical and cuboidal forms were very rare. Chemical analyzes in the micro-area (SEM-EDS) showed differentiation of the elemental composition. The grains of chemical composition dominated: Fe, Al, Si and P, next to which there were grains containing Ca (Fig. 4–6). Typical components for fly ash, however significantly different in morphology - no spherical spheres. The remaining ingredients: sodium, magnesium, and potassium were found in minor amounts.

Figure 7 shows the XRD pattern for the tested fly ash FA. The mineral composition of ash is dominated by quartz and anhydrite, which have been identified by the characteristic inter-plane distances - dhkl (20.5; 26.4; 35.6; 54.3 for quartz) and (9.1; 13.5; 19, 8; 28.7; 46.8 for anhydrite). The mineral composition of ash from the incineration of sewage sludge is complemented by phosphates in the form of apatite and fluoroapatite. The first of them was recognized by the strongest reflections dhkl = 20.6; 30.3; 40.2; 49.8. Fluoroapatite was identified after dhkl = 21.2; 24.8; 34.8; 47.7. These mineral phases are mainly carriers of P_2O_5, which is present in higher amounts in relation to the content known from conventional ashes.

Determining the pozzolanic activity of materials, including fly ash, used in concrete and cement production technology is a difficult task. The presented research shows that the activity is manifested in the influence of pozzolan on cement strength, in the amount and speed of binding calcium hydroxide by active ingredients, it may also depend on the type of binder to which it will be added (Kurdowski&Peukert, 1970, Osiecka, 1983, Roszczynialski, 2003). Pozzolanic activity of fly ashes according to the ASTM C379-65T standard is determined on the basis of their SiO_2 and Al_2O_3 content (ASTM C379-65T). These components, as a result of heating at the temperature of 80 °C in a 0.5-molar NaOH solution for 1.5 h, will be leached from the test sample. The parts of alumina and silica that react with calcium hydroxide and dissolve in NaOH are considered active. The parts of alumina and silica that will dissolve in NaOH are considered reactive with $Ca(OH)_2$. The total content of SiO_2 and Al_2O_3. over 20% indicates the pozzolana character of the studied sample (Małolepszy&Tkaczewska, 2007, Tkaczewska, 2008) (Table 5). Table 6 presents the values of the obtained results. Based on the studies of pozzolana activity, it was found that the total content of reactive silicon and aluminum oxide in the fly ash from the sewage treatment plant in Kraków was 21.49 and was the highest, in Łódź it was 20.94%, while in Warsaw it was 20.41% and was thus the smallest. Taking into account the requirements of ASTM C379-65T, the tested ash from the sediments was fulfilled (ASTM C379-65T).

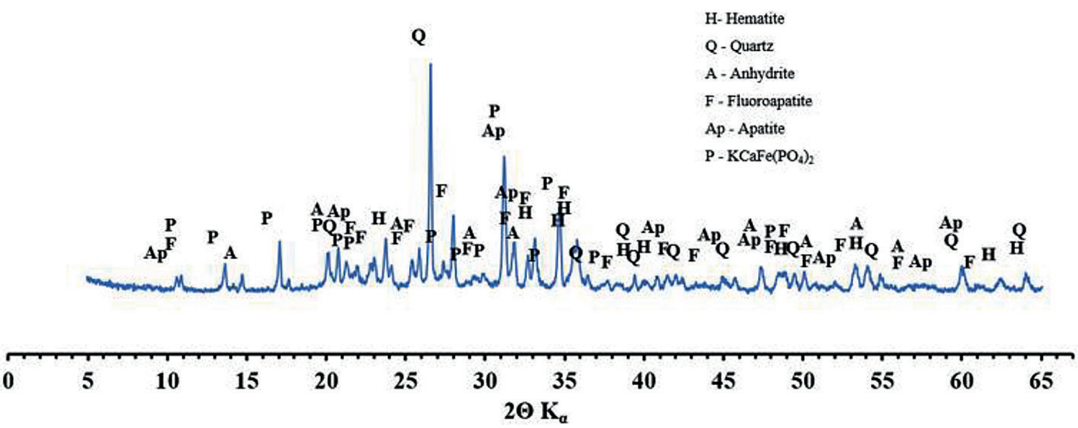

Figure 7. XRD pattern of the applied sludge fly ash

Table 5. Pozzolanic activity of fly ash (ASTM C379-65T)

