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ВПЛИВ ОРГАНО-МІНЕРАЛЬНОГО МОДИФІКАТОРА НА ОСНОВІ УЩІЛЬНЕНОЇ ФОРМИ МІКРОКРЕМНЕЗЕМУ ТА (PNS+PCE)-СУПЕРПЛАСТИФІКАТОРІВ НА ВЛАСТИВОСТІ Й МІКРОСТРУКТУРУ ЦЕМЕНТНОГО КАМЕНЯ

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Анотація. Мікрокремнезем є однією и найбільш важливих мінеральних добавок при виробництві високоякісних портландцементних бетонів. Висока реакційна здатність мікрокремнезему до портландцементу обумовлена його дуже високою питомою поверхнею та високим вмістом аморфного діоксиду кремнію. Попереднє інтенсивне перемішування ущільненої форми мікрокремнезему, що утворюється при висиханні шламу мокрого газоочищення феросплавних заводів у розчині ПНС-суперпластифікатора з наступним додаванням одержаної органо-мінеральної суспензії (модифікатору) до суміші портландцементу, води замішування та полікарбоксилатного суперпластифікатора (роздільне перемішування) забезпечує підвищення текучості цементної пасти та міцності при стиску цементного каменя в порівнянні зі складом цементної пасти, що приготовлена за звичайною технологією. Ці ефекти обумовлені диспергуванням крупних агломератів мікрокремнезему в розчині ПНС-суперпластифікатора та виключенням конкурентної адсорбції аніонних суперпластифікаторів на позитивно заряджених продуктах гідратації цементу.

Ключові слова: мікрокремнезем, суперпластифікатор, цементна паста, текучість, мікроструктура, міцність при стиску.

ВЛИЯНИЕ ОРГАНО-МИНЕРАЛЬНОГО МОДИФИКАТОРА НА ОСНОВЕ УПЛОТНЕННОЙ ФОРМЫ МИКРОКРЕМНЕЗЕМА И (PNS+PCE)-СУПЕРПЛАСТИФИКАТОРОВ НА СВОЙСТВА И МИКРОСТРУКТУРУ ЦЕМЕНТНОГО КАМНЯ

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Аннотация. Микрокремнезем является одной из наиболее важных минеральных добавок при производстве высококачественных портландцементных бетонов. Высокая реакционная способность микрокремнезема к портландцементу обусловлена его очень высокой удельной поверхностью и высоким содержанием аморфного диоксида кремния. Предварительное интенсивное перемешивание уплотненной

формы микрокремнезема, образующегося при высыхании шлама мокрой газоочистки ферросплавных заводов в растворе ПНС-суперпластификатора с последующим добавлением полученной органо-минеральной суспензии (модификатора) в смесь портландцемента, воды затворения и поликарбоксилатного суперпластификатора (раздельное перемешивание) обеспечивает повышение подвижности цементной пасты и прочности при сжатии цементного камня в сравнении с составом цементной пасты, приготовленной по обычной технологии. Эти эффекты обусловлены диспергированием крупных агломератов микрокремнезема в растворе ПНС-суперпластификатора и исключением конкурентной адсорбции анионных суперпластификаторов на положительно заряженных продуктах гидратации цемента.

Ключевые слова: микрокремнезем, суперпластификатор, цементная паста, текучесть, микроструктура, прочность при сжатии.

INFLUENCE OF ORGANIC-MINERAL MODIFIER ON THE BASE OF CONDENSATED MICROSILICA AND (PNS+PCE)-SUPERPLASTICIZERS ON THE PROPERTIES AND OF CEMENT PASTE STONE MICROSTRUCTURE

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Abstract. Silica fume is one of the very important mineral additives for producing the high-performance Portland cement-based concretes. The high reactivity of silica fume with Portland cement is primarily due to its very high specific surface and its high content of amorphous silicon dioxide. The preliminary intensive agitation of densified form of silica fume from aged slurry of Ferro-alloy works in the solution of PNS superplasticizer and the subsequent addition of the received organic-mineral suspension (modifier) into the mixture of Portland cement, mixing water and PCE superplasticizer (separate mixing procedure) provides increasing fluidity and compressive strength of cement paste in a comparison to the formulation of cement paste prepared by the traditional mixing procedure. These effects are conditioned by dispergating large agglomerates of silica fume in the solution of PNS superplasticizer and exclusion of competitive adsorption of the anionic superplasticizers for the positively charged surface area of cement hydration products.

