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# Hydraulic activity of crushing screenings of waste open-hearth slag

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**Abstract.** Theoretically and experimentally studied the hydraulic activity of crushing screenings of waste open-hearth slag and methods for intensifying the hydration hardening of slag stone during operation. During the hardening of slag stone, three periods of pronounced syneresis with gel new formations strengthening the material were established at the ages of 28 – 60, 90 – 180 days and 1 – 2 years. The first period of syneresis, in contrast to the subsequent ones without changing the strength of the samples, is recorded by the linear shrinkage of the samples, the squeezing out of bound water and the increase in pH. The introduction of two percent of cement dust or lime into the crushing screenings of waste open-hearth slag leads to the strengthening of the slag stone at 28 and 90 days of age by 3 – 4 times. However, by the age of two years, the strength of the samples stabilizes. This fact indicates that the additions of cement dust or lime are not additional binders, but hardening accelerators.
**Keywords:** industrial waste, secondary raw materials, mineral waste of soda production, metallurgical slags, composite clinkerless mineral binders, stabilization and strengthening of soils, road bedding layers

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## 1. INTRODUCTION

In accordance with the main directions of economic development of the Russian Federation until 2030, one of the most effective ways to increase national wealth is to ensure rational, economical use of natural and material resources. For this purpose, it is planned to widely use complex processing of raw materials, resource-saving equipment, low-waste, non-waste and energy-saving technologies, to fully involve local types of raw materials and materials into circulation, and to utilize secondary resources [1].

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Implementation of the national project «Safe and high-quality highways», in particular, to bring the proportion of roads that comply with established standards to 85 % in large agglomerations and 50 % of regional highways with hard surfaces, and in the future it is possible to increase the volume of construction only with the development and implementation of advanced technologies for the production of road building materials for the construction of road pavements with maximum use of local materials and industrial by-products [2], including the widespread use of low-viscosity organic binders and ferrous metallurgy slags [3]. Every year in the Russian Federation, for the purposes of road construction, over 65 million tons of road asphalt concrete mixtures are produced, the production process of which is energy-intensive and is accompanied by significant environmental pollution [4].

One of the effective low-energy-intensive and environmentally friendly technologies for the production of asphalt concrete mixtures is the production and use in road construction of wet organic-mineral mixtures, which are mixtures of moistened mineral materials: products of processing waste open-hearth slag into crushed stone and low-viscosity organic binders. At the same time, the values of adhesion and cohesion of liquid organic binders lead to unsatisfactory values of strength, water resistance and resistance of wet organic concrete (GOST 30491-2012) to shear deformations, as well as thermal-oxidative aging at technological and operating temperatures [5].

It should be noted that the hydraulic activity of open-hearth slag has not been sufficiently studied [6], which does not allow for targeted regulation of the intensification of hydraulic hardening of the slag component with intensifiers of the hydraulic activity of particles of the 0.071 – 10 mm fraction [7].

The purpose of the study is to study the mechanism of hydration hardening of the 0.071 – 10 mm fraction of open-hearth slag.

## 2. METHODS AND MATERIALS

The 0.071 – 10 mm fraction of waste open-hearth slag, represented by grains of a crystalline structure, which have a texture from tricky-porous to dense with a basicity modulus $M_0$ = 1,89, was taken as the material under study [8].

The chemical composition of open-hearth production slag is variable, depends on the steel grades being smelted and contains up to 30 elements, among which the sum of four elements (CaO, $SiO_2$, $Al_2O_3$ and MgO) is up to 98 %. Impurities of MnO, S, FeO and a large number of micro impurities (Ti, V, G, Ni, Cu, etc.) have a noticeable effect on the properties of slags. Their content in thousandths of a percent, according to V.S. Gorshkov, significantly affects quality indicators, including the astringent properties of slag [9]. Open-hearth slag is not granulated, but is poured into dumps, where it slowly cools. When cooled, due to their high basicity, they almost completely crystallize and, as a rule, contain almost no glass. The material of slag dumps can be dense, porous, clogged with an admixture of fireclay bricks from the furnace lining. Open-hearth slag is characterized by a high content of iron in the form of oxides and metal inclusions – beads and scrap. The iron content ranges from 15 to 20 % [10].

