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Abstract. The article investigates the disintegration of perlite production waste in a vortex jet mill; the regularities of milling were established. Binding compositions were obtained at different ratios of cement vs. perlite sand production waste in the vortex jet mill in various milling regimes. The peculiarities of milling processes were studied, and technological and physicomechanical properties of the binding compositions were determined as well. The microstructure of the cement stones made of activated Portland cement and binding compositions in the vortex jet mill was elucidated by electron microscopy. The open pores of the cement-binding compositions prepared using perlite fillers were found to be filled by newgrowths at different stages of collective growth. The microstructure of the binding compositions is dense due to rationally proportioned composition, effective mineral filler— perlite waste — that creates additional substrates for internal composite microstructure formation, mechanochemical activation of raw mixture, which allows obtaining composites with required properties.

1. Introduction

The increase of energy efficiency and energy conservation currently are priority fields of the energy policy in Russia. When designing an energy saving house, one should primarily think about heat loss prevention through walling and only then about the optimal operation of engineering systems in the building, cutting expenses for lighting and introduction of non-conventional energy sources. Heat insulating materials, which main characteristic is heat conductivity, play a decisive role in creating optimal microclimate in rooms. Today, the problem of creating heat insulating mortars with improved heat-shielding characteristics is very urgent. The decrease in the density of heat insulating mortars demands elaboration of an effective composite binder, which is the objective of the present work.

2. Materials and methods

The initial materials were represented by cement CEM I 42.5H that complies with GOST 31108-2003 (Belgorodsky tsement, JSC, Russia) and perlite sand production waste. The composite binder was produced in a vortex jet mill VSM-01. The microstructure of the hydrated cement specimens and hydrated binding compositions cured for 28 days was studied on a TESCAN MIRA 3 LMU high-resolution scanning electron microscope. Physicomechanical properties of binding compositions were determined as per regulatory requirements.

3. Elaboration of binder composition

Currently, composite binders are widely implemented to rationalize cement consumption in concrete preparation and to obtain high-quality building materials for various purposes [1–20].

Earlier works have established high efficacy of perlite production waste usage in composite binders for heat insulating mortars. For instance, the introduction of perlite production waste obtained in ball and vibration mills in an amount of 5–10% and plasticizer into composite binders almost doubled the compression strength [20]. In this connection, the authors have stated the problem of producing composite binders in a vortex jet mill and discovering the peculiarities of milling processes and scrutinizing the properties of produced composite binders.

A vortex mill is a gas-dynamic disintegrator, in which cascaded impact disintegration occurs at low collision speeds that are close to the material fracture threshold.

The vortex jet mill VSM-01 has the following characteristics: working chamber lining is "lunar surface"; working pressure is 10 bar; air consumption is 1 m^3/min; the productivity depends on the properties of a material. Technological and economical advantages: no local heating; no moving elements; small size, easy servicing, safety and reliability; low input air pressure (for typical jet mills, the compressed air pressure is 0.7...1.4 MPa; for vortex mills, the similar results can be achieved under 0.2...0.6 MPa) [17].

