

## DEVELOPMENT OF THE COMPOSITION OF LIGHTWEIGHT CEMENT SLURRY USING MICROSPHERES AND MICROSILICA

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### ABSTRACT

It is well known that Portland cement has long been used in the construction of oil and gas wells. In this case, cementing slurries based on Portland cement are placed in the annular space, and after setting, they provide long-term isolation of the formations for subsequent operation. Therefore, the cementing process and the quality of these operations are of significant importance. To minimize of complications due to circulation loss, effective lightweight additives, including microspheres and microsilica, are widely used. However, when using microspheres, they may float when the water-cement ratio increases, and the resulting slurries exhibit high water yield. Microsilica in the cement slurry reduces water yield and does not float, but it thickens the slurry. The aim of this work is to develop lightweight cementing slurries for moderate temperatures with the rational use of the aforementioned additives. The authors of this article have developed a composition of lightweight cementing slurries for various climatic conditions using these additives. It has been specifically demonstrated that by adding 10% microspheres and 10% microsilica, effective lightweight cementing slurries can be obtained, where the various properties of the additives produce a positive outcome. The addition of 0.1% anhydrous sodium silicate to the cement slurry further improves the primary technological parameters of the slurry and, in general, the cement stone. Based on the conducted studies, an increase in the compressive strength of the cement stone to 7.04 MPa with a water-cement ratio of 0.7 was observed.

**Keywords:** cement; microsphere; microsilica; lightweight; slurry.

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### Introduction

The use of Portland cement in well casing, known for over 100 years, is due to its ability to transform from a suspension into a strong, impermeable stone, ensuring long-term sealing of the annular space [1, 2].

At the same time, a serious problem in cementing is the loss of circulation as a result of absorption of cement slurries, which is quite expensive [3]. According to the authors of the work [4], the costs of combating this complication and its consequences in some cases reached up to 2 billion or more dollars per year. In the article [5], devoted to the problem of the quality of well wall cementing and the assessment of the risks of emergency situations due to poor cementing, a methodology for assessing the quality of well wall cementing and risk is given, and based on the results of processing the data of acoustic logging, cementometry and frequencies and the corresponding consequences in the form of qualitative and

quantitative magnitude of cementing, curves of «constant risk» are constructed, allowing to determine the magnitude of risk

Naturally, before cementing casing strings, well preparation is mandatory which various methods must be used to eliminate the absorption that occurred while the well deepening. If it is impossible to eliminate highly permeable layers composed of sandstones, cavernous limestones and fractured dolomites, the other options must be used.

One of these options is to reduce the pressure in the annular space during its squeezing in another case, they use colmatage materials that seal permeable rocks.

The pressure behind the casing, including hydrostatic and hydrodynamic components, can be controlled by the properties of the solution, reducing the height of the cement mortar lift in the annular space, and also by reducing the share of the hydrodynamic component regulating the rheological characteristics of the cement slurries. In particularly difficult conditions, it is advisable to use a combined method that includes both of the above-mentioned technological

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methods [1]. It is worth noting the attempts of researchers aimed at improving the properties of cement mortars. Among the recent works, we note the works devoted to the use of nanotechnology [6-9].

For the fields of the Republic of Kazakhstan, the issue is further complicated by the fact that, during the cementing of gas and gas condensate wells with depths ranging from 2500 to 3000 meters, it is often required to ensure that the cement slurry rises to the wellhead. For example, at the Amangeldy gas condensate field, multi-stage cementing was applied, and full cementing was achieved. The formation pressure of the Upper Tournaisian reservoir at the Amangeldy field is 24.82 MPa, while the Lower Tournaisian reservoir has a formation pressure of 23.7 MPa and a formation temperature of 63 °C. The gas composition of the Upper Tournaisian-Lower Viséan deposits includes 67.25–86.2 % methane, 4.52–10.53 % ethane, 2.86–5.2 % propane, 0.42–0.57 % isobutane, 0.82–0.95 % n-butane, 0.74–1.12 % pentane, 4.6–14 % nitrogen, 0–2.22 % carbon dioxide, and up to 0.19% helium.