As waste open-hearth slag cools, it forms masses with uneven porosity, varying ratios of glassy and crystalline phases, mineralogical composition, core metal content and other parameters. In this regard, spontaneous decomposition processes occur in the slag at different rates: silicate, lime, magnesium, manganese and iron [11]. Open-hearth slags with an increased amount of CaO and dust particles have cementing ability [12].

To study the fine structure of the emerging aggregates during the hydration of fine particles of the 0.071 – 10 mm fraction of open-hearth slag at the interface between the «slag – new formations» phases of hydrated slag minerals, an ISI-60A scanning (raster) microscope from the English company «UNI-EXPERT» was used at an operating voltage of 30 kV and a current of $2 \cdot 10^{-5}$ A. A powdered sample of slag concrete was applied to a table with conductive glue and sprayed with a layer of gold $2 \cdot 10^{-6}$ m thick. The resolution of the microscope was from 1 to $3 \cdot 10^{5}$ times [13].

Infrared spectroscopy of concrete samples from wet slag concrete mixtures to identify atomic groups of hydrated screenings was performed on a UR-20 «Specord» spectrophotometer in the range of 500 – 3900 $cm^{-1}$. To obtain IR absorption spectra, samples were prepared in the form of KBr tablets or suspensions in liquid paraffin.

X-ray spectral (microprobe) analysis of new formations (mutual arrangement of atoms, degree of perfection and orientation of crystals, and on this basis changes in the structure of slag concrete over time) on the surface of slag particles was performed on a Cameca M-46 microprobe at a voltage of 25 kV, sample current of 80 mA and electron beam diameter $1.5 \cdot 10^{-6}$ m, with recording of the wavelength of characteristic X-ray radiation using a recorder. In addition, for the same purpose, a URS-50IM diffractometer with a UR-4 attachment with an operating voltage of 45 kV and a current of $1 \cdot 10^{-5}$ A with a scintillation counter using the polycrystalline method at a sample rotation speed of one degree per minute, as well as microanalyzers of the «EDAX» and «Link-system» based on YSM-35CF with fixation of the chemical composition by radiation energy with beam locality equal to 1 [14].

Microanalysis of mineral new formations of slag concrete was carried out using a laser micro mass analyzer «LAMMA-1000» from Leybold Heraeus (Germany) using sodium aluminum garnet as radiation with $\lambda = 2.65\ 10^{-8}$ m and energy 15 mJ at a pulse duration of $15\ 10^{-3}$ s. The diameter of the crater of evaporation of the substance under study varied from $1 \cdot 10^{-6}$ m with a depth, respectively, from $4 \cdot 10^{-6}$ m to $7 \cdot 10^{-5}$ m. In this case, both atomic ions and molecular ions up to a mass of 600 at.u. were determined.

## 3. RESULTS AND DISCUSSION

The data given in Table. 1 indicate that the hydraulic activity of open hearth slag is manifested slowly. X-ray patterns of the initial sample (one day of hardening) and the final sample (two years of hardening) of the slag stone show that there are no fundamental changes in the positions of the reflections in d/n and intensity (Fig. 1). The exceptions are carbonates - calcite and calcium manganese. Two-year-old samples are generally characterized by an increased level of X-ray background, which indicates an increase in the X-ray amorphous phase of neoplasms.

The increase in carbonate content during slag hydration is also proven by an increase in the intensity of endothermic effects with extremes at 810 – 850 °C (Fig. 2). A gradual shift of the extremum to the region of higher temperatures indicates the compaction of carbonates and the improvement of their crystal structure. At 100 °C, an endo-effect was recorded, which characterizes the removal of physically bound water upon heating [15]. A wide exothermic effect at temperatures of 100 – 750 °C shows the transition of slag from a gel to a crystalline state: the number of extrema corresponds to the number of density levels and gel transition temperatures. Negative deflections between them can be interpreted as endothermic effects as a result of dehydration of bound water to varying degrees [16].

