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Lightweight cement mortar with inorganic perlite microspheres for equipping oil and gas production wells

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Abstract. In this study lightweight cement mortar was researched. Low-density mortar is used for cementing high temperature, heavily watered oil and gas wells. ISO standard methods, X-ray diffraction analysis and electron microscopy confirm that hollow microspheres of perlite are an effective lightening component for cementing slurry. The results show that admixing microspheres reduces slurry density to 1400 kg/m³. Cement stone with the 3% perlite microspheres has an increased strength of 1.9 MPa due to actual interaction of the cement matrix with the aluminosilicate substance of microspheres, the self-reinforcement of the cement matrix by use of ettringite crystals. Cellular multi-chamber structure of perlite microspheres with the reactive surface enhances water-retention of cementing slurry. Use of the perlite microspheres as a facilitating additive for grouting slurry is preferable in comparison with glass microspheres. Low-density cement mortar with perlite microspheres is recommended a lightweight solution for cementing oil and gas wells.

1. Introduction

In the oil and gas industry in the extraction of minerals, one uses well cementing [1–4]. The process consists in cement slurry injection in annular space on design height and displacement of drilling mud in a casing column (Figure 1). Well cementing aims to isolate productive objects, to reinforce wells, and to separate beds. Poor quality process leads to wrong assessment of oil and gas reserves, their flows to other beds with less pressure, and flooding of productive horizons. Reliable isolation of beds opened during drilling wells simultaneously ensures the protection of mineral resources that is important from environmental point of view and relevant at present.

Up to now, the world practice of well cementing uses the method of two separate plugs proposed by A. A. Perkins, “Perkins Oil Well Cementing Co.,” Calif [5]. As result of the ongoing development of drilling, as well as taking into account well construction in new complicated conditions, it is required both to improve cementing process and the technical means for its implementation and to develop compositions of cement slurries with adjustable setting times [6–10]. For instance, to cement highly watered areas in the wells with high-temperature, it is required a low-density cement system that can reduce hydrostatic pressure in the column with fluid when injecting cement slurry. In this case, standard cement cannot be used because the pressure at the bottom of the well will exceed the gradient pressure.

The increase of well depth leads to the fact that the necessary interval for the separation of beds increases, and cross-section contains a large number of horizons that must be separated. In particular, when drilling out gas and gas condensate fields to reduce the possibility of gas movement, it is required to raise grouting slurry on significant height. This can be done using the following methods:

- use of two-stage cementing with some breakage of the cement ring continuity at a height;
- reverse circulation and its combination;
- use of grouting slurry of reduced density, i.e., reducing the difference between the density of cement and one of drilling mud with simultaneous regulation other properties of grouting slurry.

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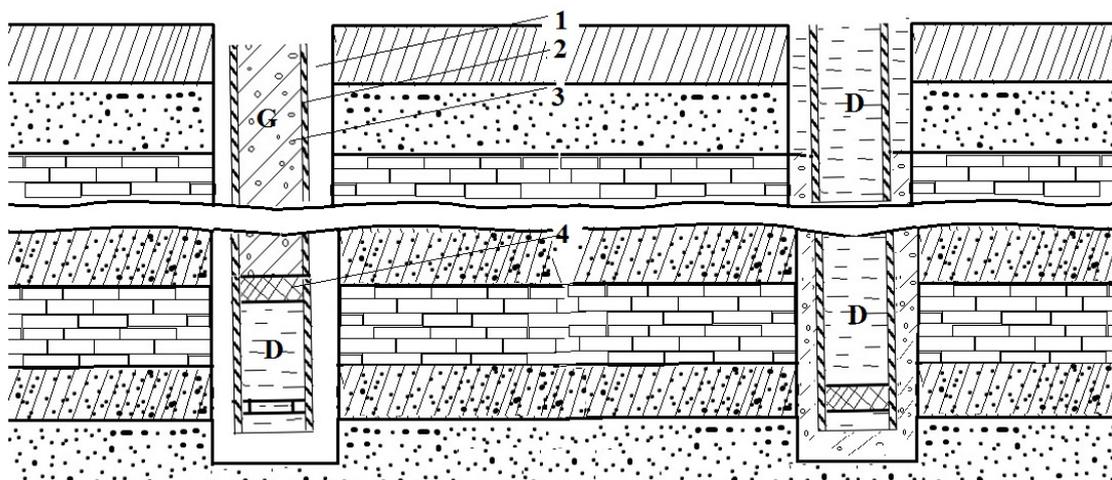


Figure 1. Flow sheet of the process of injection of cement slurry into hole-casing clearance:
1 – hole-casing clearance; 2 – casing column; 3 – cementing slurry; 4 – cement plug;
D – drilling fluid; G – cementing slurry.

Lightweight grouting slurries are widely used in oil production practice. The most well-known additions to reduce cement slurry density are bentonite, perlite, pozzolan materials, diatomite earth, and gilsonite [10, 11]. When using the majority of fillers, reduction of cement slurry density consists in retaining excess water amount with additions, i.e. in an increase of water-cement ratio or introduce of air with filler. In all cases, the introduction of fillers promotes the reduction of mechanical strength of hardened cement paste. At the same time, grouting slurries must have low and stable (regardless of the pressure in the well) apparent density, high uniformity, certain consistency (cement spreadability of 20–25 cm), tensile strength at the bend of at least 1 MPa, and heat-shielding properties in solidified state. Such properties of cement slurries can be obtained by introducing the microspheres that are characterized not only by low density and size but also by high specific strength in bulk compression into their composition [12–24]. As a result, slurries have become practically incompressible in spite of low density, which allows them to be pumped on any depth for one operation with a reverse rise to ground surface. Increased strength and crack resistance of a stone allow excluding repeated insulation work during perforation of a column, and sufficiently strong grip ensures the tightness of annular space [25].

