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METHODOLOGY OF EXPERIMENTAL RESEARCH OF THE PROPERTIES OF CEMENT-SAND MIXTURE WITH THE ADDITION OF MECHANICALLY ACTIVATED FLOW ASH FROM THE THERMAL POWER PLANT

Abstract. A methodology for experimental research on the properties of cement-sand mixture (CSM) with the addition of mechanically activated thermal power plant (TPP) fly ash has been developed. The preparatory stage consisted of mechanical screening of fly ash on a multi-frequency vibrating screen (MVS) to obtain a minus 40 μm fraction, which is the most valuable for cement production and provides a dense microstructure. Mechanical activation of fly ash was carried out by grinding the minus 40 μm fraction in the chamber of a vibration mill with multi-frequency vibration and impact excitation. The process of mechanical activation of fly ash in a vibrating multi-frequency ball mill is based on the transfer of kinetic energy of the grinding bodies (balls) of the material to destroy its crystal lattice and increase the specific surface area. The effectiveness of activation is determined by the energy intensity of the unit and compliance with the regulated parameters. Mechanical activation of the 40 μm fraction was carried out in two time regimes: mechanical activation lasting 15 min. and 30 min. The storage time of mechanically activated ash until the samples were manufactured was also varied. To determine the optimal content of fly ash, the percentage of its addition to the cement-sand mixture (CSM) was varied. The cement mortar was prepared with a cement to sand ratio of 1:3 by weight with the addition of water and fly ash in a specified amount. The mixture was placed in special molds measuring 100×100×100 mm for the manufacture of samples. The load on the sample was applied smoothly and continuously at a constant rate of 140 kg/cm² per minute, in accordance with standards. The maximum load reached at the moment of failure and the nature of the failure were recorded. The highest strength of the samples was observed at a 10% fly ash content, which coincides with the content standards regulated by the DSTU. The ratio of the strength of samples with the addition of mechanically activated ash to the strength of samples with ordinary ash at its 10% content was 1.35, for ash content from 20 to 50% this ratio was on average 1.08-1.2. The results of research using the developed methodology confirmed the feasibility of using a vibrating multi-frequency mill with vibration-shock excitation of the grinding chamber for grinding and mechanical activation of fly ash.

Keywords: methodology, experimental studies, properties of CSM, mechanically activated TPP fly ash.

1. Introduction

CSM is considered a key material used in modern construction. One of the most important factors in the reliability of a concrete slab is its strength. Strength largely affects the load-bearing capacity of structures. It is with the strength indicator that a large number of other physical and mechanical properties of CSM are associated. There are several methods for determining the strength of CSM samples. The most relevant, accurate, and popular research method is considered to be destructive. This method involves testing specially manufactured specimens of a specific shape and size for compression using a hydraulic test press.

After that, the research results are analyzed and a conclusion is made about the strength.

Improving the properties of CSM and building composites based on it is one of the main tasks facing technologists. This is an extremely important and urgent problem, to solve which various methods are used, among which promising are mechano-chemical methods of activating the binder, the use of surfactants and mineral fillers. The issues considered are related to determining the effect of adding mechanically



activated fly ash on the properties of cement-concrete mixtures and building compositions based on them. The activation of fly ash is relevant for this study. The amount of fly ash was adjusted in the range from 0% to 20% of the cement mass. The use of this technology ensures acceleration of cement hydration processes, preservation of the required level of mobility of compositions with lower mixing water consumption, and intensification of exothermic heating.

The introduction of ash into the concrete mix, unlike other active mineral additives, usually does not worsen, but improves workability, which is explained by the spherical shape of the ash particles. The introduction of ash helps reduce the water separation of the concrete mix.

The plasticizing and water-retaining ability of ash contributes to its use in lightweight concrete. Concrete mixtures with an optimal ash addition have a fairly high workability and are suitable for transportation over long distances.

The main standards that regulate the determination of the strength of concrete using control samples and the determination of the strength of cement mixtures are [1, 2].

