WARSAW UNIVERSITY OF TECHNOLOGY Index 351733
FACULTY OF CIVIL ENGINEERING
COMMITTEE FOR CIVIL AND WATER ENGINEERING
POLISH ACADEMY OF SCIENCES ISSN 1230-2945

DOI: 10.24425/ace.2020.135215
ARCHIVES OF CIVIL ENGINEERING
Vol. LXVI ISSUE 4 2020

INFLUENCE OF DIFFERENT TYPES OF NANOSILICA ON COMPRESSIVE STRENGTH DEVELOPMENT OF CEMENT MATRIX COMPOSITES

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This article presents test results of cement paste and binders with admixture of hydrophilic or hydrophobic nanosilica. The aim of the study was to determine the influence of nanosilica type and mixing method on compressive strength, porosity, and bulk density of cement paste, also on hydration heat of cement binders. The binder compounds were mixed in high speed mixer in order to provide the highest possible dispersion of nanoparticles in the binder before adding it to mixing water. Two mixing methods were studied. The admixtures increased the reactivity of cement binders. Both nanosilica types increased early compressive strength by 25% in comparison with control series. The increase in 28-day compressive strength was observed with the admixture of hydrophilic nanosilica. The differences in dynamics of binders rate of hydration and development of cement pastes compressive strength denote different reaction mechanisms of both types of nanosilica. Application of higher rotation speeds does not guarantee satisfactory mixing of the binder components. For compressive strength enhancement of cement paste prolonged mixing time occurred to be more important.

Keywords: cement, nanosilica, admixture, nanoparticles, paste

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1. INTRODUCTION

Nanotechnology made its place in various industries, especially electronics. With the development of scientific instruments there are more technological applications of nanoscience that are viable and affordable for other industries, less commonly associated with high precision. Breakthroughs have been made in the construction industry as well [1, 2]. There is a growing amount of research around nanoadmixtures application in cement composites with the purpose to modify the structure and properties of concrete [2–5]. Varieties of nanoadmixtures are investigated with the aim to evaluate their utility, the most studied are nanosilica, nanoalumina, carbon nanotubes, or nanotitania [2, 6].

The most popular one is nanosilica. It is an amorphous artificial silica with high specific surface and high chemical purity. Its pozzolanic reaction has the potential to substantially influence the compressive strength of concrete and enhance its durability [4]. The diameter of nanosilica particles is about 12-20 nm, but can easily agglomerate into clusters [7, 8]. Nanosilica particles can contribute to the speed of cement hydration by providing additional nucleation sites for reactions. The unreacted nanoparticles work as a nanoscale filler, thus they strengthen the cement matrix and reduce its porosity. Nanoparticles also promote formation of smaller and denser C-S-H crystals [1, 5].

Nanosilica can be produced by different routes and in different forms, the most popular being high temperature pyrolysis and chemical precipitation. It can be provided in colloid or powder form, in many ranges of granulation, or with surface modification. Different types of nanosilica have different effectiveness in altering the properties of cement composites [9]. A 0.5% admixture of nanosilica in a colloidal-sol can accelerate the peak of hydration of cement paste [10]. Colloidal nanosilica has a severe impact on rheology of cement paste and has a higher free water retention capacity than powdered nanosilica [11]. High temperature calcination can furtherly enhance the maximum temperature achieved during the hydration process and retard its peak [12]. A 0.8% admixture of nanosilica can decrease the slump test results of cement paste by 75% [13]. A 5% admixture of nanosilica can decrease the initial setting time of cement paste by 25% [7]. The resistance to sulfate attack of cement mortar can be diminished with a decrease in particle size of colloidal nanosilica due to higher agglomeration [14]. SCC concrete with colloidal admixture of nanosilica can achieve higher early and 28-day compressive strengths than SCC concrete with powdered nonosilica admixture. On the other hand, higher standard deviation of concrete series with colloidal admixture suggests higher agglomeration tendency of this type of nanosilica. The series with powdered and colloidal type of nanosilica had similar 91-day compressive strength. Both types of nanosilica decreased chlorine penetration [15]. Coarse nanosilica obtained as a by-product of silicon production and collected via flotation method can enhance 15-day compressive strength by up to 40% [16].

