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Thermal activation of Karakalpakstan margels and pozzolan properties in their low-carbon composite Portland cement

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ABSTRACT

This study explores the possibilities of using carbonate-aluminosilicate marl raw materials typical of Karakalpakstan as an additional cementing material in low-carbon composite Portland cement systems. The main goal of the study is to identify the physicochemical changes occurring during thermal activation of the marl and to evaluate its influence on hydration kinetics, microstructural development and mechanical properties of cement systems. Under control conditions, the pozzolanic activity of crude corl was enhanced by calcification and partial amorphization of aluminosilicate phases as well as structural changes. Phase structure, morphology and thermal characteristics were investigated using X-ray diffraction (XRD), scanning electron microscopy (SEM-EDS) and differential thermal and thermogravimetric analysis (DTA/TGA). Mechanical properties were evaluated using compression strength tests at various hardening times, and hydraulic activity was determined using the Student t-test. The results show that natural marl has low reactivity due to its crystalline structure, while thermally activated marl has high activity, accelerating the formation of secondary hydration products and leading to the collapse of the cement matrix. Research results show that thermally activated marl as an effective mineral additive reduces clinker consumption, increases resource efficiency, and reduces carbon emissions.

Keywords: thermally activated marl, additional cement materials, composite cement, hydration kinetics, pozzolan activity, low-carbon binders.

INTRODUCTION

Global demand for cement and environmental problems

The rapid development of the construction industry, the acceleration of urbanization, and the implementation of large-scale infrastructure projects are leading to a consistent increase in the global demand for cement. Cement is widely used in the modern construction industry as a key connector for residential buildings, industrial constructions, transportation systems and engineering infrastructure. As a result, global cement production is growing every year[1].

The cement manufacturing process, especially the firing stage of Portland cement clinker, is energy-intensive and requires high temperatures (≈1450 °C). This process releases large amounts of carbon dioxide due to higher fuel consumption and thermal breakdown of carbonate raw materials. It is estimated that the cement industry accounts for a significant proportion of global anthropogenic CO_2 emissions [2,3]. Therefore, the cement industry is one of the main anthropogenic factors influencing climate change.

From an environmental point of view, cement production causes problems such as landscape degradation, dust emissions, and intensive energy consumption, which are not only associated with gas emissions, but also from the extraction of raw materials [4]. The scarcity of natural resources and the global priority of sustainable development are pushing the industry to adopt resource-saving technologies. Therefore, reducing the share of clinker, increasing energy efficiency, and the use of alternative mineral additives are recognized as strategic directions for the development of cement technology [5].

In recent years, scientific research has been actively developing innovative approaches aimed at reducing the environmental impact of cement production. These approaches include the development of low-carbon binders, partial replacement of clinker, waste recycling, and the use of natural mineral supplements. These approaches not only reduce CO_2 emissions, but also reduce production costs and ensure efficient use of resources [6]. Therefore, environmental safety, energy efficiency, and material sustainability in modern cement technology are interrelated priorities for both scientific and practical applications.

Low carbon binders and sustainable material strategies

One of the priority areas of the concept of global sustainable development is the implementation of technologies aimed at ensuring environmental safety of industrial production processes and reducing carbon emissions. The building materials industry, especially the cement sector, is one of the sectors with the highest environmental impact due to its high energy consumption and large-scale production. Therefore, the development and practical application of low-carbon binders has become an important area of modern materials science and engineering.

In recent years, strategies aimed at reducing carbon emissions by reducing the share of clinker in cement systems have been widely used. One such approach is the use of additional cemented mineral materials. All these materials include active silicon and aluminum components, which during hydration react with portlandite and form additional bonding phases. As a result, the microstructure of the cement stone becomes denser, its mechanical properties are improved, and its durability increases. At the same time, reduced clinker consumption reduces energy expenditure during production and reduces the overall carbon load [7–9].

Thermally activated alumininosilicate mixtures are among the most promising mineral components. The partial destruction of the crystal structure and the formation of amorphous phases due to heat treatment enhance the chemical activity of mineral compounds. Such mixtures serve as nucleation centers during the cement hydration, contributing to the faster formation of hydration products. This makes it possible to accelerate the development of consistency at an early age and increase the density of the cement matrix [10,11].

Furthermore, the use of natural mineral additives not only increases the process efficiency, but also allows for the rational use of raw materials. Many studies have highlighted the possibility of producing highly active additives by processing carbonate-aluminosilicate materials obtained from natural deposits. This makes it possible to develop energy-efficient and environmentally sustainable cement compositions based on the use of local mineral resources [12]. In this context, research into the creation of low-carbon bonding systems is important not only from a scientific but also from an industrial and environmental perspective.

The potential of carbonate-aluminosilicate raw materials

In recent years, the efficient use of natural mineral resources and the use of alternative raw materials in the cement industry have become important areas of scientific research. Carbonate-aluminosilicate rocks are particularly attractive as a natural mineral system with high technological potential. The presence of clay phases in these rocks, in addition to the composition of carbonate minerals, significantly increases their physicochemical properties and makes it possible to increase the reactivity through various thermal and mechanical treatment methods. Therefore, these materials are considered promising additives in the creation of composite bonding systems [13–15].

Similar approaches have also been reported in studies on hybrid mineral additives based on industrial waste used to produce environmentally friendly cement composites [16].

During the processing of carbonate-aluminosilicate raw materials, changes in their structural condition and the formation of active phase are observed. Thermal treatment results in the

destruction of the crystal structure and partial amorphation occurs, which increases the surface energy of the mineral particles and their ability to enter the chemical reactions. As a result, such material is actively involved in the process of cement hydration, forming additional bonding phases and leading to densification of the matrix structure. Therefore, thermally activated carbonate-aluminosilicate materials are considered to be natural additives with high pozzone activity [17, 18].

Another major advantage of natural mineral supplements is their environmental effectiveness. By replacing a specific portion of clinker, these materials reduce energy consumption in cement production, reduce carbon emissions, and reduce the need for natural raw materials [19]. In addition, the use of mineral raw materials from local deposits reduces transportation costs and increases the cost efficiency of the production process. Therefore, the study of additives based on carbonate-aluminosilicate rocks is a key research focus in the development of environmentally sustainable building materials.

Jump to search Their natural hybrid composition allows the production of highly reactive active mineral additives through thermal activation. The addition of such materials to the cement system enhances hydration processes, enhances microstructural compression, and improves mechanical properties [20–22]. Therefore, the study of the properties of carbonate-aluminosilicate raw materials, in particular marl rocks, is of important scientific and practical importance in the development of low-carbon cement technologies.

