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DEVELOPMENT OF LIGHTWEIGHT GROUTING MATERIALS BASED ON BY-PRODUCTS OF UKRAINIAN INDUSTRY

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Abstract. A new competitive class of grouting materials has been created. These lightweight grouting materials (LGM) with a density of 1370–1650 kg/m^3 consist of oil-well Portland cement PCTI-100, fine powder of PE grade (a by-product of fireclay production from kaolin), acidic fly ash from state district power plants, and lime dust (a by-product of lime production). The composition of the products formed during LGM hydration was clarified by X-ray phase and differential thermal analysis. The rational optimal ratios of ingredients and temperature regimes for the LGM hardening were determined according to the criteria of cement stone strength.

Keywords: oil-well cementing, lightweight grouting material, cement hydration, fly ash, water-cement ratio, cement stone.

1. Introduction

When cementing deep wells with long casing strings in one stage, complications in the cementing process are observed: hydraulic fracturing of reservoirs and absorption of the grouting mortar. This leads to a decrease in the cementation quality and insufficient lift of the cement mortar to the design height behind the casing.

The cause of hydraulic fracturing and absorption is usually an increase in the static pressure of the grouting mortar column, which does not allow prompt preventive measures during the cementing process. Therefore, the problem of complications occurred during cementing of long casing strings is solved by using cementing compositions with reduced density.

The issue of the need and feasibility of reducing the grouting mortar density arose with the increase in the depth of exploration and production wells for oil and natural gas. The main reason for the use of lightweight cementing materials was the need to lift the cement to a high height in one step. The possibility of widespread use of lightweight materials became real only after the revision of requirements for the mechanical strength of the cement stone.

Theoretical studies carried out by domestic and American scientists have shown that a stone with a compressive strength of 3.5–4.0 MPa has a sufficient margin of safety and can be recognized as a reliable grouting material for demarcating layers and horizons in oil and gas wells.

For the cementing of casing strings in exploration and production wells in the presence of absorbing layers and when it is necessary to lift the grout to high altitudes, a group of researchers at the Poltava branch of the Ukrainian Research Geological Exploration Institute developed a number of lightweight grouting materials for use in various mining and geological conditions.

The purpose of this work is to study the physicochemical characteristics of lightweight grouting materials hydration and the regularities of their settings. To achieve this goal, the following research tasks were set:

– studying the composition of hydration products of lightweight grouting materials – mixtures that have been set for different periods at high temperatures;

– studying the dependence of the operational properties of stone based on lightweight grouting materials on physicochemical factors, in particular, the composition of the grouting mixture, temperature, setting tome and pressure.

2. Experimental

2.1. Materials

A modern trend is the use of industrial waste and natural minerals in construction and environmental protection processes, in particular in adsorption technologies.[1-3] Therefore, in this research we also used a number of industrial wastes as components. Several mixtures with different types of waste were prepared and investigated:

a) grouting mixture consisting of Portland cement PCTI-100, finely dispersed powder of the PE brand (a by-

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product of fireclay production from kaolin), and technical (tap) water;

b) grouting mixture consisting of Portland cement PCTI-100, acidic fly ash from state district power plant (SDPP) with a very high water-cement ratio, oxyethyl cellulose (OEC) stabilizer, and technical (tap) water;

c) grouting mixture consisting of lime dust (a by-product of lime production), acidic fly ash from SDPP, and technical (tap) water.

Oil-well Portland cement PCTI-100 produced by PJSC "Ivano-Frankivskcement" had the following composition: clinker of standardized mineralogical composition (75-95%); special impurities (up to 20%); calcium sulfate (up to 5%).

Powder lime, which is a finely dispersed powder collected from dust deposition chambers and gas cleaning facilities, was used without additional drying and grinding. Sieve composition of lime dust is the following: 0.04-0.05 (22.9%); 0.05-0.08 (36.9%); 0.08-0.10 (15.3%); 0.10-0.15 (14.0%); 0.15-0.25 (4.0%); 0.25-0.50 (3.8%), 0.50+ (2.0%). The density of lime dust is 2890 kg/m^3, and the specific surface is 361.0 m^2/kg.

Data on the chemical composition and specific surface of powder lime are given in Table 1.