Keywords: silica fume, superplasticizer, cement paste, fluidity, microstructure, compressive strength.

1. Introduction

Silica fume is a by-product of the manufacture of silicon or of various silicon alloys. Silica fume, which contains more than 80-85 % SiO2 in amorphous form, is suitable to use in the cement and concrete industries. Most particles of a typical silica fume are smaller than 1 micron. The high reactivity of silica fume with Portland cement is primarily due to its very high specific surface and its high content of amorphous silicon dioxide. So, due to results of the filling effect to reduce porosity of cement paste and the pozzolanic reaction that consumes calcium hydroxides to yield calcium silicate hydrates (C-S-H), the silica fume is one of the very important mineral additives for producing the high-performance Portland cement-based concretes [1, 2].

On the other hand small particle size and low bulk density of silica fume makes it difficult to trans-

Table 1. Chemical composition and properties of the materials used.

Composition (%) Properties	OPC	SF-1	SF-2
SiO_2	21.4	91.8	81.8
Al_2O_3	5.8	1.1	1.6
Fe_2O_3	3.4	0.65	3.0
CaO	61.5	2.4	1.1
MgO	1,7	0.05	0.2
K_2O	0.7	0.1	0.6
SO_3	2.5	0.35	3.6
Loss on ignition	1.2	3.6	7.2
Bulk density ($kg \cdot m^{-3}$)	1310	215	655
Fineness ($m^2 \cdot kg^{-1}$)	365 (Blaine)	18600 (BET)	0.14-20 mm

Fig. 1. SF-images: SF-1 undensified form (left); SF-2 agglomerated form (right).

port, distribute and handle. So, commercial suppliers are obliged to process silica fume using different methods of compaction in order to agglomerate the small individual particles into relatively large clumps containing millions of particles and measuring up to several millimeters in size (densified or pelletized) [3-6].

Pelletized silica fume is always interground with cement before use, thereby eliminating large clumps. Densified silica fume is usually added to concrete as it is. The agglomerated silica fume particles cannot be easily broken up when the concrete mixture is mixed, especially at low water-to-cement ratio even in the presence of superplasticizer. In this case the effectiveness of silica fume as microfiller and as pozzolanic additive reduces [3, 4]. Besides relatively large, nondensified clumps of silica fume may affect substantially the mechanism of concrete deterioration. According to [7] these clumps were observed to react in a manner similar to reactive aggregates with cement alkalies (ASR) to form silica gel that is often associated with expansion whereas in [8] it is reported that such reactions may induce ASR damage only under especially unfavourable conditions.

These problems become especially significant when agglomeration of ultra fine particles of silica fume takes place during the drying process in sludge collectors (wet method of gas cleaning in a Ferro-alloy production) and is stipulated by the condensation polymerization. This reaction is accompanied with the formation of siloxane linkage, growth of colloid particles and gel formation. So, aged agglomerated aqueous suspensions may need to be redispersed before use in concrete. Nevertheless the difficulty of redispersion, for example grinding in a boll mill, is stipulated due to the high interparticle forces (forces of the electrostatic charging, Van-der-Waal's forces and forces due moisture) [4]. In this case with the aim of increasing effectiveness of milled aged agglomerated silica fume the special procedures are needed to achieve an adequate dispersal in particular the preliminary preparation of silica fume aqueous slurry. However this procedure is effective when concrete mixing is carried out by the special sequence. V. Marchuk [5] reported that only one mixing sequence - (cement + water + silica fume) with the silica fume added as slurry - can be recommended for the fullest possible dispersion and homogeneous distribution of the ultrafine particles during production of the cement paste in a mortar mixer.