The glass microspheres have several advantages for lightweight grouting compositions. For example, the microspheres 3M™ have ideal spherical shape, apparent density from 0.125 to 0.6 g/cm³, a thermal conductivity of 0.05–0.26 W/(m K) at 0 °C, particle size from 30 to 120 μm, 90 % of undisturbed microspheres with isostatic crushing resistance according to ISO 9001:2000. Due to possessing the complex of properties, the glass microspheres are widely used in different branches. Traditional production of the glass microspheres is based on high-temperature processing of frit. Frit powder is obtained by melting glass of certain chemical composition at temperatures about 1400 °C with following size reduction down to set value. The chemical composition is mainly borosilicate glass [16–18]. The technology relates to processes with high-energy consumption and significant material consumption.

Production of the perlite microspheres from natural raw materials excludes the stage of glass melting, is less energy- and material-consuming and hence cost-effective. Technology is based on thermal swelling of a certain fraction of perlite rock [25]. The microspheres of expanded perlite are successfully used in the composition of lightweight heat-insulating concrete and construction ceramics [26, 27]. There are also known some studies on use of the perlite microspheres as facilitating additions in the composition of grouting slurries for well cementing [12]. However, these studies do not reflect the influence of the perlite microspheres on the formation of structure and basic properties of grouting materials.

Consequently, the aim of the work is to establish the influence of the microspheres of expanded inorganic perlite on the processes of the formation of structure and the basic properties of lightweight grouting slurry.

2. Materials and Experimental method

2.1. Materials

The microspheres. Perlite is a widespread volcanic rock with a high content of glassy phase. The largest reserves of perlite are located in Greece and the United States, the major producers of perlite grit, microspheres and sand currently are Turkey, Hungary, Italy, and Mexico. The main factors affecting the process of swelling perlite rocks are temperature and swelling interval, the content of hard-to-remove moisture in volcanic glass, the chemical composition of the rock and the content of alkalis, and the method and mode of firing.

The perlite hollow microspheres used in this study are obtained in the industrial vertical furnace at a temperature of 1500 °C from the perlite rock of the Khasyn deposit (Russia). The chemical composition in oxides form of initial perlite rock and the microspheres is presented in Table 1. According to the chemical composition, perlite belongs to the group of aluminosilicate glasses, has a high content of SiO₂ – about 75 %, Al₂O₃ – 13 %, and a significant amount of alkaline oxides. The phase composition of perlite microspheres consists of orthoclase. The radiograph of the microspheres contains the amorphous halo of the glass phase (Figure 2).

Table 1 The oxide composition of perlite, microspheres.

Material	Content of oxides (wt. %)							Loss on ignition, (wt. %)
	SiO ₂	Na ₂ O	CaO	Fe ₂ O ₃	K ₂ O	Al ₂ O ₃	MgO	
Initial perlite rock	65.0–75.0	5.0–6.0	0.5–2.0	1.0–3.0	5.0–6.0	10.0–16.0	0.2–3.0	1.0 – 4.5
Microspheres of perlite	74.7	5.3	0.6	1.2	5.1	12.8	0.3	–

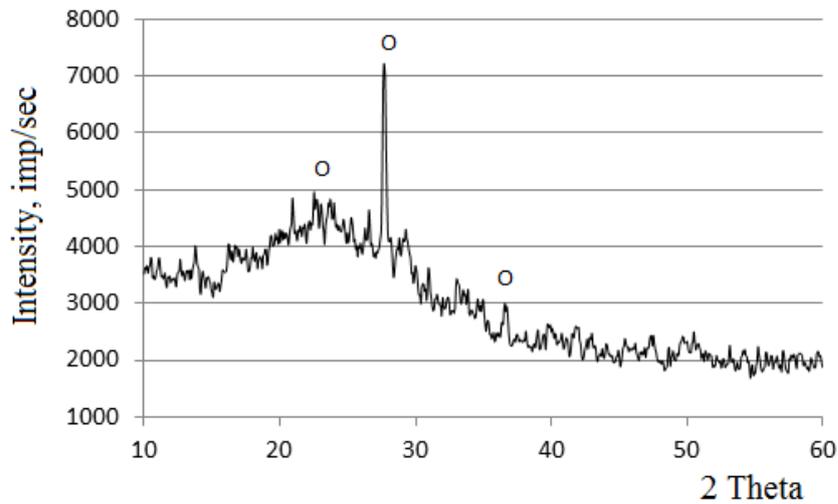


Figure 2. XRD patterns of perlite microspheres: O – orthoclase.

According to electron microscopy, the average size of the perlite microspheres is up to 150 µm with wall thickness of about 2 µm (Figure 3), and the shape is uneven, elongated and asymmetric, unlike the spherical shape of the borosilicate glass microspheres. The photomicrographs show that the perlite microspheres have a multi-chamber cellular structure that must help to increase the strength characteristics of the compositions.

The technical characteristics of the perlite microspheres are given in Table 2. The values of the properties indicate the principal possibility to use the perlite microspheres as facilitating addition in grouting slurries. According to the main indicators, the perlite microspheres correspond to the glass microspheres 3M™ obtained from sodium borosilicate glass.

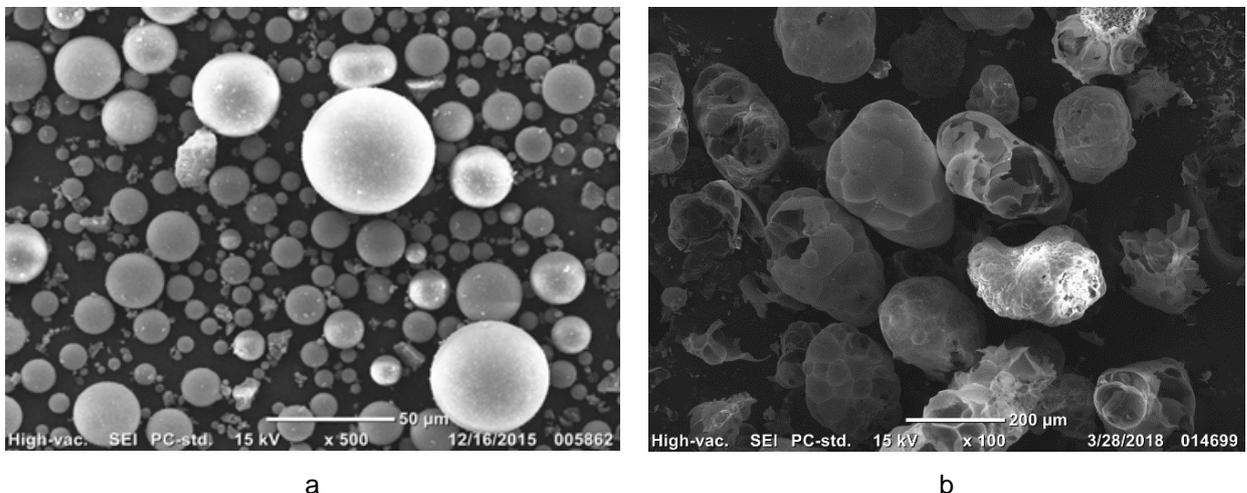


Figure 3. Electronic microscopy of the glass microspheres (a) and the perlite microspheres (b).

