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Cement-ash compositions with ash - fluidized bead removal

Abstract. The results of studies of ash-fluidized bed removal by X-ray structural analysis, differential thermal analysis and IR spectrometry are presented. Ashes and ash-slags of the fluidized bed have slightly different properties due to the fact that in fluidized bed boilers the fuel is burned for several seconds longer than in traditional boilers. During this time, the dispersed part of the mineral component of the fuel has time to melt and transform into a glass phase, as a result of which their internal energy increases and they become capable of chemical reactions. The results of the research allowed us to establish that the fly ash of anthracene coal from domestic deposits when burned in a fluidized bed can be classified as medium-calcium and sulfate, which may hide the manifestation of hydration properties. The results of the IR spectrometry study confirmed the presence of calcium sulfate compounds of the gypsum and anhydride type in the fly ash composition. Sulfate minerals in the presence of aluminates and moisture can contribute to the formation of the ettringite mineral during the hardening of cement-ash compositions, which can contribute to the emergence of expansion stresses in cement stone. However, the study of cement-ash stone by X-ray structural analysis methods at the age of six months does not confirm the formation of ettringite. Testing of cement-ash compositions showed that the strength of the compositions at the vintage age is somewhat lower than that of cement stone. However, with a further increase in the hardening period, the rate of strength gain of the compositions is somewhat greater than that of cement stone.

Keywords: fly ash, fluidized bed, cement-ash compositions, IR spectroscopy, X-ray diffraction analysis, differential thermal analysis

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Introduction.

As is known, fly ash is a product of coal combustion in the furnaces of thermal power plants. For complete combustion, coal is ground to a powdery state and blown into the furnace. Coal combustion lasts only 3-4 s. at a temperature of about 1600 ° C. In this case, non-combustible minerals are liquefied at the outlet of the furnaces during rapid cooling and form glassy structured spherical ash particles, the properties of which depend on various factors, such as the composition of the coal, the source of coal, grinding, combustion environment, boiler shape, types of added minerals, processing conditions, etc. When coal is burned, about 80% fly ash and 20% bottom ash are formed. [1]. Fly ash is covered by flue gases and is carried outside, deposited on mechanical or electrostatic filters, after which stored in dumps. The normal size of ash particles ranges from 2.5 to 35 mm, so storing ash requires additional capital investments to keep it moist.

Ash - fly ash, formed from coal combustion at TPPs, is a high-tonnage waste. Construction and operation of a technological facility at TPPs for the collection, transportation and storage of fly ash waste require significant capital expenditures, as well as costs for its maintenance. The formation and storage of waste has a negative impact on the ecosystem of the adjacent territory of the area of their location due to:

- diversion and disturbance of land for the construction of ash dumps and their engineering infrastructure;
- ingress of soluble compounds from the dump bowls into surface and groundwater with their subsequent saturation;
- sawing of ash from the surface of the dump bowls, especially when a significant amount of them accumulates and the free capacities on the dump are completely exhausted.

To date, about 360 million tons of ash and slag have been accumulated in the dumps of Ukrainian thermal power plants on an area of over 3,200 hectares. Their average annual output reaches 14 million tons and, due to the deterioration of fuel quality, tends to increase [2]. This creates technological and environmental problems, since production costs and the cost of environmental protection measures increase. The urgency of the problem of ash and slag accumulation is also determined by the fact that the share of thermal power plants in the state's energy strategy is increasing. It is known that even during normal operation of ash and slag dumps, manifestations of unfavorable geoecological processes and phenomena occur that disrupt the ecological balance in the areas where they are located.

At the same time, ash and slag materials are largely identical in chemical and mineralogical composition to natural mineral raw materials. Their use in industry, construction industry and agriculture is one of the strategic ways to solve the environmental problem in the area of operation of thermal power plants. The real solution is to use ash and slag as a secondary raw material in the construction sector: from the production of concrete, cement, bricks to laying roads. Ukrainian partners have experience - the Polish Union for the Utilization of Fuel Combustion Products is actively working in this direction. Today, ash and slag are considered waste, but in our country we have already reached the understanding that this is a product, a valuable material resource. In recent years, scientific research, especially by domestic scientists, has shown new directions for the effective use of ash-slag mixtures in the creation of slag-alkali binders, relevant regulatory documents have been developed and are being implemented in practice. At the same time, not enough attention has been paid to the development of rational ways to use ash-slag mixtures in modern economic conditions for road construction in Ukraine.