The fact is certain that dispersive ability of silica fume clumps will be much higher in the solution of any surfactant (due to the Rehbinder effect). The superplasticizers which provide dispersion and deflocculation in colloidal systems are the

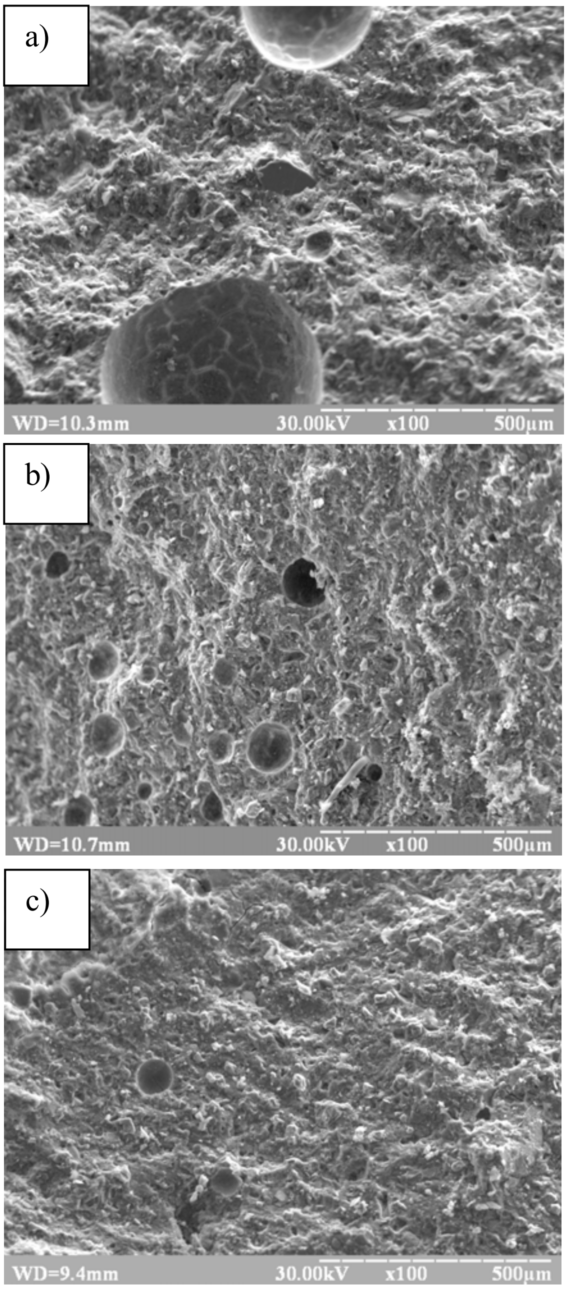


Fig. 2. SEM images of cement paste (a, b, c – formulation No 1, 2, 3, accordingly).

most suitable surfactant for this purpose because in the combination with silica fume they form effective organic-mineral modifiers of high performance concretes.

Although, the superplasticizer (water reducing agent) can help improve the properties of cement composites, some unpredicted interactions among water, water reducing agent, microsilica and cement are anticipated to complicate the study on the chemical mechanism of such composite material [9]. According to J. Plank and C. Winter [10] in complex admixture systems (especially PNS+PCE) the processes on the mineral binder surface can be explained as a competition of the anionic superplasticizers for the positively charged surface area - adsorption of molecules with higher anionic charge density (PNS) is preferred. On the other hand the influence of the cement chemistry on superplasticizer performance is attributed in part to the intercalation of the superplasticizers into hydration products. The intercalated polymer is lost for dispersion purposes and this decreases efficiency. In other cases, the efficiency of some polycarboxylates can be lowered because of competitive adsorption from sulfate ions [11]. The investigation of the effect of organic-mineral modifier on the base of densified silica fume and (PNS+PCE)-superplasticizers on the properties and microstructure of cement paste is the aim of this research.

