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Analysis on Effect of Fullerene Soot on the Chemical and Physical Properties of Cement Mixtures

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ABSTRACT

This article reports on the relevance and the necessity of the introduction of various additives in the composition of grouting mixtures. The analysis of cement compositions with various modifications of carbon is briefly outlined. A method for obtaining fullerene soot and an analysis of its effect on the chemical and physical properties of grouting compositions made of alumina cement is presented. The physical, mechanical and operational properties of the modified grouting mixture are considered. The optimal content of carbon nanoparticles in the binder is 0.1-0.5% by weight of cement (BWOC). The introduction of fullerene soot makes it possible to obtain high mechanical properties of cement stone (an increase in uniaxial compression strength by about 15% and a decrease in porosity by about 20%) in comparison with cement mix without additives. It has been determined that use of carbon materials is environmentally friendly. Addition of fullerene soot to cement system does not affect the cement hydration processes, which provides micro-reinforcement of the cement stone, and prevents the propagation of cracks in it at nanoscale.

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1. INTRODUCTION[1]

Nowadays, new approaches of solving the problem of technologically successful well cementing are being studied all over the world. During the construction of wells for various purposes (geological, oil and gas, hydrogeological, geotechnical, technical, etc.), special attention is paid to the quality of the walls of the mine operation. Lack of wellbore cementing leads to migration of formation fluids and gases through the annulus, that do not correspond requirements, standards and safety rules of facilities' operation.

To meet all the necessary technological requirements, various additives are introduced into the grouting mixtures that satisfy the drilling conditions [1-4]. The introduction of additives into plugging systems allows to increase the quality of reservoir isolation [5-6], to solve the problem of cracks [7-10] at various aggressive temperatures and pressure conditions [11-13], to increase the reliability and durability of rock separation [14-16]. In recent decades, a large number of scientists have tried to improve the properties of cement-based materials using various nanomaterials due to their excellent chemical, physical and mechanical properties [17-19]. The influence of a sufficiently small amount of 0.5% BWOC nanosynthetic graphite on grouting slurries was determined [20]. Another modification of graphite, graphite nanoplates, was used [21-22] in order to improve the physicomechanical characteristics and nanoscale reinforcement of cement stone. As reported by Alkhamis and Imqam [23], the presence of graphene nanoplates significantly improved the chemical and physical properties of the cement composition and the long-term reliability of oil wells. Al-Awami [24] carried out the

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effect of carbon nanotube on the rheological and physical properties of cement slurry under high pressure. Rahman et al. [25], Khan et al. [26] showed the properties of cement modified with multi-walled carbon nanotubes under HPHT conditions, and changed its mechanical and thixotropic properties. Mendoza et al. [27] studied the effect of multi-walled carbon nanotubes up to 0.50% BWOC on the hydration of cement paste at different temperatures. Smirnyagina et al. [28] presented the results of thermodynamic modeling in the cement-water-carbon nanomodifier system. Wang and Song [29] reported that ash-cement pastes have a higher adsorption capacity and the degree of adsorption increases with an increase in the degree of ash substitution. Termkhajornkit et al. [30] investigated the self-healing ability of ash cement stone. The influence of different amounts of fly ash replacing cement on the compressive strength of cement stone is presented in literature [31-33]. When replacing 15-50% BWOC with fly ash, the advantages and disadvantages of the resulting mixture were determined.

The analysis of the experimentally obtained results of world research on the introduction of carbon materials into the grouting mixtures showed the ambiguity of their influence on the quality of the cement stone. Despite the lack of research on this topic, it can be assumed that there is a possibility of the influence of carbon materials on the quality of grouting mixtures and cement stones for high-quality well casing.