Requirements for chemical composition, physical properties (fineness, loss on ignition), as well as criteria for compliance of fly ash used as an additive in concrete, are established by the standard [3]. The standard [4] details the requirements for fly ash obtained from thermal power plants for its use in the production of concrete.

There are a number of standards for admixed cements. The requirements for mineral additives, including fly ash, added to cement are specified in the standard [5].

It limits indicators such as loss on ignition and free calcium oxide content.

The standard [6], harmonized with the European one, defines the classification, composition and technical requirements for various types of cements, including cements containing fly ash, in particular, as a pozzolanic additive.

There are a number of standards for concrete and mortars.

The standard [7] applies to concrete mixtures, and the use of fly ash in them must meet the requirements for composition, properties and preparation technology.

The standard [8] establishes requirements for heavy concretes, which may include mineral additives, in particular fly ash, to improve certain properties. Standards for road construction. The Standard of the Organization of Ukraine (SOU) [9] regulates the use of fly ash and ash-slag mixtures as materials for road construction and repair.

The standard [10], harmonized with the European one, regulates the use of lime fly ash as a binding component for road construction materials.

Mechanical activation consists of increasing the specific surface area of the starting ash by grinding it. For grinding, the following are used: ball mills, vibratory mills, and hammer mill disintegrators, which are one of the best installations for ultra-fine grinding, including for grinding ash.

This contributes not only to a quantitative increase in the reactivity of reagents, but also has a qualitative effect: the formation of new active surfaces of the aluminosilicate phase containing microdefects, which are characterized by high surface energy and, accordingly, reactivity. At the same time, increasing the specific surface area of ash above 700 m²/kg leads to a decrease in strength due to increased water consumption. Separation of the finer ash fraction (up to 45 or 90 microns) is possible not

only through grinding, but also through separation. This approach helps save energy during the grinding process, but does not allow for the utilization of 100% of the ash.

The phenomenon of mechanical activation of fly ash is investigated in the following works [11–30]. The physicochemical characteristics of mechanically processed fly ash were investigated; dispersion optimization was performed to maximize the strength of composites based on ground fly ash with a high calcium content and Portland cement; the effect of mechanical activation of fly ash on the properties of geopolymers was investigated; the improvement of the reactivity of fly ash and the properties of the resulting geopolymers by mechanical activation was investigated; the mechanical activation of precipitated fly ash from brown coal in a mill with a stirred medium was investigated; the geopolymerization behavior of size-fractionated fly ash was investigated; the effect of mechanical grinding on the physical and chemical characteristics of circulating fluidized bed fly ash from a coal-fired idle power plant; reactivity of fly ash ground in different devices; the effect of mechanical grinding of fly ash powder on the compressive strength of geopolymer; improved processing of high-calcium ash for increased reactivity and improved high-value-added application possibilities; the reactivity of ash was assessed and the role of particle size on the mechanical and microstructural properties of ash-based geopolymer was determined; a comparative study of increasing the pozzolanic activity of ash by dry and wet grinding was performed; the influence of the mixing ratio of precursors and the rotation speed of mechanically activated ash on the properties of geopolymer foam concrete was investigated; activation of high-alumina ash using ball milling, etc.

For mechanical activation of fly ash, traditional ball, planetary, and jet mills with different grinding modes are usually used.

At the same time, the energy and time costs for the process of mechanical activation of ash are significant. Also, a certain strength limit of products from CSM with the addition of fly ash has been achieved. This requires improving methods and means of grinding and mechanical activation to increase the strength and improve the properties of CSM with the addition of fly ash.

The authors propose to carry out the process of grinding and mechanical activation of fly ash in a vibrating multi-frequency mill [31], which will help reduce energy and time costs and improve material quality indicators.