Table 2. Physical properties of the perlite microspheres.

Parameters	Indicator values	
	Perlite microspheres	Glass microspheres 3M™ Glass Bubbles HGS
Bulk density, kg/m ³	150	50–280
True density, kg/m ³	2414	150–410
Median particle size, μm	125	30–120
Microsphere wall thickness, μm	2 ± 0.5	0.52–1.29

Grouting cement. As the basis of the grouting composition, we have used grouting addition free cement of the class I-G oil well cement brand, intended for fastening oil and gas wells at moderate temperatures during exploration and production drilling (API Spec 10A/ISO 10426-1 2009). Chemical composition of grouting cement and its technical characteristics are presented in Tables 3 and 4.

Table 3. The main chemical and phase composition class I-G oil well cement.

Content (wt. %)									
SiO ₂	CaO	Fe ₂ O ₃	Al ₂ O ₃	CaO _{free}	MgO	C ₃ S	C ₂ S	C ₃ A	C ₄ AF
20.2–20.7	66.2– 67.0	3.5–4.0	6.0–6.7	1.2	1.4–2.0	58.0–67.0	8.0–15.0	10.0–12.0	10.5–12.5

Table 4. Technical characteristics class I-G oil well cement.

Measured parameters	Values of parameters
Compressive strength test (8 h curing time), MPa	4.5–6.1
Free-fluid content, ml	6.4–8.5
Specific surface, m ² /kg	350–370
Spreadability, mm	200–215
Thickening time, min	180–192
Sulfur trioxide (SO ₃), wt. %	2.8–3.0

2.2. Experimental method

Lightweight grouting mixtures were prepared by mixing components following the regulatory requirements (API Spec 10A/ISO 10426-2 2003). The Amount of the perlite microspheres have been varied from 1 to 3 % by weight.

Properties of grouting slurry. Spreadability, the property of liquid to spread over a solid surface, is an indicator of pumpability of grouting slurry. During injection into the annular space of a well, it is necessary to preserve the mobility of the slurry for a certain time. Spreadability has been determined using the spreadability cone KR-1 (Figure 4). The truncated cone – ring of the device has dimensions: inner diameter of the upper basis is 36 mm, one of the lower bases is 64 mm, height is 60 mm, and volume is 120 cm³.

The density of grouting cement slurry has been determined with lever balances. The measuring range of slurry density are: on the upper scale from 0.8 to 1.5 g/cm³, on the lower scale from 1.6 to 2.6 g/cm³; measurement error ± 0.01 g/cm.

Water segregation of grouting slurry has been determined according to API Spec 10A/ISO 10426-2 2003 «Petroleum and natural gas industries—Cements and materials for well cementing—Part 1: Specification». The cement slurry is poured into two cylinders with a volume of 250 cm³ up to mark of 250 cm³. Settling time is 2 h ± 5 min. The liquid that has been separated from a cement slurry under static conditions on the surface of cement paste is taken with a pipette, and its volume has been measured with the help of a cylinder 20 cm³.

The thickening time of grouting cement slurry has been determined with the atmospheric consistometer OFITE 80 in accordance with API Spec 10A (Figure 5). This property characterizes possibility to pump grouting slurry during a given time interval when well casing. The principle of operation of the consistometer is based on the determination of torque at thickening cement slurry in the body rotating at a given speed. Determination has been performed at atmospheric pressure and speed of rotation of chamber with cement slurry of 150 rpm. The working temperature of the device is up to 93 °C.

Strength tests of the samples of grouting stone at bending have been performed on the samples of 40×40×160 mm using the MATEST machine of model E160 (Figure 6). The maximum load of the device is 500 kN for the compression test and 15 kN for the bending test. The accuracy class is 1. Tests of molded samples have been performed after 2 days of storage in the thermostat at 75 °C.



Figure 4. Grout flow cone KR-1.



Figure 5. The atmospheric pressure consistometer, OFITE 80.



Figure 6. Machine for strength tests, MATEST E160.

3. Results and Discussion

The use of high-quality lightweight grouting materials must solve the problem of one-stage grouting the well. Therefore, such properties as the density of slurry and cement paste and its strength are of particular importance. Hardened cement paste takes a part of loads falling on the column. The increased values of mechanical strength of the stone increase bearing capacity of a casing pipe. In addition to strength characteristics, grouting slurries must have certain values of technological parameters such as spreadability, water segregation, and thickening time.

The basic characteristics of grouting slurries from Portland cement of I-G brand without additions obtained at water-cement ratio (W/C) of 0.5 and 0.8 have been preliminary determined. Results (Table 5) have shown that slurry correspond to normative requirements on all indicators, except one of density and water segregation. At $W/C = 0.5$ slurry density exceeds permissible values, such slurry will have low pumpability. If W/C increases up to 0.8, the density decreases, however, that is connected with violation of sedimentation stability of cement paste and increase of water segregation. As one can see from Table 5 at $W/C = 0.8$, the water segregation is 9.5 that exceeds the permissible values.

The introduction of the perlite microspheres into the composition of grouting slurry allows reducing the slurry density while maintaining the sedimentation stability of cement paste. To this end, the perlite microspheres which its amount varied from 0.5 to 3.0 % by weight have been added to the slurry. The slurries differing in water-cement ratio and amount of the introduced microspheres have been investigated.

Table 5. Values of parameters of grouting slurries without microspheres.