2. Materials and methods

Ordinary Portland cement CEM I 42.5 N (OPC) and two types of silica fume were used as raw materials. Silica fume SF-1 is undensified (as produced powder) while silica fume SF-2 is agglomerated during the drying process of aqueous slurry (wet method of gas cleaning in a Ferro-alloy production) (Fig. 1). The chemical composition and physical properties of raw materials are given in Table 1.

It is clear from the data of Fig. 1 and Table 1 that the initial agglomerated microsilica (SF-2) is represented by large clumps. That's why we had to grind the material using a boll mill.

Two types of superplasticizers with different mechanism of dispergating were used:

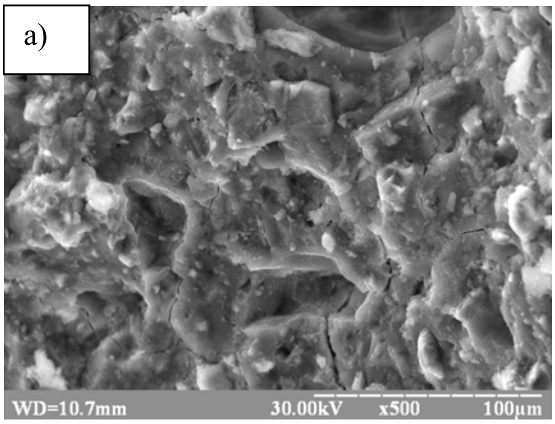
– polynaphthalene sulphonate condensate (PNS) – Mapefluid N 200 ("Mapei");
– modified polycarboxylic ether (PCE) – Woerment FM-794 ("DEGUSSA").

Mix proportions of cement pastes as well as the mixing sequence are as follows:

Cement paste 1 – OPC (240 g) + SF-2 (40 g) + W (110 ml), w/b ratio is 0.39;

Cement paste 2 – OPC (240 g) + SF-2 (40 g) + (W+PNS) (45 ml) + (W+PCE) (45 ml), w/b=0.32;

Cement paste 3 – [OPC (240 g) + (W+PCE) (45 ml)] + [SF-2 (40 g) + (W+PNS) (45 ml)], w/b=0.32.



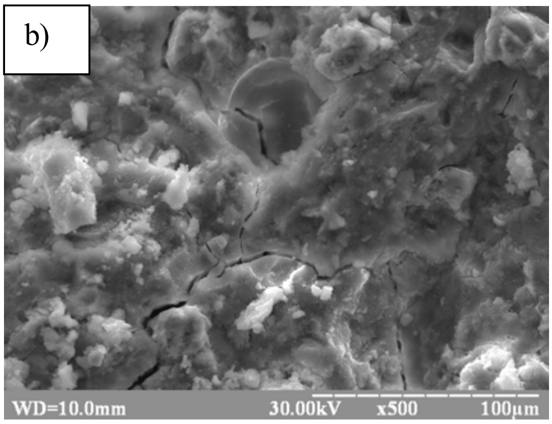


Fig. 3. SEM images of cement paste (a, b – formulation No 1, 3, accordingly).

The following investigations were carried out: the scanning electron microscope (SEM) with EDS and XRD examination of cement paste as well as determination of the compressive strength on 50 mm cubes. The flow of cement paste was measured in accordance with [12]. The flow pipe was 50 mm in diameter and 100 mm in height. Flow (F) was measured by averaging two crossing diameters of the spread. The relative flow area ratio (G) as the index of fluidity was calculated by the Eq: $G = F^2/50^2 - 1$.