Scientific interest in compounding as a hybrid mineral additive

Among the carbonate-aluminosilicate rocks, marl as a natural hybrid mineral material is of particular scientific interest. The combination of carbonate components (mainly calcite and dolomite) and aluminosilicate clay minerals makes it a unique multicomponent system. This complex mineral composition significantly enhances the physicochemical properties of marl and expands its application in various technological processes [23, 24]. Especially during thermal activation the minerals of this rock can undergo phase changes and form highly reactive components.

During the heat treatment, partial decay and amorphization of the crystalline aluminosilicate phases within the marl is observed. The process increases the surface area of the particles, the number of structural defects increases and chemically active areas are obtained. This significantly increases the pozzolan reactivity of the material, which intensifies active reactions during cement hydration [25, 26]. Such additives interact with portlandite, forming additional phases of calcium silicate hydrate (C-S-H) and calcium aluminosilicate, which leads to improved microstructural density of cement stone and improved mechanical properties [27].

Another important feature of the marl is its natural hybrid nature. The presence of carbonate components stimulates the formation of carboaluminate compounds during hydration, which increases the structural stability of the cement matrix. At the same time, the activation of clay minerals increases the number of nucleation sites in the cement system, which promotes faster and uniform formation of hydration products [28]. This leads to a reduction in porosity, reduced water absorption and improved overall mechanical properties.

Although recent studies have confirmed the efficacy of thermally activated mineral additives in cement systems, the properties of natural carbonate-clay merger rocks remain insufficiently studied. In particular, scientific data on the influence of heat treatment parameters on changes in mineral composition and whether these changes are related to cement hydration are limited [29]. Therefore, an in-depth study of the mechanisms of joint-based additives is an urgent scientific task from the point of view of cement chemistry and material science.

Current research achievements and limitations

In recent years, a lot of scientific studies have been conducted on the effectiveness of the use of additional mineral components in cement systems. These studies have shown that thermally activated aluminosilicate additives accelerate secondary hydration reactions in cement systems, leading to microstructural compression and improving long-term mechanical properties [30–32]. These additives react with portlandite to form extra C-S-H phases and calcium aluminosilicate hydrate, which increases the structural strength of the cement matrix [33].

Some researchers have argued that thermally treated mineral additives cause a slight decrease

in consistency at the initial stage of hardening. This is explained by the liquefaction effect that occurs when a part of the cement is replaced with an inert or low-reactive component [34]. However, in such systems, a stable increase in consistency is observed as a result of long-term hydration processes, indicating an activation of compounds over time [35].

Microstructural studies show that thermally activated additives reduce the volume of capillary pores in the cement matrix and produce a denser structure. This process is related to a uniform distribution of the products of the hydration and an increase in the surface area of reactive particles [36]. As a result, the permeability of the cement paste decreases, frost resistance increases, and the overall consistency improves.

However, an analysis of the available scientific literature shows that most studies have focused on additives based on pure aluminosilicates or industrial waste, while natural carbonate-clay raw materials, in particular marl rocks, have been less studied [37].

Research proposal (in the context of the Aral Sea region)

An analysis of the available scientific literature shows that research on the use of mineral additives in cement systems is mainly focused on industrial waste, artificial putsolan aggregates, and pure alumininosilicate materials. However, research on natural carbonate-aluminosilicate rocks, particularly marl raw materials, is limited, and their performance properties in cement systems have not been adequately studied [38,39]. In particular, phase changes that occur during thermal activation, the degree of amorphosis, and the mechanisms of reactive phase formation are not yet fully established.

In addition, existing studies have not well studied the effects of marl-based compounds on cement hydration kinetics, microstructure development, and mechanical properties using an integrated approach. Most studies are limited to only the analysis of mechanical or chemical parameters, and do not consider phase, microstructural and thermal analyses in an integrated way. Therefore, the functional role of such materials in the cement system and their activation mechanisms remain insufficiently studied [40].

It should be emphasized that the Aral Sea region is one of the regions that is experiencing the global environmental crisis. Efficient use of natural resources and the introduction of environmentally sustainable technologies in this region are important scientific and practical tasks [41,42]. Carbonate-aluminosilicate marl deposits found in the Republic of Karakalpakstan represent the mineral resource potential of the region; However, the possibilities of their application in industry have not yet been sufficiently studied. The production of environmentally friendly building materials based on local raw materials is also directly related to the sustainable development strategy for the Aral Sea region [43].

Therefore, increasing pozzolane reactivity of natural marl raw materials by thermal activation, detection of phase changes and a comprehensive study of the mechanisms of operation in composite cement systems is an urgent task not only from a scientific point of view, but also from the point of view of ecological and regional development.

Purpose and scientific novelty

The main purpose of this study is to study the processes of thermal activation of local marl raw materials in the Republic of Karakalpakstan, determination of their mineral and phase changes and a comprehensive evaluation of the mechanisms of action in composite Portland cement systems. This study systematically analyzes the effect of thermal treatment conditions on the degree of amorphization of aluminosilicate phases in marl, pozzolan reactivity, and cement hydration kinetics. The effect of this additive on the microstructural development and mechanical properties of the cement matrix will also be evaluated experimentally.

The practical significance of the study lies in substantiating the possibility of creating low-carbon, resource-efficient, and environmentally sustainable composite cement systems based on local mineral raw materials. Especially in the Aral Sea region, rational use of natural resources, reduction of industrial emissions and the development of environmentally sustainable building materials are important scientific and practical areas for the regional development strategy. In this context, the results of this study have important theoretical and practical significance not only for the science of cement materials, but also for the development of environmentally friendly technologies.

MATERIALS AND METHODS

Materials used in the study

Experimental studies were carried out using two main types of raw materials: Portland cement clinker produced by Karakalpakcement LLC and marl obtained from the Dout-ata deposit in the Republic of Karakalpakstan. Portland cement clinker served as a key matrix component in the production of composite cement compositions. Before use in the experiments, the chemical and mineral composition of the clinker was determined and described according to the requirements of the current standard.

Marl raw material is a natural carbonate-aluminosilicate rock, composed mainly of carbonate minerals and clay phases. In the process of laboratory preparation, the raw material was first dried, then ground in accordance with GOST 8269.0, and then turned into fine powder in a ball mill. Fraction with a particle size of less than 0.075 mm were separated using standard screens GOST 6613, which ensured the even dispersion and reactivity of the mineral additive.