Measured parameters	Values of I-G parameters at W/C ratio		Requirements for lightweight grouting cement at operating temperatures	
	0.5	0.8	low and normal	moderate and higher
Paste density, kg/m^3	1800	1600	1400–1600	1400 – 1600
Spreadability, mm	245	250	not less than 200	not less than 200
Free-fluid content, ml	0.1	9.5	not more than 7.5	not more than 7.5
Thickening time, min	95	180	not less than 90	not less than 90
Strength at the age of 2 days, MPa	4.3	3.7	not less than 0.7	not less than 1.0

It has been found that regardless of the water-cement ratio, the density of grouting slurry decreases with an increase in the amount of the microspheres being introduced into composition due to low density and high porosity of addition itself (bulk density 150 kg/m^3) (Figure 7).

Water segregation of cement slurries is an indicator of filtration properties characterizing their water-holding capacity. In stationary state, grouting slurry is divided into phases of water and cement. Water, rising up, can wash in hardening slurry channels that will not be overgrown in the process of further hardening and are able to pass through hardened cement paste formation fluids. Therefore, to improve properties of cement paste, additional additions that increase the sedimentation stability of grouting mixture are introduced into the composition of grouting slurry. It has been established that at the W/C ratio of 0.5, the water segregation is at zero level and does not change with the introduction of the microspheres into the slurry (Figure 7). At the W/C ratio of 0.8, the water segregation at the content of the microspheres from 0.5 to 1.5 % exceeds permissible values (more than 5.9 ml), while at the content of the microspheres of 2 and 3 % it meets the requirements (API Spec 10A/ ISO 10426-1 2009) (Figure 8).

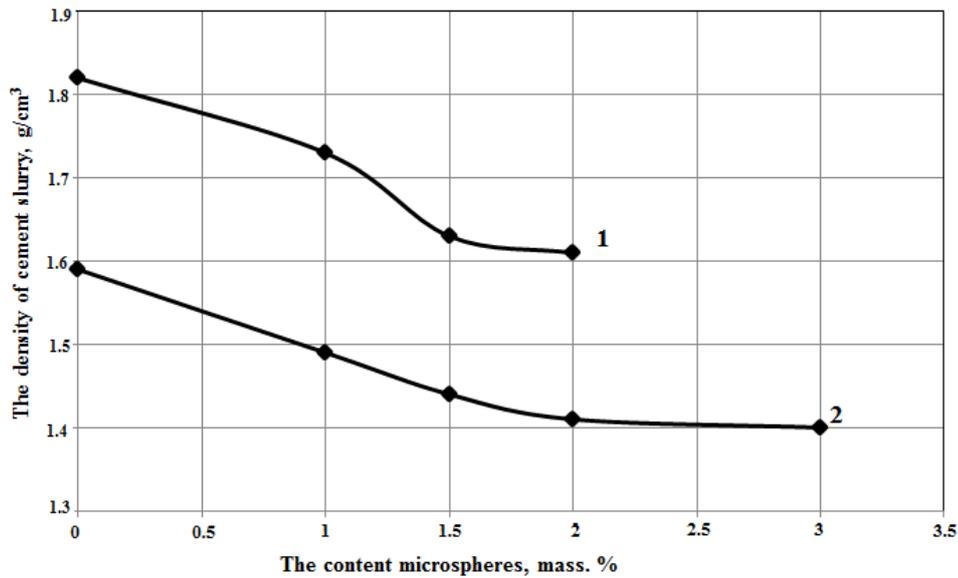


Figure 7. The density of cement slurry with different proportions of content microspheres at a water-cement ratio: 1 – 0.5; 2 – 0.8.

The thickening time of the paste without microspheres is more than 90 minutes. When adding microspheres, the paste obtained with the W/C ratio of 0.5, does not meet these requirements, unlike the W/C ratio = 0.8. Similar dependence is observed for spreadability index (Figure 9), which the value for the paste with the W/C ratio of 0.8 does not change and is at standard level for the cement slurries (250 mm), for the paste with the $W/C = 0.5$ spreadability value decreases sharply with an increase of amount of the microspheres being introduced. As a result, the grouting slurry obtained at the W/C ratio of 0.8 has optimal values of indicators for density, water segregation, and thickening time.

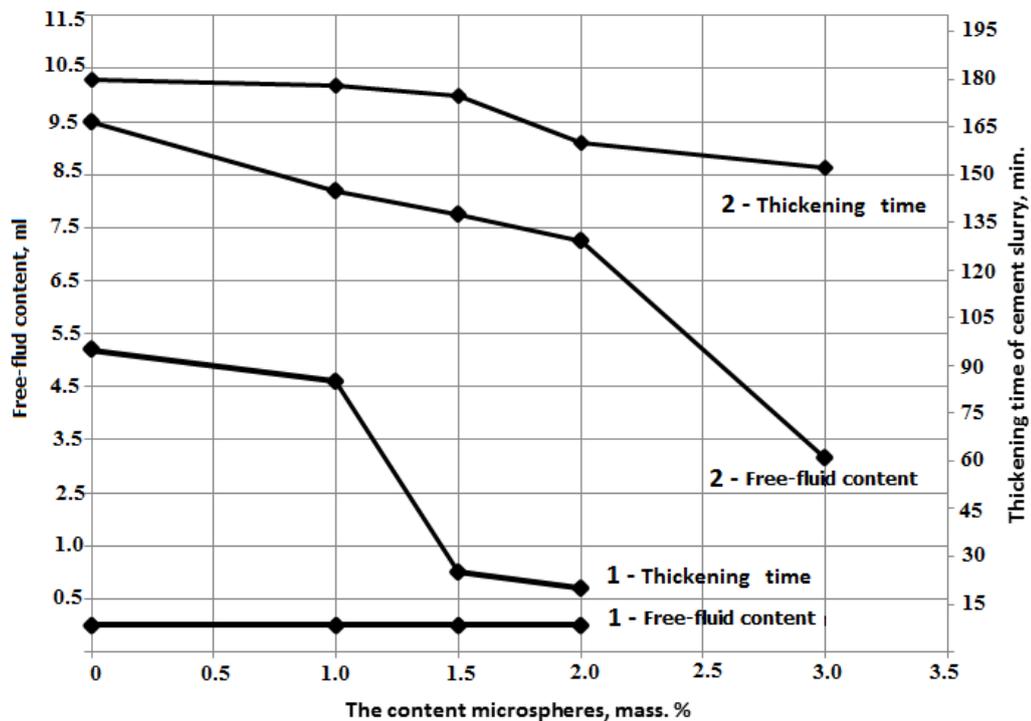


Figure 8. Free-fluid content and thickening time of cement slurry with different content of perlite microspheres at a water-cement ratio: 1 – 0.5; 2 – 0.8.