3. Results and discussion

3.1. The flow of cement paste

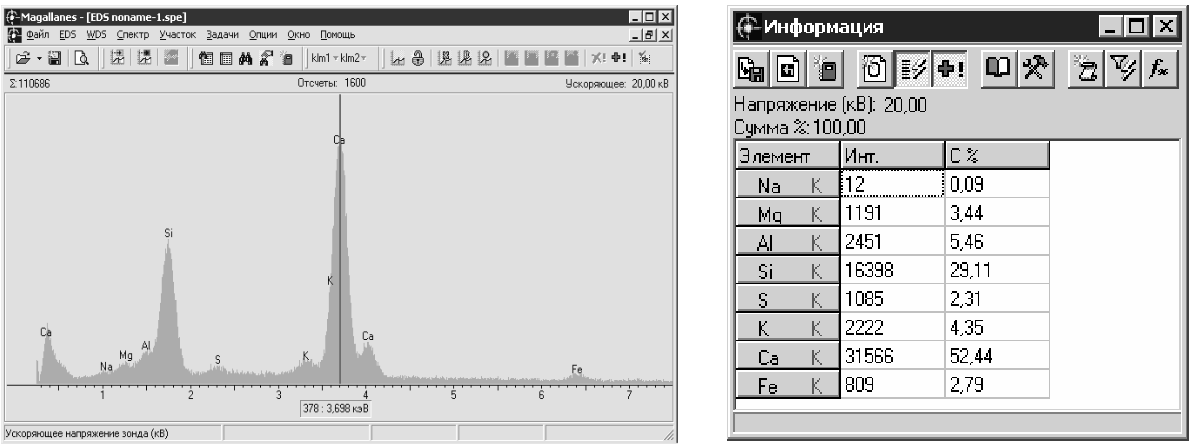
It has been found that cement paste with water-to-cement ratio w/c=0.27 is characterized by the relative flow area ratio G=0.9. In the case of partial replacement of Portland cement with silica fume SF-2 (formulation No 1) the water demand of cement paste substantially increases. When the water-to-binder ratio is 0.39 the index of relative flow area ratio reaches only G=0.85. The addition of two superplasticizers (N-200, 0.5 wt.% + FM-794, 0.5 wt.%) into the composition of cement paste, w/b=0.32 (formulation No 2) provides increasing the index G to 1.1. While a separate preparation of cement paste, foreseeing preliminary intensive mixing of silica fume in the solution of PNS-superplasticizer with the subsequent adding the obtained organic-mineral suspension into the cement paste, containing PCE-superplasticizer (formulation No 3), provides increasing the index G to 3.4. This effect can be explained from the next consideration.

We found earlier [13] that adsorption according to the data of ultraviolet spectroscopy of polynaphthalene sulfonate condensate on the surface of positively charged minerals of Portland cement clinker (C3A, C4AF) and mineral additives (limestone, dolomite) is much higher in comparison with modified polycarboxylic ether of equal solution concentration. Besides, when minerals contact with the binary solution of superplasticizers (PNS+PCE) adsorption of polynaphthalene sulfonate condensate is preferred.

So, in cement paste with comb-type superplasticizers (PNS+PCE) on the surface of hydration products (adsorptive layers) the molecules of PNS will dominate. In this case the fluidity of cement paste is not high because the steric hindrance between cement particles is not big enough. Preparation of cement paste by the separate mixing procedure when the polymer solutions are added sequentially was adopted to promote the adsorption of PCE molecules onto the cement particles, since the PNS chains are preferentially adsorbed due to strong electrostatic attraction. Ultra fine particles of agglomerated microsilica previously dispersed in the solution of PNS-superplasticizer will be distributed homogeneously in the structure of cement paste and will promote ball bearing effects increasing the fluidity of cement paste (concrete mixture).

