

STUDIES OF THE PHASE COMPOSITION AND STRUCTURE FORMATION OF POROUS AGGREGATE

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Abstract: The article presents the study of the phase composition and structure formation of porous aggregate based on quartz porphyry and carbonized clay.

Key Words: porous filler, quartz porphyry, carbonized clay, X-ray analysis, differential thermal analysis, cristobalite, mullite.

1. INTRODUCTION:

The phase composition was determined by X-ray analysis of the finished product to fully represent the physico-mechanical properties of porous aggregate annealed at a temperature of 1100 °C [1].

As can be seen from figure 1, the reflexes are $d = 0.424; 0.334; 0.245; 0.223$ NM, characteristic of quartz. In addition to the lines of β — quartz and newly formed mullite, diffraction lines of the polymorphic form of silica — β Crystalite with d/n are found.

$\beta = 4.03; 3.13; 2.87; 2.49; 2.12; 1.59; 1.52; 1.33; 1.27$ etc. The peaks characteristic of neoplasms in the radiograph of porous aggregate from quartz porphyry obtained by agglomeration by a somewhat larger value compared with the sample obtained by expanded clay technology. They cause a more complete chemical interaction between Angren clay, carbonates, and Fe_2CO_3 anhydride. Fine grinding of raw materials contributed to an increase in neoplasms.

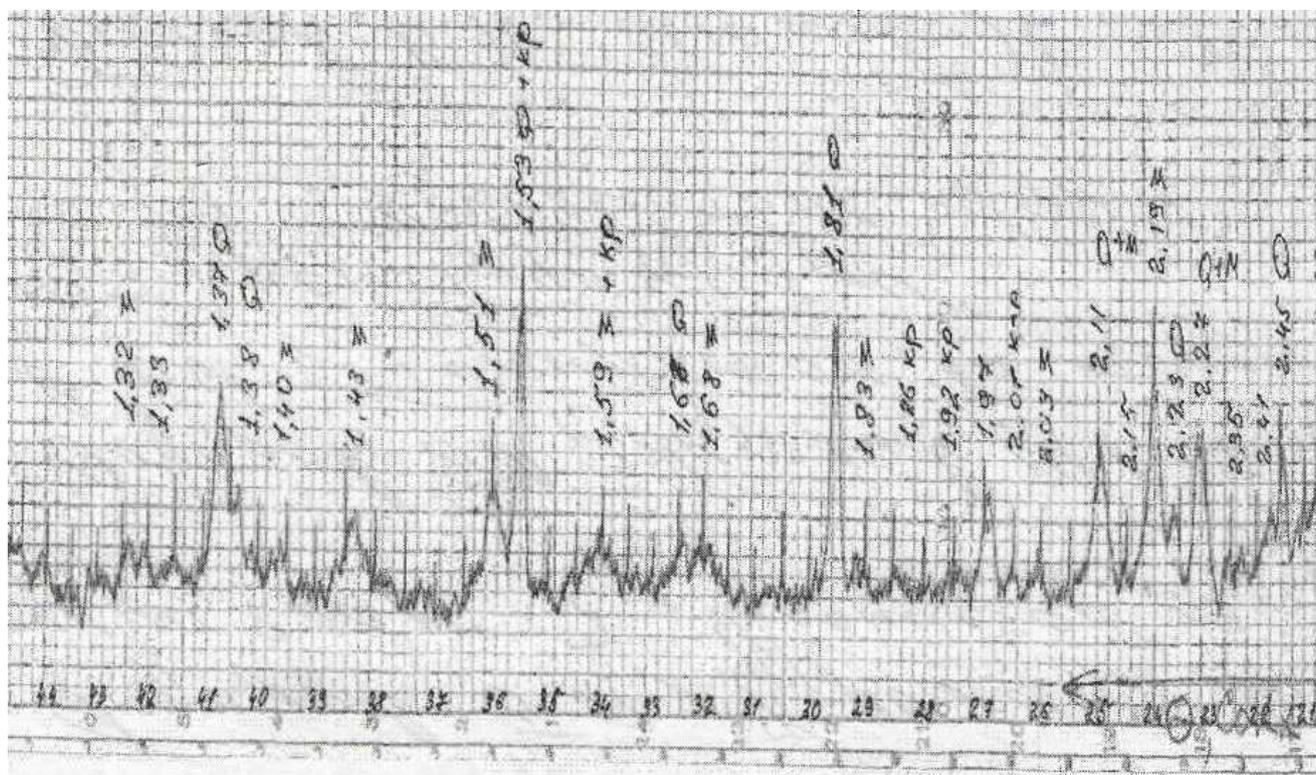


Fig.1. X-ray diffraction pattern of a porous gravel-like aggregate burnt at 1100 °C and a shutter speed of 30 minutes

