APPLICATION OF NANOMATERIALS AND NANOTECHNOLOGIES IN CONSTRUCTION

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Modification of portland cement with nanoadditives

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ABSTRACT: Introduction. Portland cement slurries (suspensions) used for well cementing have high water-cement ratios (0.45–0.6). They also contain a minimum amount of inert fillers, must have zero water separation and controlled hardening with a minimum time between the start and end of setting. **Literature review.** Most of the scientific work on the use of nano-additives in binder systems relates to the construction industry. Nanosilicon, nanotitanium, nanocarbonate, nanoclays, carbon nanofibers, etc. were widely used as modifiers of cement systems, which showed an increase in the strength characteristics of the resulting concretes. Literature review showed that there is a very wide range of concentrations of nanoadditives in cement systems from 0.001 to 10.0%. An increase in the strength of cement with high concentrations of additives in a number of publications is explained by a decrease in its capillary porosity due to clogging of the pore space. However, nanoadditives should not play the role of microfillers in the hardened stone. They should work in cement slurry at the stage of cement hydration and cement structure formation at concentrations less than 1.0%. **Results and Discussion.** The paper presents the results of experimental studies of the rheological properties and early strength of stone based on Portland cement with additives (0.01%) of nanocarbonate and nanoiron. The role of nanoadditives is to increase the rate of cement hydration by reducing the activation energy, and accelerating the dissolution of the solid phase in the liquid. Nanoadditives can be a “substrate” on which two-dimensional nuclei of a new phase are formed. The probability of the appearance of two-dimensional nuclei on the substrate is much higher than for the formation of three-dimensional nuclei of a new phase in the bulk of the solution. **Conclusion.** The results show an ambiguous effect of additives on the tested parameters, which indicates the need to optimize the amount of additives. One of the reasons for the ambiguity of the results may be high water-cement ratios, which reduce the likelihood of the formation of “constrained” conditions in cement slurries. At the same time, the effects of accelerated cement hydration are “levelled” and the number of contacts between hydration products is reduced.

KEYWORDS: nanoadditives, cement hydration, structure formation, rheology, compressive strength.

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INTRODUCTION

The quality of cementing materials is one of the main factors affecting the quality of well completion, since the tightness and reliability of the cement sheath depends on the cement stone obtained in the well [1, 2].

The variety of geological and technical conditions for well construction determines the use of a wide range of modifying substances in plugging materials and solutions (hardening accelerators or retarders, plasticizers, fluid loss reducers, gas blockers, expanding and reinforcing substances, etc.) [3–5].

It is very important that additives of nanomaterials can significantly improve the quality of cements.

LITERATURE REVIEW

Features of the use of Portland cement in well cementing are that the applied slurries (suspensions) have high water-cement ratios (0.45–0.6). Oil well cements contain a minimum amount of inert fillers, but the resulting slurries must have zero water separation and controlled hardening with a minimum interval between the start and end of setting.

Among the first works devoted to the use of nano-additives in oil-well cements is the scientific work [6], which presents the results of using nano-iron in an amount of 0.01%, and shows an increase in the strength of the resulting cement stone by 25–35%.

B.A. Suleimanov and E.F. Veliyev [7] studied the effect of particle size distribution of nanosized additives on the quality of cementing. They showed the superiority of nanoadditives SiO_2 (0.3–1.5%) and TiO_2 (0.5–2.0%) in terms of their effect on the strength of the stone compared to increasing the fineness of Portland cement.

The articles [8, 9, 11] should also be noted. In particular, in [8], the use of nanoclay in an amount of 1–3% in cement slurries for casing wells at high temperatures was discussed, and it was shown that the strength increases in compression and improves the rheological properties of the slurry. The article [9] considered the possibility of using a patented nanoadditive to increase the elasticity of the stone. The authors believe that nanoadditives contribute to the creation of long acicular crystalline structures. These structures crosslink, block capillary pores, increase the flexural strength of the cement stone, and also enhance the dynamics of cement hydration. The "modification of the cement hydration process" should take place during the first 48 hours of thickening. This is consistent with the results of a study of expanding cements [10]. This article shows that the processes of hydration of expanding additives must occur during the period of the "elastic" structure of the stone, capable of healing the formed defects.

According to article [11], an increase of the corrosion resistance (acid resistance) of a cement stone containing 1% nanosilica was experimentally proved.