Dispersion of nanoadmixtures is crucial for practical application of such small admixtures in concrete production [4, 8, 17]. Agglomerated nanosilica can provide imperfections in cement matrix [9], and different forms of nanosilica exhibit different tendency for agglomeration. While stable in its sol form colloidal nanosilica tends to form flocks in cement mixture [11]. Precipitated nanosilica tends to form more agglomerates in the cement matrix than fumed nanosilica [9]. The delayed addition of mixing water is one of the proposed procedures for improving workability and dispersion [13]. Premixing nanosilica with cement in acetone solution was also tested [18]. Immersing concrete in nanosilica solution was also evaluated [19]. Usually, nanosilica is dispersed in water and superplasticizer via mixing or ultrasonification before being added to the cement and aggregate [14, 20–22].

There is some research on the admixture of microsized hydrophobic and hydrophilic silica to cement composites [23], but there is scarcity of literature on hydrophilic and hydrophobic nanosilica. The authors decided to investigate the potential of cement-nanosilica high speed dry-mixing.

This article presents the results of the cement paste and binders tests with addition of hydrophilic and hydrophobic nanosilica. The aim of the study was to determine the influence of nanosilica type and mixing method on rate of hydration of the cement binder and compressive strength development, water saturation porosity, and bulk density of the cement paste.

2. Materials and test methods

Two types of nanosilica were used – hydrophobic (hphob) and hydrophilic (hphil). Their properties as declared by the producer are presented in Table 1. CEM I 42.5R cement in accordance with PN-EN 197-1 standard was used. Distilled water and superplasticizer with polycarboxylate chemical basis were used in the research. The density of the superplasticizer was 1,06±0,02 kg/dm^3 and the pH 8±1.

Table 1. Properties of the hydrophilic and the hydrophobic nanosilica

Type of nanosilica	Specific surface [m^2/g]	SiO_2 content [%]	Bulk density [g/dm^3]	pH	Loss on ignition [%]
Hydrophobic	170±30	99.8	50	3.5÷5.5	2
Hydrophilic	300±30	99.85	50	3.6÷4.3	2

Ten series of cement paste were prepared, their mixes are presented in Table 2. All cement pastes had a set amount of binder (500 g for a batch). Nanosilica was used as a cement replacement. The amount of superplasticizer of each series was determined experimentally with consistency test – samples of each series had to have a similar test result. Consistency was measured with a cone (volume of 0.345 dm^3) and diameter of spread was measured in two perpendicular directions. The water to binder ratio (w/b) was set and equal to 0.5. The amount of nanosilica in the binder changed within series and was either 0%, 1% or 2% of binder mass.

Two binder mixing methods in high-speed mixer were adopted. The aim was to provide the highest possible uniformity of dry components of the binder before adding water. Uniform dispersion of nanoparticles in the mixture of cement and nanosilica might hinder the tendency of nanoadmixture to reagglomerate and provide its satisfying dispersion in cement paste.

Table 2. Cement binders mixtures for preparation of cement pastes (w/s = 0.5) with consistency tests results

I mixing method					
Series number	I	II	III	IV	V
Series code	I mm 0%	I mm 1% hphil	I mm 2% hphil	I mm 1% hphob	I mm 2% hphob
Cement [g]	500	495	490	495	490
Hydrophilic nanosilica [g]	0	5	10	0	0
Hydrophobic nanosilica [g]	0	0	0	5	10
Superplasticizer [g]	0	2.5	3.75	1.35	3
Consistency [mm]	145	142.5	140	142.5	137.5
II mixing method					
Series number	VI	VII	VIII	IX	X
Series code	II mm 0%	II mm 1% hphil	II mm 2% hphil	II mm 1% hphob	II mm 2% hphob
Cement [g]	500	495	490	495	490
Hydrophilic nanosilica [g]	0	5	10	0	0
Hydrophobic nanosilica [g]	0	0	0	5	10
Superplasticizer [g]	0	2.25	3.75	1.35	3.75
Consistency [mm]	147.5	140	142.5	145	140

