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Cement paste stratification at critical cementing point

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Abstract. The relevance of this research is driven by the necessity to investigate and predict the technological parameters of grouting mixtures in conditions that closely resemble real-world scenarios. The subject of this study is a grouting compound used in well construction, which serves to ensure the adhesion between the casing and the formation, strengthen the borehole walls, and prevent the leakage of underground fluids. The objective is to develop a methodology for investigating the cement mixture formation based on both theoretical and empirical data, with the aim of most accurately representing the actual behavior of the grouting solution within the annulus of a wellbore. The authors have proposed a model for two types of cement mortar structures. A method for examining the sedimentation stability of cement mortars using a specially designed experimental setup and monitoring protocol is presented. The analysis of samples collected at the proposed site allows us to study changes in the density of cement slurry over time, while simulating the behavior of cement in the annulus during the first hour of pumping. The findings indicate a tendency towards thickening and hardening of the cement mixture, as well as highlight potential issues that may arise when the cement composition does not meet the requirements set by downhole conditions. The statistical analysis of measured data demonstrates good reproducibility with low error, allowing us to simulate deposition of cement under various conditions. The results and the proposed recommendations for improving cement stability will be of value to technical experts and researchers, enabling them to achieve the objectives of environmentally friendly, time-efficient, and economically viable well construction.

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1. Introduction

During the construction of wells, cement-based formulations are primarily used, which must comply with the requirements outlined in relevant regulatory documents such as Russian State Standards GOST 1581-2019 and GOST 34532-2019, as well as API Specification 10A:2019 and API RP 10B-2-2024. These standards specify the type and range of properties for both cement paste and the resulting artificial stone. The main purpose of grouting mixtures is to fill the annular space between the casing and the borehole.

The problem of the lack of a homogeneous cement stone structure creates the need for a more detailed study of the issue of quality violations during well construction [1, 2]. The active stratification of the grouting composition by the depth of cementation negatively affects the quality of rock insulation and the tightness of the borehole, and also forms a highly porous cement stone, significantly reducing the operational lifespan of the well [3]. The appearance of water in the upper zones of the cementing sites is one of the observed consequences of uneven filling of annular space with cement paste during drilling. The aim of this study is to develop a technique for determining water separation in cement slurry, free from the shortcomings of the standard method, as well as identifying correlations with key factors influencing water separation in grouting mixtures.

It is known that the denser the packing of Portland cement particles, the higher the ability of cement paste to hold a certain volume of water. The cohesion of the structure is crucial when designing a cement mix with specified rheological properties. If the normal density of the cement paste is significantly exceeded, the particles do not come into direct contact with each other, as they are sufficiently removed and separated by layers of water. This type of system is sedimentationally unstable, leading to water separation. This process can also be described as the stratification of the cement dispersion system. Stratification occurs throughout the entire height, forming a material density gradient. Naturally, the density in the upper layers will be lower than in the deeper layers. This density gradient, after hardening, will naturally lead to differences in porosity and strength in the cement stone.

Water separation is a structural heterogeneity of the cement paste, and, as a result, a negative factor leading to the formation of a cement stone with gradients in its density and properties, particularly during the construction of deep boreholes, where there is a difference in the height of the cementing intervals. To eliminate this inhomogeneity, various methods have been proposed, including the introduction of filler additives such as chalk, quartz, clay, fly ash, asbestos, and others, which reduce water segregation (plasticizing and hydrophilic surfactants), as well as the use of aqueous solutions containing chloride and sodium carbonate [4–6].

The water-cement (W/C) ratio of a cement paste of normal density is dependent not only on the mineral composition of the clinker and the content and properties of mineral additives but also on the conditions, under which the structure forms. For normal Portland cement, this parameter is approximately 0.25 under hardening conditions. It is known that when filling borehole spaces with a grouting mixture, the initial W/C ratio specified may differ significantly from the actual concentration of the cement gel, depending on the depth of the mass distribution. By analogy with Boltzmann's barometric formula [7], we can consider a problem regarding the effect of forces on a cement particle evenly surrounded by water in a state of equilibrium. In this case, the downward gravitational force will be balanced by the resistance forces of the medium and Archimedes. This model accurately describes the issue of uneven filling of the annulus of the well with cement slurry, followed by strata formation and possible absorption or migration of the cement mix into nearby heterogeneous formations. As a consequence, the resulting anisotropic cement rock will not meet the standards for high-quality isolation of the borehole from corrosive subsurface fluids (liquids and gases), which could lead to industrial incidents.

The analysis of standard requirements reveals that to assess the stability of grouting mixes (cement slurry or mortar) against sedimentation, the characteristic "water separation" is used, defined as the volume of water that separates. In this instance, a measuring cylinder with a capacity of 250 cm^3 is employed, which significantly differs from the depth of the well being cemented. This disadvantage could be overcome by simulating the sedimentation process of cement-water systems. Nevertheless, addressing this issue involves overcoming challenges related to the interaction of settling particles and the formation of agglomerates, whose structure and properties vary significantly over time. Consequently, experimental statistical models have gained popularity, the production of which entails conducting experimental studies using equipment that avoids the shortcomings of known methods [8–10].

The rheological properties of cement slurry are influenced by various factors, including the concentration, average size, and volume distribution of cement particles. These factors also include the shape, structure, surface properties, and physical properties of the liquid and solid components [11, 12].

Additionally, water segregation in cement slurries is influenced by physical and chemical processes such as the sedimentation, aggregation, and hydration of cement particles. This process leads to the formation of a dispersed phase, which can be observed in the chemical equation for the hydration of tricalcium silicate C_3S, a major component of cement [13]:

$$2\left(3\mathrm{CaO}\cdot\mathrm{SiO}_2\right)+6\mathrm{H}_2\mathrm{O}\rightarrow 3\mathrm{CaO}\cdot\mathrm{SiO}_2\cdot 3\mathrm{H}_2\mathrm{O}+3\mathrm{Ca}\left(\mathrm{OH}\right)_2. \quad (1)$$

It follows from this equation that the ratio of the volumes of the solid phases of the hydration products $\mathrm{V_g}$ and the starting material $\mathrm{V_{ng}}$ is $\mathrm{V_g}/\mathrm{V_{ng}}=1.65$. Some of these processes are considered in the standards for determining the water separation rate of a cement mix using a measuring cylinder [14]. A cement paste is placed in a cylinder and allowed to set for a specific period of time. After this, the stratification of the mixture is visually examined, and the extent of stratification is calculated based on the volume of water displaced in the upper portion of the cylinder [15–17]. However, this method of water separation measurement does not consider many geological, technical, and other significant factors, including the influence of external environmental conditions (geological characteristics of rocks in contact with cement), physical and mechanical parameters (pressure, temperature), and technological aspects of well construction (geometrical parameters of the well, construction method, type of flushing fluid, etc.).

