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## Burnt rock of the coal deposits in the concrete products manufacturing

### Горелая порода угольных месторождений в производстве изделий из бетона

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**Key words:** burnt rock; slagheaps ash; mine dumps; coal deposits; active mineral addition; concrete; cement; cement stone; fly ash

**Ключевые слова:** горелая порода; зола терриконов; шахтные отвалы; угольные месторождения; активная минеральная добавка; бетон; цемент; цементный камень; зола уноса

**Abstract.** The paper presents the results of the comprehensive study of the composition, properties and structure of the burnt rock found at the mining dumps of Cheremkhovo coal deposit (Irkutsk region, Russia). In the course of laboratory research, which included optical crystallography, there have been established the reasons accounting for the extent of burnt rock activity when in contact with cement during the cement stone formation. The benefit of the burnt rock as an active mineral additive, compared to the fly ash used by the cement plants, was confirmed as well. The optimal ratio of cement and burnt rock in concrete mixtures was determined experimentally. Likewise, the most effective method of using burnt rock as an active mineral additive was developed as the result of semi-industrial tests when the sample was subjected to the pressing and steam treatment. Finally, the impact such an additive can have on the production cost have been calculated.

**Аннотация.** В работе представлены результаты исследований состава, свойств и структуры горелой породы шахтных отвалов Черемховского угольного месторождения, расположенного в Иркутской области. В ходе лабораторных исследований и кристаллооптического анализа установлены причины, объясняющие степень активности горелой породы в контакте с цементом, при формировании цементного камня. Показано преимущество использования горелой породы в качестве активной минеральной добавки, по сравнению с золой уноса, традиционно применяемой цементными заводами. Опытным путём определены оптимальные соотношения цемента и горелой породы в бетонных смесях. В ходе полупромышленных испытаний в режиме прессования и тепловлажностной обработки определён наиболее эффективный способ использования горелой породы в качестве активной минеральной добавки. Установлены границы её влияния на себестоимость продукции.

### Introduction

At present time, concrete products lay the foundation for the construction industry. Hence, improving the quality of the concrete products while reducing the cost of their manufacturing is a task of the utmost importance. An extensive number of theoretical research as well as applied studies are dedicated to this issue [1–4].

The prime cost of the concrete (up to 70%) depends on the price of its main component – the cement, as it is the qualities of the cement that account for the strength and durability of the concrete constructions [5].

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The use of active mineral additives in the cement production can reduce the energy and material costs associated with the commercial output. Moreover, it allows to improve both construction and technical properties of the product [6]. When cement is mixed with water, active mineral additives (AMA) react with calcium hydroxide, which is released as the result of hydration of tricalcium silicate, and form water-insoluble calcium hydrosilicates. Thus, together with conservation of the clinker part, mineral additives provide the cement with a number of special properties such as increased impact strength, reduced water permeability and efflorescence, and so on [7].

It is also worth noting that as technogenic AMA are inherently wastes and by-products of various mining and processing industries, they are of considerable interest in terms of accessibility, cost and environmental safety [8, 9].

Based on the extent of AMA activity, special attention should be paid to the burnt rock of the coal deposits, as that is one of the most widespread types of technogenic waste. During the mine exploitation of the coal deposits, dead rock containing traces of coal is stored in the artificial mounds called the slag heaps [10, 11] (Figure 1).



**Figure 1. Slag Heap**

Spontaneous coal combustion in slag heaps with temperatures ranging from 600°C to 850°C results in the formation of ash rich in active ingredients [6,12,13]. Such combustion processes inside the slag heap tend to last for 35–40 years. During that time, the entire internal massif reaches a homogeneous state (i.e. the chemical composition of the samples is identical to those collected at all sampling points of the slag heap).

Nine slag heaps of a total volume of about three million cubic meters are located in the Cheremkhovo area of Irkutsk region, Russia. Multiple slag heaps of similar appearance and chemical composition are located in the regions where coal was extracted via deep mining technique (Russia, Ukraine, China, Czech Republic, Spain, etc.) [14–17].

The paper presents the results of the profound study of the burnt rock of the Cheremkhovo coal deposit. It also sets to propose technologies for the use of the burnt rock of the aforementioned mine dumps as an acidic active mineral additive to the cement and the concrete products based on it.