The first mixing method (I mm) started with spoon mixing of cement with nanosilica for 90 s. For 30 s the binder was mixed in high-speed mixer on its first gear (11000 rpm) and for 15 s on its second gear (14500 rpm). Mixing was finished by additional 30 s of spoon mixing. The second mixing method (II mm) combined spoon mixing of cement with nanosilica for 90 s, 90 s of mixing in high-speed mixer on its first gear (11000 rpm) and final 90 s of spoon mixing.

The preparation of cement paste started with 5 min of mixing distilled water with superplasticizer with a magnetic stirrer. Water with superplasticizer had been added to the binder and during 10 s after that mixing of cement paste in laboratory mortar mixer started. The first step involved 90 s of slow rotation mixing (100 rpm). Next, 30 s of spoon mixing involving scrapping the cement paste from the bottom and the walls of the container followed. Finally, 90 s of additional mixing in the mixer with slow rotations ended the mixing process.

Consistency tests were performed after mixing. Cement paste specimens for compressive strength, density and water saturation porosity tests were prepared in cubic forms of 20x20x20 mm dimensions. Each series contained 36 specimens. The tests were performed after 2, 7 and 28 days from mixing. At each test time 6 specimens were used for compressive strength test and 6 for water saturation porosity test and density test. Specimens were placed in water after demoulding up until performance of the tests. Water saturation porosity was calculated as the amount of water absorbed by the hardened paste related to the volume of the specimen, assuming that the volume of pores accessible to water can be treated as identical to bulk water saturation. Samples were dried to constant weight in 105°C for their dry-weight and measured. After that they were saturated to constant weight in water for their wet-weight. Using conduction calorimeter designed by the Institute of Physical Chemistry of the Polish Academy of Sciences [24] the change in the rate of hydration in the first 48 h from mixing was observed.

4. Test results and analysis

4.1. Hydration heat tests

Analyzing the results of hydration heat tests it can be observed that the difference in the mixing method had no influence on the results of control binders and binders with 1% admixture of hydrophobic nanosilica (Fig. 1 and Fig. 2).

The admixture of hydrophilic nanosilica accelerated the hydration peak of cement binder. It occurred an hour earlier than in the case of control binder and its maximum value was 6% higher. 1% admixture of hydrophobic nanosilica retarded the occurrence of the hydration peak by an hour comparing it with the results of the control binder. Its value was also 3% higher than the value of the peak for the pure cement paste specimen. Pastes with 2% admixture of hydrophobic nanosilica had the highest hydration peak value. Their values were 11% higher than for the control paste hydration peak in the case of I mixing method (Fig. 1) and 8% higher in the case of II mixing method (Fig. 2).

Both nanoadmixtures had a different influence on hydration heat of cement paste. Hydrophilic nanosilica accelerated the occurrence of the hydration peak, whilst hydrophobic retarded it. The results are the indication of different interaction mechanisms of both nanoadditives with cement.

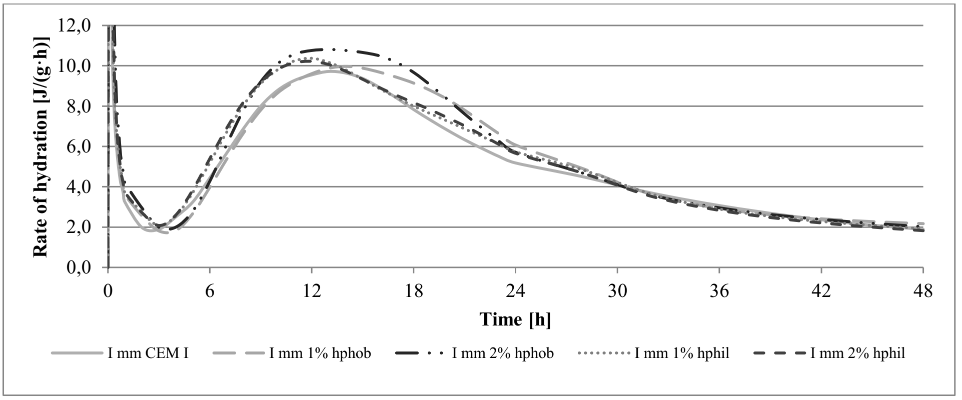

Fig. 1. Hydration heat of cement pastes mixed with a) I mixing method (I mm)

