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The influence of ashes from the combustion of municipal sewage sludge on the strength properties of concrete containing recycled ceramic aggregate

Wpływ popiołów ze spalania komunalnych osadów ściekowych na cechy wytrzymałościowe betonu zawierającego recyklingowe kruszywo ceramiczne

Original research paper (Oryginalny artykuł naukowy)

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Abstract. The ecological awareness of society and, above all, the need to base the country's economy on sustainable development mean that the issue of impact on the natural environment is an issue that is increasingly discussed by concrete technologists. At the same time, it fits into the broadly understood trend of a closed-loop economy, in which the possibility of generating waste, also in construction, is minimized. Numerous studies are being undertaken to reduce the energy consumption of solutions for the construction sector and to reduce the amount of waste generated. The article presents selected results of tests on the mechanical properties of concrete containing waste ceramic aggregate and fly ash from the combustion of sewage sludge. The concrete was designed based on Portland cement CEM I 42.5 R. The results of compressive strength tests after 28 and 90 days of curing and Young's moduli were summarized. The influence of increased temperatures of 300, 450 and 600°C on the strength properties of concrete was also determined. The conducted research clearly demonstrated the possibility of using recycled aggregate from the crushing of fine ceramics and fly ash from the combustion of sewage sludge in construction concrete.
Keywords: ash from sewage sludge incineration; ceramic aggregate; compressive strength; high temperature.

Streszczenie. Świadomość ekologiczna społeczeństwa, a przede wszystkim konieczność dążenia do zrównoważonego rozwoju w gospodarce powoduje, że wpływ betonu na środowisko naturalne jest coraz częściej omawiany. Wpisuje się to jednocześnie w szeroko rozumiany trend gospodarki o obiegu zamkniętym, w której ogranicza się do minimum możliwości powstawania odpadów także w budownictwie. Podejmowane są liczne badania mające na celu zmniejszenie energochłonności rozwiązań dla sektora budowlanego oraz zmniejszenia ilości wytwarzanych odpadów. W artykule przedstawiono wybrane wyniki badań cech mechanicznych betonu zawierającego odpadowe kruszywo ceramiczne i popiół lotny ze spalania osadów ściekowych. Określono także wpływ podwyższonej temperatury 300, 450 i 600°C na właściwości wytrzymałościowe betonu. Przeprowadzone badania jednoznacznie wykazały możliwość stosowania kruszywa recyklingowego pochodzącego z przekruszenia ceramiki szlachetnej oraz popiołów lotnych ze spalania osadów ściekowych w betonach konstrukcyjnych.
Słowa kluczowe: popiół ze spalania osadów ściekowych; kruszywo ceramiczne; wytrzymałość na ściskanie; wysoka temperatura.

The dry weight of sludge from industrial and municipal sewage treatment plants amounted to 1,025.8 thousand tons in Poland in 2021 [1]. One of the available methods for neutralizing this type of waste and reducing it significantly is its thermal transformation. This method includes combustion, co-combustion and gasification. As a result of using this method, a significant reduction in the mass and volume of waste is achieved, which in the case of combustion may amount to 90% of the volume and 65% of the initial mass [2]. The percentage share of thermal methods in the management of municipal sewage sludge in EU countries is approximately 18% [3]. The Netherlands dominates among European countries, where 100% of sewage sludge is neutralized using this thermal method. Belgium takes second place with a score of 90% [4]. As a result of burning municipal sewage sludge, we obtain significant amounts of ash. This ash is increasingly used, among others, in construction as a substitute for cement. Research on the use of ash generated from the incineration of sewage waste (Sewage Sludge Ash – SSA) is increasingly being conducted around the world.

Review of the literature

Sewage waste is characterized by different chemical composition, which results in ash of differing composition. The authors [5] used the additive (SSA) in the amount of 10 – 40% as a replacement for cement in concretes based on Portland