The study [18] showed the technological limitations of the current standards for water separation analysis using a measuring glass cylinder in revealing the true picture of cement particle sedimentation in the annulus. The work also identified and described cracks and voids (migration pathways) that may be filled during cement injection.

W/C ratios are among the key cement parameters that affect the mixture characteristics and mechanical properties of grouting materials. Micro-cement compositions with W/C ratios ranging from 0.8:1 to 2:1 were examined in [19]. It was found that density, workability, rate of cement-stone formation, and bending and compressive strengths of cement stones gradually decreased with increasing W/C ratio. Conversely, the spreadability and fluidity of micro-cement mixes gradually increased.

A method for investigating the water separation properties of cement mixtures using low-field nuclear magnetic resonance (NMR) has been proposed in the study [20]. The findings indicate that the NMR technique in a weak magnetic field can be successfully employed not only to measure the extent of water separation, investigate the microstructure of cement materials but also to provide information on the concentrations of substances present in the separated water. Furthermore, it has been determined that the migration of a substantial amount of water from the porous structure of a maturing cement slurry gives rise to significant alterations and deterioration in the microstructure of the resulting cement stone.

To address the issue of static segregation, it is common practice to select formulations with the lowest possible W/C or water-binder ratio, as well as to utilize microsilica to reduce water segregation while maintaining the structural properties of the cement matrix in studies of high-performance mobile concrete mixes [21]. At the same time, it has been established that superplasticizers, which increase the flowability (workability) of the cement paste, adversely affect the mechanical characteristics of mortar mixtures and reduce the durability of molded cement products [22]. In order to explore the correlation between cement properties and the propensity of cement mixtures to water segregation, the authors of [23] discovered that with a lower specific surface area and higher alkali content, there is a greater tendency towards intensive water segregation.

The authors of [24] investigated the deposition of cement mixtures by visual observation, which revealed two stages of delamination: an initial phase of rapid water separation followed by a stage with a decreasing sedimentation rate, where the upper layer gradually became transparent.

In predicting the sedimentation of cement mixtures, the laws of suspension dynamics may be applied. Specifically, article [25] addresses the issue of particle sedimentation in a stationary fluid. The authors conclude that the concentration of solids in the flow and the density increase linearly with depth, while the pressure increase follows a weakly quadratic trend.

One of the most significant criteria for assessing the quality of concrete is the density of the concrete mixture. The researchers in [26] developed a method for determining the density of settling concrete suspensions by sampling from specific heights at different times. This study demonstrated that the characteristics of the concrete mixtures, including their uniformity and flowability, vary significantly depending on the height and time of sampling. Further research into the relationship between concrete mixture properties and their tendency to separate revealed that mixtures with a smaller specific surface area have a higher tendency for water displacement. It has also been shown that due to obvious separation, the water-to-cement ratio and phase content in the settled suspension layer do not match the initial concrete composition parameters.

The main limitation of previous studies is the significant gap between laboratory testing of grouting materials during the initial phase of cement-stone structure formation and the measurement of the state of the cement annulus after hardening in the borehole.

The classical approach to the sedimentation process involves determining the sedimentation rate of a particulate, which is influenced by the forces of Stokes and Archimedes, as well as the gravitational force of the Earth. However, the actual rate of deposition of grouting mixtures may differ from the calculated value, due to both assumptions made in determining the geometric properties of the particles, and the influence of adjacent particles [27]. The Lyashenko method has been used to study the constrained deposition rate of particles with different geometric shapes. However, this method has the disadvantage of relying solely on experimental measurements, which are used as the primary method for determining the properties of sedimentation. As mentioned above, and in other studies [28–30], completely experimental dependencies are used to calculate the parameters of suspended deposition.

The quality of grouting material cannot be determined solely based on average laboratory test results. In practice, there are always variations from the obtained results. Changes in mixing and injecting the mixture, composition of clinker, activity and normal density of cement, dosage of improving additives, and other technological and geological factors affecting the formation and hardening of the structure have an impact on the quality of well construction. To improve the quality of cementing operations, it is necessary to modernize existing laboratory methods for simulating borehole conditions.

In accordance with the objectives set by the authors, this paper presents the findings of a study on the sedimentation of cement particles and stratification of cement slurry prior to cement setting when it is injected into a borehole. A method has been devised to analyze the heterogeneity of cement matrix formation based on a density analysis of grouting mixture at various depths after a specific period of time. To implement the method for measuring cement slurry density, an experimental setup is proposed, and the process of sample preparation and experimentation is described in detail. The results obtained indicate consistent trends in the behavior of the cement mixture in simulated conditions of cement structure formation in a wellbore.

2. Materials and Methods

For the experiments, casing Portland cement I-50 brand similar to Portland cement I-G-CC-1 brand was used, which meets the requirements of GOST 1581-2019. The properties of the cement mixtures were investigated using methods specified in GOST 33213-2014, GOST 34532-2019, and GOST 30744-2001. Storage, preparation, labelling and sample preparation were conducted in accordance with GOST 30515-2013. The standard specifications and actual characteristics of the cement are presented in Table 1 below.

Table 1. Characteristics of the cement mixture with a W/C ratio of 0.5.

Physical and mechanical characteristics	GOST requirements	Actual performance
Bending strength, MPa at the age of 2 days, not less than MPa	2.7	3.1
Spreading capacity of cement paste, not less than mm	200	220
Bleeding capacity, no more than ml	8.7	2.5
Thickening time to consistency of 30 Bc, not less than min	90	320

To meet the requirements for pumpability and workability of standard grouting cement, it is common to use W/C = 0.44…0.5. However, for lightweight and heavyweight cements, this ratio can vary between 0.3 and 1.3. In this study, we will consider compositions with W/C = 0.5…1.0.