At the same time, nanoadditives have not yet found wide application in well cementing. In our opinion, this is due to the specific conditions of the work. There is also a large uncertainty in the evaluation of the results obtained regarding the quality of the resulting stone in downhole conditions. The technology of using nanoadditives limits their widespread use in production.

It should be noted that the bulk of research work on the use of nano-additives in binder systems relates to the construction industry.

The most complete review of works of the using of nanoadditives in binder systems used for the preparation of concrete is given in reviews [12–14].

Among the nanomaterials used in the modification of cement systems, nanosilicon [15–16], nanotitanium [17], nanoiron [15, 16, 18], nanocarbonate [19–21], nanoclays [8, 13], carbon nanofibers [22] and some others.

At the same time, most authors noted an increase in the strength parameters of the resulting concretes.

While analyzing of works devoted to the using of nanosized additives in cement systems, there is the question about the mechanism of their action and their rational amount.

Some papers present the results of studies using nanoadditives in the amount of 3.0–10.0% [15, 19, 21]. Explaining the improvement of stone properties by reducing its capillary porosity, the authors are actually talking about microadditives that perform the specified role in concrete. It seems to us that nanoadditives should work at the stage of hydration and structure formation of cements, and the amount of additives should be tenths or hundredths of a percent.

The effect of nanoadditives on the hydration kinetics of cements was discussed in [23, 24], in which these additives were considered as seeds for activating the process of cement hydration.

When studying the effect of various nano-additives on cement hydration, it was shown that many of them accelerate cement hydration by 10–30 times due to a decrease in activation energy by approximately 2–3 times. At the same time, a stone made from modified cement at a concentration of (0.01%) after 28 days of hardening had an increase in strength characteristics by 45–65%, depending on the type of additives. The use of SiO_2 as a nanomodifier ensured the completion of the hardening process on the first day due to the crystallochemical structure of the neoplasms [25].

The article [19] describes the acceleration of the hydration process of binders in the presence of additives of nanomaterials. The products of cement hydration settle on nanoparticles due to their high surface energy and begin to grow. In this case, a conglomerate containing nanoparticles as a "core" is formed. The earlier such nuclei form, the sooner they can grow to larger hydration crystals and accelerate the hydration of the cement. Due to the high dispersity, nanoparticles have a very large specific surface area, are highly reactive, and can act as crystallization centers.

To control the kinetics of hydration, the calorimetry method was used, which showed a significant acceleration of the hydration of Portland cement by the addition of nanocalcium carbonate. At the same time, according to the data of thermogravimetric analysis, the amount of $CaCO_3$ decreased with hydration, although no new products were detected by XRF.

The process of hydration of Portland cement consists of two opposite processes – dissolution of the cement substance in a dispersed mixture and crystallization of solid products from a supersaturated solution [26, 27]. The formation and crystallization of hydration products occurs spontaneously and is due to the chaotic movement of molecules in solution, as well as the release of "constrained" conditions [28, 29].

Based on the consideration of articles [28, 29, 30], a possible mechanism of action of nanoadditives on the processes of structure formation of cement mortars seems to be as follows.

The probability of the appearance of a new phase nucleus is described by the Folmer formula:

$$w_i = w_0 \exp\left(-\frac{A_i}{kT}\right),$$

где w_0– pre-exponential factor; A_i– energy of formation of a nucleus of a new phase; k – Boltzmann's constant; T – absolute temperature.

To increase the probability of the emergence of a new phase, ceteris paribus, it is necessary to reduce the energy of formation of nuclei in the volume, which is proportional to the surface area of the nucleus and the specific surface energy of the neoplasm:

$$A_з = \frac{1}{3} S\sigma,$$

где S – surface area of the nucleus; σ – specific surface energy of neoplasm.

The work of nucleation on the substrate is much smaller and equal to $^1/_2$ of the peripheral energy:

$$A_2 = \frac{1}{2} U æ \sim \frac{1}{2} \delta\sigma,$$

где U – nucleus perimeter; δ – thickness of the monomolecular layer; $æ \sim \delta\sigma$ – specific edge energy.

Due to the lower expenditure of energy for the formation of nuclei at the phase boundary, the probability of their formation on the substrate is higher than when they are formed in the volume of the slurry.

The size of a stable nucleus of a new phase is determined by the Thomson-Kelvin equation:

$$r_{кр} = \frac{2M\sigma}{RT\ln\alpha},$$

где $r_{кр}$– stable critical size of the nucleus; M – molar mass; σ – surface tension at the interface; R – universal gas constant; T – temperature; α – degree of supersaturation of the solution.