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cement CEM I. The obtained results confirmed that replacing 10% of cement with the addition of SSA has a positive effect on the compressive and tensile strength when bending, as well as porosity. total, ability to transport chloride ions and resistance to sulfate ions. In [6] an extensive literature analysis was carried out on the use of SSA in concrete. Due to its structure and chemical composition, it was assessed that this material can be used as a raw material for the production of cement clinker and lightweight aggregates, fine aggregates, filling aggregates and in ground form as a component of cement. In the article [7] the influence of SSA on the flowability and strength of ultra-high-strength concretes (UHPC) was investigated. The results showed that the compressive strength of UHPC samples with 10% SSA after 7 and 28 days of maturation was slightly higher compared to the base samples. The SSA content contributed to reducing the heat of hydration and increasing the pore volume of the modified concrete. In the study [8], the acid extraction method of phosphorus from SSA was used. Phosphorus-free ash (AW-SSA) was used as a partial replacement for sand or cement in the concrete mix. With a 10% cement replacement, the mortar had a similar 28-day compressive strength to the base mortar. It was found that AW-SSA probably has a latent hydraulic character, positively influencing the late development of strength. The compressive strength of the mortar with 10% cement replacement after 42 days was higher than the reference one by about 5 MPa. The work [9] assessed the behavior of cement slurries containing sewage sludge ash (SSA) at elevated temperatures (600 – 1000°C). The results showed that the slurry containing 10% SSA (S10) showed higher residual compressive strength than the plain cement slurry (S0). Increased temperatures caused the decomposition of hydrate products, thickening of pore structures and the formation of cracks. The addition of SSA promoted greater formation of glenite at temperatures of 900 – 1000°C, and such a phase can fill the pores in the matrix. The influence of SSA addition on the properties of cement mortars was determined in articles [10 – 12]. Research has identified the mechanisms behind some of the beneficial effects of SSA on the strength development of mortars through a comparative study with sewage sludge fine ash (FSSA). The results of this study indicate that the presence of SSA accelerates the rate of heat release of cement hydration. Replacing cement with SSA or FSSA up to 10% did not cause significant changes in the pore structure of the slurries. The formation of bruschite in SSA or FSSA cement mortars contributes to the increase in the strength of the mortars after a longer setting period. It was found that the addition of SSA caused the formation of a larger amount of ettringite in mortars under water immersion conditions and the mortars expanded already before the 7th day of maturation. Research has shown that replacing 15% of Portland cement with SSA and its fractions: coarse-grained (SSAC) and medium-grained (SSAM) results in an increase in compressive strength. In work [13], the properties of mortars with the addition of ash from septic tank sludge (SA) were examined. Cement and sand were used in mass proportions of 1 : 3. Percentage additions of sludge ash were 5 – 30% in relation to the weight of cement. The obtained results indicate that the addition of SA improves the general properties of mortars, and the addition of 20% SA was considered the most optimal. The possibility of using aggregates from the crushing of sanitary ceramics as full-value substitutes for natural aggregates in mortars and concretes was confirmed in [14]. The procedure for producing aggregate (crushing, dividing grains into two groups – fine and coarse, and determining their proportions) and designing the concrete mixture are described. Tests on the properties of this aggregate and the properties of concrete containing this aggregate were presented. The tested concrete was characterized by high strength and high abrasion resistance. Particularly favorable results were obtained when using clay cement and samples subjected to thermal loads up to 1000°C. Based on the conducted research, it was confirmed that aggregate for sanitary ceramics can be recommended for making special types of concrete: abrasion-resistant concrete and concrete intended for elements operating at high temperatures. The possibility of using sanitary ceramics in ordinary concrete was also confirmed in work [15], in which concrete mixtures were designed using CEM 32.5R cement and only aggregate from crushed sanitary ceramics of various fractions.

Materials and methods

The tests used standard cubic (10 cm x 10 cm x 10 cm) and cylindrical concrete samples (30 cm high and 15 cm in diameter) in accordance with the standard [16]. The average values of compressive strength and Young's modulus of a given series of samples were obtained by testing at least three samples. Compressive strength tests were performed on an Advantest 9 testing machine, and Young's modules were tested using the WalterBai system, which cooperates with extensometers located on cylindrical samples. The samples were made of the following ingredients: waste ceramic aggregate of the 0 – 4 mm and 4 – 8 mm fraction, which was obtained from products that did not meet the quality criteria of one of the leading domestic manufacturers of sanitary fittings, and ash obtained from thermal treatment of sewage sludge from the Płaszów II sewage treatment plant in Kraków and Portland cement CEM I 42.5 R from Cementownia Ożarów SA in Ożarów. Four series of samples were made: base samples and three series containing 5, 10 and 15% of ash replacing cement. The research aimed to determine the impact of the addition of ash on the physical and strength characteristics of the modified concrete. Compressive strength after 28 and 90 days of setting and the influence of temperatures (from 300 to 600°C) on compressive strength were determined. Table No. 1 lists the proportions of materials used to prepare the samples.