Table 1. Chemical composition and specific surface area of powder lime

Chemical composition, %							Specific surface area, m^2/kg
CaO	MgO	SiO_2	Al_2O_3	Fe_2O_3	SO_3	LOI	
62–64	12–14	2.0	–	–	1.1	19.1	361.0

Note: LOI is loss on ignition

PE powder is a by-product of kaolin roasting and has the following chemical composition: Al_2O_3 (40 wt. %), SiO_2 (50 wt. %), Fe_2O_3 (2 wt. %), CaO (1 wt. %), the rest is MgO, K_2O, Na_2O, and others. The density of the PE powder is 2520 kg/m^3, and a volume weight is 750 kg/m^3.

Acid fly ash from Kurakhivska SDPP is a dark gray powder with the density of 1980–2000 kg/m^3, specific surface area of 350–420 m^2/kg, and a bulk weight of 1100–1150 kg/m^3. The chemical composition of ash is: SiO_2 (50–52 wt. %), Al_2O_3 (15–24 wt. %), Fe_2O_3 (17–22 wt. %), MgO (2.0–3.0 wt. %), CaO (2.2–2.8 wt. %), SO_3 (0.4 wt. %), LOI (23.4 wt. %).

Acid fly ash from Ladyzhynska SDPP is a greenish-gray powder with the density of 2400–2500 kg/m^3, a specific surface area of 210–220 m^2/kg, and a bulk weight of 1500–1600 kg/m^3. The chemical composition of ash is the following: SiO_2 (57 wt. %), Al_2O_3 (23 wt. %), Fe_2O_3 (11 wt. %), CaO (2.0 wt. %), and MgO (2 wt. %).

To improve the sedimentation stability of unstable dispersed systems based on compositions of oil-well Portland cement and acidic fly ash from SDPP at a very high water-cement ratio, the OEC stabilizer was used.[4]

2.2. Methods and Instruments

The grouting mortar was prepared in a standard way according to DSTU BV.2.7–86–99 using a paddle mixer at a shaft rotation speed of 1500±100 min^{-1}. The amount of water taken from the water supply network was calculated depending on a certain water-cement ratio (W/C).[5-7] This ratio was determined based on the flowability of grouting mortar with the help of a flow cone according to DSTU BV.2.7–86–99.

The mortar properties were evaluated by sedimentation stability and bleeding rate, which were determined according to the standard method.[8]

The density of grouting mortar was determined using a 100 cm^3 pycnometer with a predetermined (clean and dry) weight m_1 in grams.[5-7]

Bleeding was determined in a standard way according to DSTU BV.2.7–86–99.[8] Autoclaving of the samples was carried out using the installation designed by the Poltava branch of the Ukrainian Research Geological Exploration Institute.[5-7] The samples were made according to the description.[5-7]

The cement stone strength was determined according to typical procedures.[5-8]

X-ray phase analysis of grouting stone was performed using the DRON-2 installation.[5-7]

The external specific surface of the starting materials was determined by the Tovarov method.[9]

3. Results and Discussion

3.1. Lightweight Aluminosilicate Grouting Materials Based on the By-Products of Fireclay Production from Kaolin

The technological properties of lightweight aluminosilicate grouting materials based on a mixture of PCTI-100 oil-well Portland cement with PE powder are given in Tables 2 and 3. The new lightweight grouting materials are well retarded by standard chemical reagents. For example, at a temperature of 393–433 K and a pressure of 50–60 MPa, with the addition of synthetic tartaric acid (STA) or aminotrimethylene phosphonic acid (ATMP) as retarders (up to 0.1%), the pumping time of the 50:50 grouting composition (PCTI-100:PE) is more than 4 h. Grouting mortars based on such mixtures are resistant to sedimentation, and their bleeding is zero.

Table 2. Technological properties of mortars based on lightweight aluminosilicate grouting compositions

Composition, %		W/C	Density, kg/m³	Flowability, mm	Setting time, h–min at T= 348 K, P = 0.1 MPa	
PCTI-100	PE				beginning	end
100	–	0.50	1860	250	2–25	3–25
90	10	0.55	1790	185	2–17	2–40
80	20	0.70	1670	230	2–30	2–54
70	30	0.71	1640	215	2–00	2–27
60	40	0.70	1650	180	1–49	2–04
50	50	0.75	1640	200	1–45	2–05
40	60	0.80	1580	192	1–35	2–05

Table 3. Technological properties of cement stone of lightweight aluminosilicate grouting compositions