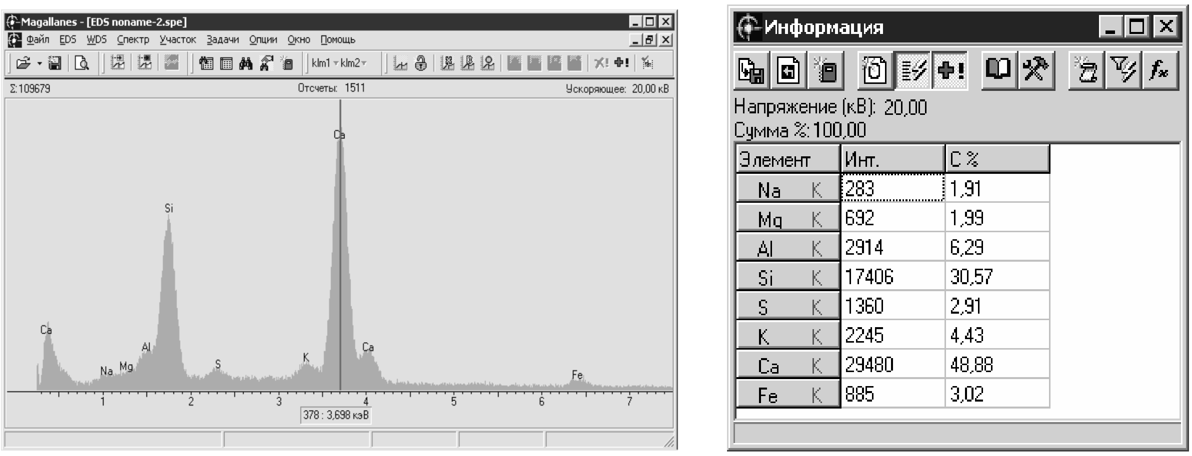
3.2. Microstructure of cement paste

Scanning electron microscope (SEM) images of three compositions of cement paste are shown in Fig. 2. Large spherical agglomerates ~500 mm were displayed (Fig. 2 a) in the microstructure of cement paste of formulation No 1 (28 days of hardening). These agglomerates in once or twice exceed the size of initial cement particles and can be identified as



Элемент		Инт.	C %
Na	K	12	0,09
Mg	K	1191	3,44
Al	K	2451	5,46
Si	K	16398	29,11
S	K	1085	2,31
K	K	2222	4,35
Ca	K	31566	52,44
Fe	K	809	2,79

Fig. 4. EDS of hydrated cement paste (formulation No 1).



Элемент		Инт.	C %
Na	K	283	1,91
Mg	K	692	1,99
Al	K	2914	6,29
Si	K	17406	30,57
S	K	1360	2,91
K	K	2245	4,43
Ca	K	29480	48,88
Fe	K	885	3,02

Fig. 5. EDS of hydrated cement paste (formulation No 2).

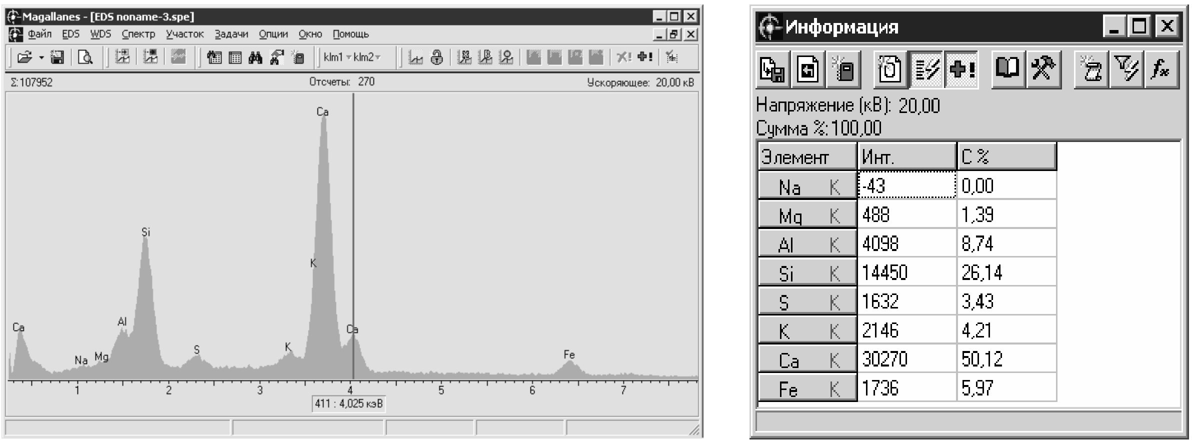
not dispersed clumps of micro silica. The addition of the comb-type superplasticizers (PNS+PCE) into the composition of cement paste (formulation No 2) provides dispergating large micro silica agglomerates, however the content of particles with the size ~50 mm remains big enough (Fig. 2 b). It is clear from the image of microstructure of cement paste, prepared by the separate mixing technology (formulation No 3), that the individual micro silica particles are highly dispersed and not agglomerated (Fig. 2 c).