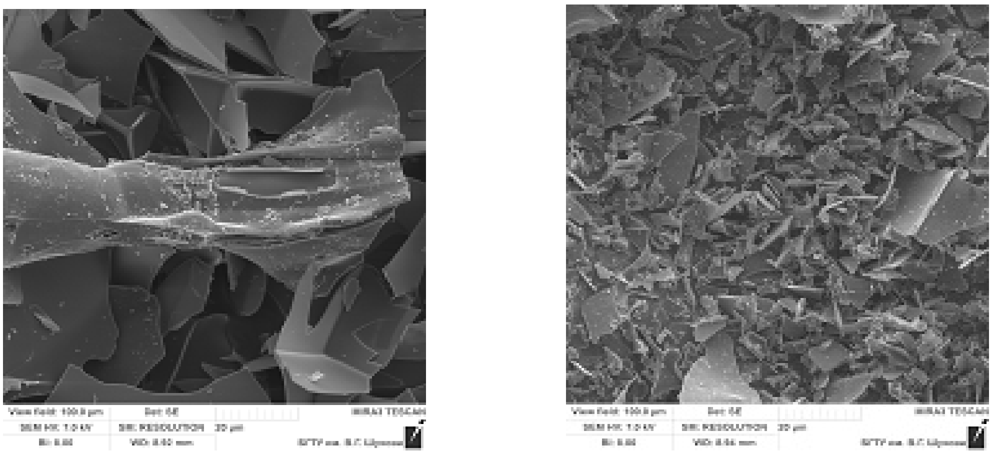
According to the stated objective, at the first stage, the peculiarities of perlite production waste milling in this mill were studied. Earlier, electron microscopy has established the distinctive features of milling of inflated perlite grains in a ball mill that take splintered laminar shape during milling, which favors the formation of a highly dispersed spatial structure of binding compositions in created dry heat insulating mortar on its basis [17]. The analysis of milled perlite grains in a vibration mill has demonstrated that the grains in the process take ball-ellipse shape, which increases the normal consistency of binding compositions due to entrapment of water by ball-shaped perlite grains. The experimental milling of perlite grains in the vortex jet mill has established that perlite grains have laminar prismatic shape (Figs. 1a and 1b), which is evident on microphotographs. Obviously, milling in the vortex jet mill has impacted the formation of perlite waste particles that has take the shape of "chocolate crumbs".

The experimental curve for finished cement in Fig. 2a demonstrates the particle size distribution from 0.1 to 100 μm with the specific area of 12637 cm^2/cm^3.

The distribution curve for perlite waste (Fib. 2b) shows the particle distribution from 1 to 100 μm; the specific particle surface area is 3099 cm^2/cm^3. At nearly equal particle distribution, the specific surface of the cement is more than 4 times larger than that of the perlite waste. This is explained by the cement particles having grain shape, while perlite waste particles have laminar shape (with considerable fineness of the particles, they have larger length and width). The microstructure of the hydrated Portland cement specimens (compositions 1–4) and hydrated binding compositions (compositions 5–16) cured for 3 and 28 days was studied on a TESCAN MIRA 3 LMU high-resolution scanning electron microscope.

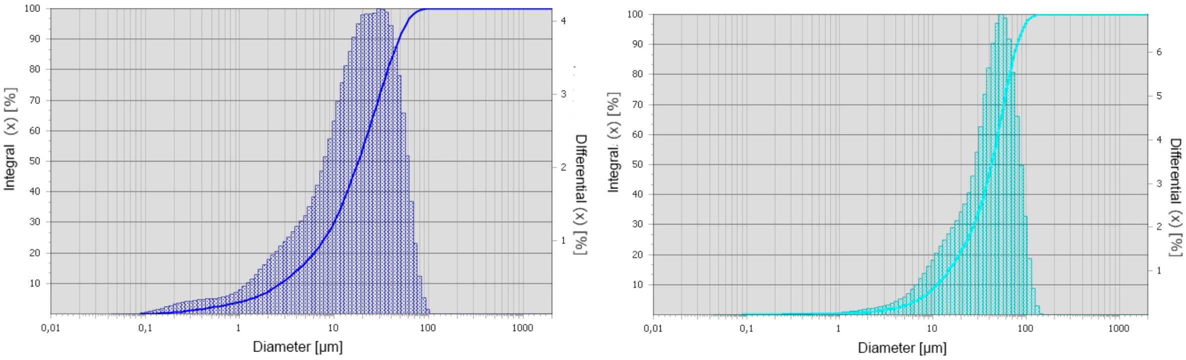
The electron microphotographs of 28-day cement stones, obtained using the scanning microscope from the surface of fractures, depict two images for each of the most typical sections with different magnification (Figs. 3–8). The microstructure of the studied hydrated Portland cement specimens (compositions 1–4) are presented in Figs. 3–6.

The hydration of cement powder is known to evolve along its surface, i.e. it obeys the Gibbs' phase rule. The heterogeneity of cement stone is determined during the initial curing period by the heterogeneity of initial cement grain and clinker, which is achieved due to the high specific area through activation of Portland cement and cement binding compositions.



a) (×100 µm) b) (×100 µm)

Figure 1. Initial perlite waste (a) and perlite waste milled in vortex jet mill (b)



a) Portland cement (PC) b) Perlite sand (PS)

Figure 2. Cement particle size distribution (a) and perlite waste particles size distribution (b)

The authors have studied the fracture surface of 28-day cement stone (Fig. 3) to compare it with the structure of hydrated specimens on the basis of binder compositions. According to conventional vision, the development of the cement stone crystalline structure occurs in two stages: at the first stage, a spatial crystalline frameworks and intergrowth contacts are formed (two types of frameworks are possible: hydrosulphoaluminate and hydrosilicate ones); at the second stage, overgrowing of the existing framework occurs.