Composition, %		W/C	Stone strength after 2 days of setting, kg/cm³									
PCTI - 100	PE		348 K 0.1 MPa		373 K 30 MPa		393 K 50 MPa		413 K 50 MPa		433 K 60 MPa	
			B	Com	B	Com	B	Com	B	Com	B	Com
100	–	0.50	60.0	150.0	38.0	115.0	26.2	55.62	13.1	42.5	–	–
90	10	0.55	48.3	88.12	27.0	85.0	26.1	68.28	22.3	62.0	10.3	20.0
80	20	0.70	33.50	70.34	25.0	78.0	26.7	97.5	23.0	69.0	16.3	48.4
70	30	0.71	20.1	38.06	22.3	74.48	30.4	105.0	30.0	103.0	22.0	59.37
60	40	0.70	21.0	40.60	24.70	56.30	35.7	117.3	39.0	103.0	37.7	88.7
50	50	0.75	17.4	39.0	15.1	37.4	14.7	47.5	32.4	79.3	34.0	77.0
40	60	0.80	9.0	20.65	7.1	16.3	10.2	16.4	15.1	20.3	11.8	26.7

Notes: B is bending; Com is compression

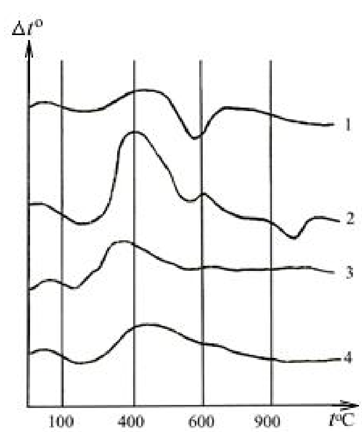

Fig. 1. Thermogram of cement samples from lightweight aluminosilicate grouting compositions based on PCTI-100 Portland cement and PE aluminosilicate admixture
1 – PE powder is not hydrated; 2 – Portland cement PCTI-100, 348 K; 3 – PCTI-100:PE – 50:50, 348 K; 4 – PCTI-100:PE – 50:50, 453 K; the hydration period is 2 days

The mineralogical composition of cement stone made of Portland cement with the addition of an aluminosilicate admixture (PE powder) was studied using a differential-thermal method.[10-12] The resulting curves are shown in Fig. 1. When this grouting mortar is set under barothermal conditions we observe the presence of ettringite, as well as calcium hydroaluminate ($4CaO{\cdot}3Al_2O_3$). They are characterized by endothermic effects in the temperature range of 433–1043 K. The height of the peaks of the endoeffect thermograms clearly shows the process of binding kaolin to kaolinite, and with increasing autoclaving temperature – to metakaolinite (endoeffect at 893–918 K).

Effects related to portlandite are insignificant, which can be explained by the adsorption and binding of portlandite in the metakaolinite core.

Powdery by-products of the fireclay production from kaolin are accumulated in large volumes at the Zaporizhzhya Plant of Refractory Materials.

3.2. Lightweight Grouting Materials with the Use of Fly Ash from SDPP

The developed lightweight grouting materials using fly ash from SDPP are widely used in geological and production drilling companies for cementing casing in wells. The theoretical basis for the research was the possibility of maximizing the sedimentation stability of unstable dispersed systems based on compositions of Portland cement and acidic fly ash (AFA) at a very high water-cement ratio using the OEC stabilizer.[13]

The grouting mixtures with the PCTI-100:AFA ratios of 70:30; 60:40; 50:50; 40:60; 30:70 were studied. Acid fly ash from Ladyzhynska SDPP (AFA_L) and Kurakhivska SDPP (AFA_K) were used for research. Bleeding of grouting mortars stabilized with 0.1, 0.2, and 0.3% of OEC was studied at W/C ratios of 0.7, 0.9, and 1.0 (Fig. 2). It was found that the introduction of 0.3% OEC allows to obtain a grouting mortar with a density of 1450 kg/m^3 with good sedimentation stability. At the same time, a cement stone with sufficient strength is formed (Fig. 3). The compositions PCTI-100:AFA$_L$ with the ratio of 60:40 and PCTI-100:AFA$_K$ with the ratio of 50:50 were found to be the most acceptable from the standpoint of technological properties.