Some researchers have proven that finer ash contributes to an increase in the pozzolanic effect, as a result of which concrete becomes denser, strong and resistant to the action of alkalis, sulfates and silica [3,4,5].

It is known that many countries of the world have found methods of using ashes and ash slag in the national economy, including in construction, because it is some material rich in mineral oxides [6,7,8]. Some researchers find that fly ash can be used not only in construction, but also in agriculture to improve the properties of land [9].

Fly ash can also be used in the production of ceramic building materials, as an additive to clays to regulate their technological properties. A more important area of use of ash remains concrete technology [10]. Due to the presence of oxides such as SiO_2 and Al_2SiO_3 in the composition, ashes become similar to Portland cement [11]. At room temperature and in the presence of moisture, the ash chemically reacts with calcium hydroxide, forming compounds similar to the hydration minerals of Portland cement [12,13]. The glass phase contained in the ash when used with Portland cement chemically reacts with calcium hydroxide, which is released during the hydration of cement minerals, and forms calcium hydrosilicates of the C±S±H type, which contributes to the compaction and strengthening of cement stone [14, 15]. It should be noted that the increase in compressive strength is observed only over a long period of time [15]. The use of fly ash as an additive or part of cement and concrete allows for some economic benefits. There is data in the literature that fly ash can be added in an amount of up to 75% of the cement mass, but there are strict recommendations for the use of fly ash in cements and concretes [16]. There is data on the use of fly ash in the production of lightweight aggregates for concrete [17,18,19]. There is information on the use of fly ash in road construction as one of the components of the earthen embankment and it is claimed that this allows you to save on earthworks [20,21,22,23,24].

There is information on the use of fly ash in the technology of producing burnt bricks. It is noted that

Objective of the research. To study the properties of the fluidized bed fly ash and the products of ash hydration with Portland cement

Research methodology: The work used the methods of X-ray structural and differential thermal analysis, IR spectrometry.

Results and discussions: Fig. 1 shows the X-ray diffraction pattern of the fluidized bed fly ash X-ray structural analysis allows us to investigate the chemical and mineralogical composition of the ash. The most common oxides in the ash are silicon oxide SiO_2, aluminum oxide Al_2O_3, calcium oxide CaO and iron oxide Fe_2O_3. Traces of MgO, Na_2O, K_2O, SO_3, TiO_2. MnO and C can also be found. The X-ray diffraction pattern shows the presence of gypsum mineral or calcium sulfate dehydrate ($CaSO_42H_2O$) in the ash with peaks 4.5191; 4.2689; 3.0408. The presence of anhydrous calcium sulfate, or the mineral anhydride ($CaSO_4$) shows peaks -3.5149; 3.1959; 2.578; 1.8711; 1.1424. Both minerals belong to calcium sulfates and, when decomposed, emit sulfur gas (SO3) and calcium oxide. The presence of silicon oxide in the ash is indicated by peaks with peaks at 3.3476; 2.4575; 2.2851; 2.1306; 1.9201; 1.6724. Thus, the crystallized part of the ash-slag consists of calcium-containing minerals and silicon oxide. The non-crystallized part of the fly ash can be observed by analyzing the noise part of the X-ray diffraction pattern. This part, as a rule, consists of amorphous oxides and is considered to be chemically active components. The hydraulic activity of the ash-slag is associated with the presence of such compounds as lime in a free state or anhydride, which are able to react with water to form a water-resistant stone without the introduction of additional activators.

According to [30], for ashes and ash-slags of the “fluidized layer”, which are the tested ash-slags, the manifestation of hydraulic properties is more characteristic than pozzolanic, due to the presence of free calcium oxide in the composition.