2. MATERIALS

Fullerene soot (FS) is a product of the first stage in the production of carbon nanomaterials - fullerenes. In the process of erosion of graphite electrodes in an electric arc in an atmosphere of a protective gas, soot is formed with a large number of fulleroid-type clusters of different shapes. It should be especially noted that the graphite used for the production of electrodes has a high spectral purity with a minimum amount of impurities (<< 200 ppm). The total content of fullerenes in soot is 8±2 wt%, of which the ratio of the number of C60 to C70 molecules is 3:1 and less than 2% of higher fullerenes. The main structural units of FS are graphite-like nanocrystallites in the form of defective and distorted bundles of hexagonal carbon networks. FS has a large specific surface (tens and hundreds of m^2/g) and a normal particle size distribution in the range of 10 nm - 150 μm [34-35].

When carrying out experimental studies, alumina cement (AC) with an additive in the form of FS were used as a basis for grouting mortars. The characteristics of the chemical composition of the cement used according to the manufacturer's passport are presented in Table 1.

TABLE 1. The chemical composition of the used AC

Cement	Content of oxides of elements, %				
	Al_2O_3	MgO	SiO_2	Fe_2O_3	CaO
AC	38,10	7,40	2,10	2,80	23,40

3. METHODS

Experimental studies were carried out in St Petersburg Mining University. The structural and chemical parameters of the materials and grouting compositions as well as the physical and mechanical properties of the cement stone were studied in accordance with international standards ISO 10426-1, ISO 10810, ISO/TR 12389, ISO 1332, ISO 29581, BS EN 196, and Standard Operating Procedures of each device. The optimal percentages of the reagents with the mixing water volume and with each others were selected empirically used in the theory of experiment planning, taking into account their compatibility and the main parameters of the resulting grouting mixture and cement stone.

3. 1. Determination of the Phase Composition Powder X-ray diffractometry was performed on a Shimadzu XRD-7000 X-ray diffractometer and was used to determine the phase analysis of carbon materials and crushed cement stones.

3. 2. Determination of the Change in Mass of a Sample Dependent on Temperature Thermogravimetry has been used to measure heat flux and the change in material weight as a function of temperature in a controlled atmosphere. Thermal analyzer SDT Q600, manufactured by TA Instruments, USA, was used for thermal analysis of cement stones. The temperature range of the research was from 0 to 1000 °C.

3. 3. Determination of the Strength of Cement Stone To determine the strength of the cement stone, after preparation and measurement of structural and rheological parameters grouting compounds were poured into special forms 40x40x40 mm in order to harden for 1, 3, 10 days. Compressive strength was calculated as the arithmetic mean of the two largest test results on three specimens. This testing was carried out using a Controls-Pilot 3 semi-automatic compression and flexural testing machine.

3. 4. Determination of Cement Stone Porosity The complex system SkyScan 1173 (X-ray microtomograph) was used to determine the porosity of the hardened cement stone.

4. RESULTS AND DISCUSSION

4. 1. Determination of the Phase Composition Diffraction curves reflecting the high amorphous phase are shown in Figure 1. It was determined that the FS contains crystalline carbon.

Next, a phase analysis of a cement stone made of AC and water without additives was carried out. The main phases of alumina cement are Al_2O_3 (28.21%) CaO (19.49%), TiO_2 (10.69%), SiO_2 (8.44%), Fe_2O_3 (4.74%), MgO (1.17%), presented in Table 2.

It can be noted that during cement hydration, the Al_2O_3 phase decreased by 35%, while the CaO phase decreased by 20%. The presence of the MgO compound also sharply decreased (by about 7 times), while Fe_2O and SiO_2 increased by about 1.7 and 4 times, respectively.

With the introduction of FS and the process of hydration, an increase in the content of the Al_2O_3 and CaO phases is observed on average by 10-15%, as well as an insignificant increase in the content of MgO, Fe_2O, SiO_2 compounds (by about 3-12%).

The main crystalline phases of alumina cement are $CaTiO_3$, $SiO_2.xH_2O$ and $KAlSi_2O_6$. It can be noted that the introduction of FS did not affect the elementary composition of the cement stone.
Diffraction curves reflecting the intensity of the peak (CaTiO3) - the phase composition of the studied cement powders - are shown in Figures 2 and 3. The low intensity of the peaks as a result of X-ray amorphous analysis indicates the amorphous state of the cement stone. Consequently, there is a large error in determining the composition of the cement stone.