Regardless of water-cement ratio, the presence of the microspheres in the grouting material reduces its strength parameters however at the lower water-cement ratio of 0.5, the strength of the samples at the age of 2 days is slightly higher in comparison with the strength of the stone obtained at the W/C of 0.8 (Figure 10). This is connected with the following processes:

- Diffusion of CO_2 in pores and capillaries of stone filled with air;

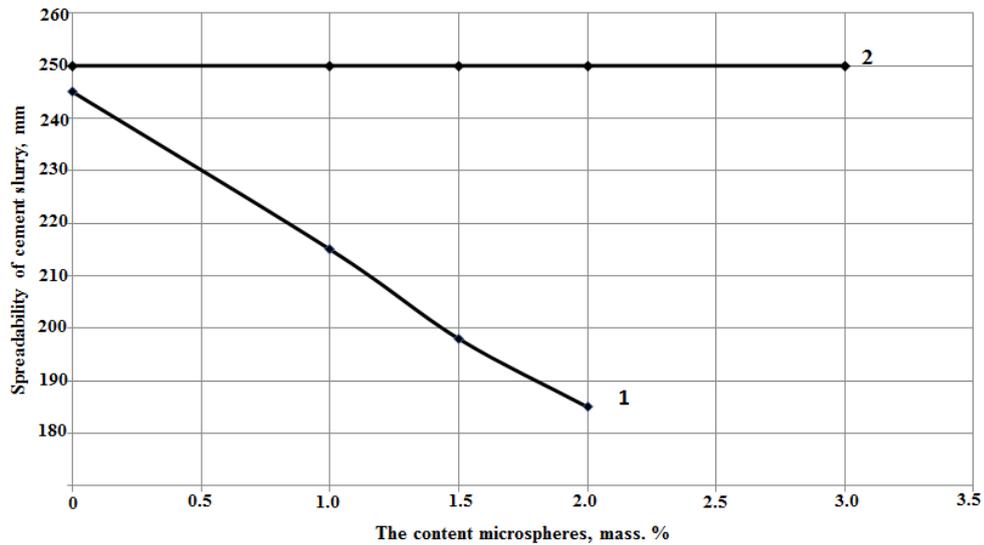


Figure 9. Spreadability of cement slurry with different content of perlite microspheres at a water-cement ratio: 1 – 0.5; 2 – 0.8.

- Dissolution of carbon dioxide in the liquid phase of cement slurry to form carbonic acid and its dissociation into hydrogen ions, bicarbonate and carbonate ions;
- Diffusion of the formed ions in liquid phase;
- Dissolution of calcium oxide hydrate, its dissociation and diffusion of Ca^{2+} and OH^- ions;
- Chemical interaction of carbon dioxide with dissolved calcium oxide hydrate with the formation of bicarbonate and calcium carbonate;
- Crystallization of calcium carbonate.

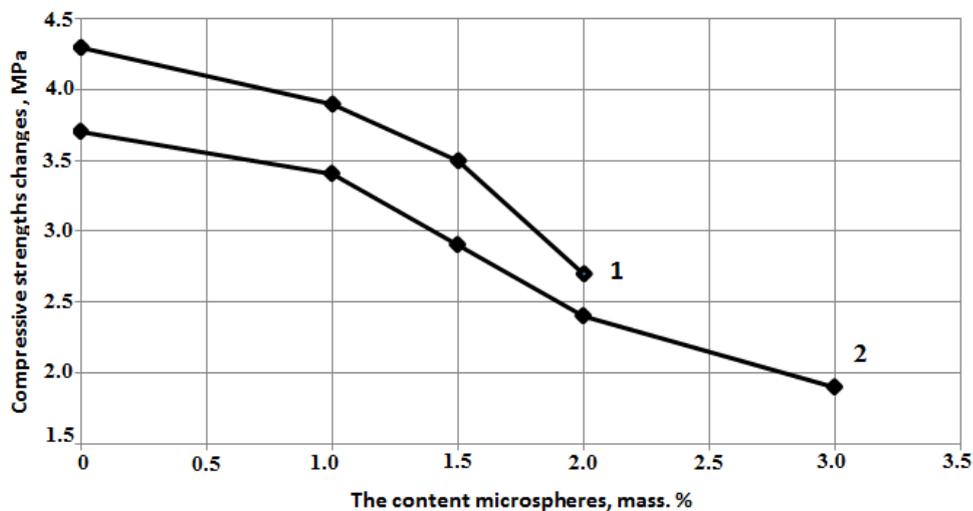
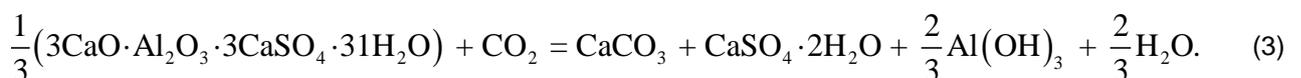
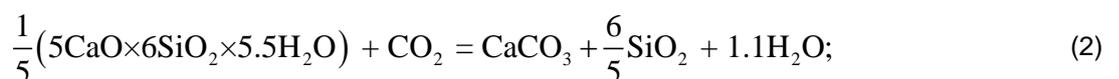


Figure 10. Compressive strengths changes after 2 days hardened cement paste with different content of perlite microspheres at a water-cement ratio: 1 – 0.5; 2 – 0.8.

Interaction of hydrate phases of hardening cement with CO_2 occurs in accordance with the equations 1 to 3:



The x-ray diffraction data have shown the presence of the following crystalline phases on the radiograph: portlandite $\text{Ca}(\text{OH})_2$, calcite CaCO_3 , and ettringite $\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12} \cdot 25\text{H}_2\text{O}$ (Figure 11). The radiograph does not contain distinct reflexes of calcium hydrosilicates because of their weak crystallization that is connected with the condition of hardening of stone (low-temperature hydrothermal processing at 75°C).