This requirement – cementing gas and gas condensate wells up to the surface (i.e., along the entire height of the annular space) – is carried out in accordance with industrial safety regulations and the need to prevent inter-reservoir gas migration (*Safety Regulations in the Oil and Gas Industry*, approved by Order No. 182 of the Minister for Emergency Situations of the Republic of Kazakhstan, dated 12.12.2014, with amendments). Therefore, full cementing to the wellhead is a mandatory requirement to ensure well integrity and to prevent gas migration in the annular space. In such cases, multi-stage cementing and reverse circulation cementing technologies can be used [10].

At the same time, the main direction for ensuring the required height of cement slurry lift in the annular space is the use of lightweight cement mortars that have the following characteristics:

- density within 1500-1600 kg/m<sup>3</sup>;
- low water loss (no more than 100 ml/30 min);
- the strength of the stone is not less than 3.5 MPa in compression and 2 MPa in bending.

In general, obtaining low-density cement slurries can be achieved by only two ways [3]:

- increasing the water-cement ratio;

- reducing the density of the solid phase through the use of lightening additives.

The first path, implemented by increasing the water-cement ratio, leads to high water loss and sedimentation instability of solutions, shrinkage deformations and a decrease in the physical and mechanical properties of the resulting stone.

To reduce the density of the solid phase of cement, and, accordingly, to reduce the density of the resulting cement slurry, clay (gel cement), industrial waste, microsilica (MS), organic additives, microspheres (MS), inorganic perlite MS, ash from coal combustion, etc. are widely used [2, 10, 11].

Gel cement often contains bentonite clay, which has good water-holding capacity. However, a number of authors [2, 12] claim that gel cement solutions are characterized by both the heterogeneity of the resulting mixture, an increase in the setting time of the solutions, and a decrease in the physical and mechanical properties of the stone.

The use of vermiculite, perlite, expanded clay and creogen which have low bulk density, can lead to technological accidents and are often considered unacceptable even in field conditions. As a rule, porous additives with a bulk density of up to 300 kg/m<sup>3</sup> do not withstand pressure during volumetric compression [13].

The use of nano-additives and cements with differentiated properties may be promising for obtaining lightweight cement slurries [14, 15, 16].

Today, several types of microspheres (polymer, aluminosilicate and glass) are used in production [17], the properties of which are given in table 1.

The authors of the work [18] claim that the use of MS is complicated by their destruction by hydrostatic pressure during cementing, high water loss, and a decrease in the strength of the stone. However, some authors believe that cement slurries with hollow MS have low density and are practically incompressible [13, 17].

Another problem with the use of cement slurries with hollow cores is their weak sedimentation instability (fig. 1), which increases with a raise of the water-cement ratio.

The problem is solved by using additives with water-retention properties, such as VINNAPAS, which reduce the segregation of solutions while simultaneously thickening them, requiring the use of plasticizers.

Properties	Values		
	Polymer MS	Aluminosilicate MS	Glass MS
Color	different	from white to light gray	white
Particle shape	spherical	spherical	spherical
Particle size, μm	50-500	40-500	10-200
Bulk density, g/cm <sup>3</sup>	0.10-0.45	0.30-0.45	0.12-0.40
True density, g/cm <sup>3</sup>	0.16-0.42	0.34-0.50	0.16-0.70
Compressive strength, MPa	up to 4.0	up to 5.0	10.0-20.0
Thermal conductivity coefficient, W/(m °C)	0.050	0.06-0.20	0.060-0.067