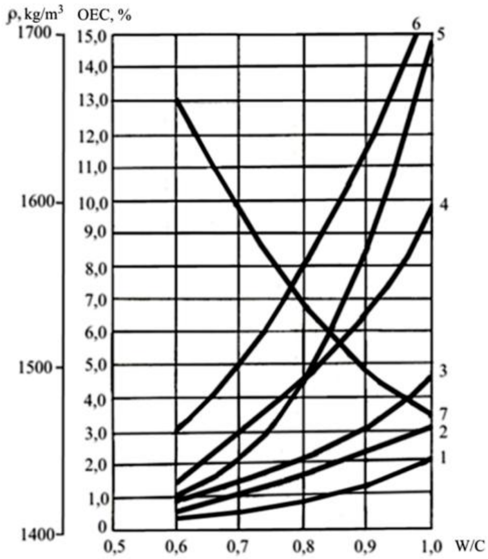

Fig. 2. Bleeding and density of cement-ash mixture *vs.* water-cement ratio (kg/m^3): 1 – cement:ash = 60:40, OEC 0.3%; 2 – cement:ash = 50:50, OEC 0.3%; 3 – cement:ash = 70:30, OEC 0.3%; 4 – cement ash = 60:40, OEC 0.1%; 5 – cement:ash = 70:30, OEC 0.1%; 6 – cement:ash = 50:50, OEC 0.1%

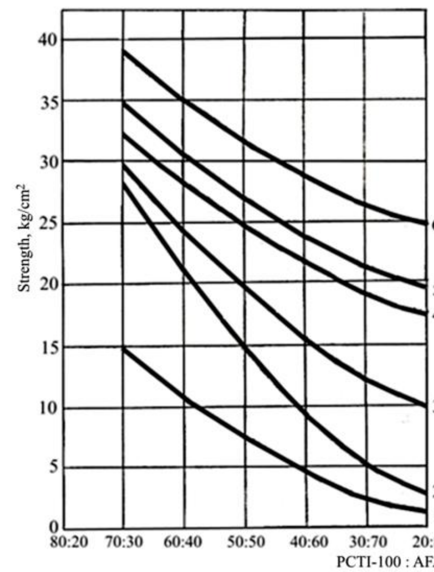

Fig. 3. Strength of cement-ash stone *vs.* mixture components ratio with 0.3% OEC (kg/cm^2) : 1, 3, 5 – W/C = 0.9; 2, 4, 6 – W/C = 0.7. Setting conditions: 1, 2 – 313 K; 3, 4 – 348 K; 5, 6 – 373 K; time - 2 days

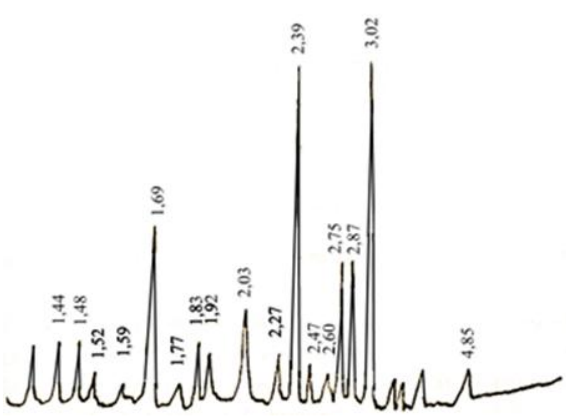

Fig. 4. X-ray of non-hydrated lime dust

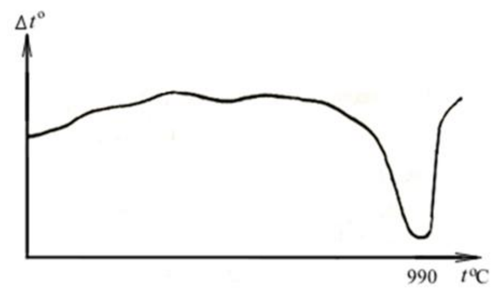

Fig. 5. Thermogram of non-hydrated lime dust

3.3. Lightweight Clinkerless Grouting Materials Based on By-Products of Lime Production and Acidic Fly Ash from SDPP

Lightweight grouting materials based on the by-products of lime production and acid fly ash from SDPP have been developed. When lime is burned at metallurgical plants, a large number of by-products are formed in the form of lime dust. "Azovstal" metallurgical plant alone accumulates 60 thousand tons of lime dust annually.