The thermal activation of marl samples was carried out in a laboratory muffle furnace. Before thermal treatment, the marl raw material was dried and ground to obtain a homogeneous powder. The powdered samples were placed in ceramic crucibles and heated in a furnace at a controlled heating rate of approximately 10 °C/min until the target temperature of 500 °C was reached. The samples were kept at this temperature for 60 minutes to ensure sufficient thermal decomposition of the carbonate phases and partial amorphization of the aluminosilicate minerals.

After the storage period, the furnace was turned off to prevent rapid structural changes caused by sudden cooling and the samples were allowed to cool naturally to room temperature inside the furnace. The cooled material was then ground in a laboratory ball mill to produce a fine powder with uniform particle size distribution. The resulting thermally activated marl powder was then used as a mineral additive in the preparation of composite cement systems.

Lime was prepared and samples kept in laboratory conditions using drinking water; Water quality complies with GOST 23732. In the preparation of lime, standard quartz sand, in terms of particle size in accordance with GOST 6139, was used. The chemical composition of the clinker and marl samples was determined based on laboratory analysis (Table 1).

Both raw materials were used without further chemical treatment. This approach allowed them to retain their natural mineralogical properties and made experimental conditions as close as possible to real industrial conditions.

Conditions for the preparation of composite cement mixtures

Composite cement systems are manufactured using standard laboratory techniques. The purpose of the study was to evaluate the effects of thermally activated marl additive on the hydration process, microstructure development, and mechanical properties of cement composites. For this purpose, experimental composition, with the addition of control composition (D-0) and mineral supplements (D-15, D-20 and D-25), was prepared. The mineral additive was introduced by partial substitution of Portland cement clinker in the 10, 20, and 30 weight ranges; The water-to-cement ratio remained unchanged for all compositions.

The mixtures were prepared in a laboratory mixer. The components were first dry and then mixed by adding water until a plastic mass was formed. The mixture was placed in two layers on prismatic molds 40 × 40 × 160 mm and compacted using a swinging table. The specimen was removed after keeping in the mold for 24 hours, and then grown in a moist environment. Pressurization tests were performed on days 2, 7, and 28. For each content, at least three parallel samples were prepared and the results were analyzed statistically based on the mean values.

Test methods

Mechanical strength tests

The tensile strength of bending and compression was determined in prismatic samples of 40 × 40 × 160 mm dimensions. Tests were carried out on days 2, 7 and 28 in accordance with the requirements of GOST 310.4 and GOST 30744. After flexural testing, compression strength was determined on the semiprismatic samples and at least three parallel results were averaged for each composition. This approach ensured the reliability of the results.

Table 1. Chemical composition (wt%) of clinker and thermally activated marl samples

№	Material	LAW	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Pr.	S
1	Portland cement clinker	0.31	18.03	6.22	3.94	58.93	1.98	5.55	5.04	100.0
2	Marl (Dout-ata)	38.56	16.10	3.47	1.80	37.10	1.71	0.40	—	99.17

Determination of hydraulic activity of a mineral additive (Student's criterion)

The hydraulic activity of the mineral additive was statistically evaluated based on the results of compressive strength tests. The evaluation was carried out in accordance with the requirements of Uz DSt 336 and GOST 25094. This method is based on determining the statistical significance of the difference between the strength values of the experimental and control compositions using the Student's t-test.

It should be noted that the activity of mineral additives in cement systems can also be evaluated by other methods widely used in cement chemistry, such as the strength activity index, the Chapelle test and the Frattini test, which are widely used to assess the reactivity of putty. In this study, the Student's test was chosen because it allows for a statistical comparison of the results of mechanical strength and an indirect assessment of the hydraulic activity of the mineral additive in the cement system.

For testing, the control composition (clinker + standard sand + gypsum) and the experimental composition with the addition of a mineral mixture were prepared. Prism samples of 40 × 40 × 160mm size were taken from each compound and the compression strength was determined after a fairly defined hardening mode. For each compound, a minimum of 12 results were obtained and mean value, dispersion, and standard deviations were calculated.

Medium strength:

$$\bar{X} = \frac{1}{n}\sum_{i=1}^{n} X_i \qquad (1)$$

where: *Xi* is the consistency of the individual specimen, *N* – Sample Sony.

Dispersion and standard deviation:

$$S^2 = \frac{\sum_{i=1}^{n}(X_i - \bar{X})^2}{n-1}, S = \sqrt{S^2} \qquad (2)$$

Mesopotamian students:

$$t = \frac{|\bar{X}_1 - \bar{X}_2|}{\sqrt{\frac{S_1^2 + S_2^2}{2}}} \qquad (3)$$

where: $\bar{X}_1 - \bar{X}_2$ – the average consistency values of the control and experimental compositions, $S_1^2 + S_2^2$ – mos ravishda dispersiya qiymatlari.

The calculated value t was compared with the normative value. According to the requirements of the standard: t< 2.07 → extra low hydraulic active, 2.07 ≤ t ≤ 15 → average hydraulic activity, t > 15 → high hydraulic active.

The actual hydraulic activity in the cement system of the mineral additive was thus statistically assessed.

X-ray diffraction analysis (XRD)

The phase composition of clinker, primary marl, and thermally activated marl samples was determined using a Rigaku MiniFlex 600 X-ray diffractometer. In this study, marl samples were thermally activated at 500 °C, after which their phase composition was analyzed. X-ray analyses were performed using CuKα radiation (λ ≈ 1.5406 Å) in the scanning range 2θ = 5–70°. Before analysis, the samples were ground and homogenized to ensure sample measurements. The crystalline phases were identified by comparing the obtained diffractograms with standard database cards. The main objectives of the study were mainly aimed at determination of crystalline phases and the evaluation of structural changes resulting from thermal treatment. The main mineral phases and their relative intensity were compared using diffraction patterns (Figure 1).