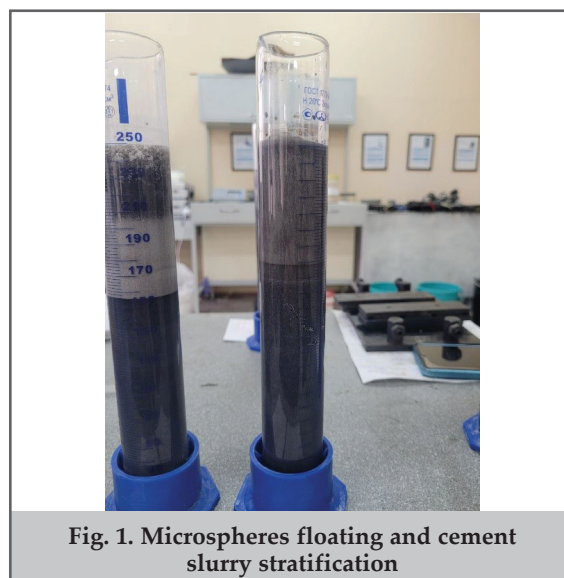


Fig. 1. Microspheres floating and cement slurry stratification

Microsilica is also a lightening additive, the use of which can reduce the density of cement slurry to 1700 kg/m<sup>3</sup> at a water-cement ratio of 0.7.

Its use allows [19]:

- reduce the density of the cement slurry;
- increase the compressive and bending strength relative to stone made without additional cement slurry;
- increase the water-holding capacity of the solution and improve its sedimentation stability.

Experience in cementing a gas hydrate reservoir has shown that the use of microsilica with a granule size of 1000 mesh makes it possible to obtain a cement slurry with a density of 1530 kg/m<sup>3</sup> with an additive consumption of 12% [20].

The authors suggest that the use of hollow aluminosilicate microspheres (ASM) together with microsilica makes it possible to obtain effective low-density cementing solutions, allowing to cement casing columns successfully in difficult geological conditions.

ASM, MS are produced in our country, in addition, the Republic of Kazakhstan has quite large reserves of production waste as slag, sludge and quarry [21].

The objective of this work is to develop lightweight cement slurries using two locally produced lightweight additives with complementary relative properties.

To achieve the set objective, the following tasks were solved:

- study of the influence of lightening additives on the properties of cement slurry and stone with a water-cement ratio of 0.7;
- study of the effect of the hardening accelerator CaCl<sub>2</sub> on the strength characteristics of cement stone at W/C 0.7;
- study of the influence of anhydrous sodium silicic acid on the properties of the solution and the resulting stone.

## 2. Materials and reagents used

The properties of lightweight cements were studied within the temperature range of 25 to 70 °C. This is due to the fact that low-density cements are mainly used at depths where

the formation temperature does not exceed the specified values. For example, at the Amangeldy gas condensate field, the formation temperature at a depth of 2.500 m is 68 °C. In addition, there are hundreds of wells in Kazakhstan where the difference between the bottomhole and the wellhead does not exceed 25-30 °C.

During the research, we used PTST-IG-CC-1 brand portland cement grade manufactured by LLC «Gejuba Shieli Cement Company» (Kazakhstan). The main condition for choosing additives is the focus on local production. Therefore, in the work we used heat-insulating inorganic aluminosilicate microsphere (ASM) manufactured by TOO «Fortuna Trade Company» Republic of Kazakhstan. Also one of the parameters when choosing ASM was high strength, which ensures pressure resistance during volumetric compression. MS is formed at thermal power plants during combined hydraulic removal of ash and slag during the combustion of coal. The properties of ASM are given in table 1.

MS-85 brand microsilica produced in the Republic of Kazakhstan (by YDD CORPORATION, Karaganda) was used as a modifying substance. The main parameters of MS-85 microsilica are given in table 2.

SMA – anhydrous sodium silicic acid salt, which is a chemical filler used as a technological additive in cement materials. This additive was used to improve the technological properties of the cement slurry and stone.

The calculated specific surface area of microspheres is approximately 1100–1200 cm<sup>2</sup>/g. The specific surface area of silica fume waste, obtained during the production of metallurgical silicon and ferrosilicon, may vary depending on the method of collection and processing but typically ranges from 15000 to 30000 m<sup>2</sup>/kg.

### 2.1. Methodology of conducting experiments

The physical and mechanical properties of the cement were determined according to GOST 26798.2-96 «Oil well cements. Test methods». The technical conditions of these materials are regulated in GOST 1581-96 «Oil well portland cements. Technical conditions».