Table 1. Identification of burned sample minerals by X-ray phase analysis

No	Angle of reflection, θ , $\text{CoK}\alpha$	Interplanar distance, d_n/n , nm	The intensity of the peak of the mineral on the file ASTM, %	Identified mineral by file ASTM
1.	9° 60'	0,535	70	Mullite
2.	12° 14'	0,424	50	β - quartz
3.	12° 78'	0,403	100	β - cristobalite
4.	15° 30'	0,338	100	Mullite
5.	15° 48'	0,334	100	β - quartz
6.	16° 54'	0,313	60	β - cristobalite
7.	18° 16'	0,286	30+70	Mullite + β - cristobalite
8.	19° 42'	0,268	60	Mullite
9.	20° 62'	0,253	70	Mullite
10.	21° 06'	0,248	80	β - cristobalite
11.	21° 30'	0,245	40	β - quartz
12.	21° 70'	0,241	40	Mullite
13.	23° 10'	0,227	50+30	β - quartz + mullite
14.	23° 54'	0,223	40	β - quartz
15.	24° 00'	0,219	70	Mullite
16.	24° 96'	0,211	50+50	Mullite + β - cristobalite
17.	26° 00'	0,201	50	β - cristobalite
18.	27° 56'	0,197	40	Mullite
19.	27° 70'	0,192	60	β - cristobalite
20.	28° 60'	0,186	60	β - cristobalite
21.	29° 20'	0,183	40	Mullite
22.	29° 50'	0,181	90	β - quartz
23.	32° 00'	0,168	60+50	β - cristobalite+ β - quartz
24.	32° 36'	0,167	50	β -кварц
25.	34° 20'	0,159	50+40	Mullite + β - cristobalite
26.	35° 50'	0,153	90+60	β - quartz + β - cristobalite
27.	36° 00'	0,151	70	Mullite
28.	38° 40'	0,143	50+50	β - cristobalite + mullite
29.	39° 68'	0,140	40+40	Mullite + β - cristobalite
30.	40° 28'	0,138	80	β - quartz
31.	40° 64'	0,137	90+50	β - quartz + β - cristobalite
32.	42° 42'	0,132	40+50	Mullite + β - cristobalite
33.	45° 50'	0,125	50	Mullite
34.	49° 40'	0,117	70+40	β - quartz + β - cristobalite
35.	51° 20'	0,114	40	Mullite
36.	55° 86'	0,108	80	β - quartz

DTA burnt porous gravel-like aggregate, burnt at 1100 °C for 30 minutes is presented in Fig.2. It was established that during Sintering of granules during calculation at temperatures above 1100 °C, the mineral mullite is formed from the amorphous decomposition products of kaolinite contained in the initial raw materials — quartz porphyry and carbonized clay (coal preparation waste) (table 1).

2. MATERIALS:

The neoplasm during firing is also cristobalite, detected by DTA analysis (fig. 2). This fact suggests that during Sintering of the investigated compounds, part of the amorphous silica formed during the thermal decomposition of kaolinite at 560 °C remains unbound in mullite, other silicates or silicate melt. As a result, recrystallization of amorphous silica into cristobalite occurs. The initial crystals of β -quartz remain in the calcined products, which, due to their redundancy, is not included in the reactions of formation of new silicates [1].

The ions of sodium, potassium, calcium, magnesium, iron, etc. in quartz porphyry and carbonaceous clay do not form crystalline silicates because their low contents, but are eutectic mixture forming modifier cations. As a result of eutectic melting at relatively low temperatures (800–900 °C), the formation of a silicate melt phase begins, the content of which increases with an increase in the firing temperature.