After curing in accordance with standard requirements [16], the samples were dried at a temperature of 105 ± 5°C to constant weight in a KC-100/200 laboratory dryer. Then they were heated in a special PK 1100/5 furnace equipped with a heating chamber. The station has dedicated ThermoPro software that allows you to program the thermal process. The heating time depended on the programmed temperature. The temperature was maintained in the oven for 30 minutes until the heating process was

Table 1. Composition of concrete mixtures [kg/m³]
Tabela 1. Skład mieszanek betonowych [kg/m³]

Ingredients	Designation of a series of samples			
	CP0c	CP5c	CP10c	CP15c
Cement (I 42,5 R)	488,0	463,6	439,2	414,8
Ash (SSA)	0,0	24,4	48,8	73,2
Ceramic aggregate (4 – 8 mm)	997,0	997,0	997,0	997,0
Ceramic aggregate (0 – 4 mm)	398,0	398,0	398,0	398,0
Water	196,0	196,0	196,0	196,0

completed. The temperature distribution in the furnace was monitored by a regulating thermocouple inserted through the rear wall. The thermocouple is located near the top of the furnace. Figure 1 shows the actual temperature distributions during heating in the PK 1100/5 special furnace recorded by the ThermoPro software. The standard „temperature-time" curve in accordance with the requirements [17] was adopted as the basis for the heating process.

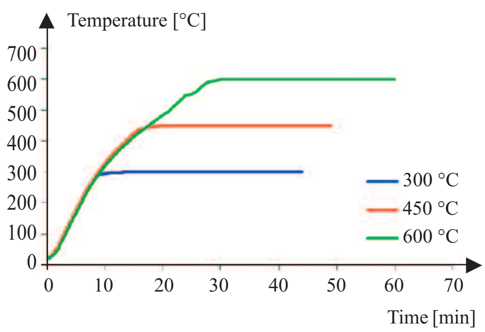

Fig. 1. Temperature distribution in the furnace according to the external thermocouple
Rys. 1. Rozkład temperatury w piecu wg termopary zewnętrznej

A FEG Quanta 250 scanning electron microscope was used to examine the morphology and elemental composition of concrete. Mechanical tests were carried out using an Advantest 9 Controls hydraulic press. The obtained results were compared with reference samples. The research was carried out at the Construction Laboratory of the Lublin University of Technology.

Results

The results of tests on the chemical composition of the component materials: waste ceramic aggregate, ash and cement are summarized below. The compressive strengths of four series of concretes were also presented, taking into account the influence of heating temperatures (300, 450 and 600°C). The chemical composition and morphology were determined using a Quanta 250 FEG SEM microscope. The chemical composition of the tested fly ashes was determined using the XRF energy dispersive X-ray fluorescence method. The mineral composition was determined by X-ray phase analysis (XRD). Measurements were made using the powder method using an X'pertPRO MPD X-ray diffractometer with a PW 3020 goniometer.

Test results for ash from the combustion of sewage sludge and 43.5 R cement and recycled ceramic aggregate. Photos 2, 3 and 4 respectively show the microstructures and EDS analysis results from the image areas of ceramic aggregate, ash from the combustion of

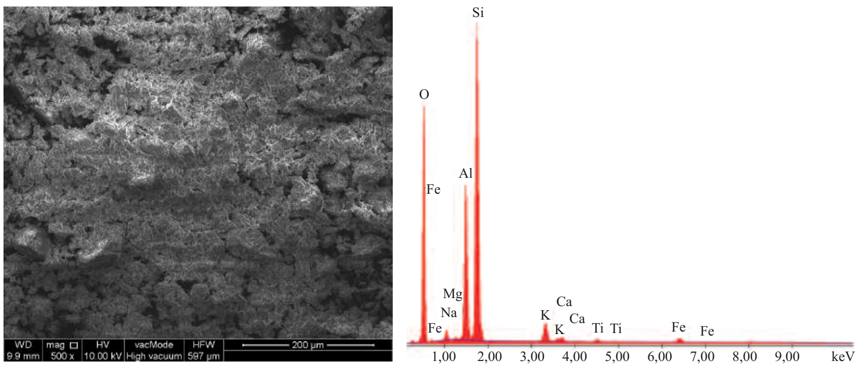

Photo 1. View of the microstructure of ceramic aggregate (500x magnification). EDS elemental analysis from the area visible in the photo
Fot. 1. Widok mikrostruktury kruszywa ceramicznego (powiększenie 500x). Analiza pierwiastkowa EDS z obszaru widocznego na zdjęciu

