




Fly ash from thermal transformation of sewage sludge as an alternative additive to concrete resistant to environmental influences

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Abstract

Concrete is currently the most widely used man-made composite material and second only to water in the entire range of materials used. It is a material with a high potential to adapt to specific operating conditions and can be made from local raw materials (aggregate, cement, water, and mineral additives), which can be selected to minimize the carbon footprint. The use of fly ash from the thermal conversion of sewage sludge in concrete is in accord with the advice on waste management proposed in the European Union. This paper presents the results of research on the effect of the partial replacement of Portland cement with this material on the strength parameters, frost resistance, and carbonation of concrete compared to reference concrete and to concrete containing a conventional additive – siliceous fly ash. In addition, the potential environmental impact of the use of sewage sludge ash was investigated by determining the leachability of heavy metals. Concrete mixtures of C20/25 ordinary concrete, based on CEM I 42.5R Portland cement, with varying ash contents comprising 0–20% of the cement mass, were produced for the experimental work. The obtained test results confirmed the possibility of producing plain concrete modified with fly ash from the thermal treatment of sewage sludge and the concrete's compliance with the environmental requirements relating to the leaching of heavy metals.

Introduction

Human activity, to a greater or lesser extent, affects the natural environment around us. Understanding the consequences of such activities for environmental protection (understood in a broad sense) contributes to the rational use of environmental resources and their proper management. The growing awareness of society about the need to protect the natural environment continues to spread to various aspects of life. In the light of changes to environmental regulations, the commercial power industry faces the challenge of meeting increasingly

strict emission requirements. From January 1, 2016, with the coming into force of the EU Directive on industrial emissions, emissions of nitrogen oxides, sulfur, and dust were reduced. For the construction sector, one of the most important issues is to make concrete a more environmentally friendly material. The search for solutions to this challenge is indispensable when designing the composition of a concrete mixture, two components of which, cement and aggregate, contribute to anthropoppression during their production. There are already areas in Poland and elsewhere in the world where obtaining good quality materials for the production

of cement is a problem. In addition, the world economy requires increasingly more cement for concrete production, for which no comparable substitute has been found to date. A significant problem is that during the production of 1 metric ton of cement, 0.5 to 1 metric tons of greenhouse gases are produced, which, according to various data, constitute 6–8% of the total anthropogenic emission of greenhouse gases (Directive, 2010; IEA, 2019). Currently, silica fly ash from hard coal combustion is widely used in cement production and above all in concrete production in Europe, including Poland. Its wide application is mainly determined by its chemical and phase composition, and in particular its pozzolanic activity and high fineness, which is similar to cement. The use of siliceous fly ash to produce concrete is only possible if the requirements specified in PN-EN 450-1:2012 are met (Gupta, 2007; PN-EN 450-1:2012).

The reduction of carbon dioxide emissions introduced by the EU has encouraged research into next-generation materials containing less clinker, the main component of Portland cement. The development of sewage networks and sewage treatment plants has led to the formation of increasing amounts of municipal sewage sludge, the neutralization and management of which is a serious ecological issue, not only in our country but across the world. In countries such as the UK, the Netherlands, Austria, and Germany, incineration constitutes a significant part of sewage sludge management. In Switzerland, Austria, and the Netherlands, it exceeded half of the total amount of sludge used, in the United States 25%, and in Japan 55% (Werther & Ogada, 1999). In Poland, sludge management is subject to the Act of 14 December 2012 on waste (Legal Act, 2012), as well as executive regulations and laws specific to the method of its production, processing, and environmental impact. Considering the ban on the storage of sewage sludge, its management has become not only an economic and technical, but also an environmental problem (Legal Act, 2013; Regulation, 2015; Sadecka, Myszograj & Suchowska-Kisielewicz, 2011). Currently, after initial stabilization, the generated sewage sludge (after treatment with lime or oxygen or under anaerobic conditions) is released into the environment. Considering the sanitary danger and hydration giving a large mass, sludge disposal has always been a technical problem (Bień et al., 2011; Borowski, Gajewska & Haustein, 2014). Due to the presence of heavy metals and toxic substances preventing its use in agriculture, thermal methods are the most appropriate method of sewage sludge disposal (Środa, Kijo-Kleczkowska & Otwinowski,

2012; Pająk, 2014). As a result of this process, heat or electricity is obtained, the volume of waste (sludge) is reduced, and the content of nitrogen and sulfur compounds in the exhaust gas is reduced (Suzuki, Tanaka & Kaneko, 1997). The secondary material generated during the thermal treatment of sewage sludge in installations is waste (fly ash) with the code 19 01 14, which also requires appropriate management.