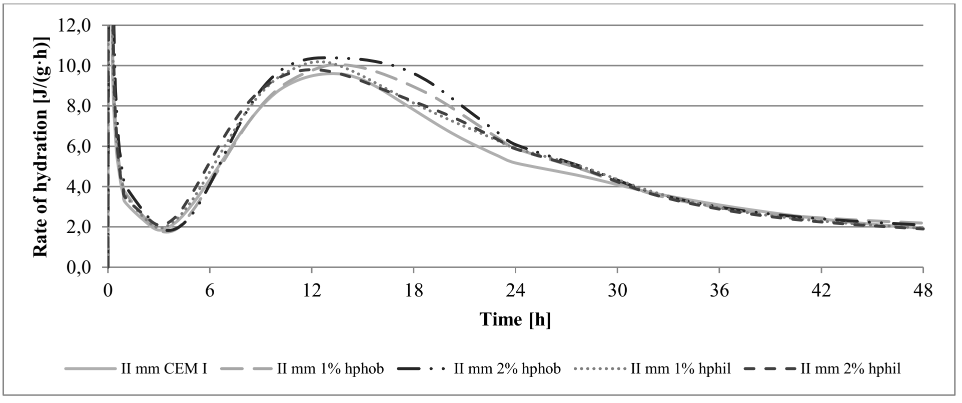

Fig. 2. Hydration heat of cement pastes mixed with b) II mixing method (II mm)

4.2. Compressive strength tests

The admixture of hydrophilic nanosilica increased the early mechanical properties of cement paste mixed by the I mixing method. The 2-day compressive strength of specimens with 1% and 2%

admixture was 25% higher than the result of control specimens (Fig. 3). 1% admixture of hydrophobic nanosilica had a similar effect on the results and 2% admixture increased them by 5%.

Analyzing the results of series mixed by I mixing method (I mm), the authors noticed that the highest increase in compressive strength was observed in the 7-day compressive strength results. The series with hydrophilic nanosilica notified compressive strength results higher by 31-35% than control specimens. The increase in compressive strength was smaller in the case of hydrophobic nanosilica. The 1% admixture increased the 7-day compressive strength values by 17% and the 2% admixture by 9%.

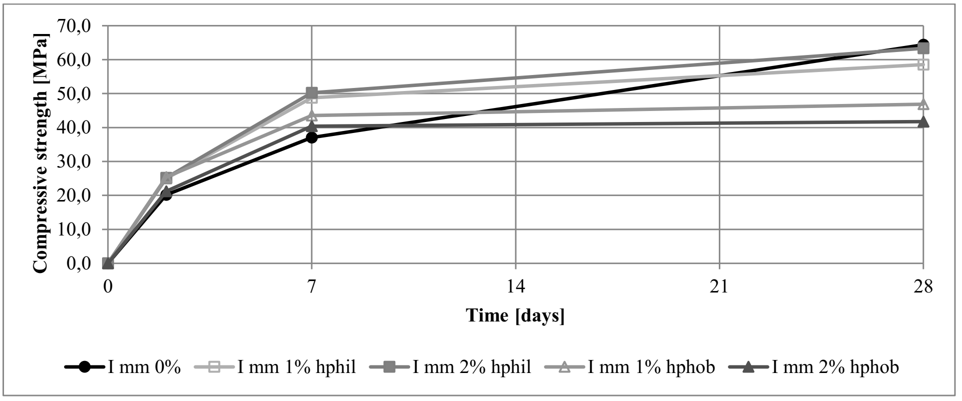

Fig. 3. Development of compressive strength of cement paste with admixture of nanosilica mixed by I mixing method (I mm)

All specimens mixed with I mixing method (I mm) had lower 28-day compressive strength results than the control specimens. In the case of hydrophilic nanosilica admixture the decrease was small: with 2% admixture 2% lower, and with 1% admixture 9% lower than the result of control specimens. However, in the case of hydrophobic nanosilica the decrease was substantial with 27% lower values than control specimens values for 1% admixture, and 35% for 2% admixture.