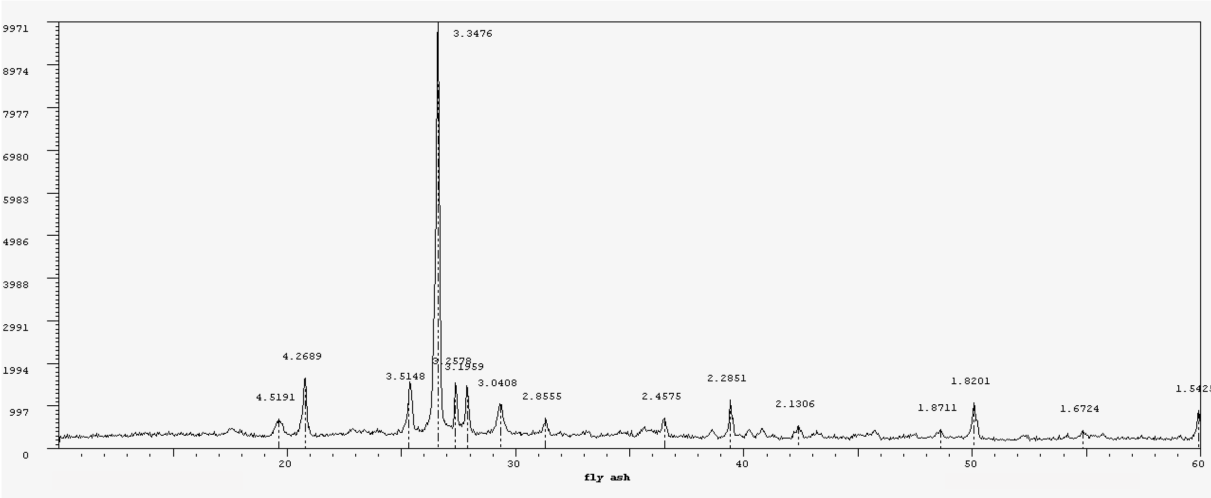

Figure 1 –X-ray diffraction pattern of fly ash.

At the same time, the presence of amorphous aluminosilicates, the amount of which exceeds 50%, also determines the manifestation of pozzolanic activity. CaSO4 remains a sufficiently active component, which reacts isothermally with water.

The composition of the ash was also confirmed by the results of differential thermal analysis, the thermogram of which is shown in Fig. 2

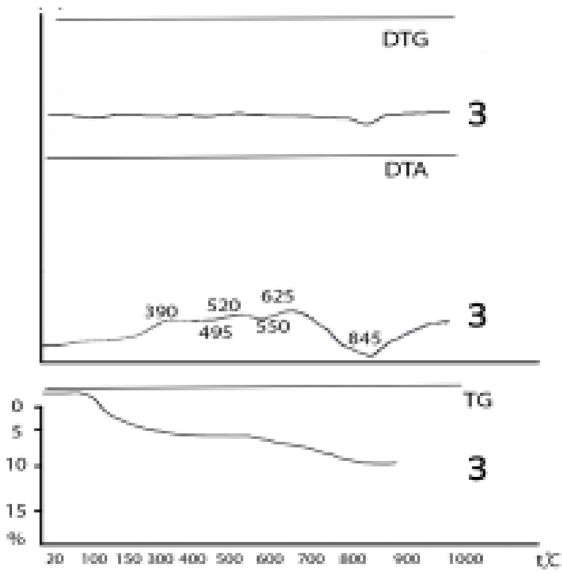

Figure 2 – Fluadized bed ash derivations

As is known, minerals and oxides of elements react differently to temperature changes. This effect is used to determine the presence of certain minerals in the composition of mineral building materials. On the derivatives we can observe the effect at a temperature of 390 which belongs to calcium sulfate; and the effect at a temperature of 550 °C belongs to CaO. The effect at a temperature of 625 °C confirms the presence of Al_2O_3 in the composition of fly ash, the effect at a temperature of 845 °C proves the presence of silicon oxide SiO_2 .