Previous studies on fly ash from the thermal treatment of sewage sludge have focused on the assessment of the possibility of using it for composite materials such as ceramics (Merino, Arevalo & Romero, 2007; Lin et al., 2016), fired tiles, bricks (Lynn, Dhir & Ghataora, 2016; Piasta & Lukawska, 2016), mortar, pastes, and concrete (Yusur et al., 2012; Chakraborty et al., 2017; Vouk et al., 2017; Rutkowska, Fronczyk & Filipczuk, 2020). The introduction of a certain amount of ash from sewage sludge as a partial replacement for cement allows concrete with comparable parameters to that made from siliceous and calcareous fly ash to be produced (Rutkowska et al., 2016; Rutkowska et al., 2020). Additionally, the physicochemical and pozzolanic properties of ash from sewage sludge indicate the possibility of using it to produce composite materials (Rutkowska et al., 2018).

So far, no guidelines or standards have been developed for the use of fly ash from the incineration of municipal sewage sludge as a raw material to produce cement-based building materials (e.g., concrete). Due to the small number of experimental studies and implemented processes involving the use of such ash, obtaining additional information about the possibilities of its application is necessary.

The main purpose of this research was to analyze the influence of the physicochemical properties of fly ash from the thermal transformation of sewage sludge on the compressive strength and frost resistance of concrete produced with its participation. As part of our pilot study, the impact of this additive on the course of carbonation and the natural environment was also determined. The obtained test results were compared to control runs using material containing no fly ash in its composition and to the samples with the addition of fly ash from the combustion of hard coal (siliceous ash).

Material and methods

For experimental work, a concrete mixture of ordinary concrete of the C20/25 class with a K2 consistency according to Bukowski was designed

according to PN-EN206+A1:2016-12 by the method of three equations (Jamróży, 2015; PN-EN 206:2016). CEM I 42.5R Portland cement, natural aggregate with a grain size 0–16 mm, water, and an additive were used for sample preparation. Fly ash from the thermal treatment of sewage sludge from the “Płaszów” sewage treatment plant in Kraków and silica fly ash from the combustion of hard coal in a heat and power plant in Warsaw were used as mineral additives. For each composition of the concrete mix, a constant granulometric composition of the aggregate selected by the method of boundary curves was maintained.

To determine the effect of individual types of fly ash on the compressive strength, carbonation, and frost resistance of ordinary concrete, three types of samples were prepared:

- CON – no addition,
- FA-I – with the addition of fly ash from the thermal treatment of sewage sludge in an amount ranging from 5% to 20%,
- FA-II – with the addition of silica ash in an amount ranging from 5% to 20%.

The composition of the prepared concrete in kg per m³ is shown in Table 1.

To determine the properties of the prepared mixtures, the following tests were carried out: consistency by the drop cone method (PN-EN 12350-2:2011), apparent density (PN-EN 12350-6:2011), and air content by the pressure method (PN-EN 12350-7:2011). According to PN-EN 12390-3:2011, after 28 and 56 days of maturation in a Matest (Italy) H011 hydraulic testing machine, a compressive strength and frost resistance test was carried out