Evaluation of the hydration process

In composite cement systems, the intensity of the hydration process was evaluated by determining the amount of chemically bound water. Samples taken at specific times (days 1, 3, 7, and 28) were dried and the amount of water bound was calculated based on mass changes. This

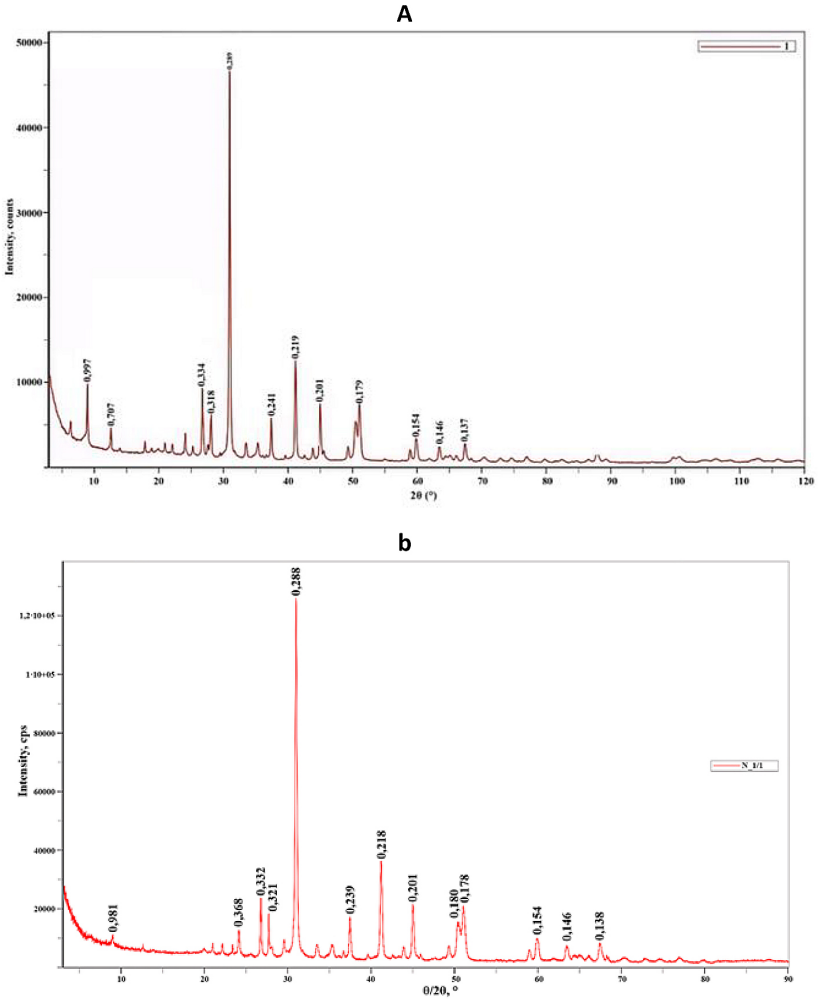


Figure 1. Initial and thermally activated XRD diffractograms of Dout-Ata mine merger

indicator indirectly characterizes the degree of hydration of cement and the pozzolanic activity of the mineral mixture.

Thermal analysis (DTA/TGA)

Synchronous thermal analysis (TG–DTA–DSC) was performed to identify the thermal stability, phase changes and fragmentation mechanisms of samples. The research was carried out on an analyzer of type STA PT 1600/LT; the device performed simultaneous measurements of TG, DTA and DSC, allowing parallel observation of mass changes and thermal effects.

During the analysis, two protective and two reactive gas streams were transmitted to the furnace chamber via software control, ensuring the gas medium change during the experiment. A vacuum system (rotor pump at 4 m^3/h, up to 10^{-2} mbar) allowed for a rapid atmosphere refresh and system cleaning.

Measurements were taken in the range from −100 °C to 1600 °C. Based on the TG curves, the mass loss stages were quantitatively evaluated and interpreted in relation to DTA/DSC signals. The results served to determine the effects of mineral additive on dehydration, decarbonization, and phase transition processes in a composite cement system (Figure 2).

Microstructural analysis (SEM-EDS)

The microstructure of the samples was studied using scanning electron microscopy (SEM) method. The analyses were performed on an electron microscope EVO 10 (ZEISS, Germany). The sample surfaces were pre-dried under vacuum conditions, covered with an electrically conductive coating and fixed on a standard holder (Figure 3). The images were recorded in high vacuum mode at an acceleration voltage of 20 kV, a working distance of 14.0 mm, and a magnification parameters of ×160, using a second-by-second circuit detector (SED).

To determine the element composition, an energy-dispersed X-ray spectroscopy (EDS) system

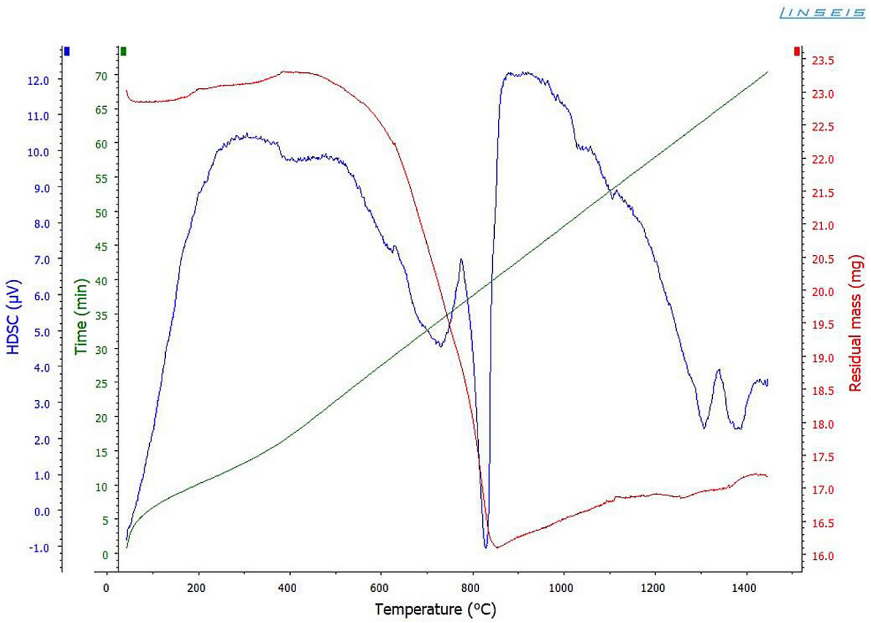


Figure 2. DTA/TGA curves of dout-father marl samples

integrated into the microscope was used. The spectra were recorded under conditions of 30 s real time, ≈3 % dead time, and ≈3928 CPS compute rate. The amount of elements was quantified based on the ZAF correction algorithm.

The results of the EDS showed the presence of O, Ca, Si, and C as the main elements in the analyzed region, as well as small amounts of Fe, Al, Mg, and K. Microstructural observations allowed to evaluate particle morphology, porosity level, and aggregation properties, and EDS data served as the basis for the identification of potentially reactive components of mineral additive in the cement hydration process.

RESULTS AND DISCUSSION

Analysis of the chemical composition of raw materials

The chemical composition of the daut-ata clinker and marl samples was determined by laboratory analysis; The results are presented in Table 1. A high CaO content of clinker indicates that it is sufficient to form the basic clinker minerals, while the presence of oxides SiO_2, Al_2O_3 and Fe_2O_3 confirms the presence of the components required for the formation of silicate and aluminum phases.