The extrema of the gel's exo-effects also tend to shift along the temperature scale to the right up to 40 °C, which indicates its compaction. It is probably the compaction of the gel that causes the slow increase in the strength of the slag stone over time.

**Table 1.** Binding properties of screening fractions 0.071 – 10 mm of open-hearth slag.

| Composition in mass parts | | | Strength of slag stone, MPa, during hydration | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Slag | Mixing water | Activator | Days | | | | | Years | |
| | | | 1 | 28 | 60 | 90 | 180 | 1 | 2 |
| Unseparated, 100 | 15 | – | 0.2 | 1.0 | 1.6 | 1,8 | 5.7 | 13.7 | 14.2 |
| Unseparated, 100 | 15 | Lime, 2 | 0.5 | 2.9 | 4.2 | 5.7 | 5.5 | 15.2 | 14.6 |
| Unseparated, 100 | 15 | Cement dust, 2 | 0.4 | 2.7 | 3.6 | 5.2 | 5.0 | 14.3 | 13.7 |
| Fraction of open hearth slag more than $5 \cdot 10^{-3}$ m, 100 | 15 | – | 0 | 0.6 | 1.9 | 2.9 | 6.4 | 12.1 | 13.1 |
| Ground slag, 100 | 15 | Lime, 2 | 1.1 | 4.3 | 6.9 | 6.8 | 6.3 | 14.3 | 14.6 |
| Ground slag, 100 | 15 | Cement dust, 2 | 0.8 | 2.2 | 4.2 | 3.5 | 3.2 | 13.9 | 14.2 |
| Fraction of open hearth slag more than $5 \cdot 10^{-3}$ m, 100 | 15 | – | 0.1 | 0.9 | 2.0 | 3.1 | 4.7 | 11.3 | 11.8 |

Continuation of Table 1

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Fraction of open hearth slag more than $5 \cdot 10^{-3}$ m, 100 | 15 | Lime, 2 | 0.3 | 1.6 | 3.9 | 5.2 | 6.2 | 10.8 | 11.3 |
| Fraction of open hearth slag more than $5 \cdot 10^{-3}$ m, 100 | 15 | Cement dust, 2 | 0.3 | 1.3 | 3.1 | 4.9 | 6.0 | 10.7 | 9.7 |

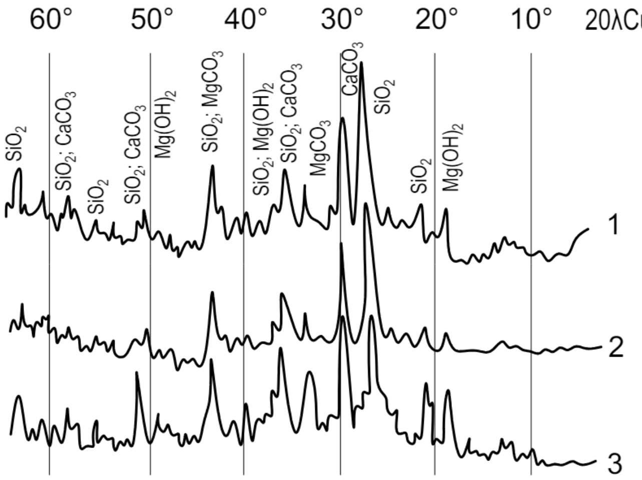



**Fig. 1.** X-ray diffraction patterns of hydrated fractions of 0.071 – 10 mm open hearth slag at the age of: 1 – 1 day; 2 –1 year; 3 – 2 years.