Table 1. Concrete mix proportions by weight

Specification	Mass of concrete ingredients [kg/m ³]			
	Water	Aggregate	Cement	Fly ash
Concrete CON	164.58	1912.73	352.26	–
Concrete with 5% fly ash – FA5%	164.58	1912.73	334.65	17.61
Concrete with 10% fly ash – FA10%	164.58	1912.73	317.04	35.22
Concrete with 15% fly ash – FA15%	164.58	1912.73	299.42	52.84
Concrete with 20% fly ash – FA20%	164.58	1912.73	281.81	70.45

using the direct method in accordance with PN-B-06265:2004 in a Toropol (Poland) chamber. The tests were carried out on samples with dimensions of 10×10×10 cm. The carbonation test was carried out in accordance with PN-EN 13295:2005 on 10×10×50 cm beams in a carbonation chamber. The depth of the carbonation front was measured at a CO₂ concentration of 3% after 56 days from placing the samples in the chamber. A phenolphthalein solution (1 g of phenolphthalein per 70 g of ethyl alcohol, diluted in 30 g of distilled water) was used for the measurement. The fly ash employed was tested to determine its physicochemical properties. Table 2 shows the research methods used.

Leachability tests were performed on a concrete sample without additives (CON) and on two concrete samples with the addition of 20% ash (FA-I 20%, FA-II 20%). It was assumed that in these tests the content of heavy metals in the eluate would be the highest. The leaching tests were carried out in

Table 2. Scheme of the methodology used in this study (Rutkowska et al., 2018)

Test	Test Method
Composition chemical material	The composition was determined by X-ray energy dispersion fluorescence (XRF) on a Panalytical Epsilon 3 spectrometer. The study was carried out in the measuring range of Na-Am using an apparatus equipped with an X-ray Rh 9 W lamp, 50 kV, 1 mA, a 4096-channel spectrum analyzer, 6 measurement filters (Cu-500, Cu-300, Ti, Al-50, Al-200, Ag), and a high-resolution semiconductor SDD detector (Be window, 50 µm thick) cooled with a Peltier cell.
Schedule grainning	The analysis was performed by laser diffraction using a Mastersizer 3000 analyzer (Malvern Instruments). The measurement was carried out in a dispersing liquid (demineralized water) in the presence of an ultrasonic probe to break up larger aggregates of the tested samples. Grains with equivalent diameters ranging from 0.1 µm to 1000 µm were analyzed.
Morphology and chemical composition in micro-areas	The determination was carried out using an FEI Quanta 250 FEG SEM scanning electron microscope, equipped with a chemical composition analysis system based on the energy dispersion of X-rays – EDS (Energy Dispersive X-Ray Spectroscopy) by EDAX.
Mineral composition	The composition was determined using X-ray phase analysis (XRD). Measurements were made by the powder method using a Panalytical X’pertPRO MPD X-ray diffractometer with a PW 3020 goniometer. A Cu lamp (CuKα = 1.54178 Å) was used as the source of X-ray emission. HighScore X’Pert software was used to process the diffraction data. The identification of the mineral phases was based on the PDF-2 release 2010 database formalized by JCPDS-ICDD.

accordance with the procedures contained in PN-EN 12457-2:2006. After strength tests, the concrete samples were ground to fractions below 4 mm. Distilled water ($EC = 0.0286 \text{ mS/cm}$, $pH = 6.7$) was added to the obtained granulate to obtain a liquid/solid phase with an (L/S) ratio = $100 \text{ ml} \cdot 10^{-1} \text{ g}$ of solid. The samples were placed in 250 ml bottles, which were shaken for 24 h at a speed of 200 spins/min. (speed 200 c.p.m.) on an Elpin+ (Poland) Type 357 rotary shaker with water bath. The tests were carried out in triplicate at a temperature of 20°C . The obtained liquid was filtered through a $0.45 \mu\text{m}$ membrane filter, and then the content of heavy metals (Cd, Cr, Cu, Ni, Pb, Zn, As, Sb, Ba, Hg, Mo, and the non-metal Se) was determined. The content of sulfates and chlorides was determined by titration, while the pH was measured using a Eutech Instruments CyberScan pH 510 pH meter with a GPX-105S pH head (Rutkowska et al., 2018).

Results and discussion

Properties of fly ash

The results of the ignition losses and the analysis of the oxygen composition of silica ash (FA-II) and ash from sewage sludge (FA-I) are presented in Table 3.