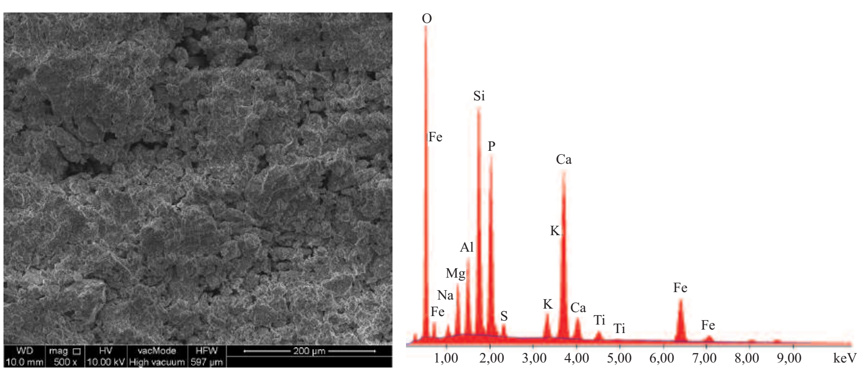

Photo 2. View of the microstructure of ash from sewage sludge (500x magnification). EDS elemental analysis from the area visible in the photo
Fot. 2. Widok mikrostruktury popiołu z osadów ściekowych (powiększenie 500x). Analiza pierwiastkowa EDS z obszaru widocznego na zdjęciu

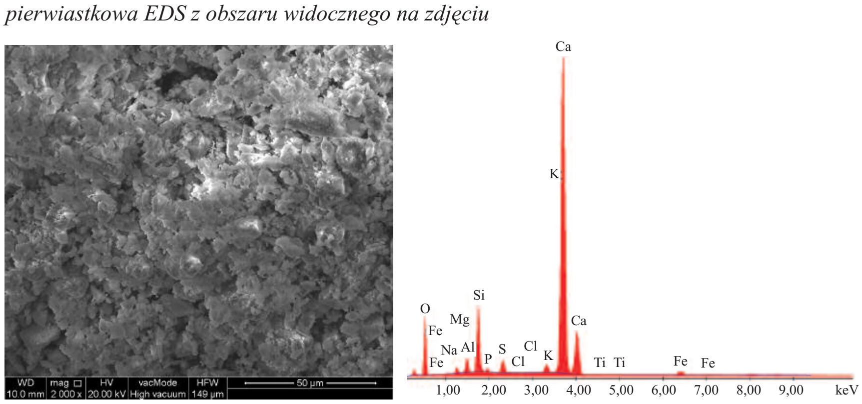

Photo 3. View of the cement 42,5 R microstructure (2000x magnification). EDS elemental analysis from the area visible in the photo
Fot. 3. Widok mikrostruktury cementu 42,5 R (powiększenie 2000x). Analiza pierwiastkowa EDS z obszaru widocznego na zdjęciu

municipal waste and 42.5R Portland cement. Table No. 2 summarizes the results of testing the oxide composition of the above-mentioned products. ceramic aggregate, ash and cement.

Table 2. Oxide composition of ceramic aggregate, ashes and Portland cement CEM I 42.5 R
Tabela 2. Skład tlenkowy kruszywa ceramicznego, popiołów i cementu portlandzkiego CEM I 42,5 R

Oxide	Fe_2O_3	Na_2O	MgO	Al_2O_5	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2
	[%]									
Aggregate	2,79	1,57	0,35	29,95	61,19	0	0	2,72	0,71	0,73
Ash	13,51	1,18	4,56	9,51	24,64	23,62	1,81	1,91	17,64	1,62
Cement	2,06	0,20	1,21	4,03	14,95	0,29	3,41	1,19	72,34	0,22