For the formation and further existence of smaller nuclei on the "substrate", it is necessary to reduce the interfacial energy between them. It will be minimal if the surface of the interfaces is large and its energy unsaturation, as well as if the crystal chemical characteristics of the material that creates these boundaries are identical to the precipitated phase [30].

According to Polak A.F. [29] the measure of crystal chemical congruence between the substrate and the new phase can be the coefficient of physicochemical inhomogeneity:

$$x = \frac{\sigma_2 + \sigma_{1-2} - \sigma_1}{\sigma_2}.$$

If the substrate and neoplasm are completely congruent, i.e. $\sigma_1 = \sigma_2$, then $\sigma_{1-2} = 0$, hence $x = 0$. If no bonds arise between them, then the interfacial energy is calculated as the sum of the interfacial energies of the substrate and the new phase in the contact area, i.e. $\sigma_{1-2} = \sigma_1 + \sigma_2$, and $x = 2$. The work of nucleation directly depends on the coefficient of physicochemical inhomogeneity ($A \sim x$), and even a insignificant decrease in the energy intensity of the process leads to the probability of the appearance of a new phase nucleus by tens and hundreds of thousands of times [29].

When the structure of the cement slurry is gaining strength, the formation of a coagulation structure initially occurs, followed by the intergrowth of crystalline hydrates and the formation of a structure from calcium hydrosilicates [26].

The addition of nanopowders to this system makes it possible to increase the number of "substrates" (crystallization centers) due to the appearance of particles related in structure and composition. This mechanism is observed in the first hours of hardening – from 8 to 24 hours. This is due to the high chemical activity and the mechanism associated with the direct participation of particles in the processes of phase formation of hydrates.

Exceeding of the concentration of nanoadditives in the cementing system can lead to the opposite effect – a slowdown in the rate of hardening and a decrease in strength characteristics. This is due to the fact that with an increase in the specific surface area and, consequently, chemical activity, nanoparticles similar in composition to Portland cement bind water with the creation of sparingly soluble crystalline hydrates, reducing the amount of water for cement hydration [31].

METHODS AND MATERIALS

During the scientific study, measurement methods were used in accordance with ISO-10426-2-2003. In particular, the measurement of the rheology of solutions and compressive strength.

The rheological characteristics were measured at 24, 30 and 60°C on a Fann viscometer model 35SA-SR12. Measurements on a Fann 420ATC ultrasonic strength analyzer were performed with similar temperature conditions and a pressure of 3000 psi.

The compressive strength of the mortar and stone structure was measured using an ultrasonic strength analyzer Fann 420 ATC.

The study was carried out using Portland cement 1-G-CC-1 manufactured by Dyckerhoff LLC with the addition of calcium carbonate and iron nanopowders. The particle size of the carbonate nanopowder is 60–80 nm, the $CaCO_3$ content is ⩾ 98%. The particle size of the iron nanopowder is 50–110 nm, the Fe content is ⩾ 99.8%, the content is 0.01% by weight of dry cement.

The choice of these powders is due to the possibility of participating in the hydration process as hardening initiators. Also, these additives can serve as a substrate for the formation of nuclei of a new phase [28, 29].

The water-cement ratio of the solutions was 0.45. The density of cement mortars was 1.87 g/cm^3. A surfactant and a defoamer were also added to the solution in an amount of 0.01% by weight of dry cement.

RESULTS

With the addition of calcium nanocarbonate, a slight increase in rheological parameters was observed. When nanoiron was added, the parameters were almost identical at different temperature conditions (Table 1).

Graphs of changes in the strength of cement stone, which were obtained on an ultrasonic strength analyzer, are shown in Fig. 1–6 and Table 2.

Table 3 shows the relative change in the strength of cement stone with nano additives relative to cement without additives.