Fly ash was also investigated by IR spectroscopy, the results of which are shown in Fig. 3.

The IR spectra of fly ash contain bands characteristic of silicates and aluminosilicates. Silicate minerals with different types of crystal lattice structures are characterized by absorption bands in the region of 1050-1200 cm-1, which are due to the stretching vibrations of the Si-O group; in the spectrum they appear as a series of weakly expressed peaks against the background of a broad band common to sulfates. In general, the spectrum shows bands characteristic of quartz. In this case, a shift of the stretching vibrations of the Si-O bond relative to the reference values characteristic of quartz is observed to the low-frequency region, which is due to the influence of the Al2O3 oxide impurity. The spectrum also contains a doublet characteristic of aluminosilicates in the range of wave numbers 770-810 cm-1, which refers to the vibrations of the Al-OH and Si-O-Al bonds. Deformation vibrations of the Si-O bond are expressed in the absorption band in the range of wave numbers 500-400 cm-1.

The presence of carbonates in the composition of ash and slag is confirmed by the presence of absorption bands, which are due to C-O vibrations: valence - an intense band at a wave number of 1440 cm-1, a narrow intense band at 875 cm-1; deformation - a weak band at 713 cm-1, as well as bands at 2516 and 1795 cm-1.

Sulfate groups are determined by the intense absorption band in the wavenumber range 1090-1180 cm-1 and 680-650 cm-1. The absorption is due to the presence of vibrations of the S-O bond. According to the position maximum, it is possible to reliably determine a mineral in the sulfate group. For example, for gypsum, the maximum of the characteristic band is located at a wave number of 660 cm-1, for anhydrite, a shift of this band to the high-frequency region is observed, in the spectrum of ash and slag this is the band at 678 cm-1. For gypsum, the presence of two types of differently bound water in the structure is identified by a strong splitting of the doublets of the bands in both the valence and deformation vibrations of the OH groups of water molecules. Judging by the spectrum, only anhydrous sulfates are present in the composition of ash and slag.