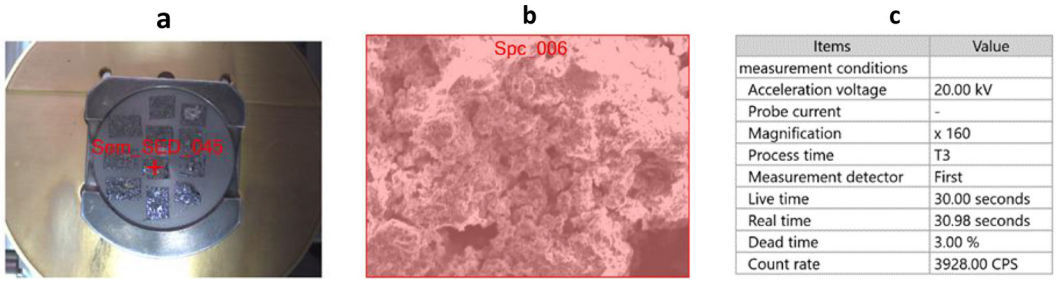
The composition of the marl is dominated by carbonate components, and a high LOI value indicates the presence of thermally degradable carbonate minerals. However, the determination of oxides of SiO_2 and Al_2O_3 shows its aluminosilicate property and increases the possibility of pozzolan properties after thermal activation. Overall, the results confirm the presence of the necessary chemical conditions for the use of marl as a mineral additive in composite cement systems.

Analysis of morphological features

The morphology of the original and thermally activated marl samples was evaluated using visual and structural analysis (Figure 4). The starting material is composed of large, dense and irregular particles, which shows a crystalline structure and low dispersion resulting in a low chemical reactivity.

After thermal treatment, the particles are finely dispersed, produce irregular shapes and a rough surface, which is explained by the breakdown of aggregates due to decarbonation and structural decay. Increased dispersion increases the surface area and number of nucleation sites during hydration. As a result, the cement matrix becomes denser, the porosity decreases, and the mechanical stability increases.

In consequence, thermal activation radically changes the particle structure and surface properties of the corl and improves its effectiveness as a putzolanic compound.



Items	Value
measurement conditions	
Acceleration voltage	20.00 kV
Probe current	-
Magnification	x 160
Process time	T3
Measurement detector	First
Live time	30.00 seconds
Real time	30.98 seconds
Dead time	3.00 %
Count rate	3928.00 CPS

Figure 3. SEM–EDS analysis workflow and representative microstructure of marl sample (a) sample mounted on SEM holder;(b) SEM micrograph of analyzed area; (c) measurement conditions and acquisition parameters

Influence of thermal activation on phase structure (XRD analysis)

The results of the XRD analysis showed that significant mineralogical changes occur between the initial and heat-treated states of the Daut-Ota merger at 500 °C (Figure 1). The diffraction pattern in the first sample is characterized by sharp to high intensity sediments which are mainly associated with well-crystallized carbonate minerals (mostly calcite) and some clay phases. Such narrow peaks indicate a high order crystal lattice and low reactivity in the material's natural state.

After heat treatment, the diffraction pattern changes significantly: the intensity of the carbonate phase peaks decreases and some reflections expand or disappear. This is explained by the deterioration of the crystalline structure, partial decarbonation and amorphization of the aluminosilicate phases as well as an increase in the chemical activity of the material. The formation of amorphous phases is important for the cement system since they function as nucleation centers during hydration due to the high surface energy and the number of active centers. As a result, the formation of C-S-H phases and calcium aluminosilicate hydrate is accelerated, the compression and strength of the cement matrix increases.

The weakening of carbonate peaks indicates partial decomposition of the phases containing $CaCO_3$, which facilitates the separation of reactive calcium components and provides for their active participation in hydration. Overall, the XRD results confirm that thermal activation converts corl from inert natural rock to highly reactive cementing material and provides a mineralogical basis for the observed increase in hydraulic activity and mechanical properties.

Hydraulic activity of the primary marl

According to GOST 25094, the activity of mineral additives is determined using the Student t-test. This indicator is determined by evaluating the statistical significance of the difference between

Figure 4. Morphological structure of primary (a) and thermally activated (b) marl samples

the compression force values of the supplement and control composition (Table 2). This approach makes it possible to determine the true reactivity of the mineral additive in the cement system.

As can be seen from the table, the grinding rate of the gravel, sand and gypsum samples is in the standard range, and clinker is relatively finely clinked, which gives it a high reactivity.

To evaluate the hydraulic activity of the mineral additive, the compression strength of the control mixture (clinker + sand + gypsum) and the marl admixture (clinker + marl + gypsum) were compared (Table 3).

According to the table data, the mean compressive strength of the compositions with the marl addition was 3.89 MPa, which is 2.68 MPa (40.8%) lower than in the control sample. This result indicates that the merger does not have sufficient hydraulic activity in the cement system in its natural state.

This situation indicates that the necessary conditions for the assessment on the basis of the Student t-test in accordance with the requirements of GOST 25094 are not met; That is, the difference in consistency values does not statistically prove that the additive works as an active mineral component. Therefore, the naturally occurring marl from the Davut-Ata mine is unsuitable for use as a mineral additive to cement.

The obtained results indicate that the aluminosilicate phases in the marl are present in the crystalline state and hence their chemical reactivity is low. Therefore, thermal activation is necessary to increase the hydraulic activity of the material. This processing leads to partial fragmentation of carbonate components and amorphization of aluminosilicate structure, which in turn increases material reactivity and speeds up pozzolanic reactions within cement system.

As a result, the hydraulic activity of the original marl samples is low and insufficient to be used directly. However, thermal treatment can significantly improve their functional properties. Therefore, the study of the properties of the marl samples after thermal activation at different temperatures is of great scientific importance at the next stage.

These results scientifically substantiate the fact that thermal activation is a necessary technological step for effective use of marl as an active mineral additive.

Hydraulic activity of thermally activated marl

To determine the hydraulic activity of heat-treated marl samples, statistical calculations were performed using the same methodology for experimental mixtures prepared using calcined marl at 500 °C (Table 4). The results of the calculation are presented in which the average compressive force of the control samples was 5.48 MPa, while for the heat-activated mixtures stored by the marl, this value was 7.29 MPa.