According to X-ray structural and differential thermal analyses, the main minerals in the composition of lime dust are calcium and magnesium oxides (interplanar distances are 1.44; 1.69; 2.75 and 1.48; 2.03), as well as calcite (endothermic effect at 1263 K, interplane distances 1.59, 2.27, 2.39) (Figs. 4 and 5).[10,12]

The lime dust products under study lack silica (silicon oxide) for the effective formation of calcium hydrosilicates in the hydration process.[13,14] The lack of silica is compensated by the addition of acidic fly ash to the binder, for example, ash from Kurakhivska SDPP. Silica contained in acid ash contributes to the formation of heat-resistant hydrosilicates in the lime-acid ash-water system.

To choose the optimal composition of the grouting material, the mentioned components were taken with different weight ratios (from 10 to 90% taking into account the densities of lime dust (2890 kg/m^3) and acid fly ash (2020 kg/m^3).

Tables 4 and 5 show the technological properties of grouting mortars and the formed stone from the investigated compositions based on lime dust and acidic fly ash.

One can see that the strength of lime-ash stone increases with an increase in the setting temperature. During setting under normal conditions, the rate of chemical interaction between lime and silica of ash is very small and practically does not increase the strength.

In autoclave conditions at a pressure of 50–60 MPa and a temperature of 373– 473 K, calcium oxides of lime dust reacts with silica of ash with the formation of calcium hydrosilicates. At normal temperature, the lime solubility is much higher than that of silica. With the increase in temperature the solubility of lime decreases and the solubility of silica increases. This leads to the fact that the interaction between them begins in a lime-saturated solution, in which a lime-rich silica phase, such as C_2S α-hydrate, is stable, which is formed at the first stage in the samples of any initial composition and exists until $Ca(OH)_2$ is completely bound, *i.e.*, until the solution is saturated with lime. After the free lime is bound into hydrosilicate, the SiO_2 concentration in the liquid phase begins to increase as a result of free silica dissolution. The increase in SiO_2 concentration continues as long as the lime-rich phase remains stable. At a certain concentration in the solution, the transition of the lime-rich phase to a less basic hydrosilicate, which is stable under new conditions, begins. A similar process is repeated until a solid phase is formed that is stable in a saturated silica solution or in a solution the concentration of which is determined by the intrinsic solubility of new formations, in particular tobermorite and other low-basic hydrosilicates.[15-19]

Table 4. Technological properties of grouting compositions based on lime dust and AFA

Composition, %		W/C	Density, kg/m^3	Flowability, mm	Bleeding, mL	Strength after 2 days (bending/compression), MPa			Setting time (at T = 343 K, P = 0.1 MPa), h–min	
Lime dust	AFA					343 K 0.1 MPa	373 K 30 MPa	393 K 50 MPa	beginning	end
Ash from Ladyzhynska SDPP										
50	50	0.8	1520	270	24.0	0.8/1.27	0.93/1.46	1.25/1.70	12–00	18–00
40	60	0.8	1520	270	40.0	0.66/0.90	1.13/1.28	1.80/1.95	15–00	24–00
30	70	0.8	1510	250	46.0	0.6/0.78	0.47/0.79	0.83/1.25	16–00	27–00
Ash from Kurakhivska SDPP										
50	50	0.8	1460	190	0.5	0.7/2.4	2.9/7.3	1.2/3.0	10	16
40	60	0.8	1440	230	4.0	0.8/2.6	3.0/8.7	2.8/5.1	12	18
30	70	0.8	1370	270	27.5	0.9/3.1	3.0/8.9	5.5/15.6	12	18

Table 5. Technological properties of grouting compositions based on lime dust and AFA from Kurakhivska SDPP

Composition, wt. %		W/C	Density, kg/m^3	Flowability, cm	Strength, MPa						
					Setting conditions – temperature (K) / pressure (MPa):						
					348 / 0.1				393 / 50	473 / 60	
					Setting time, days						
Lime dust	AFA_K				2	7	14	28	2	2	7
10	90	0.65	1450	18.5	0.47	0.71	1.19	0.83	2.52	3.93	3.15
30	70	0.75	1480	19.5	0.95	3.28	3.78	3.16	5.44	3.78	5.75
50	50	0.85	1480	19.0	0.71	1.65	2.84	2.01	1.18	1.65	1.95