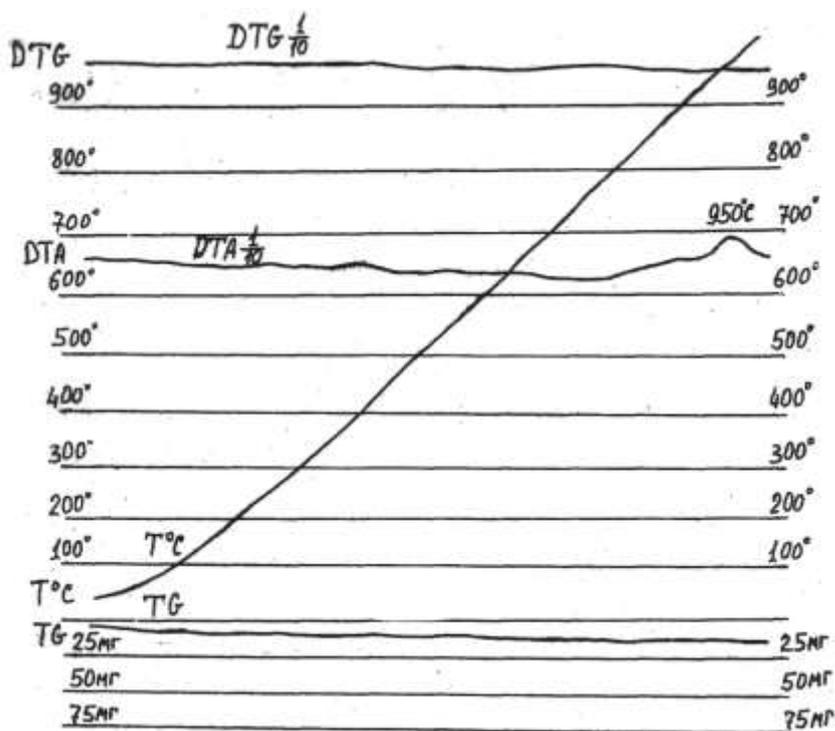


Fig.2. Differential-thermal analysis of baked porous aggregate at a temperature of 1100 °C

As a result of the mutual germination of newly formed mullite crystals, which has a needle-like shape of crystals, and gluing them together to each other and quartz crystal grains, forming a hard granulated granule during cooling into solid glass phase by dropping a silicate melt.

Due to the formation of a relatively small amount of silicate melt at an established firing temperature of 1100 °C, a large volume shrinkage does not occur during Sintering and a large porosity of the granules arises due to the burning out of the carbonaceous substance.

As a result, the porous structure of the granule is formed, which is characterized by a combination of mechanical strength and high porosity, which determines the required performance of the aggregate.

3. METHOD:

According to the results of differential thermal analysis (Fig. 2), it was established that the Sintering of granules occurs at temperatures above 940–980 °C, i.e. beginning with the formation of the crystalline phase of mullite, the crystals of which form as they grow a rigid framework. The porous structure is caused by the presence of a large amount of carbonaceous substance burning out during roasting, the content of which in clay-according to the results of differential thermal analysis is almost 34 %. The removal of substantial amounts of kaolinite crystallization water from the volume of the granule also contributes to the formation of the porous structure which is confirmed by the study of the pore structure of the aggregate.

4. DISCUSSION:

The porosity of the quartz porphyry and carbonized clay aggregate was determined by indentation of mercury [2, 3, 4]. Quartz porphyry aggregate, and expanded clay gravel are fundamentally different in the nature of their porous structure. It is possible to schematically represent these structures in the form of cellular in expanded clay and capillary-porous in gravel of quartz porphyries. The structure of expanded clay aggregate is characterized by the presence of mostly closed pores of rounded shape, and gravel of quartz porphyry interconnected pores of various shapes and sizes. Even with a small increase, it can be seen that the cellular structure of expanded clay granules with separate closed pores significantly differs from the porous structure of a quartz porphyry granule consisting of irregularly shaped pores, and even cracks

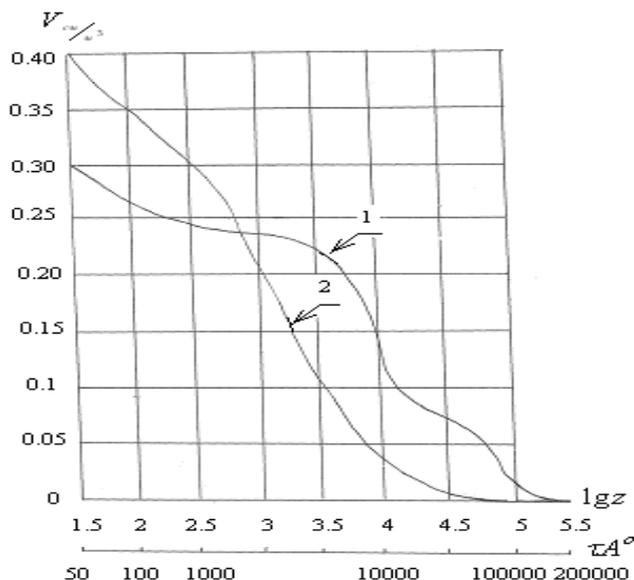


Fig.3. Integral curves of porosity of aggregates: 1 - gravel of quartz porphyry and carbonized clay; 2 - expanded clay gravel