The multi-frequency vibrating mill is designed for fine and ultra-fine grinding of mineral raw material particles with a size of 2.5–3.0 mm. In the vibration system of the mill, resonant vibration-impact multi-frequency oscillations are implemented, while the intensification of nonlinear multi-frequency oscillations of the grinding chamber with the technological medium is implemented to the values of their accelerations up to hundreds of m/s^2 with accelerations of the drive frame of the mill with vibration exciters of oscillations of tens of m/s^2 . This ensures the durability of the vibration exciters that are most vulnerable to dynamic loads and the dynamic system of the vibrating mill as a whole [32].

In this regard, the development of a methodology for experimental research on the properties of CSM with the addition of TPP fly ash mechanically activated in a vibration multi-frequency mill and the determination of the effect of adding mechanically

activated fly ash to the composition of CSM and on its strength is an urgent scientific task, which is essential for reducing energy and time costs and improving product quality.

The purpose of the study is to identify the influence on CSM strength the adding of mechanically activated fly ash in a multi-frequency vibration mill.

2. Methods

According to DSTU B V.2.7-214:2009 "Building materials. Concrete. Methods for determining strength using control samples", the following basic requirements are imposed on concrete samples for testing [1]:

1. Shape and size of samples.

Shape: Specimens are typically shaped like a cube (for compression tests) or a prism (for bending tensile tests).

Nominal size: the main (base) size of a cube face or prism cross-section is 150 mm (standard size).

Permissible sizes: it is permissible to use samples of other sizes (for example, 100 mm, 200 mm, 250 mm, 300 mm), if this is provided for by the regulatory documentation for a specific type of concrete or by the project. In this case, the sample size must be at least three times larger than the maximum grain size of the coarse aggregate.

Deviations: the actual dimensions of the working faces of the samples should not differ from the nominal ones by more than $\pm 1\%$.

Thus, in accordance with the above requirements, samples of cubic-shaped CSM with dimensions of $100 \times 100 \times 100$ mm were selected for the study.

2. Number of samples.

To determine the strength of concrete depending on the time of its manufacture (for example, 7 or 28 days), a series consisting of at least three identical samples is produced. The final result is taken as the arithmetic mean value of the strength of these samples.

3. Manufacturing and hardening.

Molds: special molds (formwork) are used to produce samples, which meet the requirements of DSTU B V. 2.8-38:2011 - Molds for producing concrete test specimens. Technical conditions (GOST 22685-89) [33].

Compaction: The concrete mix in the forms is compacted by tamping or vibrating (depending on the mobility of the mix) to ensure uniformity of the sample.

Hardening conditions: after manufacture, the samples are stored in conditions that provide the required temperature and humidity regime (as a rule, in a humid environment) or under a layer of moist material at a temperature of $20 \pm 2^\circ\text{C}$ until the time of testing (usually 28 days to achieve design strength).

Compliance with these requirements is critically important for obtaining reliable concrete strength test results.

The mold for manufacturing CSM samples is shown in Fig. 1. The proportions of components for preparing CSM of the M150 brand with replacement of 10 to 50% of cement with mechanically activated fly ash are given in Table 1.



Figure 1– Metal mold (3FK-100) for manufacturing cubic samples of CSM with a face size of 100 mm

Table 1 – Proportions of CSM components

Cement brand		M500				
CSM brand		M150				
Cement	gram	900	800	700	600	500
Water		400–600				
Sand		3000				
Fly ash		100	200	300	400	500

3. Theoretical and experimental parts

The developed methodology for conducting experimental studies of the CSM is as follows.

Preparation of fly ash for mechanical activation.

To conduct these studies, fly ash was selected from the Priydniprovskya TPP.

The preparatory stage consisted of mechanical sieving on a MVS screen [34, 35] to obtain a minus 40 μm fraction, which is the most valuable for cement production, as it provides a dense microstructure.

The feed of the starting ash was carried out in a dosed manner. The sub-grid product with a size of minus 40 microns and the super-grid product with a size of plus 40 microns were discharged into the corresponding containers through conical chutes.

Mechanical activation of fly ash.

Mechanical activation of fly ash was carried out by grinding the minus 40 μm fraction in the chamber of a vibrating mill with multi-frequency vibration and impact excitation [31, 32].