The study is intended to provide a comprehensive explanation of the increased extent of the burnt rock activity, develop a technology for its use as an active mineral additive in the concrete production, and determine the optimum quantity of the cement to be replaced with the burnt rock without reducing the strength parameters of the end product.

To achieve this goal, accomplished tasks include the following:

- research of the physical properties and chemical composition, and the structure of the burnt rock;
- measurement of the extent of the burnt rock activity as well as its place in the range of active mineral additions;

– determination of the effect the burnt rock has on the formation of the cement stone as well as its subsequent qualitative characteristics; establishing of the optimum degree of replacement of cement by burnt rock under normal conditions of hardening;

– conducting crystal optical studies of the structure of the cement stone formed with the addition of the burnt rock to identify the possibility of new formations, namely, the forms and the types of replacement as well as interaction of the formed phases in the general structure;

– conducting semi-industrial tests with a view to replace the cement with the burnt rock at the operating plant; determination of the optimum degree of cement substitution under the conditions of vibrocompression and stream treatment.

### *Materials and Methods of research*

Crystal-optical studies of the burnt rock as well as the cement stone with the burnt rock additive were performed with a digital polarizing microscope Altami Polar 1. X-ray diffraction analysis was conducted to study the phase composition of the burnt rock, with the help of the Shimadzu X-ray diffractometer XRD-7000. Qualitative and quantitative analyses of the chemical composition of the samples was carried out by the Bruker AXS S4 PIONEER X-ray fluorescence spectrometer.

To conduct laboratory tests of the burnt rock activity as well as its mechanical properties in the cement composition, the following regulatory documents were referred to:

- Russian State Standard GOST 310.1–76\* “Cements. Test methods. General” (EN 197–1);
- Russian State Standard GOST 310.2–76\* “Cements. Methods of grinding fineness determination” (EN 196–6);
- Russian State Standard GOST 25094–94 “Active mineral additions for cements. Methods of testing” (EN 934–2);
- Russian State Standard GOST 310.4–81 “Cements. Methods of bending and compression strength determination” (EN196–1).

All samples were dried to achieve stationary weight. Burnt rock drying was carried out at the temperature of  $105 \pm 5$  °C; gypsum stone drying (as to prevent its dehydration) was completed at the temperature of  $68 \pm 2$  °C. Further, the materials were pebbled together. To study their grindability, the grinding time in the mill was altered (30, 60 and 100 min). The grinding quality was evaluated based on the specific surface and screen sizing (sieve No. 0071 was used). The surface area of the grains was 5500–6500 cm<sup>2</sup>/g (to compare, the surface area of the cement grains subjected to the same grinding technique lies in the range of 3000–4000 cm<sup>2</sup>/g).

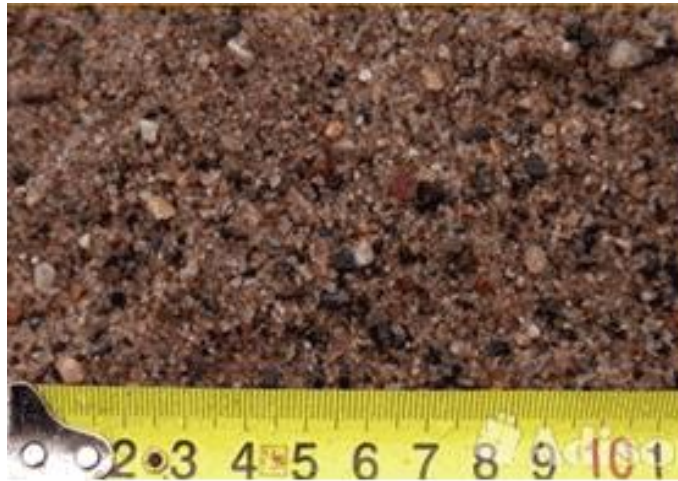
The materials milled to the desired fineness were dried again for one hour period and then placed in hermetically sealed vessels. The vessels with the powdered substance, prior to their use in the compositions, were stored in a chamber where calcium chloride was layered on the oven-tray to maintain the low level of relative air humidity.

Water-to-cement ratio was determined in accordance with Russian State Standard GOST 30744 “Cements. Methods of testing with using polyfraction standard sand”. The samples were prepared from the mortar mixtures based on Russian State Standard GOST 310.4–81 (EN 196–1). Until the testing, the samples were stored in the chamber with a relative air humidity of 95–98 %.