1. Average value (arithmetic mean):

For each content:

$$\bar{X} = \frac{1}{n}\sum_{i=1}^{n} X_i \tag{4}$$

where: $n = 22$

- Nazorat (Garbage No1): $\bar{X}_p = 5.48$ MPa
- Extra (Smec No3): $\bar{X}_a = 7.29$ MPa

2. Og‘ish (deviation):

$$\Delta X_i = X_i - \bar{X} \tag{5}$$

3. Deviations square:

$$(\Delta X_i)^2 = (X_i - \bar{X})^2 \tag{6}$$

4. Dispersion (Equation Dispersion):

$$S^2 = \frac{\sum_{i=1}^{n}(X_i - \bar{X})^2}{n-1} \tag{7}$$

Your scheduled gatherings:

- For Smec No1: $\sum(X_i - \bar{X})^2 = 0.5904$
- For Smec No3: $\sum(X_i - \bar{X})^2 = 0.2206$

Table 2. Starting material grind indicators

№	Material name	Residual in sieve No008, % (norm)	Actual value
1	Marl	13–15	14.0
2	Portland cement clinker	13–15	8.0
3	Standard sand	13–15	14.0
4	Gypsum stone (grade 2)	4–6	5.0

Table 3. Comparative indicators of strength of binder compounds

Pattern ID	Control mixture (sand), MPa	Marl mixture, MPa
1	5.44	4.07
2	7.36	5.14
3	6.20	4.24
4	7.20	4.01
5	6.64	4.00
6	6.60	4.00
7	5.44	4.24
8	6.64	4.01
9	6.60	4.00
10	7.36	4.07
11	6.20	5.14
12	7.20	4.00
S	**78.84**	**46.72**
Average	**6.57**	**3.89**

Of these:

$$S_p^2 = \frac{0.5904}{11} = 0.0537 \tag{8}$$

$$S_a^2 = \frac{0.2206}{11} = 0.0201 \tag{9}$$

5. Standard deviation:

$$S = \sqrt{S^2} \tag{10}$$

$$S_p = \sqrt{0.0537} = 0.232, \quad S_a = \sqrt{0.0201} = 0.142 \tag{11}$$

6. Student mezoni(t-kriteriy):

For two independent selections (Smec No1 and Smec No3):

$$t = \frac{\left|\bar{X}_a - \bar{X}_p\right|}{\sqrt{\frac{S_a^2}{n} + \frac{S_p^2}{n}}} \tag{12}$$

With your values:

$$t = \frac{|7.29 - 5.48|}{\sqrt{\frac{0.0201}{12} + \frac{0.0537}{12}}} \approx 23.09 \approx 23.10 \tag{13}$$

7. Interpretation (according to criterion 336 of the UzMSt):

- T<2.07 → Low Hydraulic Activity
- 2.07≤ t ≤15 → average hydraulic activity
- High hydraulic activity → T>15

You have: t ≈ 23.10 > 15 → thermally activated marl (500 °C) has a high hydraulic activity.

Based on the results obtained, it turned out that in the calculation of the student t-test value t = 23.10, which is significantly higher than the standard limit (t ≥ 15) according to the requirements of the Uzbek T 336. This result confirms that the difference in strength indicators is statistically significant and that the joint exhibits real hydraulic activity in the cement system. Therefore, based on its hydraulic activity, the thermally activated marl sample is classified as a highly active mineral compound.

A comparison of the results of natural and heat-treated marl shows that the heat treatment process significantly increases the reactivity of the mineral additive. This is explained by the partial decay of carbonate components, the release of structural water and the transition of aluminosilicate phases into the amorphous state. As a result, the number of active reaction sites on the material surface increases and the formation of secondary binders during cement hydration is accelerated.

Analysis of mechanical strength results

To assess the effect of average clinker replacement on the hydration process and mechanical properties of composite cement systems, the content of thermally activated marl used as a mineral additive was selected as 15%, 20% and 25% by weight of cement. Such replacement levels are usually used in the study of mixed cements and allow determining the optimal amount of the additive without significantly reducing the mechanical strength of the binder system. The results of physical and mechanical tests of the studied compositions are presented in (Table 5).

During the initial hardening phase (2 days), relatively low consistency values were observed in compositions containing mineral additives compared to the control sample. This phenomenon is explained by a decrease in the proportion of clinker phases and a relatively low reactivity of the additive in the initial period. However, as the hydration process continued, a significant increase in the rate of increase in consistency was observed (Figure 5).

Results after 7 days show that thermally activated marl undergoes active pozzolanic reactions in the cement system. During this period,

Table 4. Results of compression of the control and thermally activated marl added compositions and statistical indicators for the Student criterion

№.	Specimens from mixture №1 (clinker + sand + gypsum)	Parameter	Parameter	Specimens from mixture №3 (clinker + marl №3 + gypsum)	Parameter	Parameter
	XP	$X_p - \bar{X}_p$	$(X_p - \bar{X}_p)^2$	X_a	$X_a - \bar{X}_a$	$(X_a - \bar{X}_a)^2$
1	5.25	0.23	0.0529	7.29	0.00	—
2	5.33	0.15	0.0225	7.90	−0.61	0.3721
3	5.25	0.23	0.0529	7.34	−0.05	0.0025
4	5.50	−0.02	0.0004	7.17	0.12	0.0144
5	5.81	−0.33	0.1089	6.77	0.52	0.2704
6	5.72	−0.24	0.0576	7.24	0.05	0.0025
7	5.25	0.23	0.0529	7.29	0.00	—
8	5.33	0.15	0.0225	7.90	−0.61	0.3721
9	5.25	0.23	0.0529	7.34	−0.05	0.0025
10	5.50	−0.02	0.0004	7.17	0.12	0.0144
11	5.81	−0.33	0.1089	6.77	0.52	0.2704
12	5.72	−0.24	0.0576	7.24	0.05	0.0025
S	**65.76**		**0.5904**	**87.49**		**0.2206**
Mean ($\bar{X}$)	**5.48**			**7.29**		

Table 5. The effect of thermally activated marl additive on physico-mechanical properties of Portland cement

No.	Additive content (Calcined marl 500 °C), wt.%	W/C	Setting time, h–min		W/C	Strength (flexural/compressive), MPa after:		
			Initial	Final		2 days	7 days	28 days
1	D-0	0.28	3–16	3–55	0.5	3.07 / 21.96	4.70 / 28.13	5.65 / 34.58
2	D-15	0.27	3–15	4–08	0.5	3.50 / 19.78	4.45 / 26.16	5.45 / 34.97
3	D-20	0.26	3–38	4–14	0.5	2.90 / 18.97	4.15 / 23.05	5.05 / 34.17
4	D-25	0.26	3–29	4–22	0.5	2.55 / 14.79	3.85 / 20.14	4.30 / 23.76

as a result of its interaction with portlandite, additional phases of calcium silicate hydrate (C-S-H) are formed, the microstructure of the cement paste becomes denser and its mechanical stability increases.