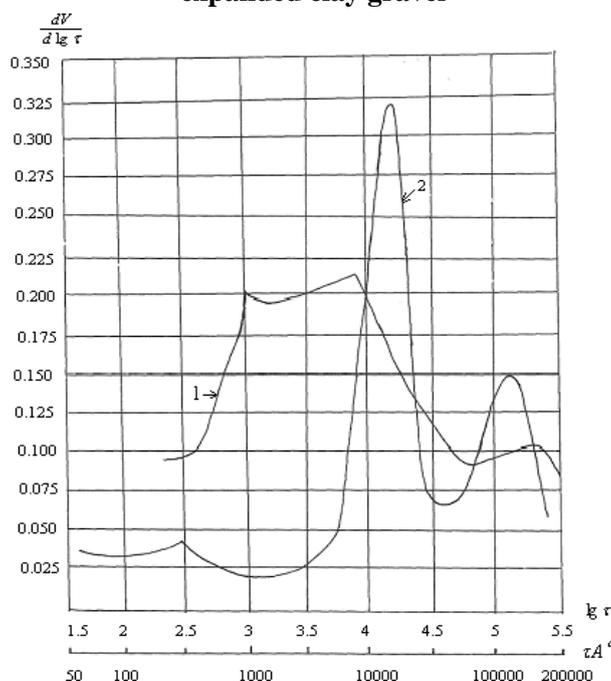


Fig.4. Differential porosity curves of aggregates: 1 - gravel of quartz porphyry and carbonized clay; 2 - expanded clay gravel

The differences in the structures of expanded clay and granules of quartz porphyry can be explained by the feature of their pore formation. Pellets of quartz porphyry acquire porosity due to the burning of coal and organic matter. Pore formation occurs with virtually no change in the volume of the initial granule grains. In claydite, the same process occurs mainly due to the expansion of the pyroplastic melt with a significant change in the initial volume of raw granules. Figure 3 shows the integral curves of mercury indentation for granules of quartz porphyry and keramisite gravel, obtained by replacing the magnitude of the pressure on the axis of the abscissas with the values of the logarithms of the radii. Figure 4 presents the differential curves of porosity of the investigated aggregate. From fig. it is seen that the porosity of expanded clay has clearly defined maxima per pores in the range of radii (1000-8500A), while for the aggregate of quartz porphyry the maximum falls on pores with radius from 12000 to 20000 A.

The main characteristics of the porous structure of expanded clay and quartz porphyry aggregate, and of carbonized clay, determined by the indentation of mercury, are summarized in Table 3.8: pore volume in the range of radii (30-500A); pore volume in the range of radii (500-5000A); pore volume in the range of radii (> 5000A); total porosity in the volume of all open pores available to this method. The average statistical pore diameter D , determined by the formula:

$$A = \frac{\sum A_i V_i}{\sum V_i}$$

Where, D_i - average pore diameter in this range, A;
 V_i - pore volume of diameters D_i , cm^3

5. SUMMARY:

An analysis of the data showed that in the quartz porphyry and carbonized clay aggregate, 65 % of all mercury-filled pores are macropores (> 5000 A radius). In expanded clay, macropores are about 35 %, and most of the pores are much smaller and are in the range of radii of 500-5000 A. The average pore diameter of gravel from quartz porphyry is 2 times greater than D expanded clay aggregate. This indicates a larger-porous gravel structure made of quartz porphyry and carbonized clay compared with expanded clay. The above indicates the specificity of the structure of gravel from quartz porphyry and its difference from the cellular structure of expanded clay. This implies an improvement in the properties of lightweight concrete on such a porous aggregate.

Physico-chemical studies (X-ray structural, DTA and mercury porometry) confirmed the optimality of the parameters for the preparation of porous aggregate. It has been established that the phase composition of the porous aggregate consists mainly of the minerals mullite and cristobalite, and its structure is capillary-porous. At the same time, the average pore diameter of gravel from quartz porphyry is 2 times larger than expanded clay aggregate, which indicates its more porous structure.

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