### *Results and discussion*

#### *Research into the composition, properties and the structure of the burnt rock*

The burnt rock is comprised of a loose granular material; the color varies from crimson to light orange. The grains of the rock are gravelly shaped and structured in a layered or lamellar way (Figure 2).



**Figure 2. Burnt rock facies**

Tables 1 and 2 show the chemical composition and the properties of the burnt rock found at the mine dumps of the Chermkhovo coal deposit.

**Table 1. Average chemical composition of the burnt rock of mining dumps of the Chermkhovo coal basin deposits**

Oxides content, (wt%)									
SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	MnO	TiO <sub>2</sub>	SO <sub>2</sub>
65.60	18.70	6.03	2.20	2.60	1.90	0.35	0.03	0.30	0.29

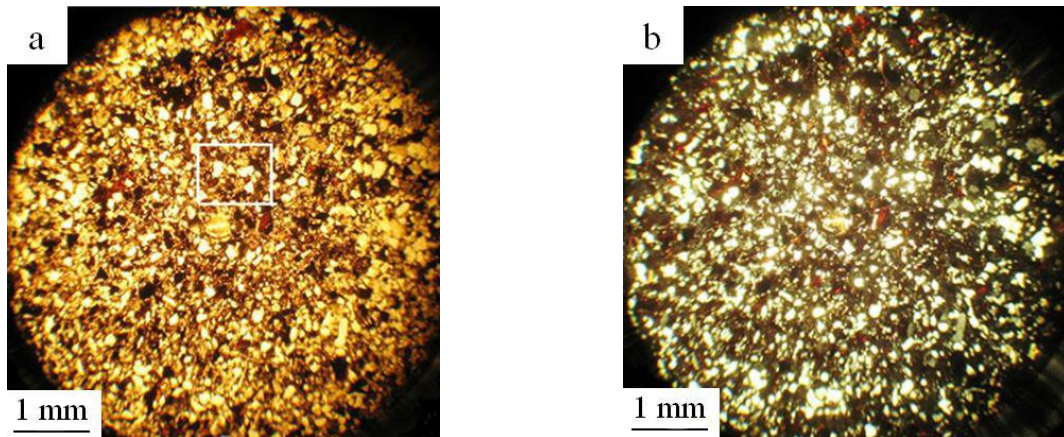
Note: the chemical composition of the slag heaps ash is reproduced from the analysis performed in the analytical department of the Vinogradov Institute of Geochemistry – Siberian Branch of the Russian Academy of Sciences

**Table 2. Averaged physical and mechanical properties of the burnt rock of mine dumps of the Chermkhovo coal basin deposits as determined by Russian State Standard GOST 8269.0–97**

Characteristics, dimension	Values
Natural humidity, (wt%)	8.80
Bulk density, kg/m <sup>3</sup>	1085
Grain content, size 0-5 mm (wt%)	58.60
Grain content, size 5-70 mm (wt%)	41.40
True density, g/cm <sup>3</sup>	2.58
Strength in a cylinder, g/cm <sup>2</sup>	17.70
Water saturation, (wt%)	15.30
Softening coefficient	0.69