To more thoroughly determine the effect of mechanical activation on the properties of fly ash, mechanical activation of the obtained 40 μm fraction was carried out in two time regimes: mechanical activation lasting 15 min. and mechanical activation lasting 30 min.

The storage time of mechanically activated ash until the samples were produced was also varied: 1 day, 10 days, and 28 days.

To determine the optimal content of fly ash, the percentage of its addition to the CSM was varied from 0% to 50%.

These parameters are given in Table 2.

Table 2 – Variable parameters of fly ash mechanical activation study

Mechanical activation time	15 min.			30 min.		
Holding time of fly ash after mechanical activation	1 day	10 days	28 days	1 day	10 days	28 days
Amount of fly ash added per 1 sample	10%	10%	10%	10%	10%	10%
	20%	20%	20%	20%	20%	20%
	30%	30%	30%	30%	30%	30%
	40%	40%	40%	40%	40%	40%
	50%	50%	50%	50%	50%	50%

The process of mechanical activation.

The process of mechanical activation of fly ash in a vibrating multi-frequency ball mill is based on the transfer of kinetic energy of the grinding bodies (balls) of the material to destroy its crystal lattice and increase the specific surface area.

The effectiveness of activation is determined by the energy intensity of the unit and compliance with regulated parameters.

The grinding medium is a cylindrical chamber filled with steel balls with a diameter of 10–20 mm, to achieve combined impact and abrasion of the material. The filling of the mill chamber with fly ash was from 30% to 40% of its volume, the rest was grinding bodies.

The chamber is hermetically sealed, installed and rigidly fixed on a vibration drive, which, together with the chamber, performs multi-frequency vibration and shock oscillations with a wide range of frequencies from zero to tens of thousands of Hz.

The obtained material after each mechanical activation mode was unloaded from the chamber, weighed, and divided into three equal parts.

The first part was added to the samples immediately after mechanical activation, in the proportions indicated in Table 1.

The second and third parts were delayed for 10 and 28 days, respectively, to determine the effect of time on the mechanically activated material.

After reaching this deadline, on days 10 and 28, mechanically activated fly ash was added to the CSM in the same proportions to produce samples.

Similar actions were carried out with the material obtained after mechanical activation in the 15 and 30 min modes.

Sample production.

To determine the compressive strength of the CSM, cubic samples measuring 100×100×100 mm were selected.

1. Preparing the mixture.

The cement mortar was prepared with a cement to sand ratio of 1:3 by weight, with the addition of water and fly ash in a specified amount (Table 1).

2. Formation.

The mixture was placed in special molds measuring 100×100×100 mm for the manufacture of samples (Fig. 1).

3. Sealing.

The mixture in the molds was compacted by vibration and/or shaking to remove air bubbles and ensure uniformity.

4. Hardening and intensive strength gain.

Active hydration, after setting, begins during the period of intensive growth of calcium hydrosilicate crystals. They intertwine, forming a strong framework (cement stone).

First 24 hours. The concrete gains initial strength sufficient for careful demoulding of the specimens.

First 7 days. Concrete gains about 60–70% of its design (brand) strength.

28 days. This is the standard period for concrete to reach 95–100% of its design (grade) strength, provided that regulatory conditions for maintenance are met.

Critical need for moisture. At this stage, a constant supply of water is necessary to maintain hydration. Loss of moisture through evaporation leads to cessation of the reaction, which critically reduces the final strength and durability of the concrete.

5. Demolding.

After demolding, the samples continue to be stored in a high humidity chamber until they are tested.

Preparation of samples for testing.

Strength tests of the samples were carried out on a hydraulic press.

1. Removal. Approximately 30 minutes before the start of the test, the samples were removed from the curing chamber.

2. Inspection and cleaning. The surfaces of the samples were carefully inspected. Any dirt, debris, or protruding parts were removed to ensure an even fit to the press platens. Surfaces in contact with the press must be smooth and even.