4. 2. Determination of the Change in Mass of a Sample Depending on Temperature DTG curves - the dependences of the change in the mass of grouting compositions on the temperature of two samples of cement stone without additives and composition with FS are shown in Figures 4 and 5.

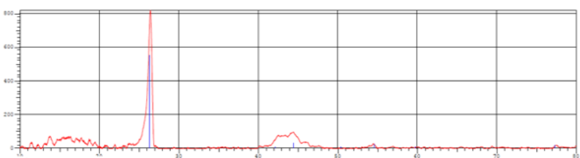


Figure 1. X-ray diffraction pattern of fullerene soot, where the peak is crystalline carbon C (graphite)

TABLE 2. Phase chemical composition of the studied samples of cement stone without and with the addition of FS

Chemical phase	Al_2O_3	CaO	TiO_2	SiO_2	Fe_2O_3	MgO
AC	28.21	19.49	10.69	8.44	4.74	1.17
AC + FS	31.35	22.35	5.11	9.39	4.92	1.28

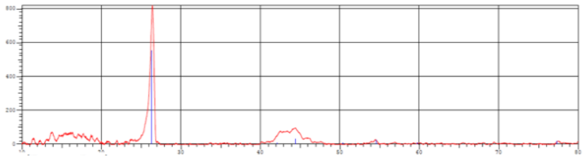


Figure 2. X-ray diffraction pattern of AC without additive, the peaks are $CaTiO_3$ phase

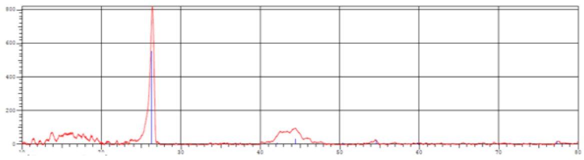


Figure 3. X-ray diffraction pattern of AC with the addition of FS, the peaks are $CaTiO_3$ phase

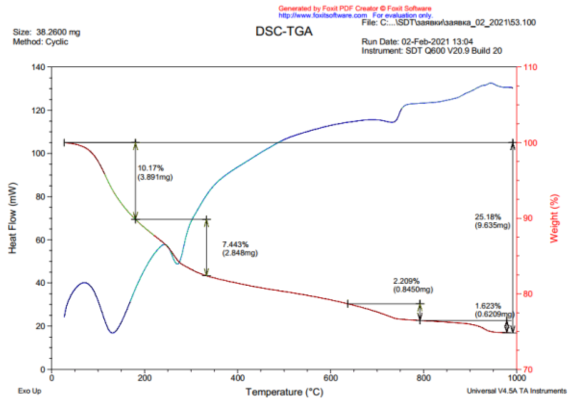


Figure 4. Thermograms of cement sample composition without additives

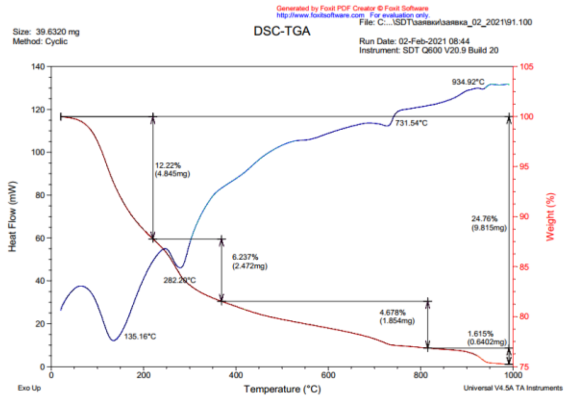


Figure 5. Thermograms of cement sample composition with FS content

Thermal analysis of AC without additives showed losses of H_2O - 5.99% and CO_2 - 19.03%. With the introduction of fullerene soot, a slight decrease in the mass of the cement stone sample is observed with the following values: H_2O - 5.04% and CO_2 - 19.70%. Thus, it can be noted that the water content of the modified grouting mixture decreased, and the release of CO_2 increased.