The loss on ignition of fly ash from sewage sludge, determining the content of unburned carbon in each sample, was lower than that of silica ash. This was mainly due to the combustion temperature being equal to 850°C and the combustion technology in a fluidized bed furnace. In the ash samples from sewage, the highest percentages were SiO_2 , CaO , Fe_2O_3 , and P_2O_5 , while in the silica ash sample they were SiO_2 , Fe_2O_3 , and Al_2O_3 . The sum of the contents of the three main oxides, SiO_2 , Fe_2O_3 , and Al_2O_3 , in FA-I fly ash was significantly lower than in FA-II fly ash and did not meet the requirements of PN-EN 450-1+A1:2012. In addition, a higher content of phosphates was also observed compared to conventional ash. This is due to the removal of phosphorus from wastewater and its accumulation in sewage sludge. The addition of this type of ash to the concrete slows down the cement hydration process. According to the literature, in the liquid phase of the slurry PO_4^{3-} ions react with Ca^{2+} , precipitating

very sparingly soluble calcium phosphate on the surface of the cement grains in the form of a fine crystalline and poorly water-permeable layer, which significantly hinders the cement hydration process. Cement containing an increased amount of soluble phosphorus compounds binds more slowly (Tkaczewska & Kłosek-Wawrzyn, 2012). The mineral composition of fly ash from the thermal treatment of sewage sludge was dominated by quartz and anhydrite, and it was also supplemented by phosphates in the form of apatite and fluorapatite. Chemical analyzes of micro-areas (SEM-EDS) showed differentiation of the elemental composition, with the dominance of grains with the chemical composition silicon, aluminum, phosphorus, and calcium, next to which there were grains containing silicon and aluminum. Determined in accordance with PN-EN 1097-07:2008, the specific density of ash from sediments was 2770 kg/m^3 , the bulk density was 820 kg/m^3 , while the fineness, determined in accordance with PN-EN 451-2:2017-06, was 46.2%. By analogy to the fluidized combustion of coal dust, it can be concluded that the fly ash grains from sewage sludge were characterized by a high content of grains (grain conglomerates) with a high open porosity, which translated into high water demand. The degree of porosity was 70.86%.

Properties of concrete mix

Based on our results obtained on the fresh concrete mix, it was found that for individual samples the density values of the mixtures were close and fitted a range from 2313 to 2378 kg/m^3 , similar to the results for ordinary concrete ($2000\text{--}2600 \text{ kg/m}^3$). The obtained results were not very homogeneous. It has been observed that applied sediment ash has a negative influence on the consistency of concrete mixtures because it is characterized by high water demand – the mixture is non-workable after a short time. A positive effect of high-water demand is to lower the effective water content in the concrete mixture, which gives higher horizontal endurance on compression (especially early endurance). In practice, to assure appropriate consistency (workability) of the concrete mixture, superplasticizers or larger amounts of water may be added, which in effect

Table 3. Oxygen composition of fly ash

Component designation	SiO_2	Fe_2O_3	Al_2O_3	CaO	MgO	TiO_2	SO_3	Na_2O	K_2O	P_2O_5	Roasting loss
FA-II	49.0	5.20	27.5	3.43	2.58	2.00	0.43	0.56	0.30	0.50	8.21
FA-I	32.21	19.25	6.25	11.90	3.14	1.20	1.82	0.80	2.11	18.91	0.52

causes lower endurance on compression, concrete shrinkage, and low durability. For samples containing silica ash, K2 consistency was obtained. The greatest content of air was 3.5%, found with FA-I, while the lowest was 2.4%, found with FA-II.