Table 1. Composition of binders

No. of composition	Name	Amount of water [ml]	Normal consistency [%]	curing [min]		density [g/cm^3]	R_{compr}, MPa	
				start	end		after 3 days	after 28 days
1	PC0	116	29	169	271	2.3	40.1	43.1
2	PC1=>(1 pass)	128	32	121	199	2.1	46.3	47.2

3	PC2=>(2 passes)	134	34	78	169	2.1	45.5	49.0
4	PC3=>(3 passes)	168	42	124	191	2.1	48.4	50.1
5	KV1.0=>PC/PS=95/5%	203	41	252	378	1.8	13.6	25.8
6	KV1.1=>PC/PS=95/5% (1 pass)	165	44	146	260	2.0	41.9	51.6
7	KV1.2=>PC/PS=95/5% (2 passes)	177	45	172	267	2.0	34.8	38.1
8	KV1.3=>PC/PS=95/5% (3 passes)	180	51	157	244	2.0	42.2	52.0
9	KV2.0=>PC/PS=92.5/7.5%	260	65	77	434	1.7	6.9	13.2
10	KV2.1=>PC/PS=92.5/7.5% (1 pass)	175	44	84	278	1.9	31.9	38.0
11	KV2.2=>PC/PS=92.5/7.5% (2 passes)	180	45	76	243	2.0	20.0	41.8
12	KV2.3=>PC/PS=92.5/7.5% (3 passes)	185	46	137	251	2.0	23.4	31.6
13	KV3.0=>PC/PS=90/10%	250	63	30	406	1.6	5.8	13.2
14	KV3.1=>PC/PS=90/10% (1 pass)	180	45	20	275	1.8	23.8	45.5
15	KV3.2=>PC/PS=90/10% (2 passes)	184	46	20	168	2.0	15.3	53.3
16	KV3.3=>PC/PS=90/10% (3 passes)	186	47	20	140	2.0	21.8	47.8

The microstructure of the Portland cement stone from normal-consistency paste cured under normal conditions for 28 days (Fig. 3) consists of separate aggregate blocks formed by parallel-oriented layers of scale-like polycrystals and bundles of crystalline hydrated compounds with the dimensions of several micrometers. The lamination of hydrosilicate structure is not always clear.

The crystalline phase of activated cement stone (1-3 passes through the mill) is presented (Figs. 4–6) by the block of crystals represented by hexagonal prisms and plates twinning as a result of geometric selection of growing crystals. In addition, in the cement stone, there are crystals and crystal aggregates, druses, at different stages of geometric selection of growing crystals in constraint environment. The figures demonstrate the overgrowing of the cement stone by hydrated compound and their diminishing, which is important for solidification of the stone and its strength formation. The same photographs illustrate well the growth of separate prismatic crystals of secondary Portlandite in the direction perpendicular to the initial surface of pore walls; the intergrowth of prismatic crystals due to geometric selection of growing crystals is evident.