In addition, cement slurries were tested for compliance with the international standard ISO 10426 «Oil and gas industry. Cements and materials for well cementing». Fresh water was used as the mixing liquid in all cement slurry compositions.

In accordance with GOST 14532-2019, water separation was measured in milliliters. For all tested lightweight cement slurry compositions, the water separation did not exceed 7.5 mL, which complies with the requirements of GOST 1581-96. This information will be included in the revised version of the manuscript to ensure regulatory completeness and clarity.

### 2.2. Experimental results

The primary objective was to reduce the density of the cement slurry. For this purpose, studies were conducted with ASM and MS at a water-solid ratio of 0.7. Their influence on the physical and mechanical properties of the cement stone was also studied. The results are presented in table 3, and the graphical interpretation is in figures 2 and 3.

The experiments showed that with an increase in the concentration of ASM from 5 to 10 %, the density of the lightweight cement slurry with W/C=0.7 decreases from 1637 up to 1558, which in turn confirms the previously obtained

**Table 2**  
Main parameters of microsilica MS-85

Color	Light gray ultrafine powder
Activity, %	95
Bulk density, kg/m <sup>3</sup>	350
Density of suspension, kg/m <sup>3</sup>	1440
Specific surface area, m <sup>2</sup> /kg	30000
Mass fraction in terms of dry product, % not less than	97
Mass fraction of silicon dioxide (SiO <sub>2</sub> %),	85
Mass fraction of calcium oxide, % no more than	3
Mass fraction of water, % no more than	3
Mass fraction of sulfur anhydride, % no more than	0.6
Mass fraction of free alkalis (Na <sub>2</sub> O, K <sub>2</sub> O), % no more than	2

Effect of Lightweight Additives on the Properties of Cement Slurry and Set Cement						
Composition	W/C	Density, kg/m <sup>3</sup>	Flexural Strength, MPa	Compressive Strength, MPa	Initial Setting Time (h:min)	Final Setting Time (h:min)
			2 days	2 days		
Neat cement (PCT)	0.7	1733	1.6	3.30	-	-
ASM						
PCT+5% ASM	0.7	1637	1.4	2.17	5:20	7:30
PCT+7% ASM	0.7	1601	1.6	2.65	5:35	7:50
PCT+10% ASM	0.7	1558	1.7	2.64	5:50	8:10
MS						
PCT+5% MS	0.7	1699	2.10	3.90	5:25	7:50
PCT+7% MS	0.7	1691	2.11	4.22	6:10	8:20
PCT+10% MS	0.7	1687	2.38	4.69	5:50	7:35