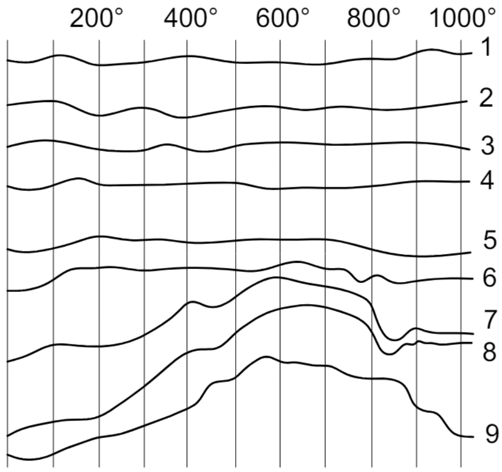
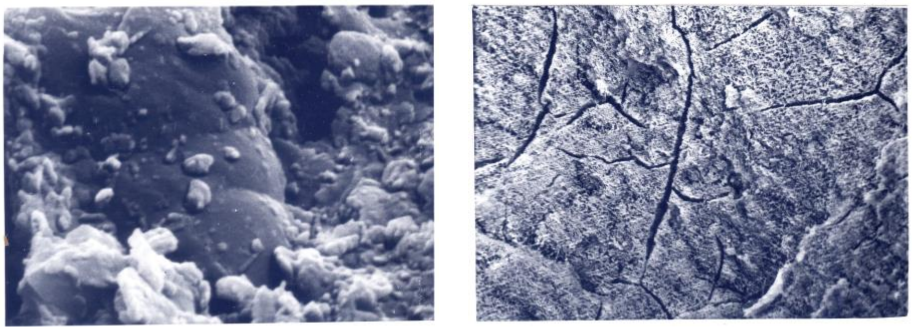



**Fig. 2.** Derivatograms of hydrated screening out fractions 0.071 – 10 mm of open-hearth slag at the age of: 1 – 1 day; 2 – 3 days; 3 – 7 days; 4 – 14 days; 5 – 1 month; 6 – 3 months; 7 – 6 months; 8 – 1 year; 9 – 2 years.

Electron micrographs show that at the age of 28 days, hydrated slag grains are almost completely covered with sintered masses of gel-like material (Fig. 3). And at the age of 60 days, individual cracks are visible on the surface of the material, characteristic of the phenomenon of gel syneresis. However, no other manifestations of syneresis were recorded for other indicators (Table 2). Obviously, the process of hydration of the slag and the formation of a second generation gel dominated due to the penetration of the alkaline solution into the resulting cracks.



a) b)

**Fig. 3.** Electronic micro photographs of the hydrated fraction 0.071 – 10 mm of open-hearth slag at the age of: a) 28 days of hydration; b) 2 years of hydration.

Using X-ray and laser micromass spectroscopy methods, the heterogeneity of the chemical composition of gel neoplasms at the microlevel was established (Fig. 4).

The nearest neighboring points of gel new formations differ significantly from each other in chemical elements and their quantities, for example, Na, Mg, Ca, etc. (Fig. 4).

The first period of gel syneresis is 60–90 days: all indicators of the slag-water system decrease, except pH (Table 2); the coefficient of linear deformation decreases (by 0.04%) and the content of bound water (from 4.91 % to 3.47 %), which can be explained by its squeezing through the capillary system during the aging process of the gel. In this case, the gel shrinks and overall compression of the sample occurs. Part of the squeezed water, together with atmospheric moisture, flows through the formed syneresis cracks to the unhydrated exposed surfaces of the slag particles, dissolves them and increases the pH of the system [17].

The coefficient of linear deformation of samples up to two years of age increases to 1.63%, decreasing only in the periods of 60 – 90 days and 180 days – 1 year (Table 2). The content of bound water, with the exception of periods of 60 – 90 days and 180 days – 1 year, increases due to an increase in the amount of gel, and, accordingly, water of varying degrees of boundness.

**Table 2.** Physical and mechanical properties of slag stone.

| № п/п | Indicators | Hardening time | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | Days | | | | | | Years | |
| | | 0 | 1 | 28 | 60 | 90 | 180 | 1 | 2 |
| 1 | Tensile strength at compression, MPa | – | 0.20 | 1.0 | 1.64 | 1.82 | 5.73 | 13.68 | 14.21 |
| 2 | Linear coefficient deformation, % | – | 0.35 | 1.44 | 1.53 | 1.49 | 1.48 | 1.48 | 1.63 |
| 3 | Total weight loss, % | 4.62 | 5.12 | 6.85 | 7.65 | 6.34 | 8.19 | 8.62 | 11.81 |
| 4 | Bound water content, % | 2.57 | 2.95 | 4.55 | 4.91 | 3.47 | 4.78 | 3.91 | 4.64 |
| 5 | Content $CO_2$, % | 2.05 | 2.17 | 2.30 | 2.74 | 2.87 | 3.40 | 4.71 | 6.17 |
| 6 | pH value | 10.13 | 10.35 | 9.28 | 9.31 | 9.38 | 9.35 | 9.41 | 8.90 |