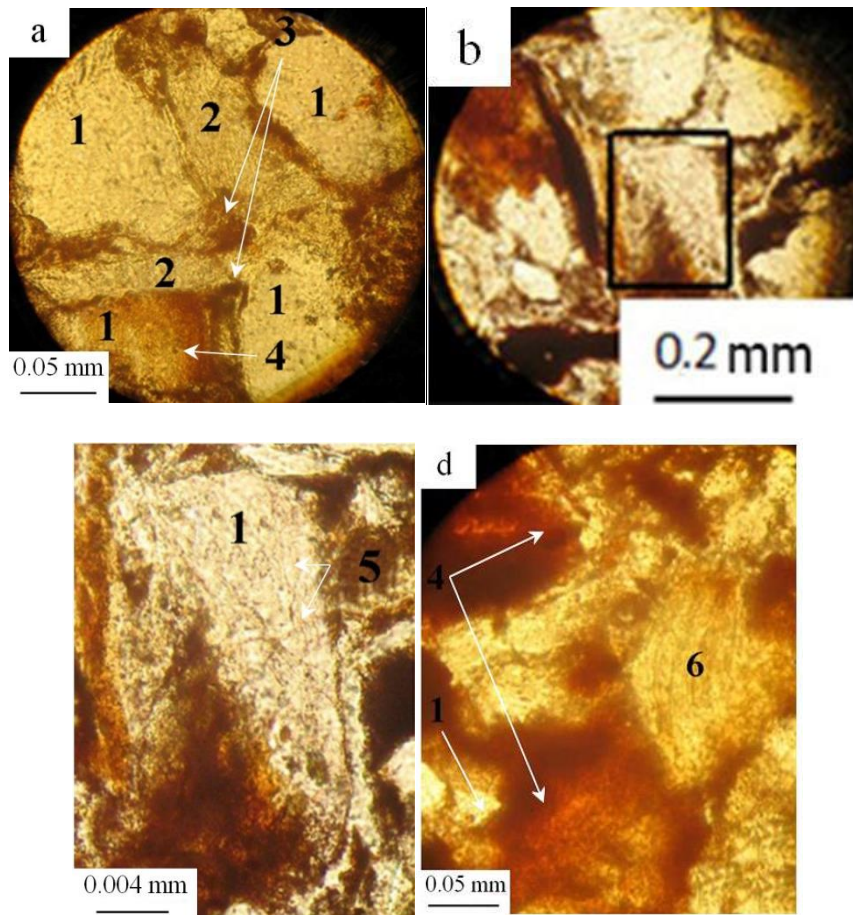
According to X-ray diffraction and crystal-optical analyses, the burnt rock is mainly comprised of crystal quartz SiO<sub>2</sub>, sillimanite Al<sub>2</sub>SiO<sub>5</sub>, gypsum CaSO<sub>4</sub>·2H<sub>2</sub>O, magnetite Fe<sub>3</sub>O<sub>4</sub>, and limonite Fe<sub>2</sub>O<sub>3</sub>·nH<sub>2</sub>O (Figures 3,4).





**Figure 3. Burnt rock view**  
a – in transmitted light; b – in polarized light

Quartz in the burnt rock is present both in the form of an independent phase and as a component of the cementing mass. Likewise, quartz is observed in the form of transparent colorless or yellowish-brown grains. The yellowish-brown shade of the quartz indicates the presence of ocherized limonite. The grains of quartz are irregularly shaped – the size of grains can vary from 0.2 to 0.4 mm while individual grains can reach 5 mm - and characterized by undulose extinction (Figure 4).

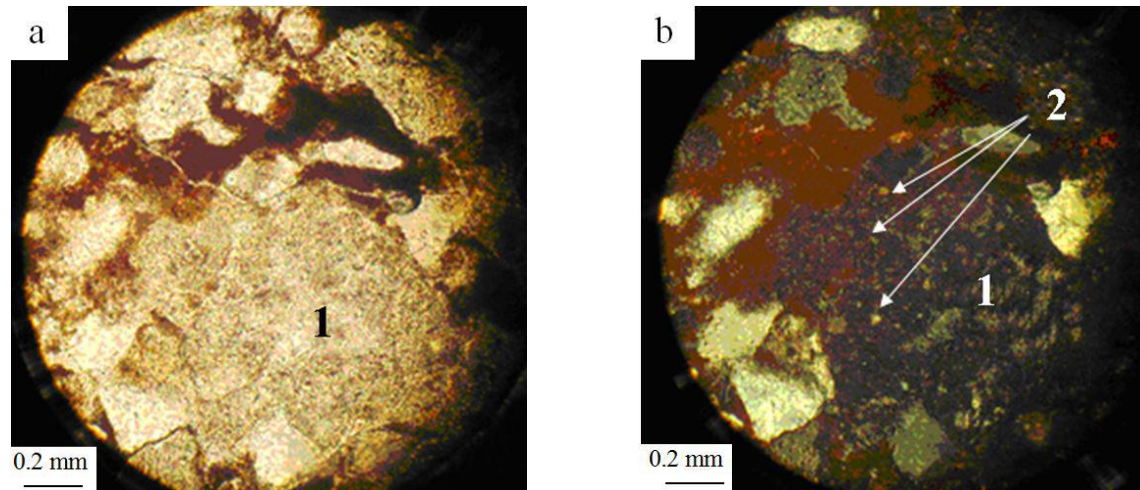


**Figure 4. Images of the burnt rock (in a transmitted light):**  
a – the selected fragment of the figure 1 (grains of quartz and fine-grained mass which cements the clastic material); b – general facies of the burnt rock; c – fragment of the Figure 2, b (quartz with inclusions of acicular and sillimanite pointlike grains); d – gypsum grains among the fragments of quartz (1 – quartz, 2 - cementing mass, 3 – magnetite, 4 – limonite, 5 – sillimanite, 6 – gypsum)