1000 µm 100 µm 10 µm

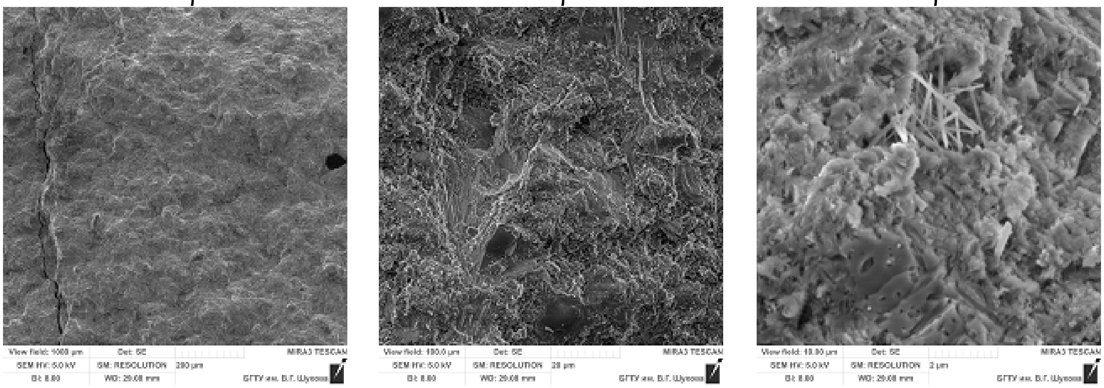


Figure 3. Cement stone microstructure cured for 28 days

1000 µm 100 µm 10 µm

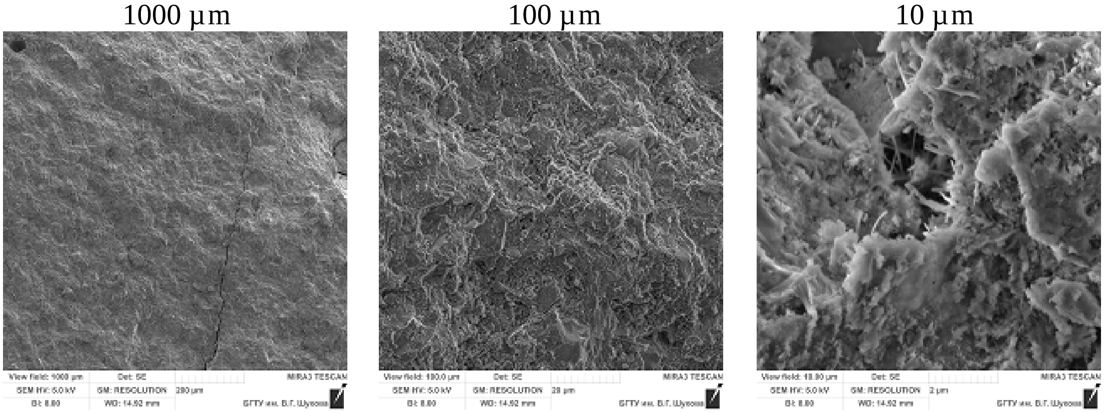


Figure 4. Cement stone microstructure from Portland cement activated in vortex jet mill (1 pass) cured for 28 days

1000 µm 100 µm 10 µm

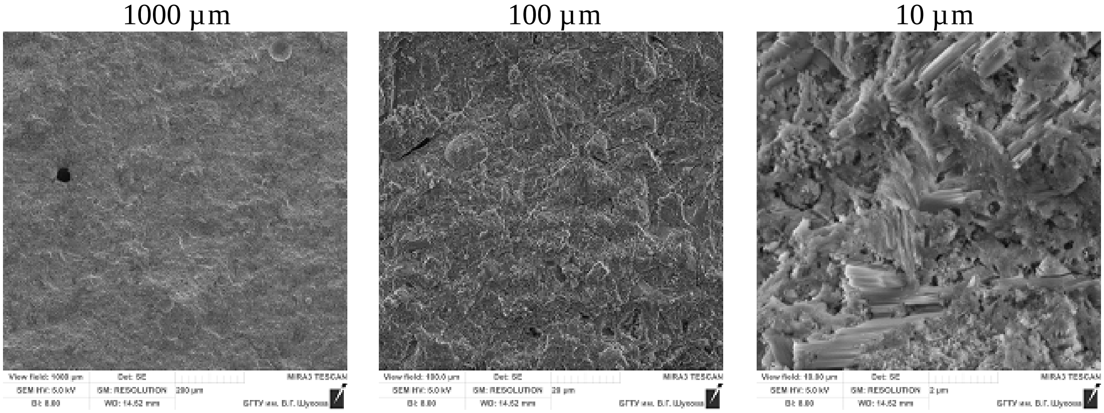


Figure 5. Cement stone microstructure from Portland cement activated in vortex jet mill (2 passes) cured for 28 days