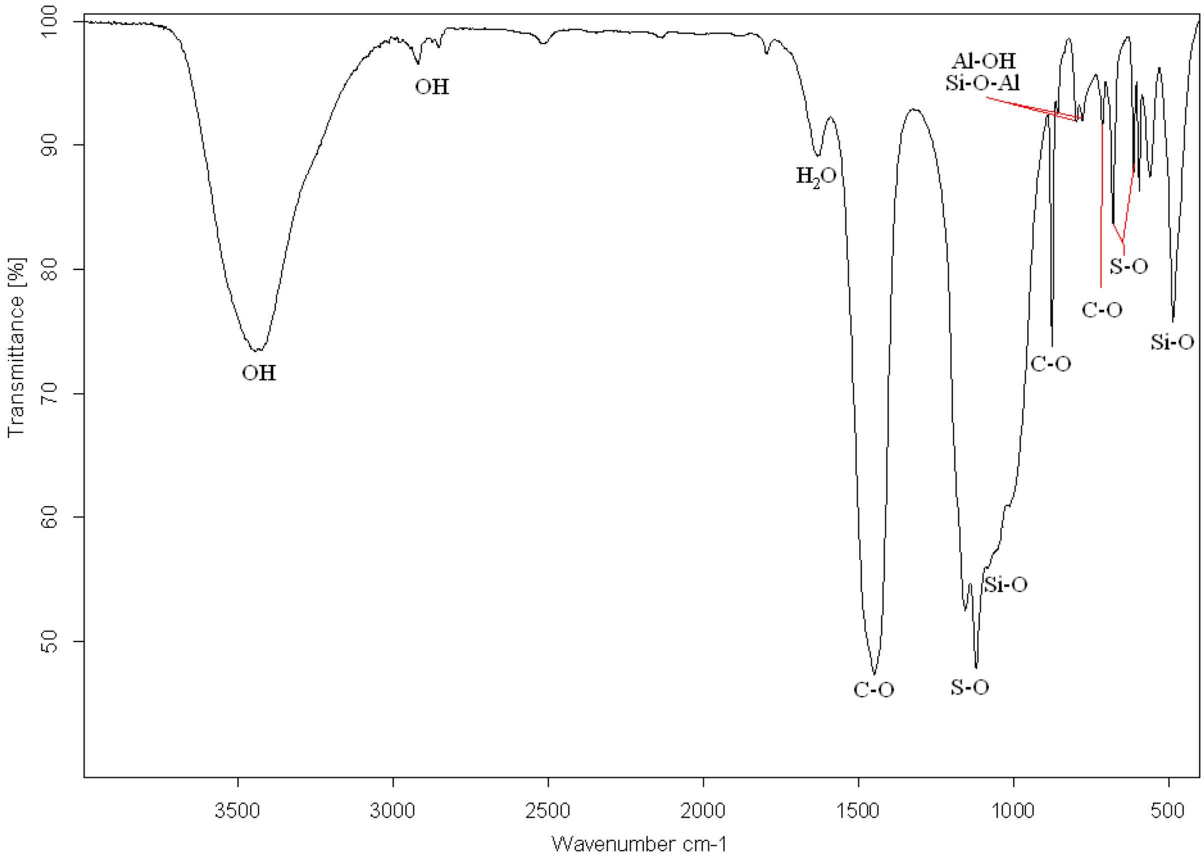

Figure 3 – Spectrogram of fly ash

The high-frequency region of the absorption bands with values: 2918; 2850 and 3443 cm-1 refers to the valence vibrations of the bonded OH groups. The absorption band with a frequency of 1630 cm-1 refers to the valence vibrations of water molecules. The presence of bands corresponding to valence, deformation vibrations of OH groups and valence vibrations of water molecules is associated with the phenomenon of moisture adsorption from the environment due to the high activity of minerals that make up the ash and slag.

Thus, studies have established that the ash and slag contains anhydrous calcium sulfate and it may be anhydrite.

In order to establish the possibility of using fly ash in cement compositions, cubes with a side of 30 mm with different fly ash contents were tested. The samples hardened at a temperature of 20 ± 2 ° C and air humidity of 80 -95%. The results of their testing at the age of 30 and 180 days are given in Table 1 and Fig. 4.

As we can see from the table and figure, the compositions harden over time. Thus, the strength of pure cement stone increased by 7.5% compared to the vintage age. Compositions with the addition of fly ash show an increase in strength from 6.0 to 10%. Apparently, silicon oxide, which is in the ash in an amorphous state, reacts with calcium hydroxide by reaction, forming calcium hydrosilicates, which contributes to the strengthening and compaction of cement stone by reaction.

$$Ca(OH)_2 + SiO_2 + mH_2O = CaO \cdot SiO_2 \cdot nH_2O \quad (1)$$

The formation of calcium hydrosilicates was confirmed by the results of the study of cement-ash and cement stones by X-ray structural analysis, the diagrams of which are shown in Fig. 5 and 6.

Table 1 – Test results of cement-ash compositions

№	Ash content, %	Strength, MPa at age	
		30 days	180 days
1	0	51	55
2	10	48	51
3	20	45	48
4	30	44	46
5	40	41	43
6	50	37	41