When discussing the structural and rheological properties of cement paste, the concept of water retention capacity is introduced [31–33]. This is the amount of water that is retained within the cement paste after it has been formed. The W/C ratio of a cement paste of normal density indicates at what water content the paste will form, without any separation of its components (phases). As the W/C increases, a more heterogeneous structure begins to form, with increased structural heterogeneity. To assess this heterogeneity, we utilize a modified version of the phase separation coefficient proposed by A.N. Bobryshev et al. [34, 35]. For cement pastes, this coefficient takes the following form:

$$\psi_{f,m} = \frac{2 \cdot v_{w,0} - v_w}{v_{w,0}}, \quad \text{for } v_w \geq v_{w,0}, \tag{2}$$

where $v_{w,0}$ – volume fraction of water for sedimentation-resistant cement paste; v_w – volume fraction of water of the studied composition.

For $v_w < v_{w,0}$, formula (2) must be converted to the form:

$$\psi_{f,m} = \frac{v_w}{v_{w,0}}. \tag{3}$$

It is evident that structural heterogeneity is the origin of water separation (phase segregation), and at $v_w = 2v_{w,0}$ structural heterogeneity reaches its maximum (Fig. 1).

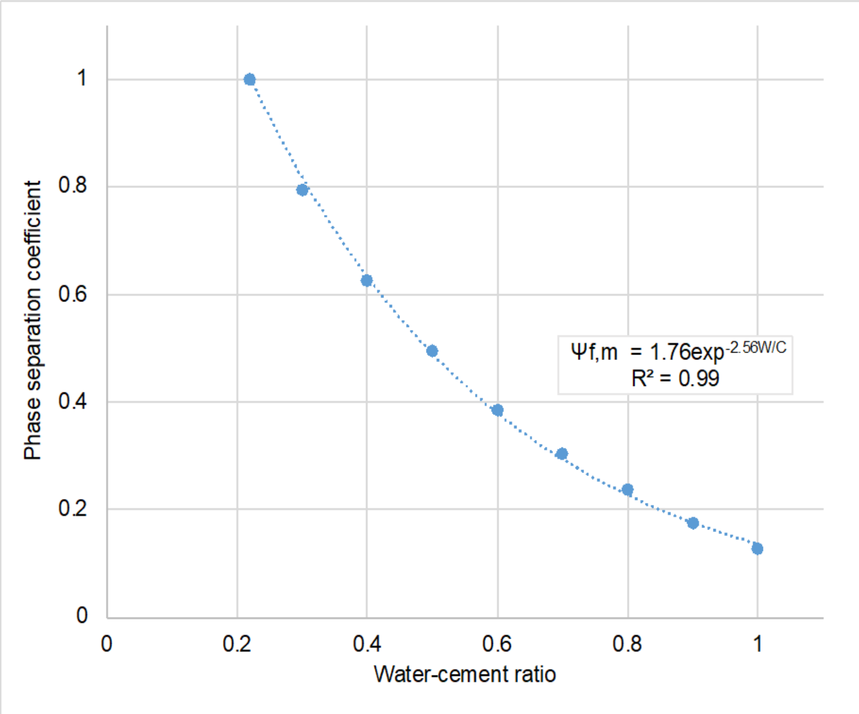


Figure 1. The dependence of the coefficient of uniformity on the W/C ratio.

As the proportion of water in a cement mixture increases, the distance between the cement particles also increases:

$$h_w = \frac{v_w}{(1 - v_w) \cdot S_u \cdot \rho_c}, \tag{4}$$

where S_u and ρ_c – unit surface and cement density.

Furthermore, the rate of increase in the thickness of the water layer increases proportionately $1/(1-v_w)^2$. Obviously, the natural process aims to minimize the potential energy of all particles in a dispersed system affected by the Earth's gravitational field. The model, which allows assessing the influence of the main factors at the initial stage, is based on the classical effect of forces on a particle as well as the limitations of: 1) the absence of the influence of other particles, and 2) the absence of compaction of the particle sediment. A particle with a diameter d_f in the initial state is surrounded by a layer of water of thickness $h_{w,0}$, and after sedimentation (steady state) – by a layer with thickness h_w. Let the height of the column where the suspension is located be H_0, and the area of the base S_0. In its initial state, this column will be divided into thin layers $d_f + 2h_{w,0}$. To reach a stable state for the particle in the first layer, that is the layer located immediately adjacent to the lower boundary, or bottom, requires that an equal distance $\Delta l = 2(h_{w,0} - h_w)$ be traversed. The particle in the second layer must traverse the distance to the interface with the first layer Δl, as well as the distance the first particle has already traversed – Δl. Thus, the total distance that particles in the second layer need to travel before reaching a stable position is equal to $2\Delta l$. Similar reasoning applies to particles in any other layer, and their distance will be equal to $i \cdot \Delta l$ (where i is the layer index). These distances are related to sedimentation rates [36–38]:

$$\frac{i \cdot \Delta l}{t_i} = \frac{1}{18} \cdot \frac{g \cdot d_f^2 (\rho_f - \rho_m)}{\mu}, \tag{5}$$

where μ and ρ_m – viscosity and density of the medium, in which occurs the sedimentation of particles with density ρ_f; g – gravitational acceleration.

In this scenario, all particles, regardless of their position, will move an equal distance over time t_i. The motion of particles in the upper regions under the influence of gravity corresponds to the creation of a layer of liquid on the surface of the system. When determining the requirements for the thickness of the liquid layer, it is feasible to assess the geometric properties of the container used in a study on the distribution of fractions of the material in various states (Fig. 2).