The increase in the 2-day compressive strength was also observable with the II mixing method but smaller than in the group of specimens mixed with I mixing method (Fig. 4). The admixture of 2% hydrophilic nanosilica increased the 2-day compressive strength by 19% comparing with the control specimens results. 1% admixture of hydrophobic nanosilica increased it by 10% and 2% admixture by 14% comparing with the control specimens results. Simultaneously, 1% admixture of hydrophilic nanosilica decreased 2-day compressive strength by 2%.

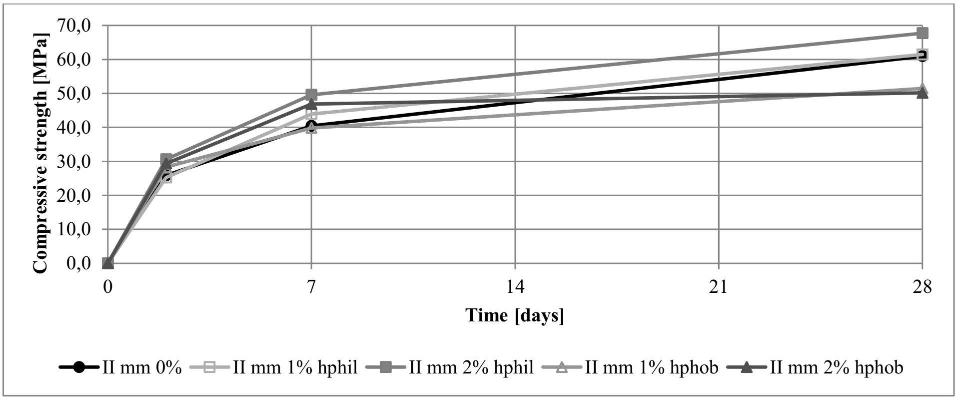

Fig. 4. Development of compressive strength of cement paste with admixture of nanosilica mixed by II mixing method (II mm)

The influence on the 7-day compressive strengths was also different in the case of II mixing method (II mm). The highest increase in compressive strength in comparison with the control paste specimens was observed in specimens with 2% admixture of hydrophilic nanosilica and it was 23% higher than the results of control specimens. 1% admixture of hydrophilic nanosilica also increased the 7-day results but by 9%. 16% increase in 7-day compressive strength results in comparison with the control specimens results was observed in the series of 2% hydrophobic nanosilica admixture. 1% admixture of hydrophobic nanosilica decreased 7-day compressive strength by 2%.

The 1% and 2% admixture of hydrophilic nanosilica increased 28-day compressive strength by 1% and 11% respectively comparing with the control specimens results. Admixtures of hydrophobic nanosilica decreased it respectively by 16% and 18% comparing with the control specimens results.

There was a slight difference in the development of compressive strengths between control specimens results mixed by different mixing methods. The I mixing method (I mm) resulted in lower early compressive strength results, especially compared with the 2-day results – it was 22% lower than the results of control specimens mixed by II mixing method (II mm) at the same test time. The 28-day results were 6% higher for the specimens mixed by I mixing method in comparison to the control specimens mixed by II mixing method. Despite that, the highest 28-day result was observed in this study in the specimens mixed by the II mixing method with 2% admixture of nanosilica. The specimens of this series also had the highest overall 2-day compressive strength. The differences in 28-day compressive strength were clearly observable. While the increase in early compressive strength observed in both types of nanosilica might be associated with filler and seeding effect of this materials. The increase in 7-days compressive strengths of pastes with hydrophilic nanosilica corresponded with the acceleration of hydration peak of its binders in hydration heat tests, which might indicate that higher solubility and reactivity in the cement solution is vital for initial strength increase of the paste. The 28-day results indicate different reaction mechanisms of admixtures with cement.