Comparing the oxide composition of cement, in which the sum of the percentages of SiO_2 and CaO, responsible for the hydration of the clinker phases, is approximately 80%, and the ash in question, in which approximately 40% of these compounds were found, it can be concluded that the addition in the form of ash does not have significant hydraulic properties. The results of testing the oxide composition of the ceramic aggregate showed mainly silica SiO_2 (61.19%) and aluminum dioxide Al_2O_3 (29.95%). In concretes that require high resistance to high mechanical loads and aggressive environmental influences, it is necessary to use aggregates with high resistance to crushing, i.e. with a low Los Angeles LA coefficient. The ceramic aggregate test was carried out on the 4÷8 mm fraction. Before testing, the aggregate was washed and then dried at a temperature of 110 ±5°C. As a result of the tests, an LA coefficient value of 22.4 was achieved, therefore the aggregate can be classified as LA25. As part of the research work, frost resistance tests were also carried out. The test results showed a weight loss during the frost resistance test of 0.29%, which is ≤ 1%, which, in accordance with the applicable requirements, allows the obtained ceramic aggregate to be classified in the F1 category. At the same time, it was determined that the average bulk density of the aggregate is 2403 kg/m³ and the average water absorption is 1.98%. This means that the density of ceramic aggregate is similar to that of natural rocks such as granite or marble, and the water absorption is similar to that of dolomite or gravel with a predominance of carbonate grains. However, the water absorption of the tested aggregate is much lower than that of other recycled aggregates, such as crushed RCA concrete, the water absorption of which in various tests ranges from 3 to 15% [18]. It is also important that the sanitary ceramics aggregate does not show any changes in structure as a result of heating at high temperatures. The tests of the aggregate structure were preceded by heating aggregate grains of the 4 ÷ 8 mm fraction at temperatures of 300°C and 600°C in a PK1100/5 chamber furnace. After reaching the assumed temperature, it was then maintained in the furnace chamber for an hour. The surface of the ceramic aggregate does not change its structure and form after annealing. Irregular pores are still visible throughout the material. Importantly, the tests did not reveal any microcracks formed during the heating of the ceramic aggregate. However, such damages were visible in tests of basalt aggregate which, after heating at 600°C, showed a number of irregular cracks. The tests carried out on the technical characteristics of recycled aggregate based on crushed ceramic cullet clearly show that its features do not differ significantly from those of natural aggregates, and in selected cases such as the resistance to high temperatures even significantly exceeds them [19].

Results of mechanical tests of concrete samples with the addition of ash from sewage sludge. Figure 2 shows the compressive strength of concrete cubic samples with edge dimensions of 10 cm. Replacing cement by 10% resulted in almost identical compressive strength values compared to the base samples after 90 days of curing.

In order to take into account the subsequent increase in compressive strength of concrete samples caused by the addition of ash (SSA), the effect of high-temperature loading of concrete was checked after 180 days (Fig. 3). Similar compressive strength results were obtained for samples with 5 and 10% SSA content compared to the base samples heated at 300°C. The highest values of compressive strength after heating at 600°C were obtained by samples of the CP5c and CP10c series.

Below (Fig. 4) are the results of Young's moduli of four series of samples tested after 28 days of maturation. The highest value of 37.66 GPa was achieved by CP0c series concrete.

Conclusions

Based on the results obtained, it can be concluded:

1. Ash from thermal processing of sewage waste in the amount of 5 – 15%

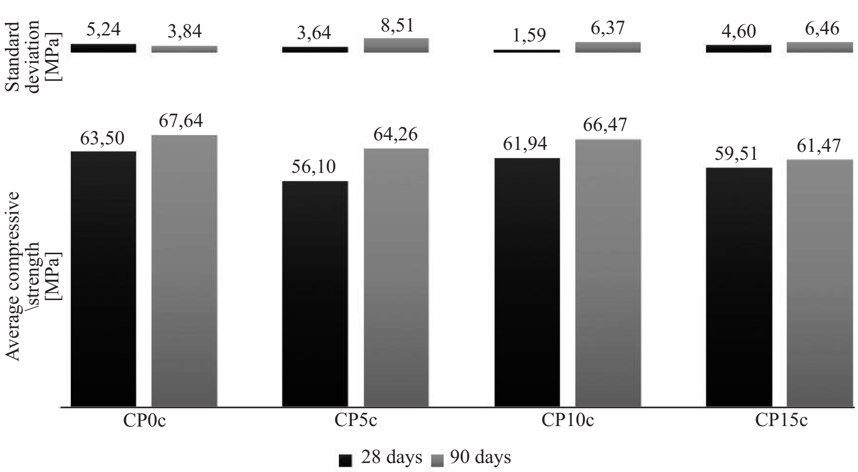

Fig. 2. Average compressive strength and standard deviation of samples with different ash content measured after 28 and 90 days of curing
Rys. 2. Średnia wytrzymałość na ściskanie i odchylenie standardowe próbek z różną zawartością popiołu po 28 i 90 dniach dojrzewania