The intensity of portlandite reflexes in the hardened cement paste obtained at the $W/C = 0.8$ is higher than that of the stone at the $W/C = 0.5$ which is explained by the introduction of a large amount of water into the system. The increased intensity of calcite reflex in the sample obtained at the $W/C = 0.5$ is probably caused by the fact that the sizes of calcium hydroxide crystals have greater dispersion and, therefore, are more active and better react with CO_2 from the air, forming calcite in large quantities.

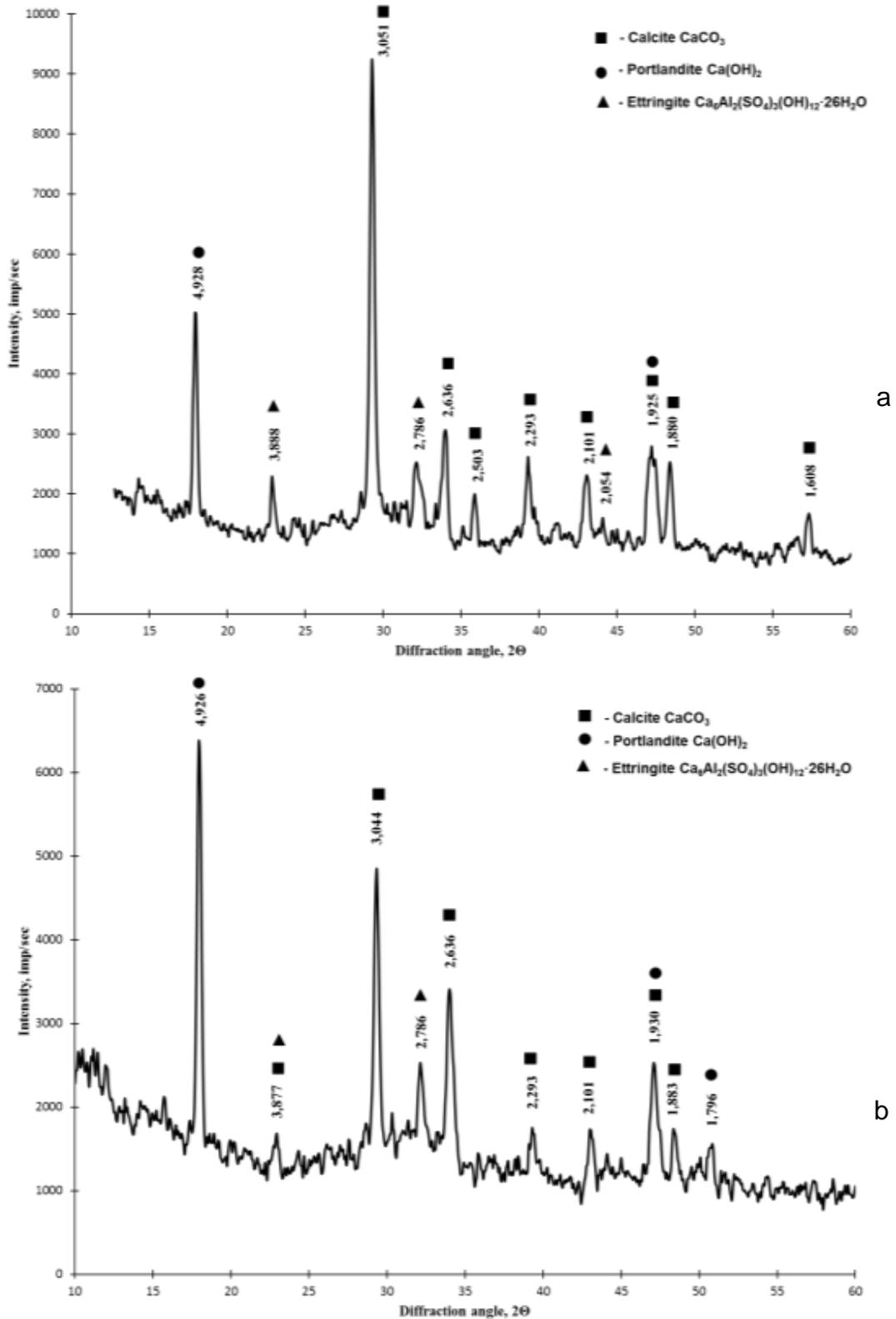


Figure 11. XRD patterns hardened cement paste one microspheres at W/C 0.5 (a), 0.8 (b).

Thus, it has been established that at the W/C ratio = 0.8, the strength decreases with an increase in the amount the microsphere, but corresponds to the requirements, more than 0.7 MPa). The density and water segregation of the slurry at this ratio do not corresponds to the requirements for compositions containing the

microspheres in an amount from 0.5 to 2.0 %. Therefore, the composition with the amount of the microspheres equal to 3.0 %, obtained at the W/C ratio = 0.8, is optimal.

The physical and technical properties of hardened cement paste are determined by the type and amounts of crystalline hydrates being its constituents, size, and shape of crystals, size and amount of pores, degree of cement hydration, and other factors. Comparative analysis of cement paste samples obtained at W/C ratio = 0.8 with the addition of the perlite microspheres in the amount of 1.0 and 3.0 wt. %, in conditions of low-temperature (75 °C) hydrothermal treatment has shown the following. According to the x-ray phase analysis (Figure 12), the radiographs show reflexes corresponding to portlandite $\text{Ca}(\text{OH})_2$ (PDF Number 000-44-1481 d-spacing 4.922; 2.627; 1.795 Å), ettringite $3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot3\text{CaSO}_4\cdot32\text{H}_2\text{O}$ (PDF Number 000-41-1451 d-spacing 9.720; 5.610; 3.873; 2.772 Å), calcite CaCO_3 (PDF Number 000-05-0586 d-spacing 3.035; 2.495; 2.095; 1.973; 1.875 Å). Moreover, the intensity of the basic calcite reflex is higher for the samples with 3.0 % of microspheres that indicates improvement of conditions for carbonization process.