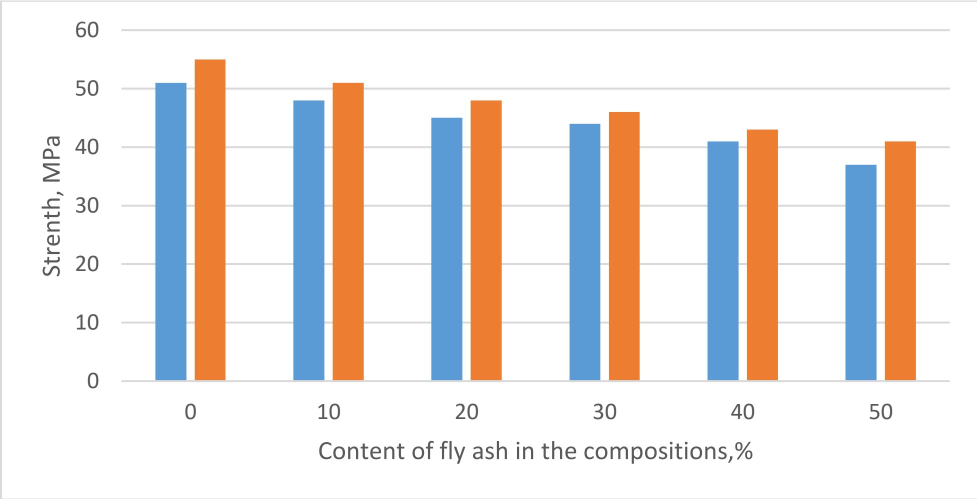

Figure 4 – Spectrogram of fly ash

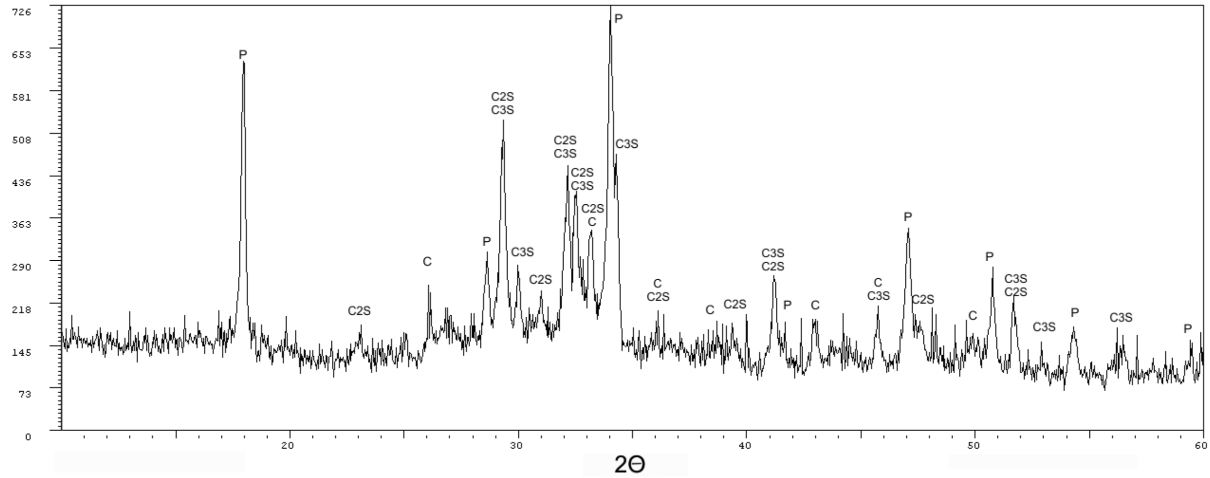

Figure 5 – X-ray image of cement stone
C3S – $3CaO{\cdot}SiO_2$; C2S – $2CaO{\cdot}SiO_2$; C – $CaCO_3$ P – $Ca(OH)_2$