The 28-day results showed that the long-term consistency of the supplement approached or exceeded that of the control samples in some proportions. This process is associated with a more active reaction of the mineral additive at a late hydration phase and the formation of a secondary connecting phase.

The results showed that with increasing extra quantity, the consistency values were maintained in an optimal range. Excessively high replacement levels can reduce the continuity of the cement matrix and adversely affect structural strength. Therefore, the optimization of the amount of mineral additives in composite cement is technologically and scientifically important.

Overall, the results obtained indicate that thermally activated marl additive activates late hydration processes in the cement system and serves to enhance long-term consistency. This provides the scientific basis for the use of this material as an effective mineral additive in the production of composite Portland cement.

Hydration process analysis

The progress of the hydration process in composite cement systems was evaluated on the basis of changes in the amount of chemically bound water over time. This parameter is an important indicator that indirectly characterizes the level of cement hydration and the pozzolanic activity of the mineral additive (Table 6).

The results of the study showed significant differences in the hydration kinetics in the samples of different compositions. The results

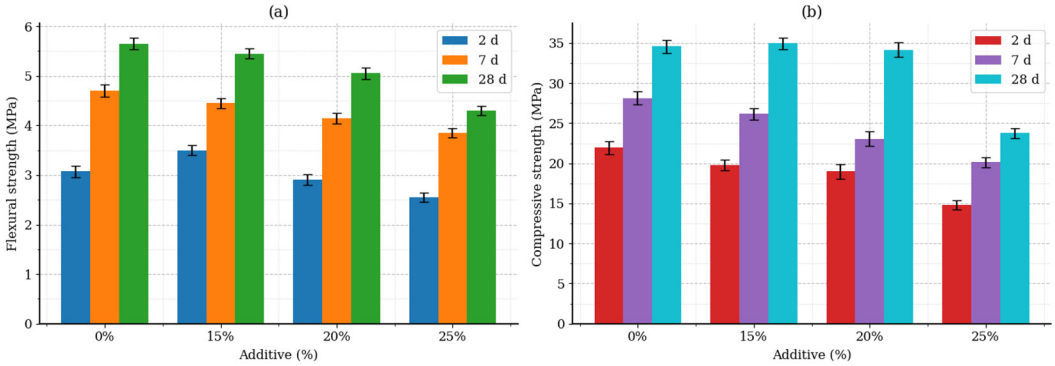


Figure 5. Effect of thermally activated marl content on the flexural (a) and compressive (b) strength of composite cement at different curing ages

obtained show a variation in the amount of water bound over time (Figure 6).

As can be seen from the graph, due to the high reactivity of the clinker phases, the hydration process in the control structure is initially faster. In compositions with the addition of mineral supplements, hydration is relatively slower at the initial stage. This is due to the fact that an additional component acts as an inert phase in the initial period.

However, over time, the rate of growth in the amount of water bound in the systems with which additives are added increases significantly. This process is explained by the formation of secondary bonding phases as a result of the reaction of amorphous aluminosilicate phases in the thermally activated marl with hydration products. The additional formation of C-S-H gel through the consumption of portlandite and pozzolanic reactions leads to density of the cement matrix.

Over a 28-day period, the difference between additive and control compositions decreased or the additive compositions predominated in some proportions. This indicates an active participation of the mineral additive in the process of long-term hydration.

Overall, the results obtained confirm that thermally activated marl additive improves the performance properties of composite material by enhancing hydration processes in cement system and providing structural compression.

Microstructure of analysis results (SEM)

Microstructural properties of composite cement systems were studied using scanning electron microscopy (SEM). The main objective of the study was to determine the effect of thermally activated marl additives on cement matrix structure, morphology of hydration products, and capillary porosity levels.

The microstructure of the original marl samples (Figure 7a) is characterized by a predominance of particles with relatively dense aggregation, large fractions, and smooth surfaces. This morphology is characteristic of the natural carbonate-clay rocks and is associated with low reactivity. The slipperiness and compactness of particle surfaces show a limited reactive surface area, which leads to lower the intensity of chemical interactions during the cement hydration.

Specimens collected after heat treatment (Figure 7b) showed a marked morphological change. Micro cracks, porous structural elements and scattered fragmentation were observed on the particle surfaces. These changes occur by partial splitting of carbonate phases, the release of structural water and the release of the crystal lattice. As a result, the active surface area of the material increases and the number of potential reaction sites increases.

Table 6. Rate of chemically bound water during hardening of Portland cement with thermally activated marl (500 °C)

№	Amount of additive (Marl calcined at 500 °C), wt.%	Chemically bound water content after (days):			
		1	3	7	28
1	D-0	13.73	16.10	16.27	21.38
2	D-15	22.25	23.20	25.05	25.62
3	D-20	21.40	26.48	26.99	26.99

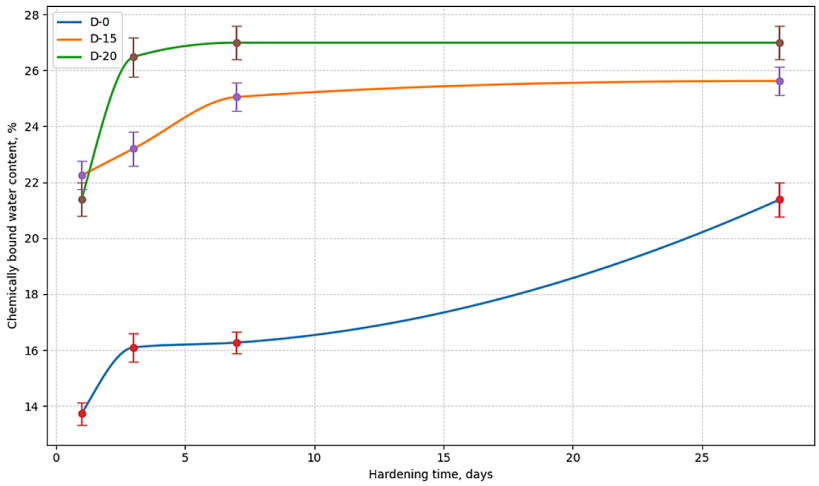


Figure 6. Changes in the amount of chemically bound water in composite cement compounds during the slurry

These microstructural modifications, which result from thermal activation, are one of the major factors that enhance the pozzolanic activity of the material. Well-developed surface structure increases the number of nucleation sites in the cement system and accelerates the formation of calcium silicate hydrate (C-S-H) and other secondary hydration products. This process results in microstructural compression of cement paste, lower porosity and increasing mechanical strength (Figure 8).