In addition to the limonite streaks in the quartz grains, the inclusions of barely discernible sillimanite acicular grains – size of less than 0.01 mm – can be observed (Figure 4, c).

Gypsum is present both in the form of an independent phase and as a component of the cementing mass. Likewise, a significant amount of gypsum is visible among the quartz fragments – in the form of irregular grains up to 0.5 mm in size (Figure 4, d). Gypsum in the burnt rock is recognized by either columnar colorless or brownish aggregates.

The cementing mass is comprised of quartz, gypsum, sillimanite, magnetite and limonite (Figure 5).



**Figure 5. Images of the cementing mass:  
a – in the transmitted light, b - in polarized light (1 – cementing mass,  
2 – quartz grains, which are part of the cementing mass)**

In the total volume of the cement mass, quartz is easily distinguished by its grains size (up to 0.01 mm). Gypsum and sillimanite form a fine-grained aggregate, which makes it difficult to identify them even at high resolution ( $\times 400$  and  $\times 600$ ). Magnetite is represented by cubic crystals of 0.2–0.5 mm in size and is characterized by an opaque brown tint (caused by the buildup of limonite).

It should be noted that such a detailed optical crystallography of the burnt rock as well as the cement stone formed with its addition was carried out for the first time. However, the very application of the burnt rock as an aluminum silicate additive to the cements has been known about since the 50s of the XX century [2, 7, 10, 18]. Both theoretical and applied research into the issue was conducted in the regions where coal was extracted via deep mining technique. The results of those studies are presented in detail [5–8].

### *Laboratory studies of cements*

For illustrative purposes, during the laboratory studies the burnt rock activity was compared to the activity of TPP fly ash generally used as an active mineral in the additive cement production. The evaluation of the mechanical properties of the laboratory grinding cement was carried out by substituting fly ash with the burnt rock. For this reason, five different compositions were prepared, in which a third, a half, two thirds and all fly ash were replaced by the burnt rock. That being said, the total amount of AMA constituted 18 % of the Portland-cement clinker weight. The addition of gypsum stone to all compositions remained constant and equaled 5.2 (wt%) of the clinker weight.

Preparation of the mortar mixtures and the samples for mechanical testing was based on the cement compositions and single-fraction sand in accordance with Russian State Standard GOST 310.4–81 (EN 196–1). Some samples were tested immediately after steaming, other samples were left to harden in water, and then tested on the 7th and the 28th days. The average values of the mechanical properties of composite cement with burnt rock and fly ash additives are presented in Table 3.

**Table 3. Mechanical properties of the composite cement with additions of the burnt rock and fly ash**

Mechanical properties		Strength of the sample, MPa
<i>Composition 1: PCC + 0 % BR + 18 % FA + 5.2 % GS</i>		
Steamed, day 1	at bending	3.57
	at compression	19.28
Hardened in water, day 7	at bending	3.61
	at compression	23.02
Hardened in water, days 28	at bending	6.35
	at compression	40.23
<i>Composition 2: PCC + 5.5 % BR + 12.5 % FA + 5.2 % GS</i>		
Steamed, day 1	at bending	3.80
	at compression	20.56
Hardened in water, day 7	at bending	3.77
	at compression	23.58
Hardened in water, day 28	at bending	6.33
	at compression	40.56
<i>Composition 3: PCC + 9 % BR + 9 % FA + 5.2 % GS</i>		
Steamed, day 1	at bending	4.14
	at compression	22.15
Hardened in water, day 7	at bending	3.90
	at compression	24.58
Hardened in water, day 28	at bending	6.64
	at compression	41.89
<i>Composition 4: PCC + 12.5 % BR + 5.5 % FA + 5.2 % GS</i>		
Steamed, day 1	at bending	4.28
	at compression	24.23
Hardened in water, day 7	at bending	4.41
	at compression	26.22
Hardened in water, day 28	at bending	6.92
	at compression	43.56
<i>Composition 5: PCC + 18 % BR + 0 % FA + 5.2 % GS</i>		
Steamed, day 1	at bending	4.37
	at compression	28.98
Hardened in water, day 7	at bending	4.58
	at compression	29.56
Hardened in water, day 28	at bending	7.22
	at compression	45.24
<i>Composition 6: PCC + 25 % BR + 0 % FA + 5.2 % GS</i>		
Steamed, day 1	at bending	4.28
	at compression	27.05
Hardened in water, day 7	at bending	4.40
	at compression	28.80
Hardened in water, day 28	at bending	7.02
	at compression	44.09