Properties of concrete

Compressive strength

The results of the measurements of the average compressive strength of concrete samples with a variable addition of fly ash are shown in Figure 1. They take into account the physicochemical composition of the fly ash used from the thermal treatment of sewage sludge and from the combustion of hard coal. It was noted that a higher concentration of SiO_2 , Al_2O_3 , and Fe_2O_3 and a lower concentration of P_2O_5 and CaO had a positive effect on the increase in the compressive strength of the produced concrete samples. The highest compressive strength after 28 days of maturation, 48.9 MPa, was obtained for the concrete sample FA-II 20%, in which the cement was replaced by 20% coal fly ash, while the lowest compressive strength, 35.6 MPa, was for the sample FA-I 5%, in which cement was replaced with ash from the sewage treatment plant. Compared to the reference concrete samples, the increase in strength was 18.3%, and the decrease was 13.9%. The highest compressive strength after 56 days of maturation, 50.8 MPa, was also achieved by FA-II 20% concrete samples, while the lowest strength, 39.4 MPa, was

found with FA-I 5% samples. According to the information presented in previous studies, the optimum amount of fly ash from thermal treatment of sewage sludge (FA-I) in cement composites ranges from 5% to 20%. The presence of this type of ash in an amount greater than 20% by weight of the cement mass delays the setting process of the slurry and gives slower growth of the compressive strength of the concrete compared to composites made with the use of ash from coal combustion or only Portland cement. However, by extending the maturation time, the strength required for structural concretes can be obtained (Monzo et al., 2003; Fontes et al., 2004; Yen, Tseng & Lin, 2011; Rutkowska et al., 2018; Rutkowska et al., 2020).

Frost resistance

The frost resistance test consisted in determining the decrease in the compressive strength of a frozen sample in relation to a non-frozen sample. The compressive strength reduction should not exceed 20%. According to the PN-88/B-06250 standard, samples subjected to freezing should not have cracks, and the weight loss should not exceed 5%. Table 4 summarizes the compressive strength results of the comparative samples and those after 150 freezing cycles.

Considering the comparative samples (without freezing), FA-I 15% had the highest compressive strength out the FA-I samples (47.8 MPa) and FA-II 20% concrete had the highest compressive strength