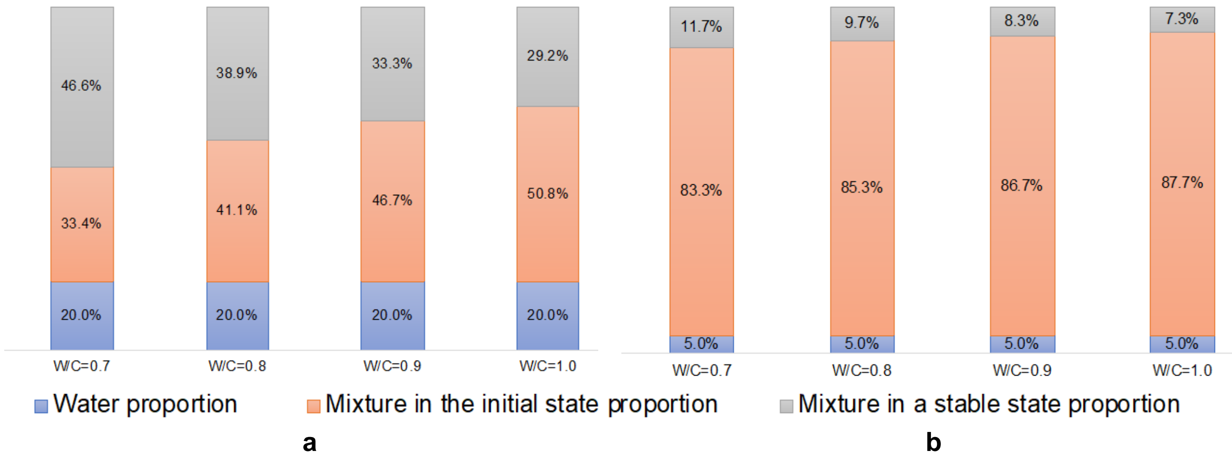


Figure 2. The effect of column height on the distribution of types of mixture states at the relative height: a) H_0:S_0 = 1:1; b) H_0:S_0 = 4:1.

The data presented in Fig. 2 illustrate that at low column heights, stratification of the mixture occurs more rapidly, specifically the formation of a stable layer of particle mixture and a layer of liquid. The time required to reach stratification is found to be proportional to the height ratio between the two containers under comparison (at $S_0 = \text{const}$). Another implication of the model is the possibility of describing the structure of the mixture as a system composed of different compositional types. Based on the presented model, it is sufficient for there to be two such structural types.

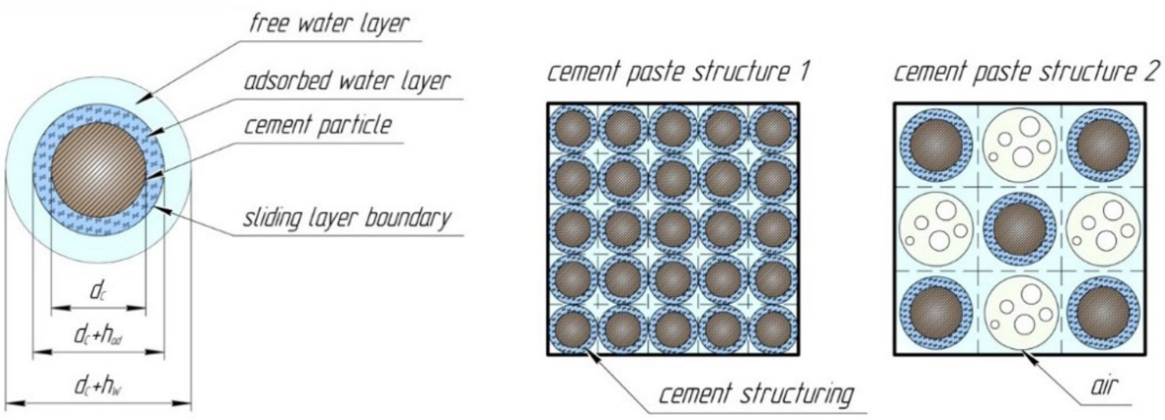
Obviously, the model under consideration is a rather crude approximation. In actual mixtures, there would not be distinct boundaries between structures. Nevertheless, it allows us to fully assess both the origin of structural heterogeneity and the impact of various factors, including the depth of well cementing.

Using the presented model, we will determine mixture density as a feature characterizing cement paste's structural characteristic at various depths. Let us imagine that the cement mixture consists of a three-phase system "cement – water – air":

$$\varphi_c + \varphi_w + \varphi_{air} = 1, \tag{6}$$

where φ_c – cement volume fraction; φ_w – water volume fraction; φ_{air} – air volume fraction.

Considering a hypothetical scenario where a cement particle is spherical in shape. At the initial point in time, the contribution from its hydration process can be disregarded, and the physical and chemical interaction results in the formation of a water adsorption layer on the surface of the cement particles. This adsorption layer differs significantly from the properties of freely flowing water (Fig. 3a). Regarding the model discussed previously, as well as the various phases that constitute the cement slurry, we can identify two main types of structures: 1. "cement – adsorbed water – free water;" 2. "cement – adsorbed water – free water – air" (Fig. 3b).



a **b**

Figure 3. Cement structuring by two types of particle arrangement:
a) model of a cement particle surrounded by water; b) types of cement paste structures, where d_c – diameter of the cement particle; h_{ad} – thickness of the adsorbed water layer; h_w – thickness of the free water layer.

The geometric characteristics of each structural type are determined by a set of parameters, which are shown in Table 2.

Table 2. Parameters for the types of structures.

Type	Number of phases	Phases	Phase Parameters			
			Cement	Water$_{ad}$	Water$_w$	Air
I	3	Cement, adsorbed water, free water	φ_c, d_c	φ_{ad}, h_{ad}	φ_w, h_w	φ_{air}, d_{air}
II	4	Cement, adsorbed water, free water, air	ρ_c, S_u	ρ_{ad}	ρ_w	ρ_{air}

The values of the parameters presented are derived from literature sources [39–41]. Under the given conditions $h_w >> h_{ad}$ and $\rho_{ad} > \rho_w$, we derive the following system of equations to calculate density according to type I:

$$\begin{cases} \rho_I = \varphi_c\rho_c + \varphi_{ad}\rho_{ad} + \varphi_w\rho_w \\ \varphi_c + \varphi_{ad} + \varphi_w = 1 \end{cases}. \tag{7}$$

For type II is valid $\varphi_c = \varphi_c - \varphi_{air}$, therefore, we obtain a system of equations:

$$\begin{cases} \rho_{II} = \varphi_c\rho_c + \varphi_{ad}\rho_{ad} + \varphi_w\rho_w \\ \varphi_c + \varphi_{ad} + \varphi_w + \varphi_{air} = 1 \end{cases}. \tag{8}$$