Reactive oxide	Content in FA - K [%]	Content in FA - L [%]	Content in FA - W [%]
SiO_2	15.24	16.14	13.32
Al_2O_3	6.25	4.80	7.09
$SiO_2 + Al_2O_3$	21.49	20.94	20.41

Determination of the pozzolanicity of fly ash according to EN 450-1 (EN 450-1: 2012)consists in the determination of the pozzolanic activity index as a proportion the compressive strength of bars made of standard mortar made with 25% by weight of fly ash and 75% by weight of Portland cement (CEM I 42.5R) to the compressive strength of bars of standard mortar made of 100% Portland cement (CEM I 42.5R). The activity index after 28 days should reach the value ≥ 75%, and after 90 days the value ≥ 85%. The activity index was determined after 28, 90 and 180 days Table 6.

As a result of the research, it was found that the pozzolanic activity of fly ash from thermal treatment of sewage sludge did not meet the requirements ofEN 450-1: 2012 after 28 and 90 days of maturation (EN 450-1: 2012). The activity index after 28 days of maturation for FA-K ash was 71.7%, for FA-W 68.5%, and for FA-L only 61.2%, while after 90 days it was 84.1% for ash with FA- K, 79.3% for FA-W and 70.7% for FA-L. The activity index exceeded the required values (85%) after 180 days of maturation.

The phosphorus contained in the used ashes from sewage sludge has a negative effect on cement hydration – it delays this process (Małolepszy & Tkaczewska, 2007).The results of studies presented in other studies confirm that the *pozzolanic activity of fly ash from thermal treatment of sewage sludge* reaches the standard value (85%) after a longer maturation period (Kosior-Kazberuk, 2011). However, it should be remembered that the mentioned standard refers to silica ash.Ashes from sludge incineration showed lower pozzolanic activity than fly ash from coal combustion in the same period, however, they reached the required values of indicators (85%). The use of ash as a substitute for a part of the cement in concrete requires an extended maturation period, which is recommended for specific applications or the introduction of admixtures accelerating the setting. In addition, it delays the maturation process of concrete, but allows to maintain the adopted consistency of the concrete mix for a longer time (Baeza-Broton et al., 2016). Table 7 summarizes the complete results of chemical characteristics of fly ash from sewage sludge and concrete samples, in which cement was replaced with 15% fly ash. The obtained results of the leachability tests were compared with the values constituting the criterion for the admission of waste of a given type to landfills. The limit values were adopted according to the ordinance of the Minister of Economy of 2015 on the admission of waste to landfills (Dz.U. 2015 poz. 1277).

Fly ash from sewage sludge can be used to prepare concrete mixtures when the concentration of heavy metals in the water extract from concrete samples in total does not exceed 10 mg/L, calculated on the basis of the mass of elements (Dz.U. 2016 poz. 108). In all the tested samples, the total concentration of heavy metals per element weight did not exceed the permissible value. On the basis of the conducted tests, it was observed that the leachability of heavy metals from concrete samples containing FA ashes was higher than from the

Table 6. Vacuum activity of fly ash (EN 450-1: 2012)

The maturation period [days]	FA - K activity index [%]	FA - L activity index [%]	FA - W activity index [%]
28	71.7	61.2	68.5
90	84.1	70.7	79.3
180	93.8	86.3	91.9

Table 7. Comparison of the leaching value of heavy metals with the values allowing for the storage of waste of a given type (in mg/kg dry weight) (Rutkowska et al., 2018)