A cement paste without addition of superplasticizer (formulation No 1) contains the very thin fibred crystals of mineral ettringite, piercing the hydrosilicate gel and the initial particles of Portland cement and microsilica (Fig. 3 a). At the same time in the microstructure of cement paste with addition of two superplasticizers (formulation No 3) on a background of amorphous hydrosilicate mass the crystalline particles of ettringite as small blocks are observed (Fig. 3 b). It testifies modifying influence of superplasticizer admixtures on the morphology of Portland cement hydratation products [14].

According to the data of Energy Dispersion Spectroscopy (EDS) calcium, silicon and aluminon are the main chemical elements in the structure of cement paste (Fig. 4-6). It should be noted the variances of sodium percentage in different formulations of cement pastes. In a standard formulation No 1 its content is 0.09%, at the same time in the standard formulation No 2 – 1.91 %.

Probably the higher sodium content in the composition of cement paste (formulation No 2) is stipulated by the presence of PNS-superplasticizer and its possible chemosorption on the Portland cement hydratation products.

However, when cement paste is prepared on the separate mixing procedure (formulation No 3) so-



Элемент		Инт.	C %
Na	K	-43	0,00
Mg	K	488	1,39
Al	K	4098	8,74
Si	K	14450	26,14
S	K	1632	3,43
K	K	2146	4,21
Ca	K	30270	50,12
Fe	K	1736	5,97

Fig. 6. EDS of hydrated cement paste (formulation No 3).