Table 1
Rheological characteristics of cement slurries

№	Additive	Temperature	PV, mPa*s	YP, Па	1-to-10-min gel strength ratio, Pa
1	Without additives	24	18	15.3	7/12
		30	36	11.5	6/7
		60	24	22	7/9
2	Nano $CaCO_3$ 0.01%	24	29.3	11	7/10
		30	27.8	13.9	6/10
		60	27.8	23.9	7/18
3	Nano Fe 0.01%	24	27.8	10.5	8/47
		30	29.3	12.5	5/7
		60	31.5	15.3	7/9

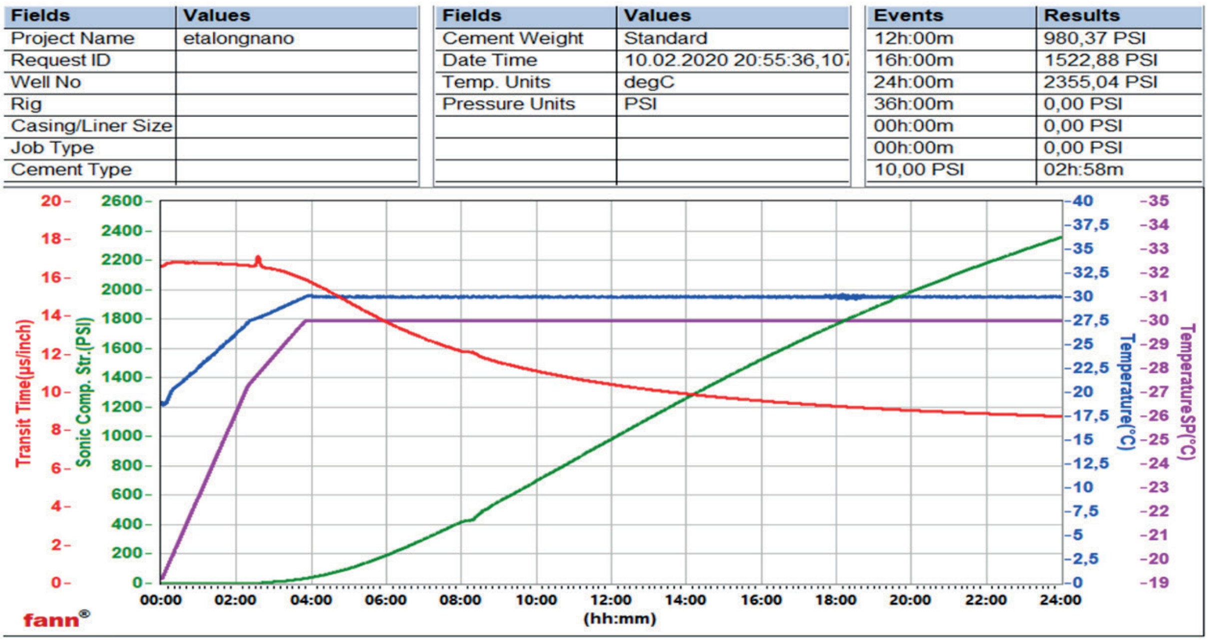
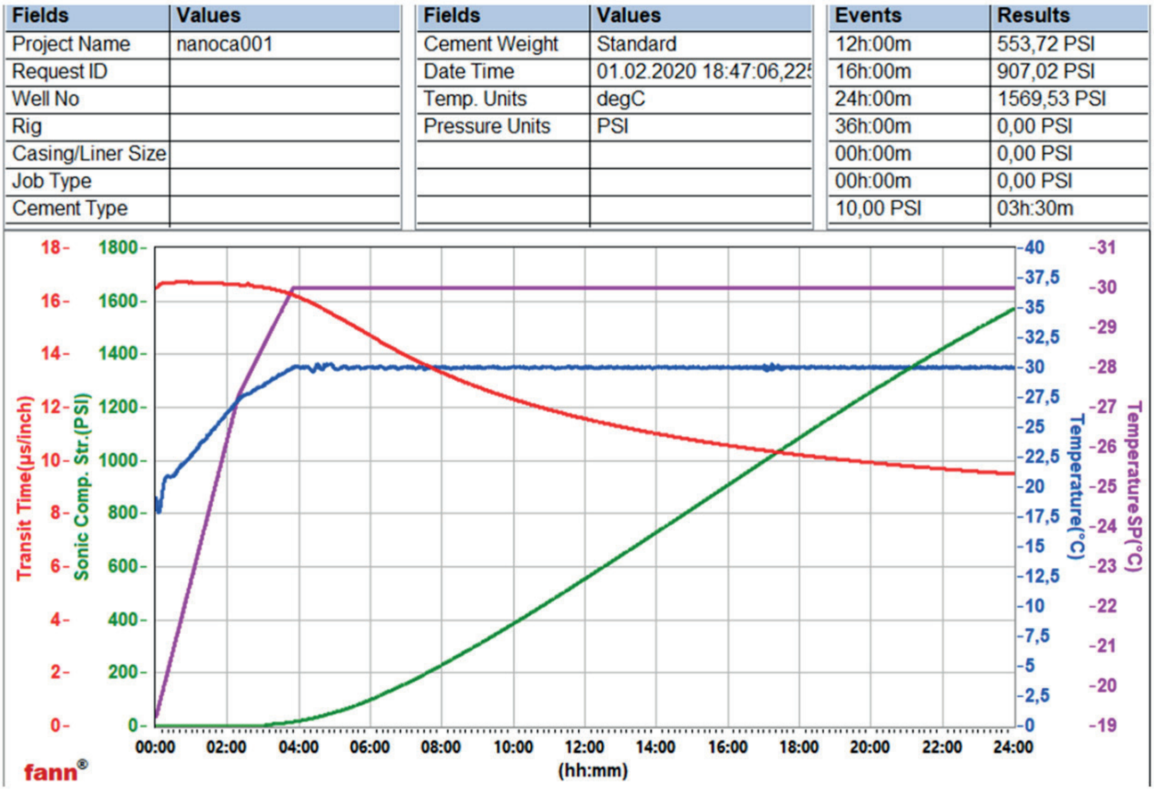