Thermal analysis showed approximately the same curves. It was determined that FS does not affect the hydration process. Thus, there is a partial compaction of the cement structure and the porosity and permeability of the cement stone decreases. A gradual decrease in the mass of the sample is associated with the loss of water (at a temperature of 85-105 °C), and then hydrates, hydroxides, and after 750 °C - the decrystallization of the cement structure.

4. 3. Determination of the Strength of Cement Stone The obtained results for the modification of AC with FS in an amount of 0.1-0.5% of the cement mass showed an increase in uniaxial compressive strength by about 15-25% compared to a solution without additives. Moreover, the most rates of strength are in the initial period of hardening. The most optimal amount of FS was 0.1-0.5% BWOC. With a decrease in the FS content, a deterioration in the strength characteristics of the grouting compounds is observed in the first week of hardening.

According to the results of the uniaxial compression strength of the cement stone samples, the most durable is the composition number 3 - AC with the addition of 0.3% BWOC FS. This sample showed the maximum increase in uniaxial compressive strength, and it also meets all the technological conditions for industrial implementation. The results of the study of strength are presented in Table 3.

4. 4. Determination of Cement Stone Porosity The next step of examining of hard-set cement was a computer micro tomography analysis to evaluate the pore structure and permeability of the pure cement stone (1) and sample with the addition FS 0.1% (2), 0.3% (3), 0.5% (4). The introduction of FS decreases total porosity of up to 30% depending on the amount of modifier. These results make it possible to believe in the reduced permeability of the cement stone and durability during operation.

The results of main obtained porosity indicators are presented in Table 4, and graphs with an illustration of the computer models of the pore space (pores - green frames) of cement stone samples with different FS contents are presented in Figure 6.

TABLE 3. Results of uniaxial compressive strength of grouting mixtures specimens based on AC

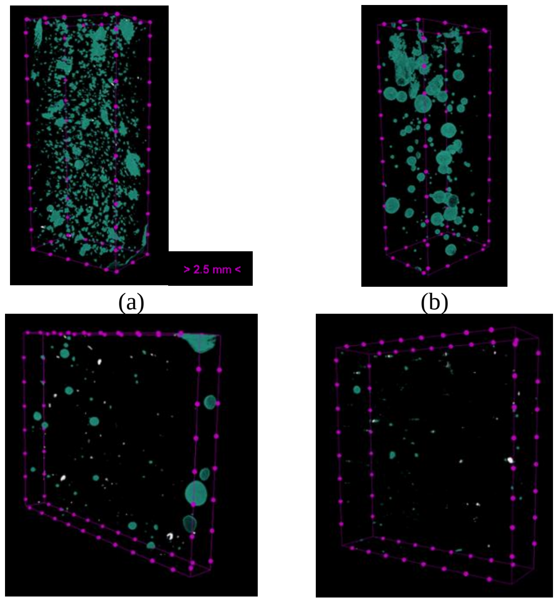
Compression strength	1	2	3	4
After 1 day, MPa	4.86	5.53	6.01	5.88
After 3 days, MPa	7.45	9.62	11.69	10.97
After 10 days, MPa	18.97	18.05	20.64	20.05

TABLE 4. Results of a study of the porosity of grouting solutions samples based on AC

Number of sample	1	2	3	4
Additive	-	**0.1%**	**0.3%**	**0.5%**
Volume of closed pores, mm^3	9.13	9.65	10.50	10.82
Volume of open pore space, mm^3	8.20	6.95	4.39	2.19
Total volume of pore space, mm^3	17.33	16.60	14.90	13.01
Closed porosity, %	2.65	2.70	2.74	2.73
Open porosity, %	1.91	1.80	0.92	0.61
Total porosity, %	4.55	4.50	3.66	3.34

As a result of studies of cement stone on a computer microtomograph, the following data were obtained: with an increase in the FS content, a sharp decrease in the number of open pores occurs with a simultaneous increase in closed pores. In general, the pore volume decreases. There is also a decrease in porosity with an increase in the content of FS in the cement slurry. Moreover, the values of open porosity decrease much more strongly with a fairly stable and small increase in closed porosity.