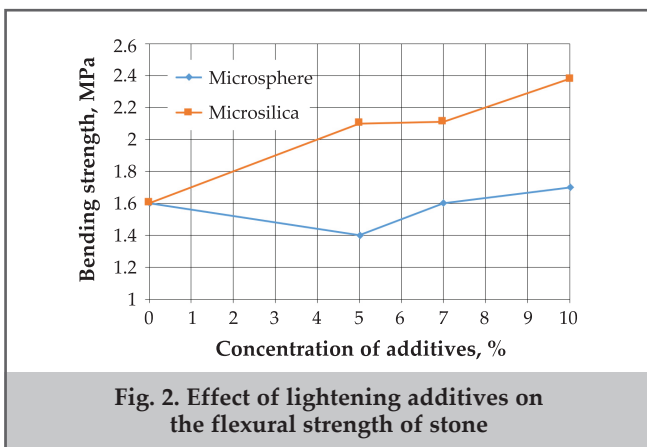


Fig. 2. Effect of lightening additives on the flexural strength of stone

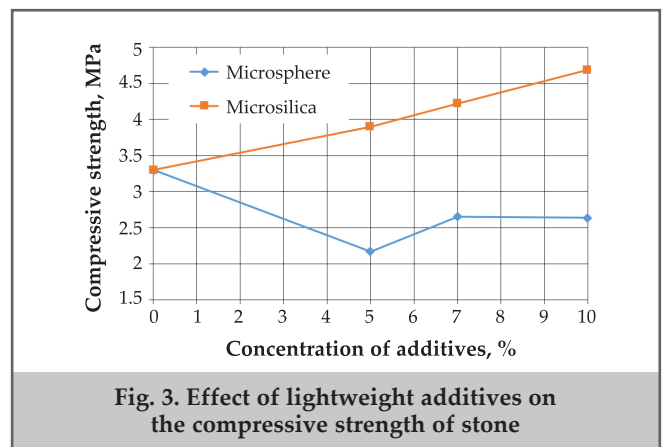


Fig. 3. Effect of lightweight additives on the compressive strength of stone

Effect of hardening accelerator CaCl <sub>2</sub> on the strength characteristics of cement stone at W/C 0.7					
Compound	Density, kg/ m <sup>3</sup>	Bending strength, MPa	Compressive strength, MPa	Beginning of, h\min	End of setting, h\min
PC+10% ASM+10 % MS+1 % CaCl <sub>2</sub>	1581	2.00	3.11	5:35	7:55
PC+10% ASM+10 % MS+2% CaCl <sub>2</sub>	1572	1.41	3.55	4:15	6:25
PC+10% ASM+10 % MS+3% CaCl <sub>2</sub>	1583	2.15	3.59	3:50	6:05

results of the authors' work [22]. When adding MS with a similar concentration to cement, the result regarding density is similar.

After a series of experiments (table 3), a cement slurry formulation was obtained that included PC+10% ASM+10% MS, which began to manifest the properties that were the goal of this work. However, this formulation did not meet the pumpability requirements, all slurry compositions remained pumpable for more than 10 hours, so the setting accelerator CaCl<sub>2</sub> was added to regulate the consistency of the cement slurry.

Further studies were conducted on the effect of the hardening accelerator CaCl<sub>2</sub> on the strength characteristics of cement stone at 10% ASM and 10 % MS (table 4, fig. 4).

The results showed that at the used concentrations of CaCl<sub>2</sub> the density of the cement slurry remains practically unchanged. However, the physical and mechanical properties increased slightly. The start and end times of setting were expectedly reduced (table 5, fig. 5).

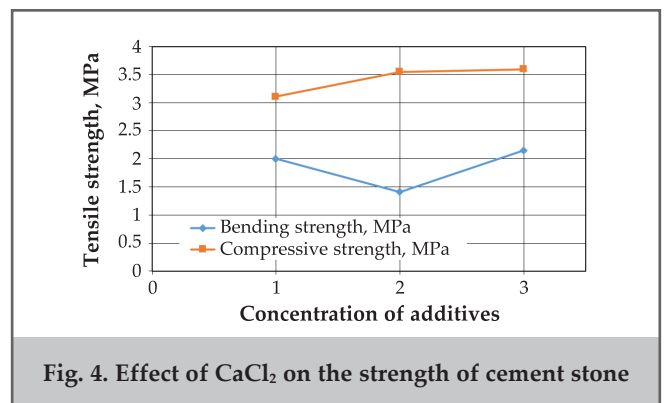


Fig. 4. Effect of CaCl<sub>2</sub> on the strength of cement stone

From the data in table 5 it can be seen that the thickening time and water yield of the solution have been significantly reduced.

Further work was carried out with formulation 3, which is given in table 5, having the composition of PCT + 10% ASM,

Effect of lightening additives and hardening accelerator on the properties of cement slurries				
Compound	Density, g/ cm <sup>3</sup>	Filtration rate cm <sup>3</sup> /30min	Flowability, cm	Thickening time, h\min
PC+10% ASM+10% MS+1% CaCl <sub>2</sub>	1.581	140.8	25	9:45
PC+10% ASM+10% MS+2%CaCl <sub>2</sub>	1.572	171.2	25	8:15
PC+10% ASM+10% MS+3% CaCl <sub>2</sub>	1.583	138.4	25	7:15