Fig. 1. **Change in compressive strength of stone from additive-free Portland cement at 30°C**

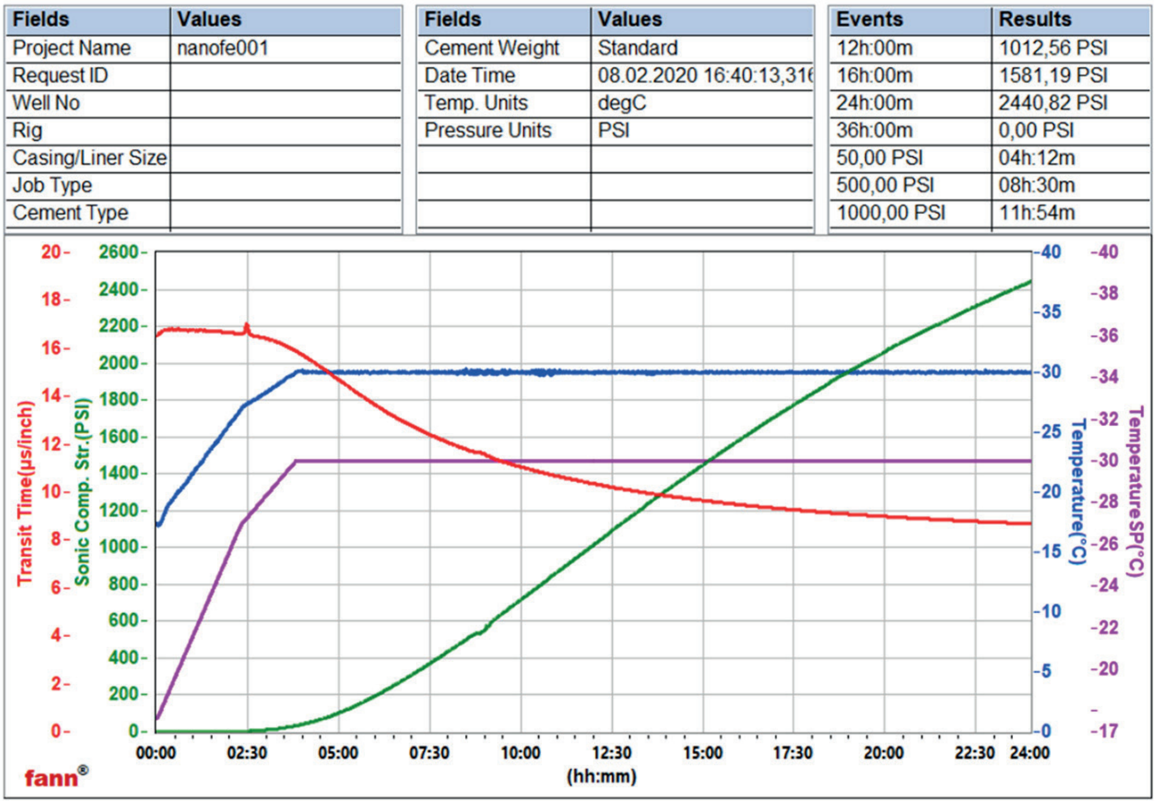


Fields	Values
Project Name	nanoca001
Request ID	
Well No	
Rig	
Casing/Liner Size	
Job Type	
Cement Type	