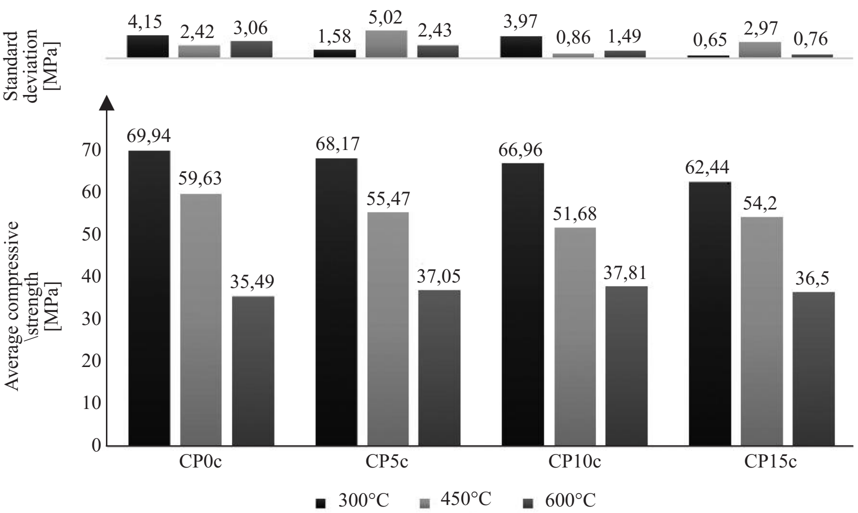

Fig. 3. Average compressive strength and standard deviation of samples with different ash content measured after 180 days of curing and heated at temperatures of 300, 450 and 600°C
Rys. 3. Średnie wytrzymałości na ściskanie i odchylenie standardowe próbek z różną zawartością popiołu mierzone po 180 dniach dojrzewania i poddanych wygrzewaniu w temperaturze 300, 450 i 600°C

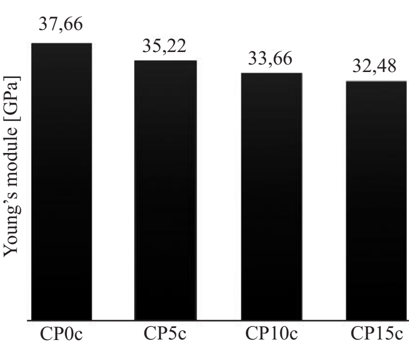

Fig. 4. Young's module of concrete samples with different ash content
Rys. 4. Moduł Younga próbek betonowych z różną zawartością popiołu

can be used as a partial replacement for cement in a concrete mix based on waste ceramic aggregate.

2. Tests of the phase composition of ash from thermal processing of waste revealed partially hydraulic components. The conducted research shows that the initial binding process itself is slower than in the reference samples. After a period of 90 days of maturation, similar values of compressive strength of the samples (CP5c, CP10c and CP15c) were obtained in relation to the base samples. The highest increase in compressive strength after 90 days of maturation, amounting to almost 15%, was observed in the CP5c series samples.

3. As a result of heating the samples to temperatures in the range of 300 – 600°C, it should be stated that with the increase in the heating temperature, the residual strength of the samples decreases. It is clearly visible that increasing the addition of ash does not affect the nature of changes in strength, because both for the reference samples and those with the addition of ash, the strength drops are comparable at all heating temperatures. At the same time, a certain anomaly was noticed – the heating temperature of 300°C resulted in a several percent increase in the strength of all types of samples in relation to the reference samples for which the strength was determined after 28 and 90 days of curing.

4. It can be clearly stated that the addition of fly ash from the combustion of sewage waste reduces the modulus of longitudinal elasticity – Young's modulus. This decrease is proportional to the amount of fly ash added. It should be emphasized, however, that the difference between the reference sample and concrete marked CP15c is approximately 15%.

5. The conducted research clearly showed that it is possible to use this type of ash in concrete with 100% recycled aggregate. The obtained mechanical properties and relatively high resistance to elevated and high temperatures make this type of concrete a relatively easy way to reduce the amount of waste generated as a result of human activity.

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