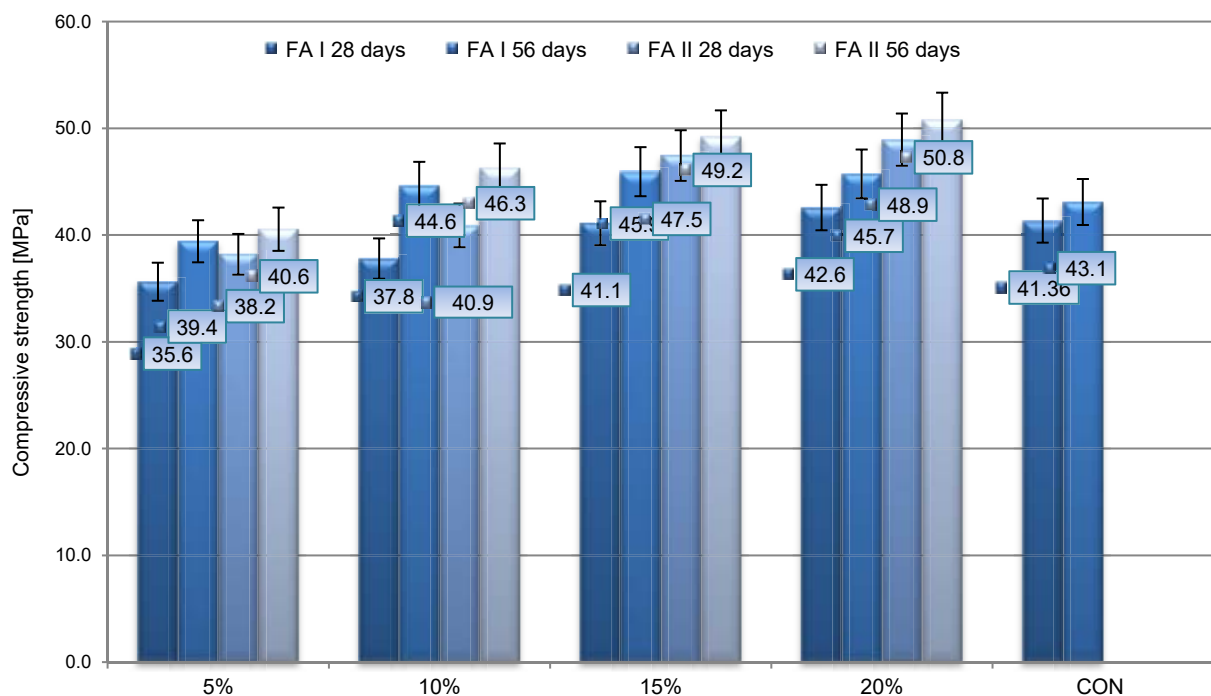


Figure 1. Compressive strength after 28 and 56 days maturation

Table 4. Summary of the average strength loss of the samples subjected to freezing and the average weight loss of the samples

Sample	Mean compressive strength		Average strength loss of frozen samples	Average weight		Average mass loss
	before freezing	after 150 cycles of freezing		before freezing	after 150 cycles of freezing	
	[MPa]	[MPa]	[%]	[g]	[g]	[%]
CON	44.1	41.4	-6.12	2366	2362	0.169
FA-I 5%	42.8	39.9	-6.78	2416	2409	0.289
FA-I 10%	45.9	43.9	-4.36	2361	2342	0.804
FA-I 15%	47.8	45.9	-3.97	2329	2331	0.086
FA-I 20%	42.9	40.0	-6.76	2312	2308	0.173
FA-II 5%	44.8	40.9	-8.71	2405	2397	0.333
FA-II 10%	47.2	45.6	-3.39	2395	2390	0.209
FA-II 15%	49.9	47.7	-4.41	2352	2350	0.085
FA-II 20%	50.1	48.0	-4.19	2349	2336	0.557

overall (50.1 MPa). The smallest strength, 42.8 MPa, was seen for FA-I 5% samples. When analyzing the compressive strength of the samples after 150 freezing cycles, it was observed that the best parameters were obtained for samples with a 20% content of ash from coal combustion (FA-II 20%), as before freezing. The lowest compressive strength, 39.9 MPa, after 150 freezing cycles was noted with concrete with 5% ash content from sewage sludge (FA-I 5%). The average strength loss of the frozen samples never exceeded 9%. The lowest decrease in strength occurred for the FA-II sample with a 10% share of silica ash, and the highest for the FA-II sample with a 5% share of silica ash. The values were 3.39% and 8.71%, respectively. The mean weight loss after 150 freezing cycles was insignificant and ranged from 0.085% for the FA-II 15% sample to 0.557% for the FA-II 20% sample. The samples used in this study were subjected to 150 cycles of freezing and thawing and the action of water, so therefore they can be assigned a degree of frost resistance of F150.

Leachability

In Poland, in accordance with the regulations (Ordinance, 2016) introducing Directive EU/2010/75 (Directive, 2010), fly ash from the thermal treatment

of sewage sludge may be used for the preparation of concrete mixtures when the concentration of heavy metals in aqueous extracts from the leachability test from concrete samples does not exceed 10 mg/L in total, calculated based on the mass of these elements. The total concentration of heavy metals in the analyzed samples did not exceed the permissible value. The results of the leachability tests are presented in Table 5.

In the case of ash from sewage sludge, the leachability of heavy metals from concrete samples was higher than from ash, but it did not exceed the limit values for hazardous waste. For silica ash, the leachability of heavy metals from ash was higher compared to the leachability of concrete samples containing this ash. Considering all types of samples, it was observed that the leachability of heavy metals from samples with the addition of fly ash was comparable or lower than that of the reference concrete without additives. According to the literature on the subject (Monzo et al., 2003), the leachability of trace elements from mature concrete does not pose a threat to the safety of the natural environment when the ash content from the thermal treatment of sewage sludge does not exceed 30% of the cement mass. Therefore, considering environmental standards and technical specifications, it can be assumed that the use of ash