Fields	Values
Cement Weight	Standard
Date Time	01.02.2020 18:47:06,225
Temp. Units	degC
Pressure Units	PSI

Events	Results
12h:00m	553,72 PSI
16h:00m	907,02 PSI
24h:00m	1569,53 PSI
36h:00m	0,00 PSI
00h:00m	0,00 PSI
00h:00m	0,00 PSI
10,00 PSI	03h:30m

Fig. 2. **Change in compressive strength of Portland cement stone with 0.01% calcium carbonate nanopowder at 30°C**



Fields	Values
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Request ID	
Well No	
Rig	
Casing/Liner Size	
Job Type	
Cement Type	

Fields	Values
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Date Time	08.02.2020 16:40:13,316
Temp. Units	degC
Pressure Units	PSI

Events	Results
12h:00m	1012,56 PSI
16h:00m	1581,19 PSI
24h:00m	2440,82 PSI
36h:00m	0,00 PSI
50,00 PSI	04h:12m
500,00 PSI	08h:30m
1000,00 PSI	11h:54m

Fig. 3. **Change in compressive strength of Portland cement stone with 0.01% nano iron powder at 30°C**

Fields	Values	Fields	Values	Events	Results
Project Name	etalongnano60gr	Cement Weight	Standard	12h:00m	2567,40 PSI
Request ID		Date Time	13.02.2020 20:59:04,71	16h:00m	2958,12 PSI
Well No		Temp. Units	degC	24h:00m	3437,98 PSI
Rig		Pressure Units	PSI	36h:00m	0,00 PSI
Casing/Liner Size				00h:00m	0,00 PSI
Job Type				00h:00m	0,00 PSI
Cement Type				10,00 PSI	01h:47m

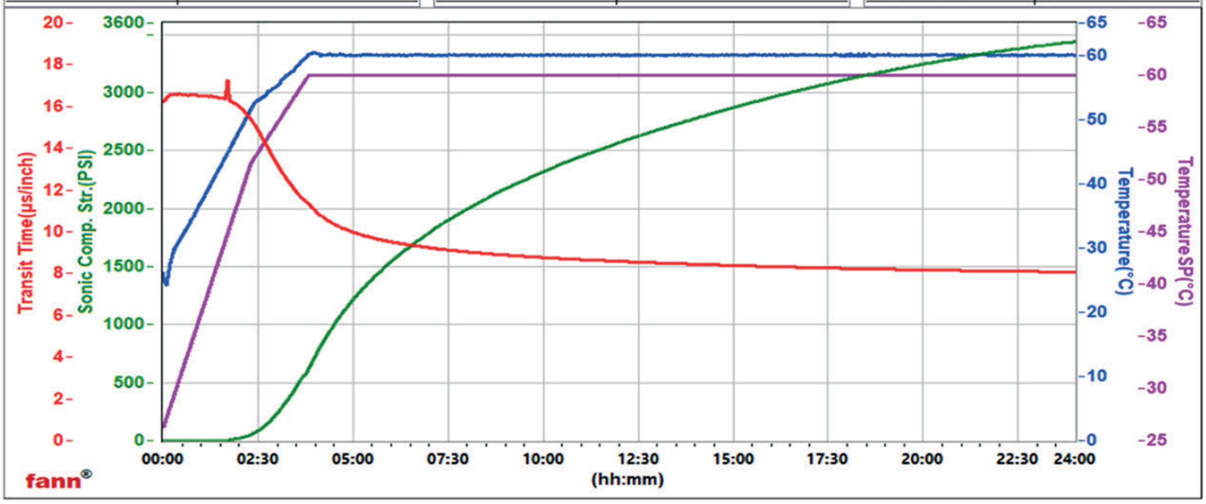


Fig. 4. **Change in compressive strength of stone from additive-free Portland cement at 60°C**

Fields	Values	Fields	Values	Events	Results
Project Name	nanoca001_60gr	Cement Weight	Standard	12h:00m	3181,06 PSI
Request ID		Date Time	02.02.2020 20:37:41,86	16h:00m	3609,46 PSI
Well No		Temp. Units	degC	24h:00m	4100,79 PSI
Rig		Pressure Units	PSI	36h:00m	0,00 PSI
Casing/Liner Size				00h:00m	0,00 PSI
Job Type				00h:00m	0,00 PSI
Cement Type				10,00 PSI	01h:15m