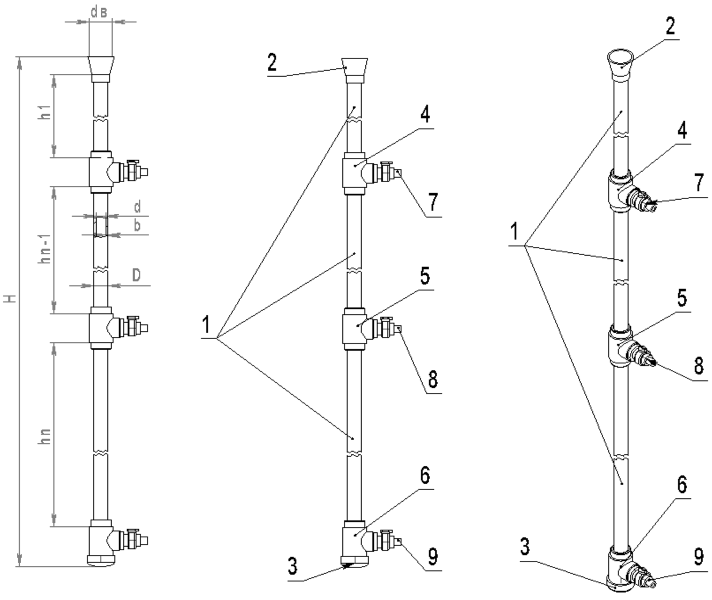
The density of the cement paste, taking into account ρ_I and ρ_{II} will be equal to:

$$\rho_{cp} = \omega_I\rho_I + \omega_{II}\rho_{II}, \tag{9}$$

where ω_I and ω_{II} – volume fractions of the corresponding types of structures: $\omega_I + \omega_{II} = 1$.

The determination of ω_I and ω_{II} is only possible through solving the inverse problem. This involves using calculated ρ_I, ρ_{II} and experimentally determined values ρ_{cp}. Furthermore, the cement slurry for determination can be chosen at any depth within the pipe column.

In order to experimentally determine the variation in the density of the cement paste, a specialized experimental setup has been designed. The schematic diagram of this setup is shown in Fig. 4.



a b

Figure 4. General view of the experimental installation for measuring the density of the cement mixture: a – side view; b – spatial view, where 1 – pipeline; 2 – funnel; 3 – removable plug; 4, 5 and 6 – tees; 7, 8 and 9 – taps.

The device is a vertically positioned housing consisting of a pipeline (1) with a diameter of d and a length of l, having a circular cross-section with a diameter that does not change in the axial direction of the pipeline. The body can be a metal or plastic pipe, industrial ceramics, plexiglass, or other material that simulates downhole conditions. A funnel (2) is fixed in the upper part of the housing for feeding the analyzed grouting mixture, and a removable plug (3) closes the bottom of the pipeline. At distances H_1, H_{n-1} and H_n from the pipe mouth, there are tees A_1 (4), A_{n-1} (5), and A_n (6), with taps X_1 (7), X_{n-1} (8), and X_n (9) connected to them. Depending on the simulated conditions, it is recommended to use at least three measuring taps. In addition, the height of the pipe column depends on the specific set conditions.

The proposed method for measuring the density of a grouting mixture is as follows. The upper edge of the stand is taken as the zero reference point, and subsequent measurements are counted down along the depth of filling the pipe column with cement paste. Cement mixture is prepared according to GOST 34532-2019. The prepared mixture is poured into a funnel to enter the pipeline, until it is completely filled with the test cement paste to the plug, after which it is tightly closed for sealing. The cement paste settles in the installation during the studied measurement time T_1, T_2 … T_n – from 15 to 60 minutes. By alternately opening the ball valves connected through tees to the pipeline, samples are taken for further measurement of the density of the cement mixture by the weighing method. Using mathematical methods of experiment planning, two variable independent factors were selected: W/C ratio and sampling depth, on the basis of which a series of experiments were conducted. The experimental density is taken to be the arithmetic mean of the results of three measurements, the discrepancy between which should not exceed 200 kg/m^3. Proper filling and opening of taps in a certain sequence helps to avoid the occurrence of air bags and water in the laboratory installation in order to obtain correct data. After the measurements, the pipe space is cleaned of the remaining cement paste by rinsing with water.

3. Results

After preparing cement slurry with various water-to-cement ratios, the initial specific gravity was measured under standard conditions. The results of the measurements of the specific gravities of grout mixtures at W/C = 0.5…1.0, and their associated errors, are presented in Table 3.

Table 3. Initial density of cement mixtures.

W/C ratio	0.5	0.6	0.7	0.8	0.9	1.0
Average initial density, kg/m^3	1790	1710	1610	1550	1500	1420
Measurement error, %	8	8	7	6	6	5

After preparing the cement slurry and filling the mold, samples were collected in a volume of 100 mL from each height level, and their density was measured using an electronic balance and the weighting method. Based on the results from three measurements, an average value for density at each height and time point (from the start of setting) was calculated. The findings of the study are presented in Table 4.

If the setting time is less than 15 minutes, the cement paste with a high water-to-cement ratio does not form a densified structure, which can lead to inaccurate density readings. Therefore, any measurements taken with a hydration time of less than 15 minutes have been excluded from the table.

When conducting laboratory tests on the cement mixture with W/C = 0.5, measurements were taken at the installation 15 and 20 minutes after the beginning of the active growth of the setting and the lack of laminar leakage from the tap. With an increase in the W/C to 0.6, the mobility of the cement system increased, allowing for sampling after 30 and 40 minutes from the start of mixing.

Table 4. Experimental density of cement slurries, kg/m^3.

W/C	Thickening time, min	Sampling depth, m		
		H_1 = 0.5	H_2 = 1.5	H_3 = 2.5
0.5	T_1 = 15	1730	1820	1900
	T_2 = 20	1700	1800	1870
0.6	T_1 = 15	1610	1750	1800
	T_2 = 20	1600	1740	1780
	T_3 = 30	1600	1730	1770
	T_4 = 40	1590	1720	1770
0.7	T_1 = 15	1550	1660	1740
	T_2 = 20	1550	1660	1730
	T_3 = 30	1540	1650	1720
	T_4 = 40	1530	1640	1720
	T_5 = 60	1520	1640	1710
0.8	T_1 = 15	1500	1600	1690
	T_2 = 20	1490	1590	1650
	T_3 = 30	1480	1610	1680
	T_4 = 40	1470	1590	1650
	T_5 = 60	1460	1580	1640
0.9	T_1 = 15	1460	1570	1620
	T_2 = 20	1460	1570	1610
	T_3 = 30	1450	1560	1610
	T_4 = 40	1450	1550	1600
	T_5 = 60	1440	1550	1590
1.0	T_1 = 15	1360	1460	1540
	T_2 = 20	1350	1450	1530
	T_3 = 30	1340	1450	1520
	T_4 = 40	1330	1440	1520
	T_5 = 60	1320	1430	1500

Experimental data have shown that when using cement slurry with a water-to-cement ratio up to 0.6, it is important to carefully monitor the initial consistency, density, and setting time of the mixture in order to prevent premature thickening in the pipeline, which would prevent the extraction of samples of the material in liquid form. Later, the density results obtained for W/C ratios of 0.5 and 0.6 were not considered due to insufficient time measurements for further analysis.