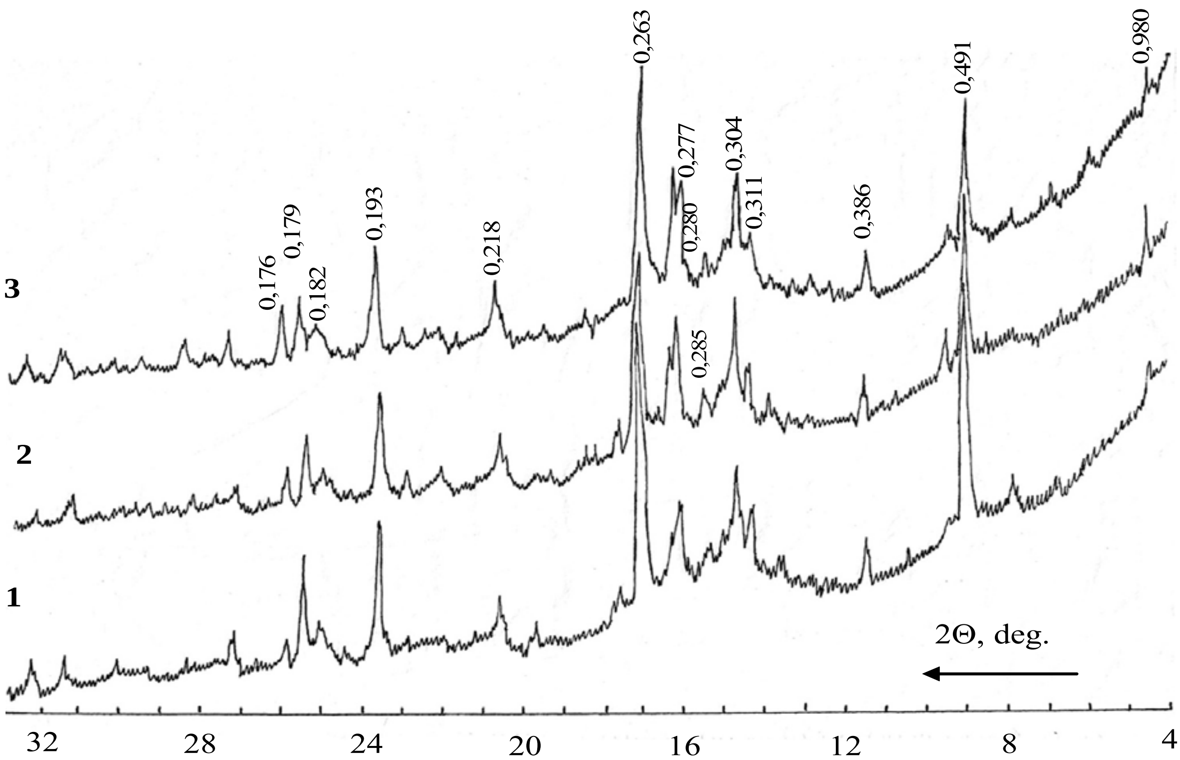


Fig. 7. XRD patterns of cement paste (formulations No 1-3).

dium content is equal to zero. At separate procedure of cement paste mixing the competition adsorption of two anionic polyelectrolytes (PNS and PCE superplasticizers) onto the positively charged aluminate phases of Portland cement is eliminated. The carboxylated polymer is mainly adsorbed onto the surface of cement particles while the polymethylenenaphthalenesulfonate molecules, partly adsorbed on-the-spot of silica fume, are in a dispersion environment as a result.

The results of XRD analysis of cement paste, hardening at normal terms during 90 days, testify to the high enough degree of Portland cement hydration - comparatively low intensity of diffraction reflections of tricalium silicate (Fig. 7). In spite of large term of hardening of cement paste and high pozzolanic activity of silica fume, there is a high content of portlandite in the microstructure of cement paste. At the same time the intensity of the most characteristic diffraction reflections of this mineral

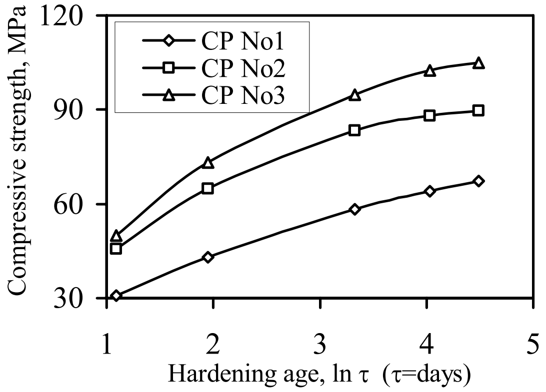


Fig. 8. Compressive strength versus hardening age for cement pastes with densified silica fume (formulations No 1-3).

(d=0.491; 0.311; 0.263; 0.193 and 0.179 nm) in the microstructure of cement paste with addition of superplasticizers considerably goes down in a comparison with the formulation No 1, especially at separate cement paste mixing procedure (formulation No 3).

The formation of calcium silicate hydrates with different basicity is a result of chemical binding of calcium hydroxide with an amorphous silicon dioxide. The presence of characteristic diffraction reflections of calcium silicate hydrates with the Ca/Si<1.5 ratio (d=0.304; 0.280; 0.182 nm) and Ca/Si=1.5-2 ratio (d=0.980; 0.285 nm) specifies on it.

3.3. Compressive strength of cement paste

The higher degree of dispersion of silica fume the higher compressive strength of cement paste is (Fig. 8). It should be noted also that growth of kinetics of compressive strength is the most intense for the cement paste of formulation No 3 which incorporates silica fume with maximal fineness. It is due to the pozzolanic reaction between amorphous silica dioxide and calcium hydroxide as well as due to result of the filling effect which reduces the porosity of cement paste.

4. Conclusion

The preliminary intensive agitation of densified form of silica fume from aged slurry of Ferro-alloy works in the solution of PNS superplasticizer and the subsequent addition of the received organic-mineral suspension (modifier) into the mixture of Portland cement, mixing water and PCE superplasticizer (separate mixing procedure) provides increasing fluidity of and compressive strength of cement paste in a comparison to the formulation of cement paste prepared by the traditional technology. These effects are conditioned by dispergating large agglomerates of silica fume in the solution of PNS and exclusion of competitive adsorption of the anionic superplasticizers for the positively charged surface area of cement hydration products.

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