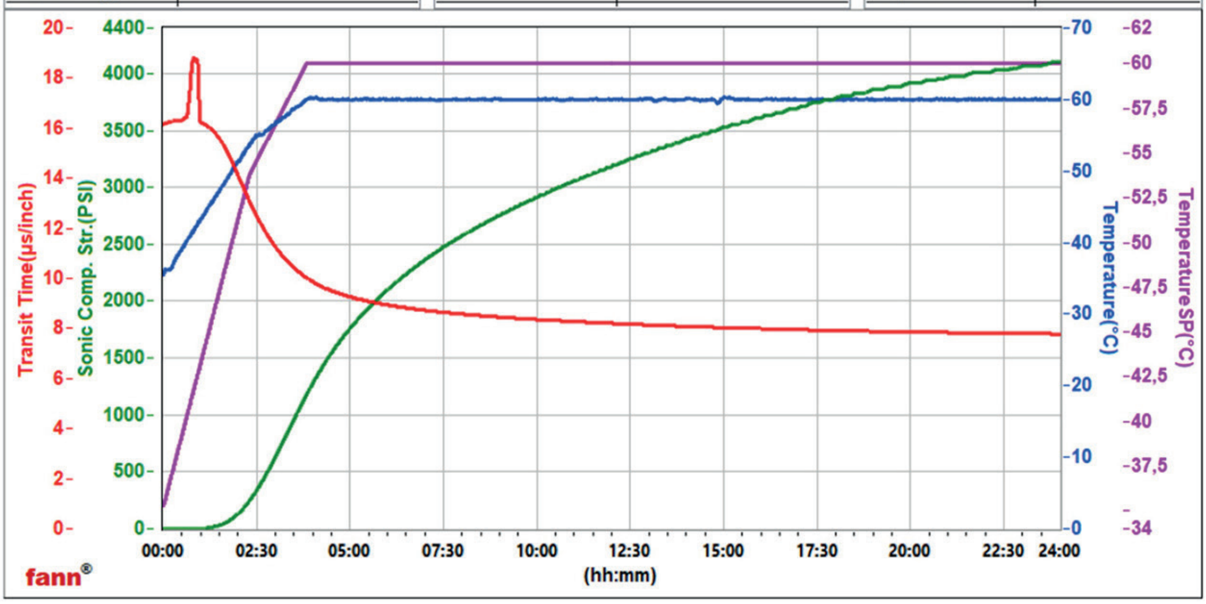
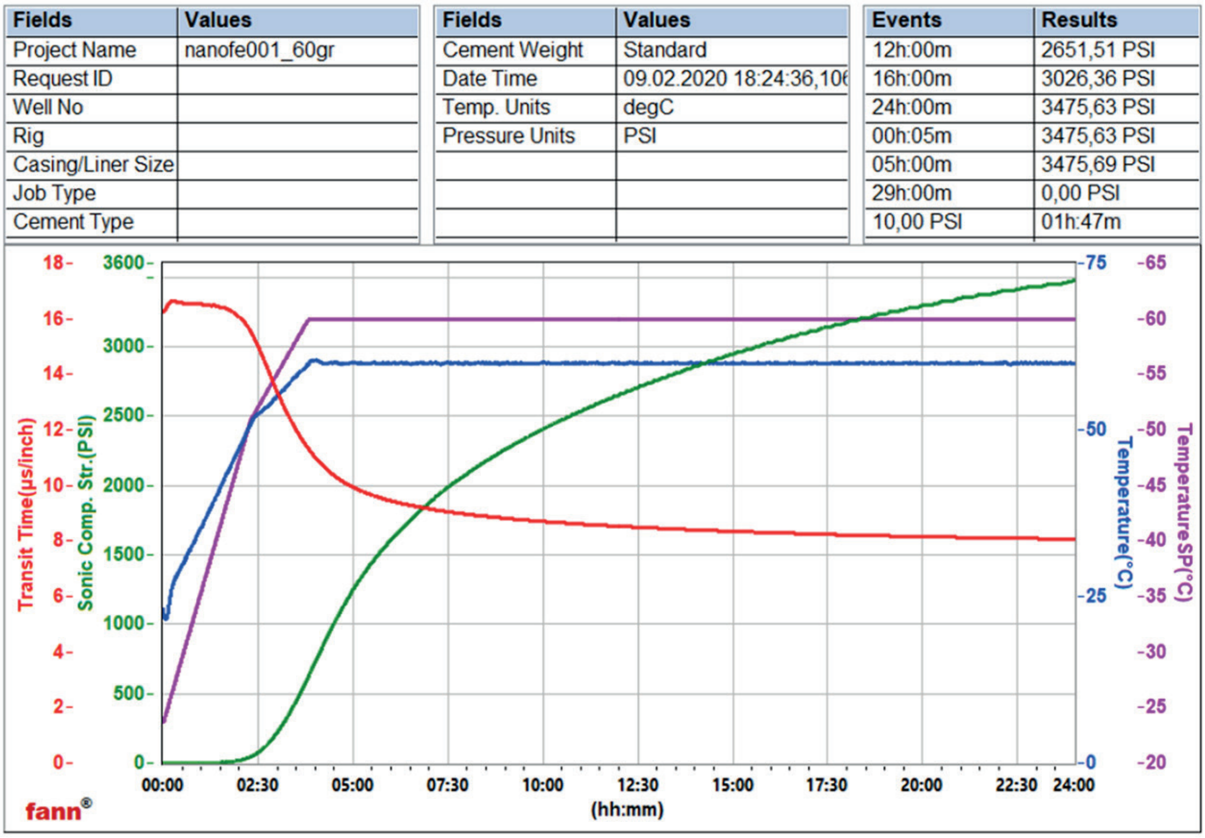