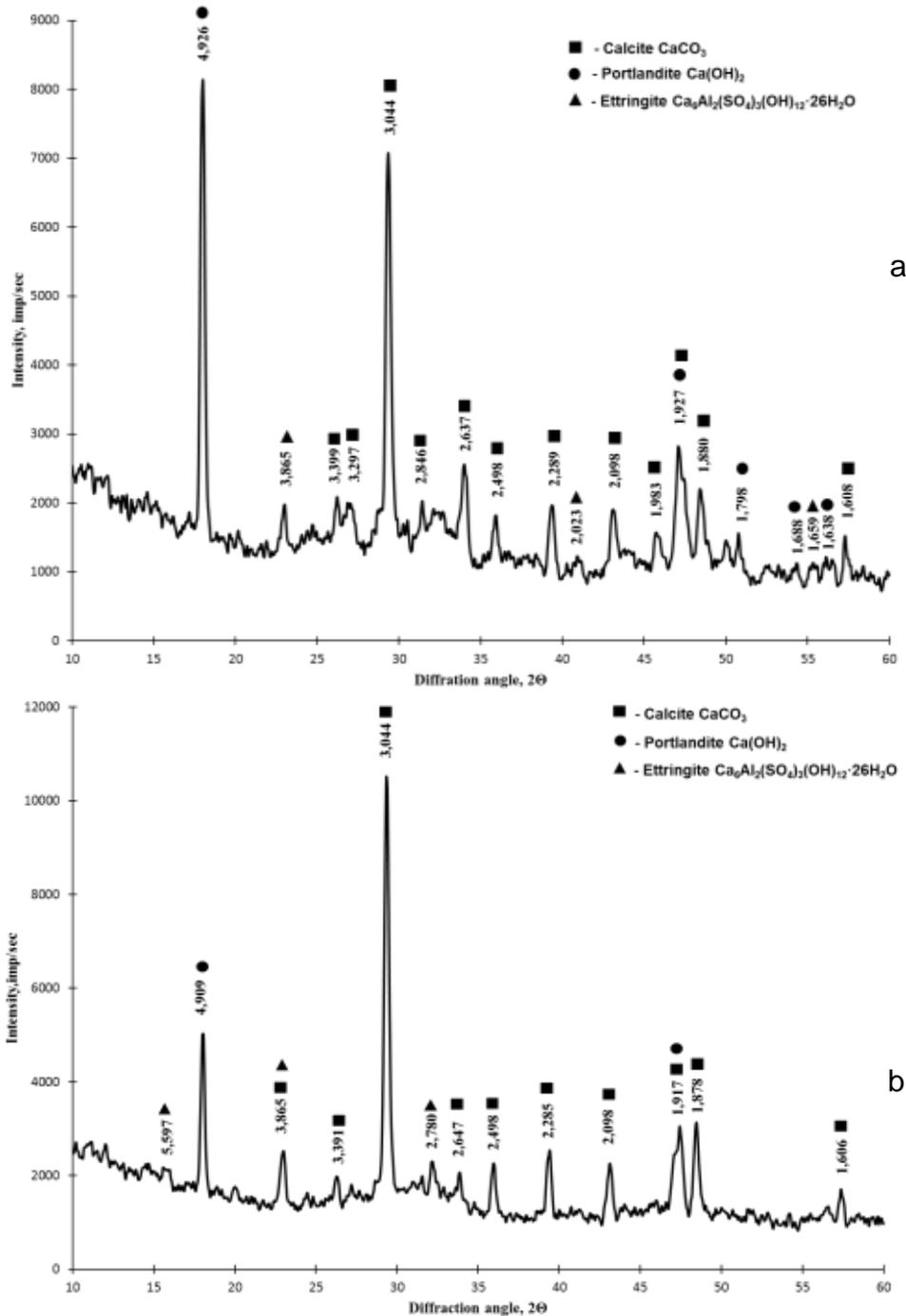


Figure 12. XRD patterns hardened cement paste at W/C 0.8 with perlite microspheres 1.0 % (a) and 3.0 % (b).

According to scanning electron microscopy (Figure 13), the grouting stone obtained with the amount of the microspheres equal to 1.0 % has a dense interfacial transition zone with a surface of the microspheres that indicates strong adhesion interaction of aluminosilicate substance of the microspheres with the products of hydration of cement paste. Cement matrix has a fairly loose porous structure composed mostly by a gel-like poor crystallized formation of calcium hydrosilicates, and hydroaluminates and carbonate formations. The presence of needle crystals of ettringite with a length from 2 to 5 μm is noted that consists with XRF data.

The microphotography of the sample obtained with 3.0 % of microspheres (Figure 14) show that cement slurry moistens well the microsphere at cement – microsphere boundary and interacts with substance of the granules to form products that harden the system as a whole. It can be noted that the surface of the microspheres is covered with highly dispersed fibrous hydrate new formations – the products of interaction of the substance of the microspheres with cement phases. Thus, the microspheres and the smallest fragments of the perlite microspheres can be centers of formation of hardened cement paste structure. The cement matrix itself has a loose structure, porosity and elongated crystals of ettringite with a length of 5–10 μm . Increased porosity is a consequence of air entrainment in the time of the introduction of granular filler. With long-term hardening, more than 2 days, porosity is compensated by the increase in the amount of crystalline phase due to the increase in the number of crystallization centers.