Effect of SMA on the properties of slurry and resulting cement stone				
Compound	V/C	Density, g/ cm <sup>3</sup>	Bending strength, MPa	Compressive strength, MPa
			2 days	2 days
Non-additive PC	0.7	1.733	1.6	3.30
Cementing mortar with the addition of CaCl <sub>2</sub>				
PC+10 % MS+10% ASM+3% CaCl <sub>2</sub> +0.1 % SMA	0.7	1.577	1.87	3.93
PC+10 % MS+10% ASM+3 % CaCl <sub>2</sub> +0.5 % SMA	0.7	1.563	2.05	4.49
PC+10% MS+10% ASM+3% CaCl <sub>2</sub> +1 % SMA	0.7	1.567	2.26	5.05
Cement mortar without CaCl <sub>2</sub>				
PC+10% MS+10 % ASM+0.1 % SMA	0.7	1.575	1.8 1	4 . 74
PC+10 % MS+10% ASM+0.5 % SMA	0.7	1.550	2. 32	5 . 72
PC+10% MS+10% ASM+1% SMA	0.7	1.559	2. 58	7 . 0 4

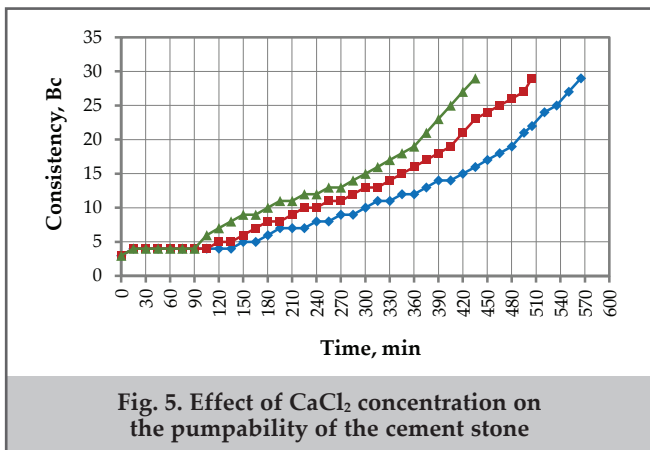


Fig. 5. Effect of CaCl<sub>2</sub> concentration on the pumpability of the cement stone

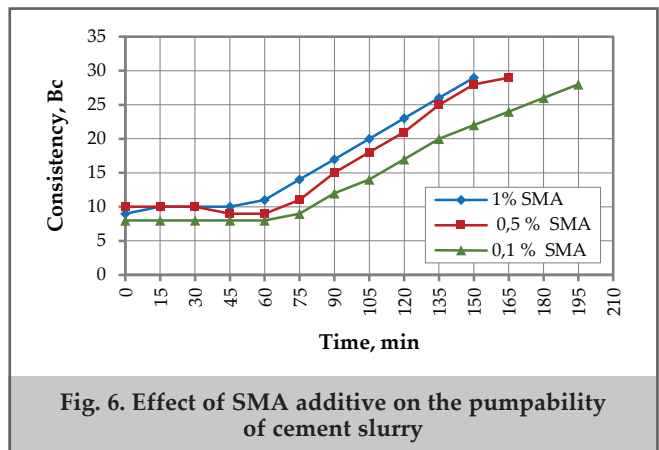


Fig. 6. Effect of SMA additive on the pumpability of cement slurry

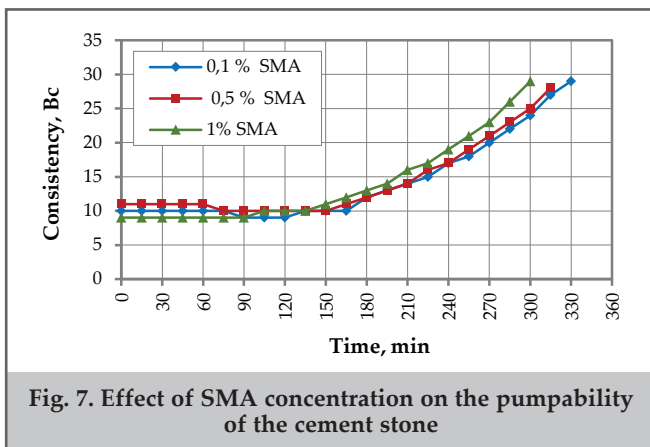


Fig. 7. Effect of SMA concentration on the pumpability of the cement stone

10% MS + 3%CaCl<sub>2</sub>. This formulation was suitable in terms of strength characteristics and thickening time.