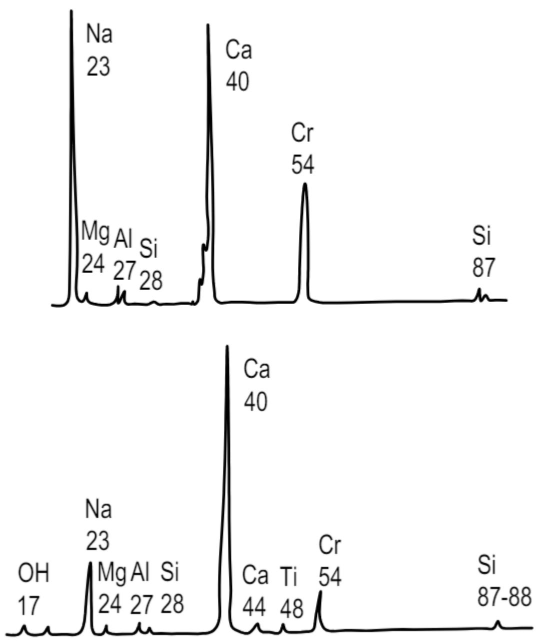



**Fig. 4.** Laser micromass spectrograms of gel new formations of fractions 0.071 – 10 mm of waste open-hearth slag at the age of 28 days.

Strengthening of the material in the period 60 – 90 days occurs noticeably slower, which is explained by the appearance and development of systems of syneresis cracks.

The second period of gel syneresis is between 180 days and one year (Table 2). Its main difference is a much smaller change in all indicators, which indicates a significant compaction of the gel and, in connection with this, a slowdown in the syneresis process.

Thus, hardening during hydration of open-hearth slag can be divided into six stages, of which three (60 – 90 days, 180 days – 1 year, 2 years or more) can be considered stages of syneresis, and the three preceding them can be considered stages of hydrolysis and hydration slag.

Hydration of slag minerals leads to the formation of a supersaturated solution and the release of a new colloidal dispersed phase, including hydrates of slag minerals. Together with the particles of the original slag, the hydrates form a coagulation structure. The saturation of the solution is maintained by the constant dissolution of new slag particles. Therefore, the coagulation structure forms and develops under conditions of solution supersaturation. An increase in coagulation contacts in the pore space of unhydrated slag particles leads to their compaction [18].

Syneresis and contraction compact the gel layer of new formations and bring the particles of the slag solid phase closer together. Both phenomena lead to the formation of microcracks in the gel. Through these cracks, an alkaline solution penetrates to the inner surface, dissolving the exposed surface of the slag particles. The resulting new generation gel "heals" cracks and strengthens the sample. At the same time, new systems of cracks are formed at other points [19]. The strength of the material increases mainly due to the general compaction of the gel, but this increase is not general, but volumetric-statistical in nature. Thus, the formation of a gel structure of new formations during the hydration of waste open-hearth slag proves the following:

– stability of radiographs;
– presence of exothermic effects in the temperature range 100 – 700 ºC;
– dominance of gel-like new formations on the surface of slag particles;
– data on the heterogeneity of neoplasms in terms of chemical composition and quantitative ratio, as evidenced by X-ray and laser micromass analysis data, the occurrence of syneresis cracks, their healing and the appearance of new ones.