Type samples	Metals heavy (mg/l)											
	Cd	Cr	Cu	Ni	Pb	Zn	As	Sat	Se	Ba	Hg	Mo
CON	<0.002	0.013	0.013	<0.005	<0.003	<0.030	<0.010	<0.010	<0.010	1.18	<0.005	<0.010
FA	<0.002	<0.010	0.008	<0.005	<0.003	<0.030	<0.010	0.020	0.047	0.112	<0.005	0.40
FA 15%	<0.002	0.016	0.011	<0.005	<0.003	<0.030	<0.010	<0.010	<0.010	1.14	<0.005	<0.010
Limit values of elution when admitting waste to landfill passing waste as fit for storage [Dz. U. 2015, item 1277]												
Inert waste	0.04	0.5	2	0.4	0.5	4	0.5	0.06	0.1	Twenty	0.01	0.5
Other waste than inert and hazardous	1	10	50	10	10	50	2	0.7	0.5	100	0.2	10
hazardous waste	5	70	100	40	50	200	25	5	7	300	2	Thirty

Note: [1] - basic test : liquid / phase constant = 10 L ·kg sm^{-1} - base test: liquid / solid phase = 10 L ·kg of dry matter $^{-1}$.

ash itself. Concentrations in the eluates of heavy metals for fly ash were below the limit values for inert waste. Carried out by Monzo et al. (Monzo et al., 2003) trace elements leaching tests from mature mortars and concretes with the addition of fly ash showed that the replacement of Portland cement with ash from thermal treatment of sewage sludge in the amount of 30% does not pose a threat to the safety of the natural environment. Chen et al.showed that of the potential impurities, only molybdenum and selenium were eluted at concentrations above the threshold values. Leaching studies on monolithic concrete have shown that concentrations of pollutants, including molybdenum and selenium, do not exceed the threshold values provided by the Environmental Protection Agency. Taking into account environmental standards and technical specifications, it was found that the use of ash from sewage sludge in building materials seems possible (Chen & Poon, 2017). Table 8 summarizes the results of physicochemical tests of eluates for fly ash samples and concrete samples.

The tested samples were characterized by high pH values. PH value was recorded for the reference concrete samples of 12.29, and the lowest value, equal to 9.75, for the sample of ash from combustion of municipal sewage sludge. In addition, for this material, the lowest values for other physicochemical parameters were also observed, such as: conductivity (2.3275 mS/cm), chloride concentration (7.8 mg Cl/l), color (24.30 mg Pt), turbidity (15,97 NTU), phenolphthalein alkalinity (1.08), salinity (1.10 (sal) (o/oo)) and TDS dry matter content (1.0935 g/l). Barbosa et al. showed that the COD in the eluate of ashes from conventional fuels combustion took higher values (Barbosa et al., 2013). The observed differences may be related to the combustion technology used, and above all, to the temperature and the composition of the sewage sludge itself. The replacement of 15% of Portland cement in concrete with fly ash did not significantly change the pH of the eluate, but it resulted in a weakening of the color intensity and a reduction in turbidity and an increase in phenolphthalein alkalinity, hardness and COD. Moreover, higher values of salinity, electrical conductivity and dry weight of TDS were observed in all eluates at a higher concentration of chlorides in FA samples of 15%. The highest values of electrical conductivity (7.8575 mS/cm), phenolphthalein alkalinity (39.24), hardness (70.42 °T) and salinity (4.25 (sal) (o/oo)) were found for reference concrete samples without additives (Rutkowska et al., 2018).

Concrete mix and concrete testing

Based on the obtained results, it was found that the fly ash from the thermal transformation of sewage sludge obtained from three different sewage treatment plants affects its individual parameters – density, consistency and air content. The density of the concrete mix was from 2,316 to 2,387 kg/m^3 for the ash from Cracow, for Łódź from 2,242 to 2,371 kg/m^{3,} and for ash from Warsaw from 2,261 to 2,399 kg/m$^{3.}$ The air content in concrete mixtures depended on theamount of added additive instead binder and it grew with the higher content of fly ash. The lowest air content was obtained for CON samples equal to 1.7%, while the highest air content was recorded in concrete mixes in which cement was replaced in the amount of 15% equal to 2.5% for ash from