It is also evident from table 5 that the water loss of the solutions is quite high, which required additional regulation

of the water content in the mixture. For this purpose, exploratory studies of the reagent were conducted and the SMA additive was determined. The studies were conducted at a concentration of 0.1 to 1% (tables 6, 7, fig. 6).

From table 6, which presents the cement slurry with the addition of an accelerator, it is evident that the bending strength limit increases with the increase in concentration from 0.1 to 1%, which reaches up to 2.26 MPa. It is also possible to see an increase in the compressive strength limit from 3.39 MPa to 5.05 MPa.

It is also evident from table 7 that the addition of SMA reduces the pumpability time from 430 minutes to 195 minutes. This makes it possible to refuse the use of CaCl<sub>2</sub> and thereby reduce the number of components used. Therefore, Tables 6 and 7 present data where the cement slurry does not contain a setting accelerator in its composition.

Addition of SMA from 0.1 to 1% showed positive results, and it was proven that the cement slurry can be made without adding CaCl<sub>2</sub> and still achieve the required pumpability (fig 7).

Table 7

Effect of SMA on the properties of cement slurry					
Compound	Density, g/ m <sup>3</sup>	Filtration index, s m <sup>3</sup> /30 min	Flowability, cm	Setting time, h\min	End of setting, h\min
Cementing slurry with the addition of CaCl <sub>2</sub>					
PC+10% MS+10% ASM+3% CaCl <sub>2</sub> +0.1 % SMA	1.577	93.6	24	3:25	5:45
PC+10% MS+10% ASM+3% CaCl <sub>2</sub> +0.5 % SMA	1.563	92.8	23	3:15	5:25
PC+10% MS+10% ASM+3% CaCl <sub>2</sub> +1 % SMA	1.567	80	23	3;10	4:55
Cement slurry without CaCl <sub>2</sub>					
PC+10% MS+10% ASM+0.1% SMA	1.575	114	21	6:10	8:30
PC+10% MS+10% ASM+0.5 % SMA	1.550	90	21	6:05	9:25
PC+MS 10%+10% ASM+1% SMA	1.559	8 4	19	5:50	8:55

### 2.3. Discussion

Aluminosilicate hollow microspheres have disadvantages when used in cement slurry, including low strength, high fluid loss and floating, which negatively affects sedimentation stability (fig. 1). The indicated negative properties were obtained as a result of experiments presented in table 3, where the stone with the addition of even 10% of microspheres had low strength relative to the cement mortar stone without additives. However, microspheres reduce the density of the cement slurry to 1500 kg/m<sup>3</sup> with a water-cement ratio of 0.7.

Another lightening additive – microsilica showed a positive result relative to the microsphere, although the density decreases only to 1687 kg/ m<sup>3</sup> at W/C-0.7. Table 3 shows that the strength of the cement stone in bending and compression is higher than that of the cement without additives.

The experiments showed that the formula of PC + 10% ASM + 10 % MS, where both lightweight additives complement each other, thereby allowing to obtain a lightweight cement slurry with a density of 1500 kg/m<sup>3</sup> at W/C-0.7. The water-holding capacity, which meets the requirements of GOST increased. This phenomenon is explained by the fact that the size of the microsilica particle does not exceed 1 nanometer, and the microspheres are from 10 to several hundred micrometers in diameter, on average about 100 μm. The ultra-dispersity of the microsilica acts as a binder and does not allow the microspheres to float.