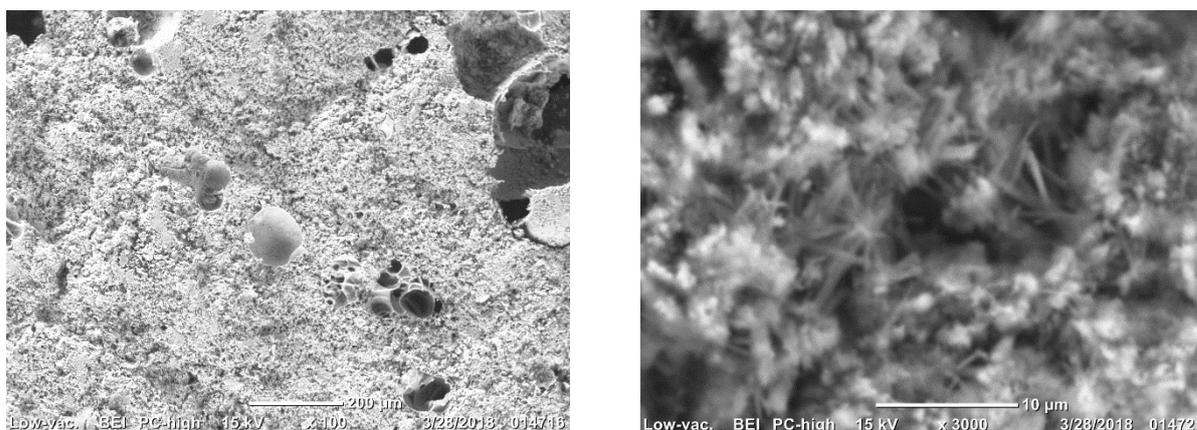


Figure 13. SEM cement slurry with perlite microspheres 1.0 % at W/C 0.8.

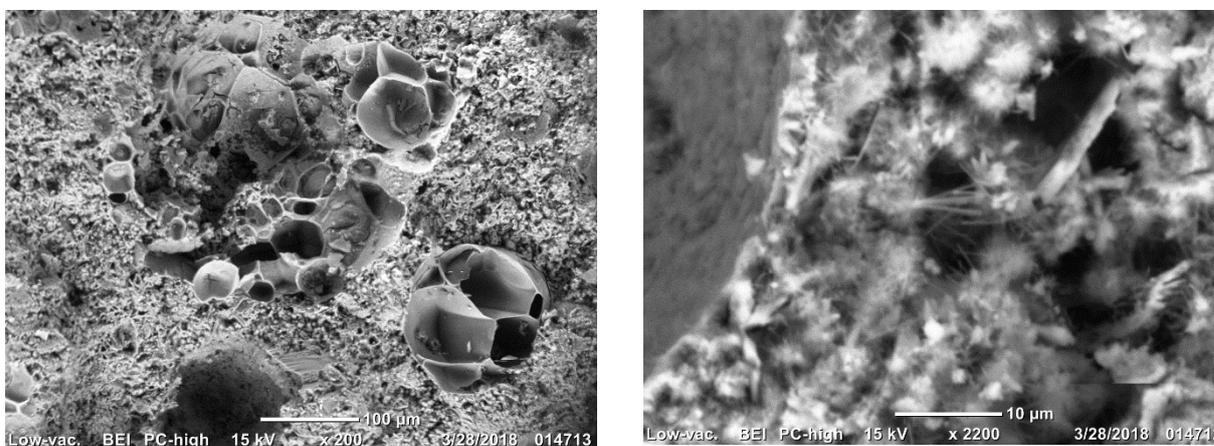


Figure 14. SEM cement slurry with perlite microspheres 3.0 % at W/C 0.8.

The hollow perlite microspheres are effective facilitating addition for grouting slurry which introduction in amount of 3.0 % reduces slurry density from 1600 to 1400 kg/m^3 , at the strength of hardened cement paste of 1.9 MPa, which is by 2.7 times higher than the requirements (not less than 0.7 MPa). The hardened cement paste with the perlite microspheres has increased strength due to both the multi-chamber structure of the microspheres and the formation of hydrated phases of cement paste with the crystallization of needle-shaped ettringite as a self-reinforcing component that is justified by data of electron microscopy and x-ray phase analysis.

4. Conclusions

The perlite microspheres can be used to reduce the density of grouting slurry that has a promising performance on basic properties. Analysis of obtained results has shown the effectiveness of use of the perlite microspheres as a facilitating addition and allowed to draw the following conclusions:

1. Use of the perlite microspheres as a facilitating addition for grouting slurry is preferable in comparison with glass microspheres, since the synthesis of the perlite microspheres excludes additional high-temperature

technological stage of glass melting and use abundant, relatively inexpensive rock for their production in contrast to specially melted borosilicate glass.

2. Developed composition of lightweight grouting slurry consisting of cement of grade I-G (97 %) and the perlite microspheres (3.0 %) at water-cement ratio equal 0.8, provides slurry density equal 1400 kg/m³, water segregation equal 3.5 ml, and thickening time equal 150 min that corresponds to the requirements of API Spec 10A/ ISO 10426-1 2009.

3. Low water segregation, i.e. good water holding capacity at the introduction of the perlite microspheres (3.0 %) in grouting cement mixture is explained by their cellular multi-chamber structure and reactive surface.

4. It is established that cement paste with the perlite microspheres in amount of 3.0 % at the age of 2 days has increased strength of 1.9 MPa in comparison with specified value up to 0.7 MPa that is caused by intensive formation of cement matrix crystals at direct participation of aluminosilicate substance of the microspheres, self-reinforcement of cement matrix with ettringite crystals and formation of durable structural frame from perlite microspheres with multi-chamber structure.

5. Acknowledgments

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Облегченный цементный раствор с неорганическими перлитовыми микросферами для обустройства нефтегазодобывающих скважин

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Аннотация. Объектом данного исследования является облегченный тампонажный раствор. Данный раствор имеет пониженную плотность и используется для цементирования высокотемпературных, сильно обводненных нефтяных и газовых скважин. С использованием стандартных методов ISO, рентгенофазового анализа и электронной микроскопии доказано, что полые микросферы из перлита являются эффективной облегчающей добавкой для тампонажного раствора. Результаты показывают, что введение микросфер уменьшает плотность раствора до 1400 кг/м³. Цементный камень с перлитовыми микросферами в количестве 3 %, имеет повышенную прочность 1,9 МПа. Это обусловлено активным взаимодействием цементной матрицы с алюмосиликатным веществом микросфер, самоармированием цементной матрицы с помощью кристаллами этtringита. Сотовая поликамерная структура перлитовых микросфер с реакционноспособной поверхностью обеспечивает высокую водоудерживающую способность тампонажного раствора. Применение перлитовых микросфер в качестве облегчающей добавки для тампонажного раствора является предпочтительнее по сравнению со стеклянными микросферами. Облегченный раствор с перлитовыми микросферами рекомендуется в качестве легкого раствора для цементирования нефтяных и газовых скважин.

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