3. Measurement. The actual dimensions of each face of the cube (length, width, height) were measured with an accuracy of at least 1% to accurately calculate the cross-sectional area. The standard size of 100×100 mm means an area of 10,000 mm² (or 100 cm²).

Equipment preparation.

1. Verification. The hydraulic press must be calibrated and meet the requirements of the standard (DSTU EN 12390-4:2024 Testing of concrete. Part 4. Compressive strength. Technical requirements for testing machines (EN 12390-4:2019, IDT) [36]).

2. Cleaning the plates. The press's load-bearing plates (upper and lower) are cleaned of dirt and residues from previous tests.

3. Centering. It is necessary to ensure that the upper movable plate can move freely and self-center for even load distribution.

Testing.

1. Setting up the sample. The sample is placed on the bottom plate of the press. It is important to install it so that the load is applied to the opposite faces, which were vertical during concreting (perpendicular to the direction of placing the mixture), unless otherwise specified.

2. Alignment. The specimen is centered exactly on the center of the lower press base plate to avoid eccentric loading, which can cause premature failure.

3. Load application. The load is applied to the specimen smoothly and continuously at a constant rate, which is typically 0.6 ± 0.2 MPa/s (N/mm²/s) or approximately 140 kg/cm² per minute, according to standards.

4. Fixation of the fracture. The operator monitors the load readings on the press display or pressure gauge. The load is increased until the sample collapses and the needle or instrument reading begins to fall.

Record the result. The maximum load (P , in Newtons or kilonewtons) reached at the moment of failure is recorded. The nature of the fracture is also noted (e.g., a cone-shaped split, which is typical of a correct test). The load–strain graph is also recorded using a recorder.

4. Results and discussion

Tables 3 and 4 present the results of the study of ordinary ash and mechanically activated ash (grinding took place for 30 minutes, addition to the CSM immediately after mechanical activation).

Table 3 – Results of the study of the strength of samples with the addition of ordinary fly ash

No. experiment	Ash content in the mixture					
	0%	10%	20%	30%	40%	50%
1	15.1	17.6	15.5	13.0	10.4	7.8
2	14.7	16.8	17.2	13.8	9.8	7.2
3	16.2	15.8	14.8	12.7	11.4	8.0
Average value, MPa	15.3	16.7	15.8	13.2	10.5	7.7

Table 4 – Results of the study of the strength of samples with the addition of mechanically activated fly ash

No. experiment	Ash content in the mixture					
	0%	10%	20%	30%	40%	50%
1	15.1	22.6	17.6	14.6	12.7	8.7
2	14.7	21.8	17.2	14.6	14.4	8.0
3	16.2	23.4	17.4	13.4	13.7	8.4
Average value, MPa	15.3	22.6	17.4	14.2	13.6	8.4
The strength of samples with the addition of mechanically activated ash exceeds the strength of samples with ordinary ash	1	1.350	1.098	1.078	1.291	1.091

Fig. 2 shows the dependences of the change in the strength of samples - mechanical load at which the samples fractured (P , t) with different fly ash contents (C , %) for ordinary ash and mechanically activated ash for 30 min. when it is added to the CSM immediately after mechanical activation.

Data analysis shows that the highest strength is observed at a 10 percent fly ash content, which coincides with the content standards regulated by the DSTU.

The excess of the strength of samples with the addition of mechanically activated ash over the strength of samples with ordinary ash at a 10 percent content was 1.35, in other cases the strength was on average 1.1 times higher.