Fig. 5. **Change in compressive strength of Portland cement stone with 0.01% calcium carbonate nanopowder at 60°C**



Fields	Values
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Request ID	
Well No	
Rig	
Casing/Liner Size	
Job Type	
Cement Type	

Fields	Values
Cement Weight	Standard
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Temp. Units	degC
Pressure Units	PSI

Events	Results
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16h:00m	3026,36 PSI
24h:00m	3475,63 PSI
00h:05m	3475,63 PSI
05h:00m	3475,69 PSI
29h:00m	0,00 PSI
10,00 PSI	01h:47m

Fig. 6. **Change in compressive strength of Portland cement stone with 0.01% nano iron powder at 60°C**

Table 2
Strength of cement stone with nano additives

№	Additive	T, °C	Compressive strength, MPa in an hour		
			12 hour	16 hour	24 hour
1	Without additives	30	6.76	10.5	16.24
		60	17.7	20.4	23.7
2	Nano $CaCO_3$ 0.01%	30	3.82	6.25	10.82
		60	21.93	24.89	28.28
3	Nano Fe 0.01%	30	6.98	10.9	16.83
		60	18.28	20.87	23.97

Table 3
Relative change in cement stone strength

№	Additive	T, °C	Compressive strength, MPa in an hour		
			12 hour	16 hour	24 hour
1	Nano $CaCO_3$ 0.01%	30	–43.5	–40.4	–33.3
		60	23.9	22.0	19.3
2	Nano Fe 0.01%	30	3.3	3.8	3.64
		60	3.3	2.3	1.1

ANALYSIS

According to the results of rheological studies, an increase in plastic viscosity with increasing temperature is observed, while the yield point decreases.

The addition of nanocarbonate content in an amount of 0.01% had a negative effect on the increase in compressive strength at 30°C (–33.35% after 24 hours), however, at a given temperature up to 60°C, the increase in strength was positive (+19, 28% after 24 hours). Similar results have been observed by other researchers. A possible reason is the formation of calcium hydrocarboaluminate, which adversely affected the strength in the initial period of hardening.

On the contrary, the addition of iron nanopowder in the amount of 0.01% shows a slight positive increase in strength both at 30 and 60°C (3.64% and 1.1%, respectively, in 24 hours).

CONCLUSIONS

1. It is efficient to provide experiments for optimization of cement with nanoadditives.

2. After identifying the optimal concentration (which shows the most level of increase in compressive strength) of nanoadditives it is necessary to conduct testing to clarify the sedimentation stability of the solution.

3. Changes in the rheological properties of cement slurries modified with nanoadditives are insignificant compared to the reference slurry.

4. The dynamics of changes in the strength of the cement slurry with the addition of iron nanopowder at 30 and 60°C is insignificant. Therefore, this additive does not participate in the hydration process and can only affect the reduction in the porosity of the cement stone, filling the voids in the crystal lattice, making it more uniform and dense.

5. The decrease in the strength of cement stone with the addition of calcium carbonate nanopowder at 30°C is explained by insufficient energy for activation. At a temperature of 60°C, the dynamics is positive, which indicates that an increase in temperature increases the probability of the emergence of a new phase nucleus. In this case, the formation of nuclei occurs on “substrates” related to hydration products.

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