SEM observations show that thermal activation significantly changes the microstructure of marl particles. After thermal treatment, the particle surface becomes rougher and more porous, which may be due to the decomposition of carbonate phases and partial structural disruption of aluminosilicate minerals. These structural changes increase the specific surface area of the particles and create additional active sites for chemical interactions during cement hydration. As a result, thermally activated marl acts as nucleation sites for the formation of hydration products such as calcium silicate hydrate (C-S-H), contributing to the densification of the cement matrix and improving its long-term mechanical properties.

In general, the SEM analysis results confirm the structural activation of thermally activated corgel and the positive influence on the formation of connective phases in the cement system. This scientifically substantiates the possibility of this additive being used as an effective functional component in the production of composite cement.

The results of extensive studies have shown that thermally activated marl additive has a

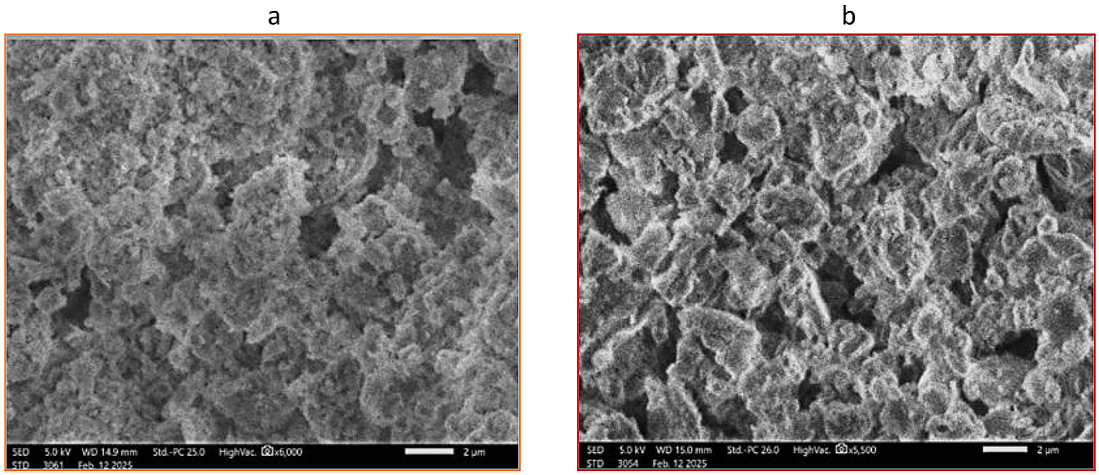


Figure 7. SEM microstructure of the Dout-Ata Merge:
(a) Initial state; (b) the thermally activated state

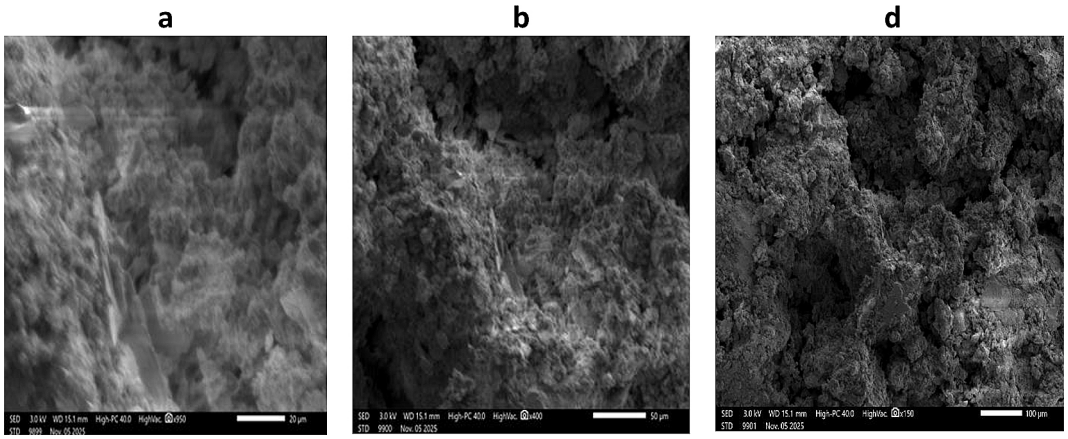


Figure 8. Microstructure of cement stone with 20% thermally activated marl additive: (a) 1 day; (b) 7 days; (c) Period of 28 days

multifactorial mechanism of action in the composite Portland cement system. Chemical analysis confirms the co-existence of carbonate and aluminosilicate components in marl and characterize it as a natural hybrid mineral additive. Increase of the percentage of amorphous phases during heat treatment was determined by X-ray survey results and is related to an increase in reactivity of material.

SEM observations revealed an increase in particle surface area and microporosity, which led to increased reactive surface area and an activation of the hydration process.

The results obtained indicate that thermally activated marl can be considered as a promising mineral additive for the production of composite Portland cement. From an industrial perspective, partial replacement of clinker with thermally activated marl can help reduce clinker consumption, which is one of the main factors affecting the environmental impact and energy intensity of cement production.

In addition, the use of locally available marl deposits can improve resource efficiency and reduce raw material transportation costs. The formation of additional hydration products observed in this study also indicates that the mechanical properties of the binder system can be sufficiently maintained even with partial replacement of clinker. Therefore, the use of thermally activated marl can be a promising approach for the development of low-carbon cementitious materials in industrial cement production.

Therefore, thermally activated marl can be evaluated as a promising mineral additive in the production of low-carbon composite cements based locally sourced.

CONCLUSIONS

As a result of the research, the following conclusions were reached:

- Dout-ata mine is a natural mineral raw material of carbonate–aluminosilicate nature and exhibits active mineral additive property after thermal activation;
- Thermal activation of marl at 500 °C led to partial decomposition of crystalline phases and the formation of amorphous aluminosilicate structures, which increased the pozzolanic activity of the material;
- Microstructural analyses showed an increase in microporosity by coalescence of the particle surface after firing, which enlarges the reaction surface and accelerates the hydration of cement;
- Introduction of thermally activated marl in an amount of 15–20% of cement composition ensured optimal consistency and formation of dense microstructure, while high content slowed down the hydration process;
- Statistical evaluation based on the student criterion showed that the differences in robustness were statistically reliable and confirmed the true hydraulic activity of the insert;
- A comparison of natural and burnt marl results showed that unthermally treated marl is low-active, while thermal activation converts it into a high-performance mineral additive;
- The results obtained confirm that the use of locally thermally activated marbles is a promising and scientifically grounded direction in the production of low-carbon composite Portland cement.

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