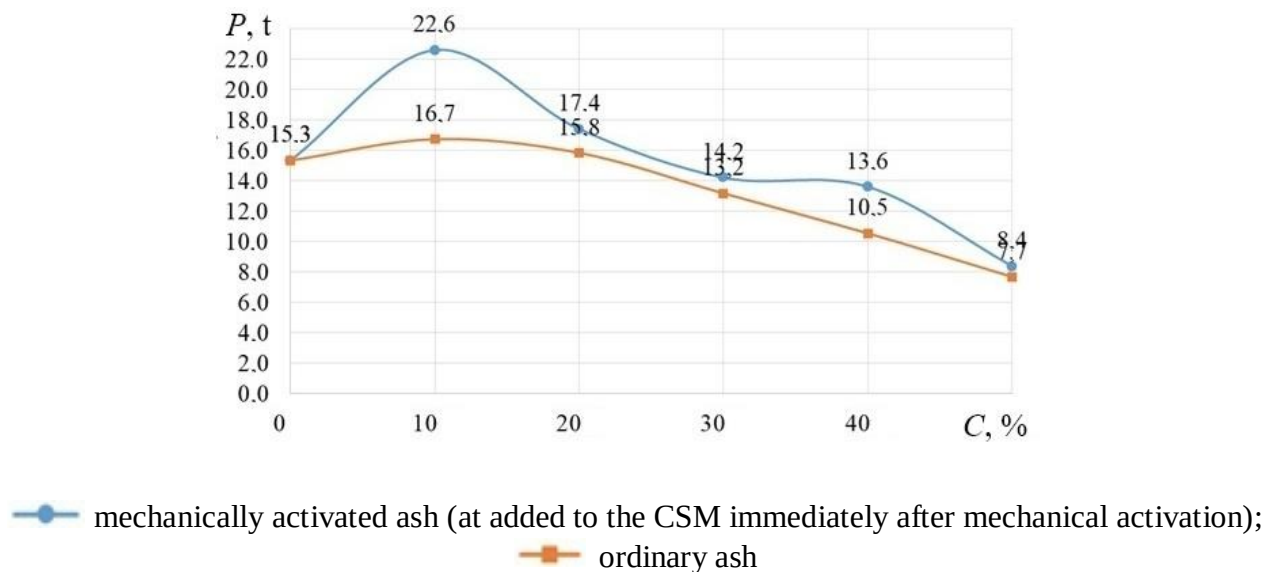


Figure 2 – Dependences of the change in strength of samples with different fly ash contents for ordinary ash and mechanically activated ash (at added to the CSM immediately after mechanical activation)

The increase in the strength of the CSM by 35% due to mechanical activation of ash in a vibration-type multi-frequency mill exceeds the data obtained with other methods of its grinding [11–30].

The increase in the strength of the CSM with the addition of ash mechanically activated during grinding in a vibrating multi-frequency mill is associated with an increase in the specific surface area, and primarily with an increase in the internal energy of the material and, as a result, the chemical activity and reactivity of the ash due to intensive mechanical grinding. This leads to plastic deformation, accelerated diffusion, and re-mixing of mixture components at the molecular and atomic levels, which also promotes further chemical reactions.

Thus, the results of research using the developed methodology confirmed the feasibility of using a vibrating multi-frequency mill with vibration-impact excitation of the grinding chamber for grinding and mechanical activation of fly ash.

5. Conclusions

1. A methodology for experimental studies of the properties of CSM with the addition of mechanically activated TPP fly ash has been developed.

To conduct these studies, fly ash from the Prydniprovskaya TPP was selected. The preparatory stage consisted of mechanical sieving on an MVS screen to obtain a minus 40 μm fraction, which is the most valuable for cement production. Mechanical activation of fly ash was carried out by grinding the minus 40 μm fraction in the chamber of a vibrating mill with multi-frequency vibration and impact excitation.

2. To more thoroughly determine the effect of mechanical activation on the properties of fly ash, mechanical activation of the obtained 40 μm fraction was carried out in two time regimes: 15 min. and 30 min. The storage time of mechanically activated ash until the samples were prepared was also varied: 1 day, 10 days, and 28 days. To determine the optimal content of fly ash, the percentage of its addition to the CSM was varied from 0% to 50%.

3. The process of mechanical activation of fly ash in a vibrating multi-frequency ball mill is based on the transfer of kinetic energy of the grinding bodies (balls) of the material to destroy its crystal lattice and increase the specific surface area. The effectiveness of activation is determined by the energy intensity of the unit and compliance with regulated parameters. The grinding medium is a cylindrical chamber filled with steel balls with a diameter of 10–20 mm, to achieve combined impact and abrasion of the material. The filling of the mill chamber with fly ash was from 30% to 40% of its volume, the rest was grinding bodies.