A characteristic feature of the hardening of fine particles of open-hearth slag is the high strength of the slag stone at the age of one year and low, for example, at the age of a month. Therefore, it is advisable to activate the process of strength gain during the initial hardening period with the help of mineral binders in small concentrations

The introduction of alkaline additives (flying dust from cement kilns) (Table 3) makes it possible to intensify the process of hydration of fine particles of open-hearth slag. OH– ions have a polarizing effect on the Me – O bonds, as a result of which, first of all, cations located in the voids of silicon-oxygen complexes and having the lowest energy of the Me – O bond pass into the solution. Calcium hydroxide is protonated by water and dissolves in it in ionic form. Alumina and silica in an aqueous alkali solution dissolve in ionic form to form hydrocomplexes that pass into the solution in the form of anions. The dissolution mechanism is reduced to the formation of an aqua complex with an oxide ion or metal ions on the surface of slag particles, its dissociation, respectively, as a base or as an acid, and the transition of the resulting ions into solution [20].

The resulting supersaturated ash colloidal solution in the pores thickens in the form of a gelatinous gel, which turns into a viscous and then into a semi-solid state. The excess strength of activated slag stone by 28 days is almost 5 times, by 60 days – 2.5 times, by 90 – 3 times. However, by 180 days the strength of the material stabilizes and then slowly increases (Tables 1, 2).

Thus, activation is effective only in the first 90 days of structure formation of the slag stone. For samples aged two years, there are no reflections characteristic of the synthesis of new crystalline phases (Fig. 5). As in the case of slag hardening without an activator, only an increase in the reflections of carbonates (calcite, manganese calcite and manganocalcite) is recorded in the diffraction patterns.

**Table 3.** Physical and mechanical properties of slag stone activated with 2 % dust entrainment from cement kilns.

| № п/п | Indicators | Hardening time | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | Days | | | | | | Days | |
| | | 0 | 1 | 28 | 60 | 90 | 180 | 1 | 2 |
| 1 | Tensile strength at compression, MPa | – | 0.50 | 2.90 | 4.18 | 5.70 | 5.49 | 15.19 | 14.60 |
| 2 | Linear coefficient deformation, % | – | 0.31 | 1.87 | 1.64 | 2,12 | 1.67 | 2.28 | 1.97 |
| 3 | Total weight loss, % | 7.35 | 7.57 | 9.28 | 9.15 | 9.64 | 10.04 | 10.71 | 10.99 |
| 4 | Bound water content, % | 5.17 | 5.36 | 6.80 | 6.19 | 6.71 | 5.34 | 6.29 | 5.81 |
| 5 | Content $CO_2$, % | 2.18 | 2.21 | 2.48 | 2.86 | 2.83 | 3.60 | 4.42 | 5.18 |
| 6 | pH value | 13.08 | 13.03 | 12.64 | 12.88 | 11.87 | 12.16 | 10.03 | 10.26 |

The fact of synthesis of the latter is also established from the DTA curves (Fig. 6). Until one month of hardening of the slag stone, DTA curves provide practically no information about the synthesis of carbonates. The extreme of the endothermic effect was recorded at 780 °C in slag stone samples aged 90 days or more. The intensity of this endo-effect quickly increases to a $SiO_2$ content of 5,1 % by 2 years (6,17 % for slag without an activator). Consequently, the introduction of an alkaline additive does not affect the growth of carbonates in the material. Over time, the structure of the carbonate improves and becomes denser, which is manifested in the slow drift of the endo-effect extremum from 780 °C to 900 °C.