It has been decided to divide the process of cement sedimentation and structure formation into three stages. In the first stage, the binder is sealed and pumped into the column, during which the most active phase of stratification occurs. The second stage, lasting from 15 minutes to 60 minutes, or the beginning of the setting of the cement mixture, reflects the main phase of structure formation. During the third stage, the cement slurry thickens until it has completely cured. The first stage is the most difficult to accurately measure due to high error and the short time period. Therefore, the most suitable stage for study is the structure formation of cement prior to its setting. Fig. 5 illustrates the kinetic curves of density versus time for W/C = 0.8.

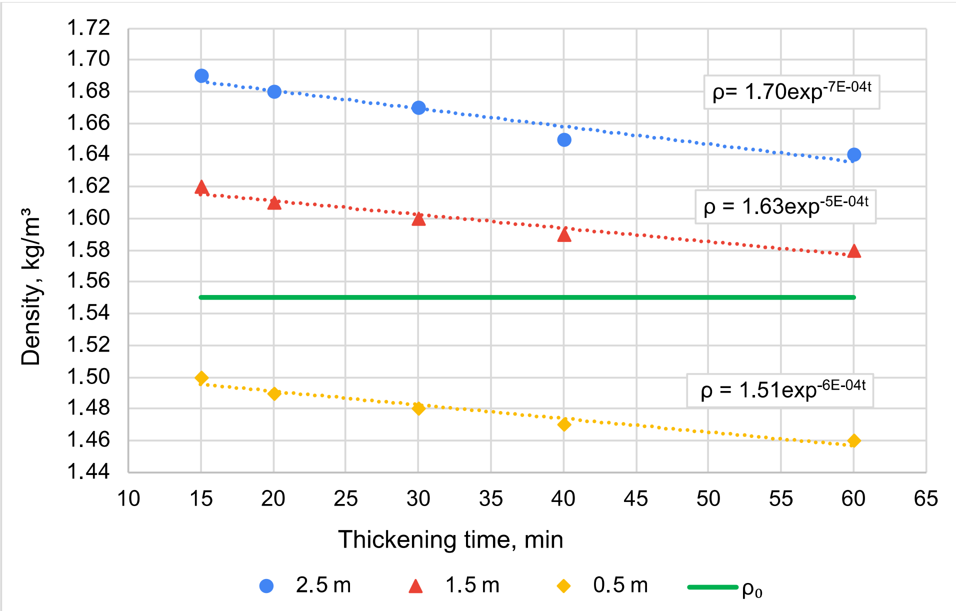


Figure 5. Kinetic curves of density as a function of time, W/C = 0.8.

It has been observed that over time, there is a decrease in density at a certain height in the column, with a maximum reduction of 2.9 %. This can be attributed to the formation of hydration products from the cement paste, as water molecules surround the cement particles and fill the empty space. Due to the small error in density measurements at each time point between T_1 and T_5, we did not consider the effect of time on sedimentation stability for the grouting mixture for up to 60 minutes after mixing. Instead, we averaged the density values of the mixture at each height for further calculations.

The results obtained allowed us to identify several trends. As the W/C ratio increases, there is a general decrease in density due to an increase in the liquid phase of the suspension. Additionally, the lower the sample, the higher the density of the cement paste, indicating a deposition of the solid phase at greater depths and, consequently, a lower sedimentation stability of the mixture within the volume of the pipe.

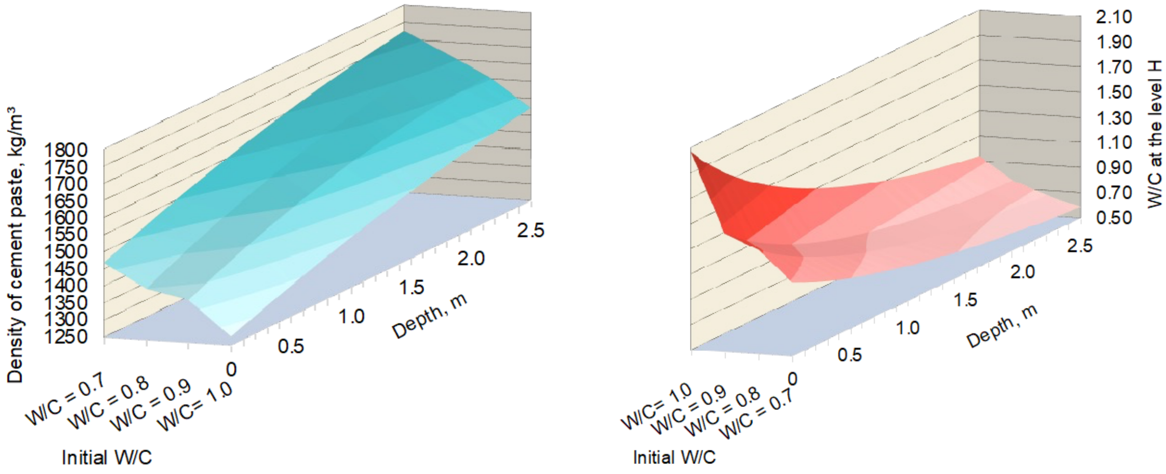
The W/C ratio also significantly influences sedimentation. As one dives deeper into the cement mixture, the density increases. This pattern is typical for suspensions, as within 40 minutes of starting the mixing process, the heaviest fractions of cement are deposited at the base of a narrow cylinder.