However, the formula of PC + 10% ASM + 10 % MS had a disadvantage in terms of water yield and pumpability, which lasted for more than 10 hours, which in turn had to be regulated by adding hardening accelerators. The standard additive is CaCl<sub>2</sub>, despite the fact that when adding this additive more than 5%, problems with casing corrosion may arise. The addition of CaCl<sub>2</sub> reduced the pumpability time to the required values according to GOST. However, the problem of reduced water yield did not disappear, and remained above 100 cm<sup>3</sup> /30 minutes (table 5).

The search for reagents and preliminary experiments showed the effectiveness of anhydrous sodium silicic acid – SMA.

The above mentioned reagent SMA is an anhydrous sodium salt of silicic acid. Sodium silicate reacts with calcium hydroxide (Ca(OH)<sub>2</sub>), formed during cement hydration, with the subsequent formation of additional CSH gel, which compacts the structure of the cement stone [23]. This leads to an increase in its mechanical strength and durability (table 6). Increasing the concentration of SMA from 0.1 to 1 % always

results in increased strength.

The use of SMA additive without CaCl<sub>2</sub> showed an improvement in strength (table 7, cement slurry without CaCl<sub>2</sub>) and a positive effect on structure formation. This result without the use of CaCl<sub>2</sub> made it possible to obtain a cement slurry with improved parameters and reduce the number of components.

In general, the peculiarity of the developed composition of the cement slurry is the use of locally produced components. The obtained results, in comparison with the existing ones, are distinguished by their low component content. For example, to obtain the cement slurry, according to GOST, water loss reducers, various fibers to increase the physical and mechanical properties, additives against the floating of microspheres, etc. are used.

The main limitations of this study are the density of the cement slurry, which varies between 1500-1600 kg/m<sup>3</sup>. The disadvantages of this study may be the lack of detailed microstructural analysis, adhesion testing, and the effect of expanding additives on this formulation. However, these studies will be reflected in future works, which will be taken into account in the future.

We acknowledge the importance of using gas-blocking agents and defoamers when cementing gas and gas condensate wells to prevent gas migration and ensure cement integrity. However, the main objective of this study was to assess the effect of lightweight additives – specifically aluminosilicate microspheres (AMCS) and microsilica (MKR) – on the density and strength characteristics of the cement slurry. The influence of gas-blocking agents and defoamers was beyond the scope of the present work but is planned for future research to provide a more comprehensive evaluation of cementing performance under gas-bearing conditions.

Since the lightweight cement slurry is primarily intended for use in the upper intervals of the well, we did not include gas-blocking additives in this study.

The developed cement slurry is not designed for high-temperature conditions that would accelerate gas release and foaming. Additionally, the formulation does not contain cement additives prone to foam formation.

Furthermore, it is worth noting that silicone-based defoamers typically incorporate hydrophobic silica particles as additives, which enhance foam destruction (e.g., Antipena 2000, AF-5, Silikonol 350). This aspect may be considered in future developments, if the slurry is to be applied in conditions with a higher risk of gas intrusion.

## Conclusions

1. The results of the experiments made it possible to obtain a lightweight cement slurry with a density of 1500-1600 kg/m<sup>3</sup> with the corresponding technological properties.
2. A rational combination of MS and ASM additives ensured a good reduction in the density of the solution and fluid loss, and an increase in the strength of the cement stone.
3. A study of the effect of the hardening accelerator CaCl<sub>2</sub> on the strength characteristics of cement stone at W/C=0.7 showed positive results relative to the solution without additives and relative to the formulation of PC+10% ASM+10 % MS, where the compressive strength was 3.59 MPa and the flexural strength was 2.15 MPa;
4. The use of SMA additive in a lightweight solution improved all the technological properties of the cement slurry and cement stone.
5. The optimal solution formulation is: PC+MS 10%+10% ASM+1% SMA, with which fluid loss decreased to 84 cm<sup>3</sup>/30 minutes; flexural strength was 2.58 MPa; compressive strength 7.04 MPa, with a spread of 19 cm. CaCl<sub>2</sub> in the future.
6. SMA additive showed a decrease in the formability of the solution which made it possible to abandon the use of CaCl<sub>2</sub> in the future.

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