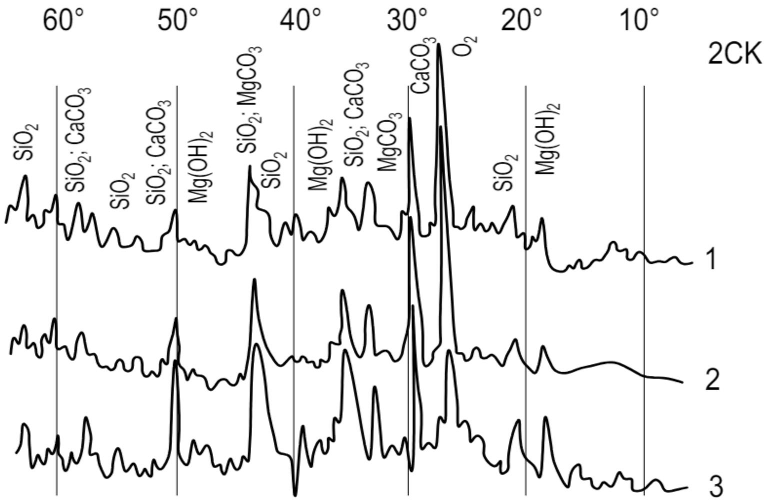



**Fig. 5.** X-ray patterns of hydrated fractions 0.071 – 10 mm of waste open-hearth slag, activated by two percent of cement kiln fly dust, aged: 1 – initial dry mixture; 2 – 1 year; 3 – 2 years.

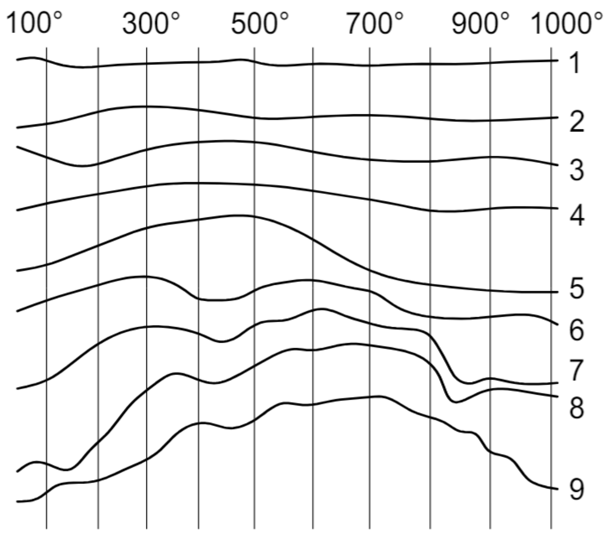



**Fig. 6.** Derivatograms of fractions 0.071 – 10 mm of waste open-hearth slag, activated with two percent lime, aged: 1 – 1 day; 2 – 3 days; 3 – 7 days; 4 – 14 days; 5 – 1 month; 6 – 3 months; 7 – 6 months; 8 – 1 year; 9 – 2 years.

When comparing the DTA curves of non-activated slag (Fig. 2) and activated 2% cement dust (Fig. 6), despite the general similarity, there are also significant differences. The main thing is a wide (but small in amplitude) exothermic effect in the temperature range 200 – 450 °C (structure formation time 3 days), reaching 600 °C by 7 days. and 750 °C by 14 days. (Fig. 6). At one month of age, a significant exo-effect was registered, which by the 8-month period of hardening is divided into two extremes at 250 °C and 600 °C. The appearance, growth and transformation of these exo-effects is explained by the synthesis of gel neoplasms of predominantly aluminum-silicate-calcium composition and their compaction [7]. In the DTA curves of non-activated slag, these processes were much less pronounced (Fig. 2), which explains the main differences in their mechanical properties.

## 4. CONCLUSIONS

Analysis of changes in the indicators of hardening slag stone over time (2 years) allows us to establish three periods of pronounced syneresis with gel new formations strengthening the material at the age of 28 – 60, 90 – 180 days and 1 – 2 years. The first period of syneresis (in contrast to the subsequent ones without changing the strength of the samples) is recorded by the linear shrinkage of the samples, the squeezing out of bound water and the increase in pH.

During the syneresis period of 90 – 180 days (except for the above-mentioned structure formation parameters), the material softens and the carbonate content ($CO_2$ content) sharply increases. The third period of syneresis (between 1 and 2 years of hardening) is also characterized by anomalous changes in strength, linear deformation, bound water content and pH.

Thus, the introduction of two percent cement dust or lime into the fine fraction of 0.071 – 10 mm of open-hearth slag leads to the strengthening of the slag stone at 28 and 90 days of age by 3 – 4 times. However, by the age of two years, the strength of the samples stabilizes. This fact indicates that the additions of cement dust or lime are not additional binders, but hardening accelerators.

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