To determine the relationship between the density of the cement mixture, the W/C ratio and depth, considering the effect of sedimentation, the actual W/C ratio was calculated at each measuring level, which was determined as a result of the sedimentation process:

$$\frac{m_w}{m_c} = \frac{\rho_w\left(\rho_c - \rho_{cp}\right)}{\rho_c\left(\rho_{cp} - \rho_w\right)}, \qquad (10)$$

where m_w – mass of water, kg; m_c – mass of cement, kg; ρ_w, ρ_c – density of water and true density of cement, respectively, kg/m³; ρ_{cp} – density of cement paste, kg/m³ [38, 42].

For a mathematical analysis of the obtained density values and an understanding of the possibility of forecasting, based on experimental data, a graph was constructed-the surface of the dependence of W/C at the H_n level and the density of cement paste on the depth of measurements at the initial W/C = 0.7…1.0 (Fig. 6).



a b

Figure 6. The dependence of the physical parameters of the cement pastes on the sampling depth: a – dependence of density; b – dependence of the W/C ratio at the level of H_n.

Fig. 7 shows the dependencies of density and W/C at H_n at the initial W/C = 0.8, representing projections of the surface graph onto corresponding planes of the Cartesian coordinate system.

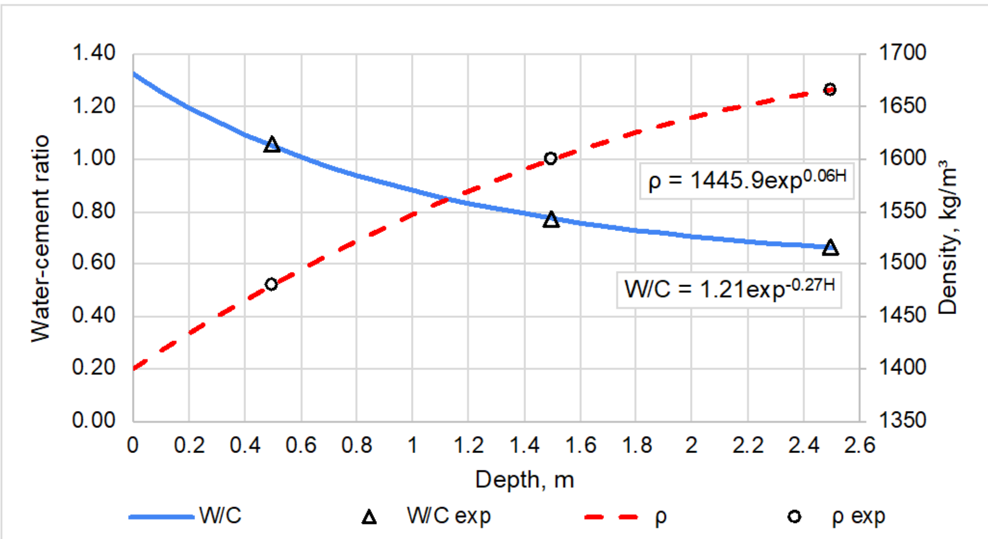


Figure 7. Dependence of density and W/C steady at the H level.

Taking into account the surface density data obtained at H = 0 m, it should be noted that as a result of sedimentation of the cement composition, the density at the bottom of the column is 14–19 % higher than that at the surface. Thus, it is possible to present a general view of the equation obtained for the dependence of cement paste density on the total depth of the column:

$$\rho_n = 2.22 \cdot \exp^{-0.42\left(\frac{W}{C}\right)_n}, \tag{11}$$

where ρ_n – density of cement paste at the level H_n; $\frac{W}{C}_n$ – actual W/C ratio calculated at the level H_n.

In order to study the stability of high-density suspensions and to test cement mixtures with W/C ratios of 0.5 and 0.6, plasticizing and/or retardant additives are recommended to be used when forming a mixture in order to solve the technical problem of well cementing under specified conditions. The results of laboratory measurements with W/C = 0.7…1.0 provide a significant level of reliability in simulating the rheology of the cement paste and offer a more accurate representation of the mixture's behavior at various readings of hydrostatic pressure and gravitational loads.

4. Discussion

Solving the inverse problem, values of a combination of indicators for two types of structures were determined from the sampling depth with initial W/C = 0.7…1.0 (Fig. 8).