The data of X-ray structural and derivatographic analyzes (Figs. 6 and 7) show that various calcium hydrosilicates are formed in lime-ash mixtures with a lime:ash ratio of 30:70 (w/w) during the samples setting. At the temperatures of 348–375 K, hydrosilicates $C_3SH(A)$ – α-hydrate are formed (temperature effect is 783–793 K), which then transforms into CSH(B) (CSH(I)) with interplane distances of 1.52, 3.02 and 5.4, as well as portlandite $Ca(OH)_2$ (temperature effect is 893 K) and calcite $CaCO_3$ (temperature effect is 1193–1233 K) (Figs. 6 and 7). The increase in the autoclave treatment time and pressure leads to the complete disappearance of effects of portlandite $Ca(OH)_2$ and α-hydrate C_2S and the formation of tobermorite, xonotlite and hyrolite (temperature effect is 1073 K). The formation of these hydrosilicates increases the strength, heat resistance, durability and other properties of the studied compositions.

The possibility of lime preliminary slaking with subsequent mixing of the grouting material with water was studied. The slaking time was chosen experimentally and was equal to one hour, W/C = 0.8. The density of the mixing water (lime-water) was 1200–1310 kg/m^3. The temperature during slaking of fresh lime was 319 K, and that of slaked lime was 302 K.

The technological properties of the grouting mortar and the stone obtained on the basis of slaked lime are given in Table 6.

The obtained results (Table 6) show the possibility of using lightweight lime-ash grouting mixtures for cementing long casing strings in one step.

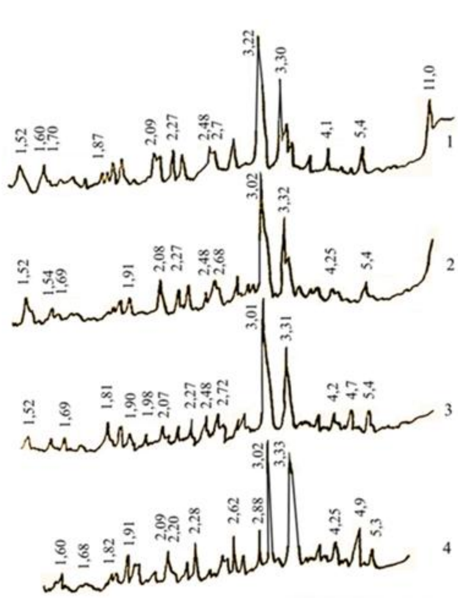

Fig. 6. X-ray diagram of lime-ash mixtures. Setting conditions: 1 – T = 348 K, P = 0.1 MPa, 2 days; 2 – T = 373 K, P = 50 MPa, 2 days; 3 – T = 473 K, P = 60 MPa 2 days; 4 – T= 473 K, P = 60 MPa, 7 days

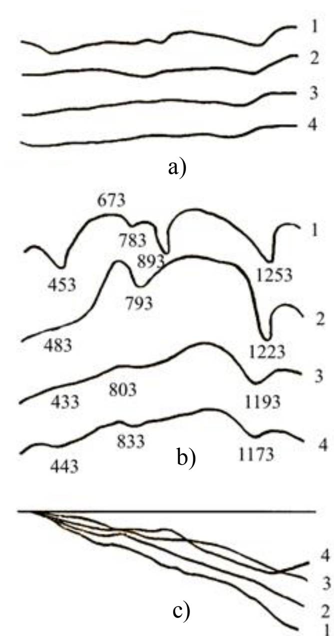

Fig. 7. Derivatograms of lime-ash mixtures: DTG (a), DTA (b) and TG (c)

Table 6. Technological properties of grouting compositions based on slaked lime and AFA from Ladyzhynska SDPP

Composition, wt. %		W/C	Density, kg/m^3	Flowability, mm	Bleeding, mL	Pumpability, h-min		Setting time, h-min		Strength after 2 days, MPa	
						Conditions		at 343 K, 0.1 MPa		Setting conditions	
lime dust	AFA_L					343 K, 0.1 MPa	373 K 30 MPa	beginning	end	343 K, 0.1 MPa	373 K, 30 MPa
50	50	0.8	1520	250	24	3-00	3-00	10-00	10-00	8.0/12.7	8.7/14.2
40	60	0.8	1520	250	40	2-10	2-00	15-00	15-00	6.7/9.0	6.7/9.0
30	70	0.8	1510	250	46	1-40	1-20	15-00	15-00	6.0/7.8	6.0/7.0