Table 5. Leaching values of heavy metals from the tested samples

Sample type	Heavy Metals (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-II	< 0.002	< 0.01	0.004	< 0.005	< 0.003	< 0.030	< 0.010	< 0.010	< 0.010	1.68	< 0.005	0.02
FA-I	< 0.002	< 0.010	0.008	< 0.005	< 0.003	< 0.030	< 0.010	0.020	0.047	0.11	< 0.005	0.40
FA-II 20%	< 0.002	0.020	0.010	< 0.005	< 0.003	< 0.030	< 0.010	< 0.010	< 0.010	0.75	< 0.005	< 0.010
FA-I 20%	< 0.002	0.018	0.015	< 0.005	< 0.003	< 0.030	< 0.010	< 0.010	< 0.010	1.12	< 0.005	< 0.010

from sewage sludge in composite materials, such as concrete, seems possible (Chen et al., 2013). The eluates of the analyzed materials were characterized by high pH values, which resulted from the presence of oxides in the samples. The lowest pH value of 9.6 was recorded for the ash sample from the combustion of municipal sewage sludge, while the highest pH value of 12.4 was obtained for concrete without any additive. For this material, the lowest values of other physicochemical parameters were also observed. The chloride concentration ranged from 7.9 mgCl/l (for sediment ash) to 75.3 mgCl/l (for sediment ash concrete samples).

Carbonation

During the carbonation process, the carbon dioxide in the air reacts in the presence of moisture with the calcium hydroxide contained in the concrete structure. As a result, water and calcium carbonate are formed. This process destroys the concrete. The measured depth of carbonation in the tested samples was 1–4 mm, which was undoubtedly influenced by the maturation of the samples in water; a smaller depth of carbonation was observed in the case of concrete samples with the addition of fly ash from sewage sludge, which can be explained by its low permeability. However, the microstructure of the concrete made with the cement with this addition did not protect against carbonation, the rate of which is faster than in the case of concrete made of Portland cement (Piasta, Sawicz & Piasta, 2008). It should be remembered that the pace of the carbonation process changes over time, and therefore the experimental research that has already started needs to be continued. The rate of carbonation advance depends mainly on the operational conditions of the concrete structure. Increased CO₂ concentration and high humidity accelerate the carbonation process.

Conclusions

The conducted research confirms the possibility of using fly ash from the thermal treatment of sewage sludge in the production of concrete. The results and their analysis allowed the following conclusions to be drawn:

1. The use of fly ash generated in sewage treatment plants from the thermal treatment of sewage sludge (waste with the code 19 01 14) brings economic benefits. The ash used to prepare the concrete mixture had a positive effect on its compressive strength and frost resistance. Additionally,

the concrete mixtures did not adversely affect the natural environment.

2. The physicochemical composition of ash from thermal transformation of sewage sludge was different to fly ash from hard coal combustion and did not meet the requirements of PN-EN 450-1:2012. The highest percentage of the ash samples were oxides of silicon, calcium, phosphorus, and aluminum. The sum of the content of SiO₂, aluminum oxide (Al₂O₃), and iron oxide (Fe₂O₃) was 57.7% for ash from sewage sludge and 81.7% for ashes from hard coal combustion.
3. The high content of phosphorus compounds significantly affected the binding properties of concrete mixtures prepared with fly ash from municipal sewage sludge. The share of the tested additives in the mixture delayed the beginning of setting and hindered its workability.
4. Concrete containing fly ash from sewage sludge as a cement substitute in an amount of up to 20% was characterized by a compressive strength comparable to that of the comparative concrete without the addition. The average compressive strength for concrete containing ash from the thermal treatment of sewage sludge was 42.6 MPa and 45.7 MPa after 28 and 56 days of maturation, respectively. These results show that strength values of concrete above the minimum of 25 MPa could be obtained.
5. Concrete containing ash from sewage sludge gave satisfactory strength parameters after 150 freezing and thawing cycles. Concrete samples made with 5% – 20% of ash were frost resistant.
6. The total concentrations of heavy metals washed out from the ash from the thermal treatment of sewage sludge and from concrete samples containing this ash were compatible with its use in the production of concrete. However, they were higher than the concentrations obtained for concrete samples containing silica and calcareous ashes.

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