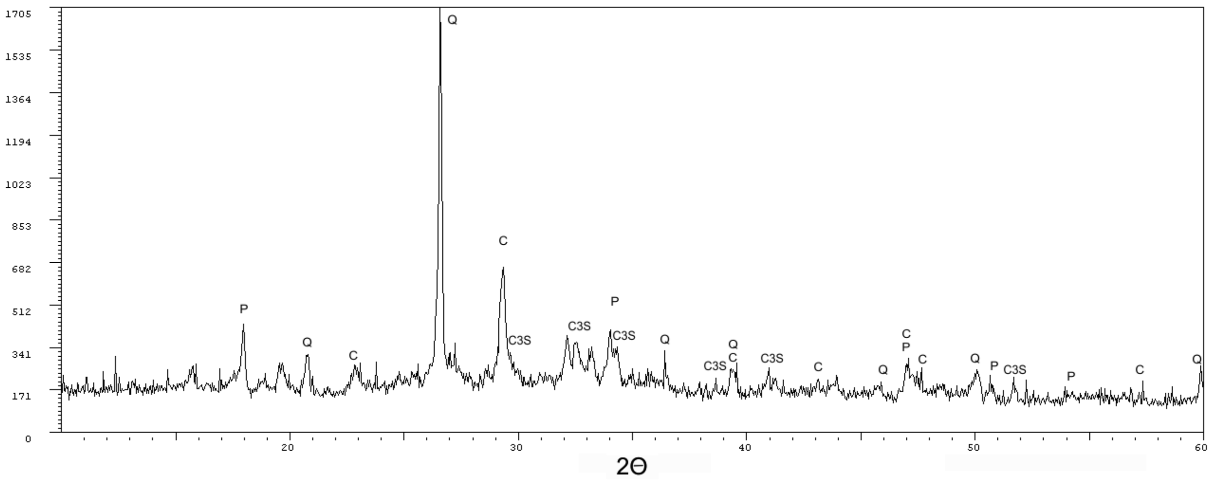

Figure 5 – X-ray image of cement-ash stone at the age of 180 days.
C3S – $3CaO{\cdot}SiO_2$; C2S – $2CaO{\cdot}SiO_2$; C – $CaCO_3$; P – $Ca(OH)_2$; Q- SiO_2

Analysis of the X-ray diffraction pattern shows the presence of additional components in the composition of the cement stone. Peaks with peaks 4.2689, 3.3527, 2.4657 belong to quartz SiO2. In addition, it should be determined that the peaks of lime Ca(OH)2 have become somewhat smaller. It can be assumed that lime begins to react with silica, which is part of the ash. But confirmation of such a phenomenon requires further studies of the stone over longer periods of hardening.

Conclusions: The results of the studies indicate that the ash-carrying fluidized bed in terms of the content of basic oxides belongs to medium calcium and sulfate.

Within the framework of the experiment, the fact of the formation of ettringite was not detected either at the stage of hardening of Portland cement with ashes, or during further hardening up to 180 days.

Cement-ash solutions have a compressive strength lower than control solutions from pure cement. During further hardening, it is observed that the kinetics of strength gain of cement-ash solutions is somewhat higher than that of pure cement stone.

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Цементно-зольні композиції з золою-винеення киплячого шару

Аннотація. Наведено результати досліджень золи-винесення киплячого шару методами рентгеноструктурного, диференціально-термічного аналізу та ІЧ спектрометрії. Золи та золошлаки киплячого шару мають дещо інші властивості завдяки тому, що у котлах киплячого шару паливо випалюється на декілька секунд довше ніж у традиційних котлах. За цей час дисперсна частина мінеральної складової палива встигає розплавитись та перетворюватись к скляну фазу, в результаті чого підвищується їх внутрішня енергія і вони становяться спроможними до хімічних реакцій. Результати досліджень дозволили встановити, що зола-винесення антраценового вугілля вітчизняних родовищ при спалюванні в киплячому шару можна віднести до середньокальційових та сульфатних, що може спряти проявленню гідратаційних властивостей. Результати дослідження ІЧ спектрометрії підтвердили наявність у складі золи-винесення сполуки сульфату кальцію типу гіпсу та ангідриду. Сульфатні мінерали при наявності алюмінатів та вологи можуть сприяти утворенню мінералу етрінгіту в процесі тверднення цементно-зольних композицій, що може сприяти виникненню розширюючих напружень у цементному камені. Але дослідження цементно-зольного каменю методами рентгеноструктурного аналізу у віці шість місяців не підтверджують утворення етрінгіту. Випробування цементно-зольних композицій показало, що міцність композицій у марочному віці дещо нижче ніж цементного каменю. Але з подальшим збільшенням строку тверднення швидкість набору міцності композицій дещо більше ніж цементного каменю.

Ключові слова: зола-винесення, киплячий шар, цементно-зольні композиції, ІЧ спектроскопія, рентгеноструктурний аналіз, диференціально-термічні аналіз.

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