4. Conclusions

4.1. The composition of hydration products of lightweight grouting materials – mixtures that have been set for two and seven days at high temperatures – was determined by X-ray phase and differential thermal analyses of cement stone. The specific peculiarities for the materials formed under barothermal conditions are following:

a) for lightweight aluminosilicate grouting materials based on by-products of fireclay production from kaolin there is a presence of ettringite and calcium hydroaluminate ($4CaO{\cdot}3Al_2O_3$);

b) for lightweight clinkerless grouting materials based on the by-products of lime production and acidic fly ash from SDPP with a lime:ash ratio of 30:70 (w/w), the formation of various calcium hydrosilicates is observed; at the temperatures of 348–375 K, hydrosilicates $C_3SH(A)$ – α-hydrate are formed, which then transform into CSH(B) CSH(I), as well as portlandite $Ca(OH)_2$ and calcite $CaCO_3$; the increase in the autoclave treatment time and pressure leads to the complete disappearance of effects of portlandite $Ca(OH)_2$ and α-hydrate C_2S and the formation of tobermorite, xonotlite and hyrolite. The formation of these hydrosilicates increases the strength, heat resistance, durability and other properties of the studied compositions

4.2. The study of the dependence of the technological properties of cement stone based on lightweight grouting materials on physicochemical factors, in particular, the composition of the grouting mixture, temperature, pressure, and setting time, made it possible to establish the following characteristic features:

a) for lightweight aluminosilicate grouting materials based on the by-products of fireclay production from kaolin, the composition with the content of PCTI-100 50-80 wt. % and PE 50-20 wt. % can be considered optimal; however, at a temperature of 413 K and higher the 2-day samples lose their strength. Taking into account the long service life of wells, these compositions can be used at the temperatures below 373 K;

b) for the lightweight grouting materials with fly ash from SDPP after 2 days of setting the highest strength is achieved at the maximum PCTI-100 content and the maximum temperature; 2-day compositions with PCTI-100:AFA ratio of 20:80 (w/w) have the lowest strength in the entire temperature range, which indicates the low activity of these binders and their suitability for application at high temperatures;

c) for lightweight clinkerless grouting materials based on the by-products of lime production and acidic fly ash from SDPP after 2 days of setting the strength of all samples with different ratios of ingredients increases with increasing temperature; for the samples set at a temperature of 348 K, the dynamics of strength gain stops after 14 days, on the 28th day a slight decrease in strength is observed, which is obviously associated with the transition of α-hydrate $C_3SH(A)$ to CSH(B).

The result of this work is the creation of a new class of lightweight grouting materials with a density of 1370–1650 kg/m^3. New materials consist of oil-well Portland cement PCTI-100, fine powder of PE grade (a by-product of fireclay production from kaolin), acidic fly ash from SDPP, and lime dust (a by-product of lime production). In terms of their operational parameters, when considered in their entirety, the obtained materials surpass all known lightweight grouting cements and are distinguished by heat resistance, high strength, and wide density change interval.

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РОЗРОБКА ПОЛЕГШЕНИХ ТАМПОНАЖНИХ МАТЕРІАЛІВ НА ОСНОВІ ПОБІЧНИХ ПРОДУКТІВ ПРОМИСЛОВОСТІ УКРАЇНИ

Анотація. *Створено новий конкурентоздатний клас тампонажних матеріалів – полегшених і легких тампонажних матеріалів (ПТМ) густиною 1370–1650 кг/м^3. Компоненти: тампонажний портландцемент ПЦТІ-100, побічний продукт виробництва шамоту з каолінової сировини – тонкодисперсний порошок марки ПЕ, кислі золи виносу ТЕС, побічний продукт виробництва вапна – вапняний пил (ВП). Рентгенофазовим і диференційно-термічним аналізом полегшеного тампонажного каменю уточнено склад новоутворень у процесі гідратації ПТМ. Визначено раціональні оптимальні співвідношення інгредієнтів і температурні режими тужавіння ПТМ за критеріями міцності цементного каменю.*

Ключові слова: *цементування свердловин, полегшений тампонажний матеріал, гідратація цементу, зола виносу, водосумішеве відношення, цементний камінь.*