Table 8. Physico-chemical parameters of water extracts (Rutkowska et al., 2018)

Parameter	FA	CON	FA 15%
pH	9.75	12.29	12.185
Conductivity (mS cm $^{-1}$)	2.3275	7.8575	6.6125
Chlorides (mgCl/L)	7.8	61.4	76.8
Sulphates (mgCl/L)	nw	nw	7.0
Color (mgPt)	24.30	17.50	36.00
Turbidity (NTU)	15.97	3.42	6.73
Basicity general ((mval / L)	1.76	1.4	1.64
Basicity phenolphthalein	1.08	39.24	33.12
Hardness (°T)	(*)	70.42	49.24
TDS dry weight (g / l)	1.0935	3.9	3.175
Salinity (sal) (°/$_{oo}$)	1.10	4.25	3.45
COD (mgO$_2$/L)	5.00 p.m.	33.50	40.00

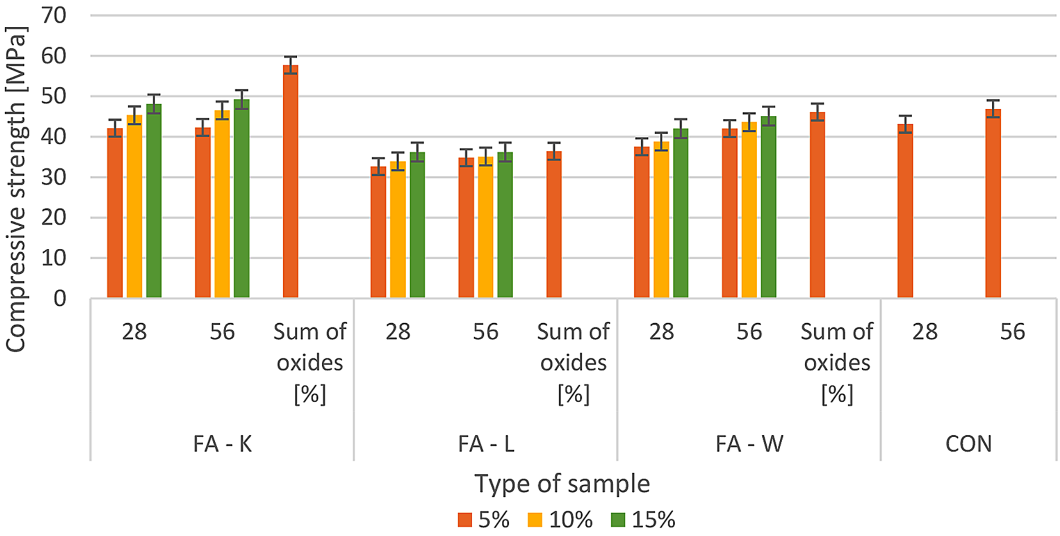

Figure 8. Compressive strength

Cracow, Łódź 2.1% and 3.1% for ash from Of Warsaw. The consistency test results obtained are very inconsistent. It is evident that the additive used is characterized by high water demand and has a negative effect on the consistency of the concrete mix - the mix is non-workable after a short time. The "positive" effect of using an additive with high water demand is the reduction of the effective water content in the concrete mix, which translates into a lower ratio the above-mentioned and higher levels of compressive strength (especially early strength) (Fig. 8). In practice, the concrete producer, in order to ensure the appropriate consistency (workability) of the concrete mix in a sufficiently long time, may add larger amounts of water - which ultimately results in lower compressive strength, high concrete shrinkage and low durability, or add superplasticizers.