4.3. WATER SATURATION POROSITY AND BULK DENSITY TESTS

The most observable difference in porosity results was noted in I mixing method (I mm) (Tab. 3). The highest porosity was noted in 2-days porosity of control specimens (51%), the lowest with 28-days porosity of specimens with 1% admixture of hydrophilic nanosilica (40%). The type of nanosilica had no influence on water saturation porosity values.

Table 3. Results of water saturation porosity and bulk density tests of cement pastes

Series code	Series number	Water sat. porosity [% vol.]			Bulk density [g/cm^3]		
		Time [days]					
		2	7	28	2	7	28
I mixing method							
I mm 0%	I	51.1	47.0	42.2	1.36	1.42	1.48
I mm 1% hphil	II	48.4	46.5	42.9	1.36	1.40	1.41
I mm 2% hphil	III	50.0	46.2	42.5	1.35	1.40	1.39
I mm 1% hphob	IV	49.5	45.2	40.2	1.31	1.37	1.35
I mm 2% hphob	V	48.8	43.8	42.3	1.26	1.29	1.30
II mixing method							
II mm 0%	VI	50.3	44.6	42.5	1.39	1.40	1.42
II mm 1% hphil	VII	49.4	47.2	44.2	1.35	1.41	1.43
II mm 2% hphil	VIII	49.6	45.5	44.6	1.35	1.38	1.41
II mm 1% hphob	IX	48.7	45.0	44.6	1.31	1.36	1.41
II mm 2% hphob	X	48.8	44.2	42.7	1.33	1.36	1.36

The differences in bulk density values were small and increased slightly with time. Especially comparing 2-days densities with 28-days densities, which reflect the increase in the amount of hydration products during this time period.

5. CONCLUSIONS

The research analysis on the influence of two types of nanosilica (hydrophobic and hydrophilic) and of the binder mixing method on the properties of cement binder can lead to following conclusions:
1.) The physical and chemical reaction mechanisms of both admixtures are different.
2.) The admixture of either hydrophilic or hydrophobic nanosilica had an influence on the rate of hydration of cement binder. Both admixitures caused an increase in the hydration peak of a cement binder. 1% admixture of hydrophilic nanosilica accelerated its occurrence comparing to the control binder result. Simultaneously, similar 1% admixture of hydrophobic nanosilica retarded its occurrence. Those observations might be attributed to different reaction mechanisms of both types of nanosilica with its hydrophilic type having higher reactivity with cement binder.
3.) Hydrophilic and hydrophobic nanosilica influenced early strength of cement paste by increasing its value. However, specimens with hydrophilic nanosilica admixture had higher 28-day compressive strength results than the specimens with hydrophobic nanosilica admixture. It could be attributed to higher reactivity of hydrophilic nanosilica.
4.) Both nanoadmixtures had little influence on bulk density and water satuartion porosity of cement paste.
5.) Application of the proposed mixing methods of cement binders with nanosilica admixture showed that utilization of higher rotation speed does not guarantee better mixing of the binder compounds. The time of binder homogenization was more important. Lower rotation speed and longer mixing time in the high speed mixer resulted in higher compressive strength results of studied binders with nanoadmixtures.