The filtration resistance of cement stone is inversely proportional to its porosity. Thus, there are no channels for the penetration of filtrate and damage to the integrity and durability of the cement stone.



(a) (b) (c) (d)

Figure 6. Computer model of the pore space of grouting compositions a) composition without additives, b) composition with FS 0.1%, c) composition with FS 0.3%, d) composition with FS 0.5%

Having analyzed all the test results obtained, it is possible to determine the boundaries for the preparation of a grouting slurry from alumina cement, which can be used for cementing the annulus in particularly difficult mining and geological conditions.

According to the results of laboratory tests of the modified grouting slurry, it was determined that the use of AC with tap water does not meet the set objectives of industrial implementation. Fast-setting AC has sufficient strength, but critically high mobility and fluid loss, especially in the early period of thickening of the cement slurry. There is an urgent need to use hardening and plasticizing additives.

5. CONCLUSION

The introduction of carbon materials into the cement mixtures contributes to an increase in strength, but it is necessary to regulate their concentration and consider the joint use of cements with FS. The grouting mixtures obtained according to the given technological parameters, containing FS, satisfy the task, each of them is easy to prepare, contains inexpensive and environmentally friendly additives.

The results obtained for the modification of AC with FS in an amount of 0.1-0.5% BWOC showed an increase in uniaxial compressive strength by about 15-25% compared to solutions without additives. With an insufficient content of FS, a deterioration in the strength characteristics of the cement compositions is observed in the first week of hardening. The increase in the strength of cement stone for uniaxial compression is associated with the mechanism of interaction of cement with FS, which at the molecular level stick cement particles together and create a dense structure during the hardening of cement slurry.

Carbon materials act as additives to increase the mobility of the cement slurry, which affects the successful achievement of the necessary technological requirements. Analysis of the results of computed tomography showed that at the molecular level, the bond between FS and cement improves the permeability characteristics, reducing the number and volume of pores in the cement stone. It was determined that FS does not affect the cement hydration processes. The introduction of carbon additives leads to a decrease in the porosity and permeability of the cement stone without losing its technological properties. The addition of FS provides micro-reinforcement of the cement stone and prevents the propagation of cracks in it at the nanoscale.

6. ACKNOWLEDGMENTS

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Persian Abstract

چکیده

این مقاله ارتباط و ضرورت معرفی افزودنی های مختلف در ترکیب مخلوط های تزریق را گزارش می دهد. تجزیه و تحلیل ترکیبات سیمان با تغییرات مختلف کربن به طور خلاصه شرح داده شده است. روشی برای به دست آوردن دوده فولرن و تجزیه و تحلیل تأثیر آن بر خواص شیمیایی و فیزیکی ترکیبات تزریق از سیمان آلومینا ارائه شده است. خواص فیزیکی ، مکانیکی و عملیاتی مخلوط گروت اصلاح شده در نظر گرفته می شود. محتوای بهینه نانوذرات کربن در چسب ۱/۰ تا ۰/۵ درصد وزن سیمان (BWOC) است. معرفی دوده فولرن امکان دستیابی به خواص مکانیکی سنگ سیمان (افزایش مقاومت فشاری تک محوری حدود ۱۵ درصد و کاهش تخلخل در حدود ۲۰ درصد) در مقایسه با مخلوط سیمان بدون افزودنی را ممکن می سازد. مشخص شده است که استفاده از مواد کربن سازگار با محیط زیست است. افزودن دوده فولرن به سیستم سیمان بر فرآیندهای هیدراتاسیون سیمان که باعث تقویت ریز سنگ سیمان می شود ، تأثیر نمی گذارد و از انتشار ترک های موجود در آن در مقیاس نانو جلوگیری می کند.