Note: PCC is a Portland-cement clinker; BR – burnt rock; FA – fly ash; GS – gypsum stone.

Based on the obtained data, it can be deduced that the optimal amount of AMA to be added to the cement composition is 18 %. This Portland cement satisfies the requirements of Russian State Standard GOST 10178-85 "Portland cement and portland blastfurnace slag cement. Specifications" (EN 196-1) and Russian State Standard GOST 2532882 "Masonry cement. Specifications" (EN 2061).

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Thus, one can conclude that the increase in the amount of the burnt rock (the AMA output is constant and equals 18%) corresponds with the increase in the cement stone strength when bending or compressing despite the hardening and the steaming time of the samples vary. The maximum strength property is observed in the Portland cement composition where the AMA consists solely of the burnt rock.

Thereby, the replacement of cement by the burnt rock in the amount of 18 % under normal conditions during the cement stone formation should be considered optimal, since in this case it is possible to achieve maximum strength of the samples. Likewise, it leads to the significant savings of the clinker.

Table 4 shows comparative quality characteristics of the cement produced with addition of the burnt rock and the fly ash of TPP.

**Table 4. Qualitative characteristics of the cement with active mineral additives in the form of burnt rock and fly ash of TPP**

Characteristics, dimension	Cement quality indicators: AMA in the form of:	
	burnt rock	fly ash of TPP
True density, g/cm <sup>3</sup>	3.00	3.03
Bulk density, g/cm <sup>3</sup>	1.08	1.10
Grinding fineness: residue on a sieve 0.08, %	13.80	13.10
Grinding fineness: specific surface area, m <sup>2</sup> /kg	326	375
Normal density, %	28.50	25.75
Setting start, min.	220	200
Setting end, min.	430	470
Water separation coefficient, %	14.1	32.0

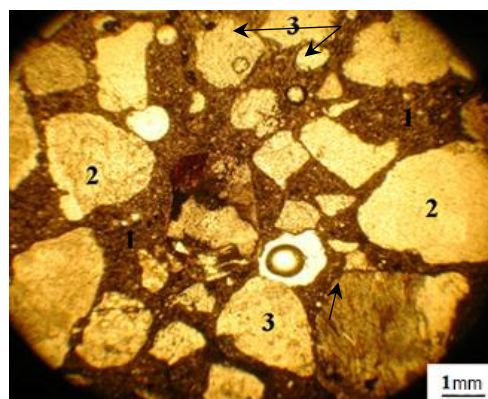
Portland-cement produced with the burnt rock additive belongs to the first steaming efficiency group while the one produced with the fly ash additive reside the third one [18]. Furthermore, the cement produced with the fly ash has a much higher water separation coefficient.

### *Crystallo-optical studies of the cements samples with AMA*

Preparation of the solution mixtures for the crystallo-optical studies was based on the use of single-fraction quartz sand in accordance with Russian State Standard GOST 310.4–81 (EN 196–1).

The samples were made with the cement containing 18 % of the burnt rock as AMA. The grinds were prepared from the plates located in three mutually perpendicular planes of the test bar. The tested sections were identical to each other with regard to their composition and structural features. The resultant description of the studied material is presented below.

Figures 6, 7, 8 show the samples consisting of the quartz sand and the cement with the burnt rock added as AMA. The samples are compounded with the clastic material. The aggregate grains are heterogeneous in composition (gypsum, quartz, plagioclase, etc.), are irregularly shaped. The average size of the grain is up to 4 mm. The clastic fragments are completely moistened with the binder material (Figure 6).