4. To determine the compressive strength of the CSM on a hydraulic press, cubic samples measuring 100×100×100 mm were selected. The cement mortar was prepared with a cement to sand ratio of 1:3 by weight with the addition of water and fly ash in a specified amount. The mixture was placed in special molds measuring 100×100×100 mm for the manufacture of samples. The load on the sample is applied smoothly and continuously at a constant rate, which was 0.6 ± 0.2 MPa/s or approximately 140 kg/cm² per minute, according to standards. The maximum load reached at the moment of failure was recorded, and the nature of the failure was noted.

5. The highest strength was observed at a 10 percent ash content, which coincides with the content standards regulated by the DSTU. The excess of the strength of samples with the addition of mechanically activated ash over the strength of samples with ordinary ash at a 10 percent content was 1.35, in other cases the strength was on average 1.1 times higher. The results of research using the developed methodology confirmed the feasibility of using a vibrating multi-frequency mill with vibration-shock excitation of the grinding chamber for grinding and mechanical activation of fly ash.

Conflict of interest

Authors state no conflict of interest.

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МЕТОДИКА ЕКСПЕРИМЕНТАЛЬНИХ ДОСЛІДЖЕНЬ ВЛАСТИВОСТЕЙ ЦЕМЕНТНО-ПІЩАНОЇ СУМІШІ З ДОДАВАННЯМ МЕХАНОАКТИВОВАНОЇ ЗОЛИ ВІНОСУ ТЕС

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Анотація. Розроблено методику експериментальних досліджень властивостей цементно-піщаної суміші (ЦПС) з додаванням механоактивованої золи виносу ТЕС. Підготовчий етап полягав у механічному просіюванні золи виносу на грохоті МВГ для отримання фракції мінус 40 мкм, яка є найцінніша для виробництва цементу та забезпечує щільну мікроструктуру. Механоактивація золи виносу проводилась шляхом помолу фракції мінус 40 мкм у камері вібраційного млина з віброударним полічастотним збудженням. Процес механоактивації золи виносу у вібраційному полічастотному кульовому млині базується на передачі кінетичної енергії помольних тіл (кульок) матеріалу для руйнування його кристалічної решітки та збільшення питомої поверхні. Ефективність активації визначається енергетичною напруженістю агрегату та дотриманням регламентованих параметрів. Механоактивацію фракції 40 мкм проводили в двох часових режимах: механоактивація тривалістю 15 хв. та 30 хв. Також варіювався час зберігання механоактивованої золи до моменту виготовлення зразків. Для визначення оптимального вмісту золи виносу змінювався відсоток її додавання в ЦПС. Цементний розчин готувався зі співвідношенням цементу до піску 1:3 за масою з додаванням води та золи виносу у визначеній кількості. Суміш поміщалась у спеціальні форми розміром 100×100×100 мм для виготовлення зразків. Навантаження на зразок прикладалось плавно і безперервно зі сталою швидкістю 140 кг/см² за хвилину, відповідно до стандартів. Фіксувалось максимальне навантаження, досягнуте в момент руйнування, характер руйнування. Найбільша міцність зразків спостерігалась при 10-тивідсотковому вмісті золи виносу, що співпадає з нормами вмісту, регламентованими ДСТУ. Відношення міцності зразків з додаванням механоактивованої золи до міцності зразків зі звичайною золою при її 10-тивідсотковому вмісті склало 1,35, для вмісту золи від 20% до 50 % таке відношення складало в середньому 1,08–1,2. Результати досліджень за розробленою методикою підтвердили доцільність використання для помолу та механоактивації золи виносу вібраційного полічастотного млина з віброударним збудженням камери помолу.

Ключові слова: методика, експериментальні дослідження, властивості ЦПС, механоактивована зола виносу ТЕС.