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LIST OF FIGURES AND TABLES:

SPIS RYSUNKÓW I TABEL:

WPŁYW RÓŻNYCH RODZAJÓW NANOKRZEMIONKI NA ROZWÓJ WYTRZYMAŁOŚCI KOMPOZYTÓW O MATRYCY CEMENTOWEJ

Słowa kluczowe: cement, nanokrzemionka, domieszka, nanocząsteczki, zaczyn

PODSUMOWANIE:

Osiągnięcia nanotechnologii znalazły zastosowanie w szeregu gałęzi przemysłu. Wraz z rozwojem instrumentów naukowych stają się one również możliwe do wdrożenia w branży budowalnej, a zastosowanie nanodrobin w formie domieszek do betonu jest przykładem tego typu prób. W tym kontekście duże zainteresowanie wzbudza nanokrzemionka. Jest to materiał składający się z nanodrobin amorficznej krzemionki o dużej powierzchni właściwej i dużej czystości chemicznej. Dzięki intensywnej reakcji pucolanowej oraz zarodkowaniu reakcji hydratacji cementu sprzyja wytworzeniu zwartej struktury matrycy cementowej, co może przełożyć się na bardzo dobre parametry mechaniczne i wydłużoną trwałość kompozytu cementowego. Problemem przy stosowaniu nanodomieszek jest zapewnienie ich jednorodnego rozprowadzenia w mieszance i stwardniałym kompozycie. W celu znalezienia satysfakcjonującego rozwiązania tego problemu badane są różne rodzaje nanokrzemionki oraz różne procedury jej mieszania ze składnikami mieszanki.

W artykule przedstawiono wyniki badań zaczynów i spoiw cementowych z domieszką nanokrzemionek hydrofilowej i hydrofobowej. Celem badań była ocena wpływu rodzaju nanokrzemionki oraz sposobu mieszania składników na rozwój wytrzymałości na ściskanie, porowatość kapilarną i gęstość zaczynów oraz rozwój ciepła hydratacji spoiw cementowych. Zawartość domieszek w spoiwach wynosiła 0%, 1% lub 2% masy spoiwa. Składniki spoiw cementowych zostały wymieszane w mieszarce wysokoobrotowej w celu zapewnienia możliwie jednorodnego rozprowadzenia nanodomieszek w spoiwie przed dodaniem spoiwa do wody zarobowej. Wykonano 10 serii spoiw: 5 spoiw wymieszanych I metodą mieszania (wykorzystującą dwie prędkości obrotowe mieszarki) oraz identycznych składów 5 spoiw wymieszanych II metodą mieszania (wykorzystującą jedną niższą prędkość urządzenia, ale z dłuższym etapem mieszania).

Domieszki zwiększyły reaktywność spoiw przy czym miały różny wpływ na czas wystąpienia ich maksymalnego efektu cieplnego – domieszka nanokrzemionki hydrofilowej przyspieszała jego wystąpienie, a domieszka nanokrzemionki hydrofobowej opóźniała. Największą wartość maksymalnego efektu cieplnego uzyskało spoiwo z domieszką nanokrzemionki hydrofobowej w ilości 2% masy spoiwa. Sposób mieszania nie miał wpływu na dynamikę emisji ciepła hydratacji spoiw cementowych kontrolnych (bez nanodomieszek).

Oba rodzaje nanokrzemionki zwiększyły wczesną wytrzymałość na ściskanie zaczynów cementowych w porównaniu do próbek serii kontrolnych. Zwiększenie wytrzymałości 28-dniowej w porównaniu do wyników zaczynów kontrolnych było widoczne w przypadku zastosowania nanokrzemionki hydrofilowej. Zaczyny cementowe z 2% domieszką nanokrzemionki hydrofilowej wymieszane II metodą mieszania uzyskały większe wyniki wytrzymałości 28-dniowej od próbek serii kontrolnej.

Nie zaobserwowano dużego wpływu nanodomieszki na porowatość wodną zaczynów i gęstość objętościową zaczynu cementowego.

Różnice wystąpienia największej szybkości wydzielania ciepła spoiw i w rozwoju wytrzymałości na ściskanie zaczynów świadczą o odmiennych mechanizmach działania badanych domieszek. Zastosowanie wyższych prędkości obrotowych nie gwarantuje zapewnienia satysfakcjonującego wymieszania składników spoiwa. W celu zwiększenia wytrzymałości na ściskanie zaczynów istotniejszym okazało się wydłużenie czasu mieszania spoiwa.

Received: 20.02.2020, Revised: 07.07.2020