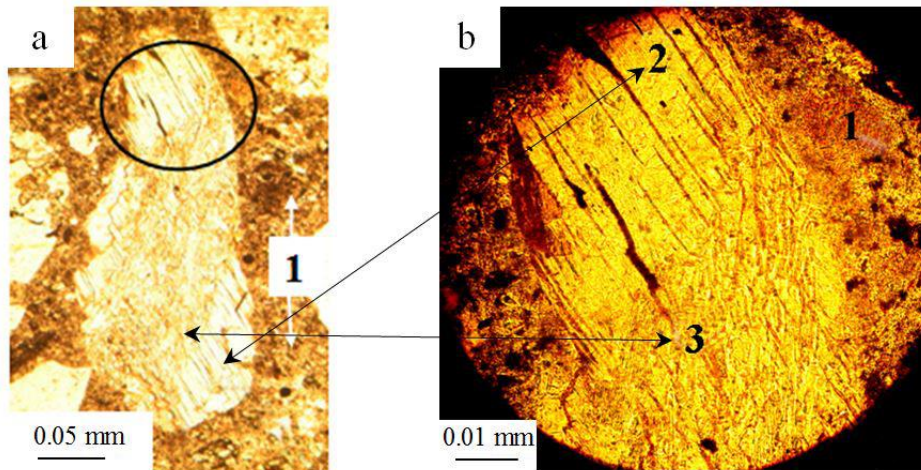


**Figure 6. Cement composition with the burned rock as AMA (general view):**  
1 – fine-grained binding mass; 2 – large clastic material; 3 – voids



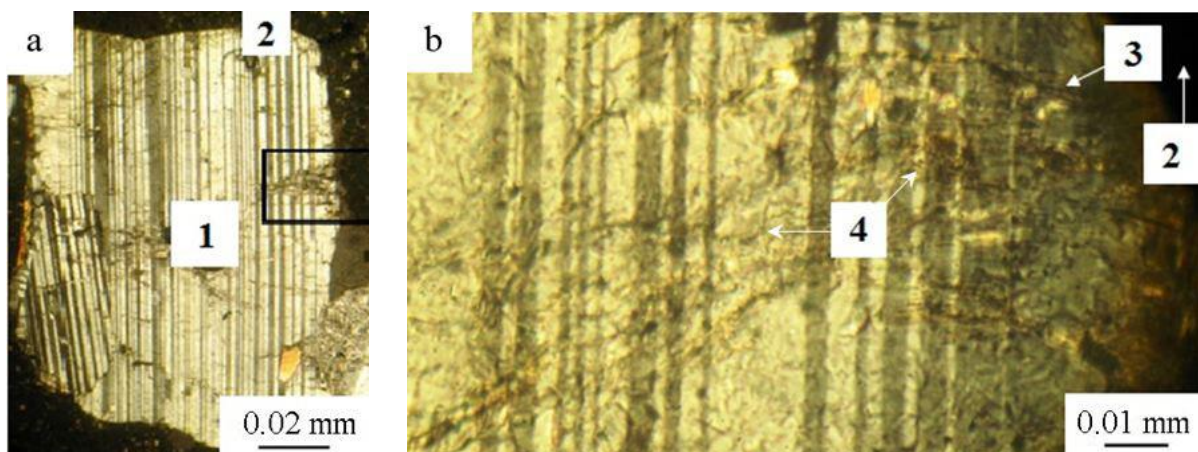
Uniformly distributed isometric voids of 0.01–0.03 mm in size, as well as acicular neoplasms, penetrating fine-grained material, were registered in the cementing fine-grained mass. Besides, the units grouped in a certain order while creating the contours of elongated new formations were found in the sample.

It is crucial to note that the new formations are present not only in the cohesive fine-grained mass, but also on its border together with the large clastic grains. This indicates the chemical interactions between the compounds. Figure 7 captures the columnar gypsum grain where its substitution by the needle-like crystals can be traced from the cementitious material deep into the grain across its columnarity.



**Figure 7. Nature of grain substitution:**  
**a – columnar-fiber grain of gypsum in cementing mass;**  
**b – detail (a) – needle-shaped new formations in gypsum, oriented across the grain columnarity (1 – cementing material, 2 – columnar gypsum, 3 – needle-shaped new formations)**

Figure 8 shows the nature of the replacement of plagioclase grains. The contours of the crystal are blurred and pitted (with the buildup of the binder material). This indicates the presence of a chemical interaction between the crystal and the binder. On some parts of the grain surface, the substitution process can be traced from the edge of the grain to its inside. There, the new formations develop across the polysynthetic twins of plagioclase.