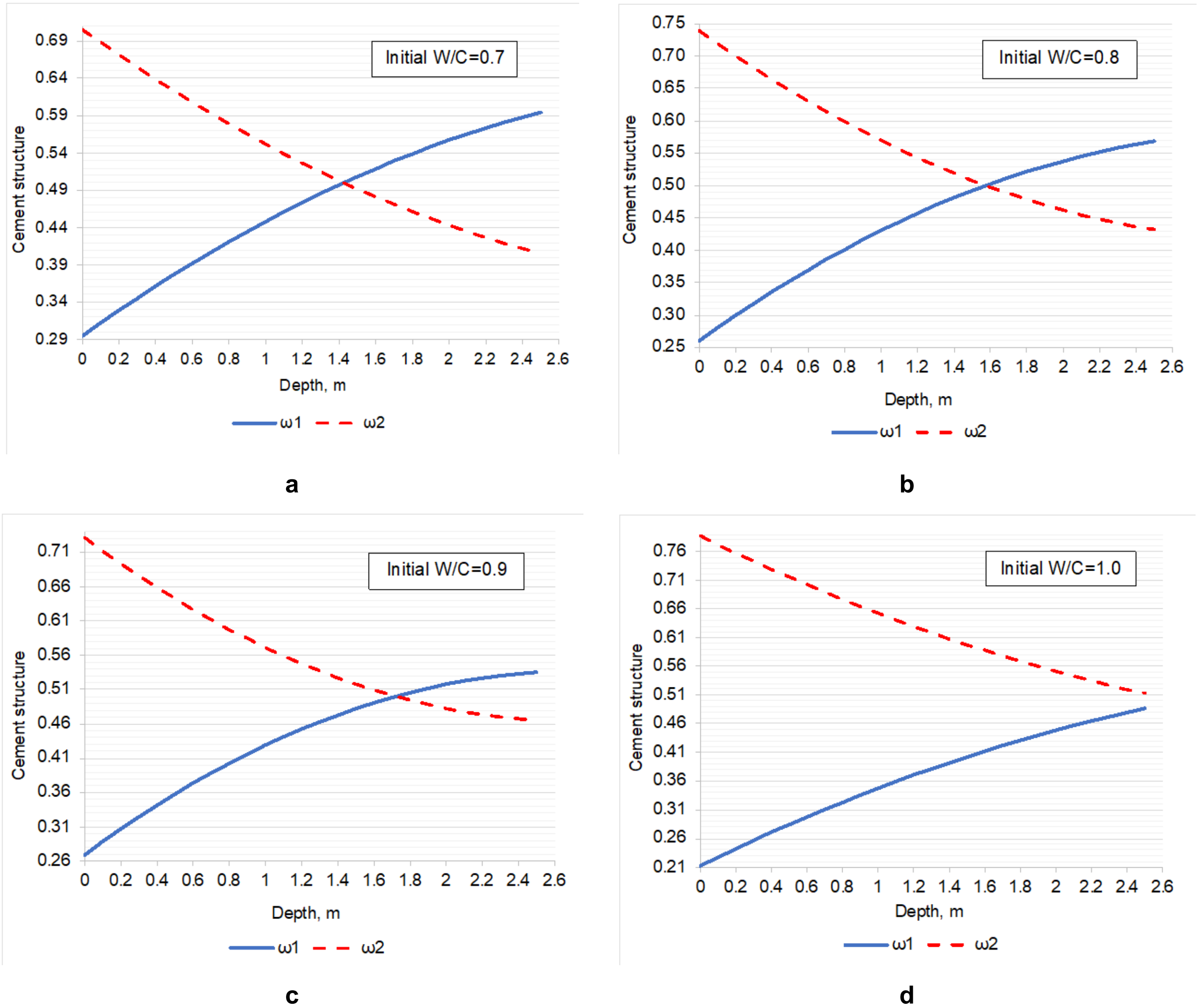


Figure 8. Dynamics of cement precipitation by two types of structures:
a – W/C = 0.7; b – W/C = 0.8; c – W/C = 0.9; d – W/C = 1.0.

Based on the calculations, it has been established that as the measurement depth increases, a phenomenon of inversion occurs, where one structural phase is replaced by a second. With increasing measurement depth, the proportion of a homogeneous structure of type I increases, while the proportion of air phase of structure type II decreases. This can be attributed to the effect of air displacement during constrained deposition of the cement slurry. It has also been determined that an increase in the water content of the cement paste leads to a structural inversion as the depth increases.

The dependencies $\omega_i = f(H)$ follow the general form:

$$\omega_i = a_{0,i} \cdot \exp\left(b_{0,i} \cdot H\right), \qquad (12)$$

where $a_{0,i}$ and $b_{0,i}$ – the coefficients, the values of which are given in Table 5.

Table 5. Dependency coefficients $\omega_i = f(H)$.

No.	W/C ratio	Structure type			
		Type I structure		Type II structure	
		$a_{0,I}$	$b_{0,I}$	$a_{0,II}$	$b_{0,II}$
1	0.7	0.33	0.27	0.70	-0.23
2	0.8	0.30	0.29	0.72	-0.22
3	0.9	0.31	0.26	0.70	-0.19
4	1.0	0.24	0.32	0.78	-0.17

When approximating the presented data with an exponential curve, a general trend of the observed relationship between the initial W/C, the structures equilibrium point ω_{inv}, and the depth at which phase inversion occurs H_{inv} can be inferred:

$$H_{inv} = 6 \cdot 10^{-5} \cdot e^{20.53 \cdot \omega_{inv}}; \tag{13}$$

$$H_{inv} = 0.54 \cdot e^{1.44 \cdot \frac{W}{C}}. \tag{14}$$

In the case of the presented experiment, the calculated point of structural equilibrium for the grouting compound with an initial W/C = 0.7 is located at ~1.53 m, and for W/C = 0.9 at ~1.84 m.

It is important to take into account the effect of sedimentation during injection of cement into a well, as it is directly related to how the composition of grouting mixtures is selected. In early stages of hydration, cement composition in upper horizons more actively penetrates adjacent rocks, causing the formation of voids, channels, and cavities until grouting mixture becomes fully manifested. In comparison with well-known standard methods for measuring fluid loss [15–17], the proposed method for measuring density of grouting mix allows simulating behavior of cement paste depending upon depth of column, solving problem of mixture penetration into rocks complicated by mining and geologic features. The results of this study confirm that the spreadability and fluidity of cement mixtures gradually increase with increasing W/C ratio, according to [19], however, the density values have an exponential character of change with depth, unlike a linear function [25]. Furthermore, the findings regarding the presence of different stages in the stratification of cement paste [24] were confirmed. According to obtained research, there is an initial phase with a high segregation rate and a subsequent one with a smoother sedimentation process throughout the depth of the column. This provides a more comprehensive understanding of the stratification process of cement paste under actual well conditions.

Due to the obtained mathematical dependencies and low errors in experimental measurements, it is possible to predict the behavior of grouting mixtures during the time period before cement sets. This study will allow us to develop an understanding of the cement hydration process in wells and avoid the negative consequences of high sedimentation of grout mixtures at the most critical time – the first hour after cement is injected into a wellbore.

5. Conclusion

Since the geometric parameters of the distribution of the grouting mixture in the annular space of the borehole are very specific, the resistance of the cement paste to deposition in the annular volume plays a significant role.

The proposed cement slurry density measurement system consists of a vertical column with sampling ports located at various sections of the pipe. Improvements to the current method of measuring cement density are required in order to predict the behavior of the material in unstable rock formations. Based on experimental data, patterns in the behavior of the cement slurry in the annulus of the wellbore were identified as a function of increasing depth. The results from these measurements indicate a direct relationship between the density of the cement sludge and its depth distribution over a given volume or, in other words, its sedimentation stability. As the water content of the mixture increases, the overall density of the final cement mixture decreases, as does the tendency for solid sedimentation to occur with air pockets. Strong density differences in the initial stages indicate a high level of water separation processes and gravitational stratification of the grouting material during the first 15 minutes following the sealing of cement clinker. As the measuring time increases to 60 minutes, the density distribution along the column becomes stable. The suggested approach for determining structure formation using two types provides a visual representation of the behavior of fully hydrated cement particles and air voids during the constrained placement of the cement mixture.

This research forms the foundation for further experiments aimed at studying the structure formation of grouting mixes with various compositions and improving their physical and mechanical properties throughout the wellbore depth to predict the technical parameters of cement rock under actual conditions.

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