On the basis of the conducted research, it was noticed that the higher concentration of SiO_2, Al_2O_3 and Fe_2O_3 and lower P_2O_5, CaO has a positive effect on the increase of the compressive strength of the produced concretes. The highest compressive strength equal to 48.1 MPa after 28 days of maturation was obtained for concrete samples (FA -K15%), in which cement was replaced with fly ash from Cracow in the amount of 15%, while the lowest compressive strength equal to 36.2 MPa of the sample (FA-L 15%), in which cement was replaced with ash from Łódź. The increase in the strength of concrete samples compared to the reference samples was 12%, and the decrease was 16%. After 56 days of maturation, the highest compressive strength was observed, equal to 49.2 MPa, was achieved by FA-K15% samples, while the lowest, equal to 36.2 MPa, for FA-L15% samples made on the basis of ash from Łódź. According to the information presented in the works, the optimal amount of fly ash from thermal treatment of sewage sludge in cement composites ranges from 5% to 15%. The ash content in the amount of 20% of the cement mass delays the process of setting the slurry and slows down the increase in compressive strength of concretes compared to concretes made on the basis of Portland cement (Rutkowska et al., 2020, Rutkowska et al., 2018, Rutkowska et al., 2020, Fontes et al., 2004, Yen et al., 2011). The use of a new, innovative additive – modifier in concrete technology, in line with the sustainable development policy, is not always associated with the improvement of its properties. In accordance with the requirements of EN 450-1 (EN 450-1:2012), when using fly ash for concrete, drops in compressive strength by 15–25% in relation to the comparative samples are allowed.

CONCLUSIONS

Fly ash from thermal treatment of sewage sludge does not meet the requirements of EN 450-1: 2012 not only by definition, but also due to physical and chemical properties. This makes it impossible to use the material as a Type II additive to concrete. The use of such ash in concrete technology will be necessary to obtain European technical approvals. On the basis of the conducted research, it was found that the fly ash from the thermal treatment of sewage sludge is a safe and valuable modifier of concretes, and the following conclusions were formulated. 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, it will be possible to further reduce the variability of the fly ash composition as a result of the application of its treatment methods and if the ash is no longer treated as waste and will be treated as a complete by-product of combustion. Ashes from thermal treatment of sewage sludge showed a different chemical composition compared to siliceous ashes. The highest percentage in the samples of ash from sewage sludge was silica, calcium, phosphorus and aluminum oxides, and in the silica ash sample - silica and aluminum oxides. The sum of the content of silicon dioxide (SiO_2), aluminum oxide (Al_2O_3) and iron oxide (Fe_2O_3) in the fly ash from sewage sludge was almost twice as high as that of conventional ashes and did not meet the requirements of the standard EN 450-1 + A1: 2012. However, there are no regulations on the chemical and physical properties of ashes obtained from the incineration of sewage sludge that would limit the possibility of their use in concrete technology. Pozzolanic activity of fly ashes from thermal treatment of sewage sludge does not meet the requirements of EN 450-1: 2012 after 28 and 90 days of maturation. The activity index after 28 days of maturation for FA-K ash was 71.7%, for FA-W 68.5%, and for FA-L only 61.2%, while after 90 days it was 84.1% for ash with FA- K, 79.3% for FA-W and 70.7% for FA-L. Thus, the innovative ash from burning sludge showed lower pozzolanic activity than fly ash from burning coal during the same period. However, they reached the required index values (85%), which allows them to qualify as active mineral additives. The leachability from the concrete sample with the addition and from the fly ash did not exceed the limit values for inert waste. The leachability of heavy metals from samples containing fly ash is comparable or lower compared to concrete without the addition of fly ash. Migration of heavy metals to the aquatic environment is insignificant and should not be a significant problem. However, it is advisable to carry out further research for variable classes and types of concrete as well as different leaching conditions. Large amounts of phosphorus compounds present in fly ashes from sewage sludge delay the beginning of binding of composites produced with their participation. It also makes their workability difficult. Concrete containing in its composition fly ash from sewage sludge as a cement substitute in the amount of up to 15% was characterized by comparable compressive strength to the comparative concrete without the addition - 43.1 MPa and 46.9 MPa. The average compressive strength for concrete containing ash from Kraków was set at 48.1 MPa and 49.2 MPa after 28 and 56 days of maturation, for ash from Warsaw at 42.0 MPa and 45.1 MPa, and for ash from Łódź at 36.2 MPa and 36.2 MPa. The results allow for the concrete strength properties to exceed the minimum 25 MPa adopted for the design.

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