**Figure 8. The Nature of grain substitution:**  
**a – plagioclase crystal (1), which is completely encircled by the cementing mass (2);**  
**b – the fragment of the figure (a) – cementing material (2), the pitted edges of the crystal (3), new formations, developing deep into the crystal across the polysynthetic twins of plagioclase (4)**

According to the data obtained by the X-ray structural analysis, the new formations in the samples made from the cement with the burnt rock addition are comprised of calcium hydrosilicate  $4\text{CaO} \cdot \text{SiO}_2 \cdot 13\text{H}_2\text{O}$ ,  $3\text{CaO} \cdot \text{SiO}_2 \cdot 2\text{H}_2\text{O}$ .

### *The use of concretes with the burnt rock additives in the manufacturing of products by methods of pressing and steam treatment in the steaming chamber*

The results of laboratory and crystallo-optical studies of the impact of the burnt rock additives on the quality of the cement stone suggest that the burnt rock additives are primarily beneficial for the samples manufactured by pressing and subsequent steaming treatment.

New formations, obtained in the result of the research, their structure and interaction with each other, as well as the nature of the replacement the grains and the binder were subjected to, prove the undeniable industrial potential of the combination (cement-burnt rock) [18–23]. Mechanical impact and steam treatment of the sample during the cement stone formation can only contribute to the case [20].

For that purpose, semi-industrial tests were carried out at the operational plant, which manufactures concrete products (“Stroitechnik”, LLC, Irkutsk, Russia).

The task of the tests was to determine the degree of the possible replacement of "M-500" cement by the burnt rock under the real conditions of the masonry units production. The main criterion to be followed was the preservation of all strength characteristics of the commercial output.

The Units (dimensions: 200×200×400) were produced by the method of vibrocompression on the Condor 1 press of “Stroitechnik Plant”, LLC. Then they were placed in a steaming chamber at a temperature of 60-80 °C for a day. The composition of the concrete mixture was prepared on the basis of the approved regulations. The standard output of the "M-500" cement was 280 kg per 1 m<sup>3</sup> of the mixture. The cement in prototypes was replaced by the burnt rock in a different percentage ratio. The compressive strength of the samples was measured on the 28 day. The results of tests of the compressive strength limit depending on the degree of burnt rock cement replacement are presented in Table 5.

**Table 5. Tests of the compressive strength limit depending on the degree of substitution of cement by the burnt rock**

Substitution of cement by the burnt rock, %	The compressive strength, day 28, MPa
0 % – standard	29.6
18	32.8
25	30.1
35	29.4
50	23.6

It can be seen that in the blocks production according to the technology described above, it is possible to replace up to 35 % of the cement by the burnt rock.

Thus, the hypothesis about the effectiveness of the burnt rock use along with cement, or as its partial replacement, was confirmed in practice. In addition, the replacement of cement by the burnt rock in the concrete products manufacturing can lead to a significant reduction of the end product price.

### *Conclusions*

1. The research of the chemical compounds, physical properties and structure of the burnt rock have shown high efficiency of its application as active mineral additive.

2. The tests of the mechanical properties of the mixture “cement – burnt rock” and “cement - fly ash” allowed to estimate the extent of the burnt rock activity and determine the optimal amount of active mineral addition (18 %). At this rate, physical and mechanical characteristics of the "M-500" cement are preserved in accordance with Russian State Standard GOST 10178–85, which complies with the 32.5 N strength class standard.

3. Crystal-optical studies with a view to determine the structural features of the burnt rock samples made it possible to establish that the burnt rock activity when in contact with the cement during the formation of cement stone is predicated by the presence of the new needle-shaped formations and their chemical interaction - both with the binder of fine-grained mass and with the large clastic grains.

4. Semi-industrial tests conducted with intent to apply the burnt rock into the concrete mixture by vibrocompression method followed by steaming treatment allowed to determine the optimum value of substitution of cement by the burnt rock. It has been established that it is possible to replace up to 35 % of cement without reducing the strength properties of the end products.

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