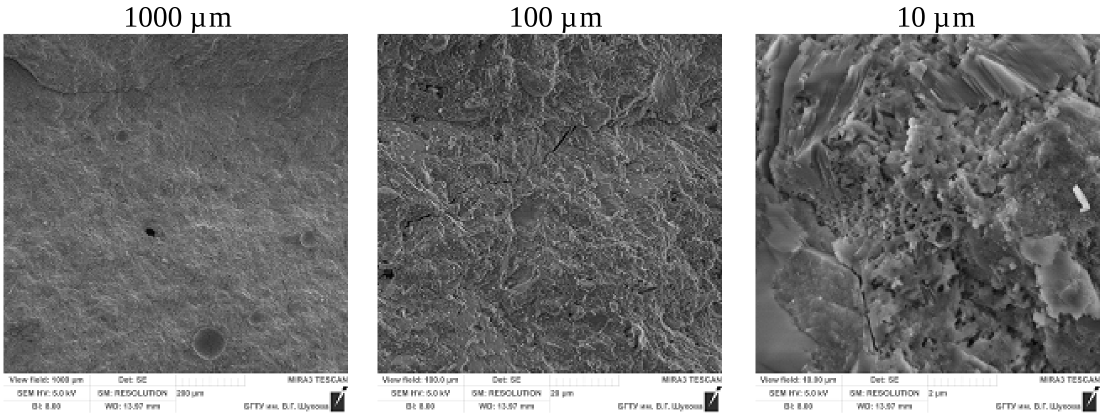


Figure 6. Cement stone microstructure from Portland cement activated in vortex jet mill (3 passes) cured for 28 days

Electron microscopy was used to study the microstructure of cement stones produced from synthesized binding compositions 5–16 (Table 1, Figs. 7 and 8). The block-rhythmic structure of the cement stone and the microstructure of separate blocks were revealed. It was established that the scales of calcium hydrosilicate have intergrown in many spots along the whole volume, i.e. there is the process of selective recrystallization conditioned by the presence of silicate component—perlite production waste. Also, the presence of perlite grain-plates is noted along the whole volume. Along the whole area of the fracture, there is active overgrowth of pores represented by net-like structures; this process evolves through dissimilar stages, which results from different mineral composition of initial clinker grains of cement and introduces perlite waste into binding compositions.

The cement stone cured for 28 days is a conglomerate consisting of blocks with different microstructures with its pores filled by single crystals and their druses. The blocks with a distinguished parallel laminar structure can consist of prismatic crystals of portlandite, hydrosulphoaluminates and hydrosulphoferrites. The size of blicks is determined by geometric shape, mineral composition, size of initial cement grains and perlite filler, and their mutual arrangement, compaction and occupation of interspace between them and water as well. The microphotographs (Figs. 7 and 8) evidence different look of the blocks: one of them has clear lamination, others are shapeless mass; this is explained by different orientation of blocks, the fracture planes in one blocks go along a layer, in others they follow the scales. At large magnification, the shapeless mass is evidently layers of scales. Both types of blocks are calcium hydrosilicates of different composition.

During the formation of the microstructure, simultaneously with the crystal growth in the structures of external rhythm and occurrence of crystal seeds in the structures of internal rhythm, the structure of internal rhythm suffers the dissolution and recrystallization of crystals and their seeds. The crystallization and recrystallization of newgrowths in the cement stone are accomplished in the presence of amorphous silica introduced by perlite waste, which by affecting the crystallization behavior can either be a part of a crystal and form solid solutions or represent inclusions in solid or liquid (mother solution) state. This is facilitated by crystallochemical peculiarities of a calcium hydroxide structure that suggest a wide range of stable and meta-stable substituting. During the cement stone structure formation, calcium hydroxide is the matrix for introduction of various elements and ions into it with consequent formation of hydrate compounds. This explains the presence of calcium hydroxide structural elements in many hydrated compounds of the cement stone. The preliminarily formed calcium hydrosilicates with the structure of calcium hydroxide suffer a solid-phase process of polycondensation of SiO^4-tetrahedrons as per scheme developed by N. V. Belov [19] with the formation of more complex calcium hydrosilicates with liberation of free calcium hydroxide. The latter after entering the solution migrates along capillaries and pores and crystallizes in different locations depending on the conditions in the system.

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1000 µm 100 µm 10 µm

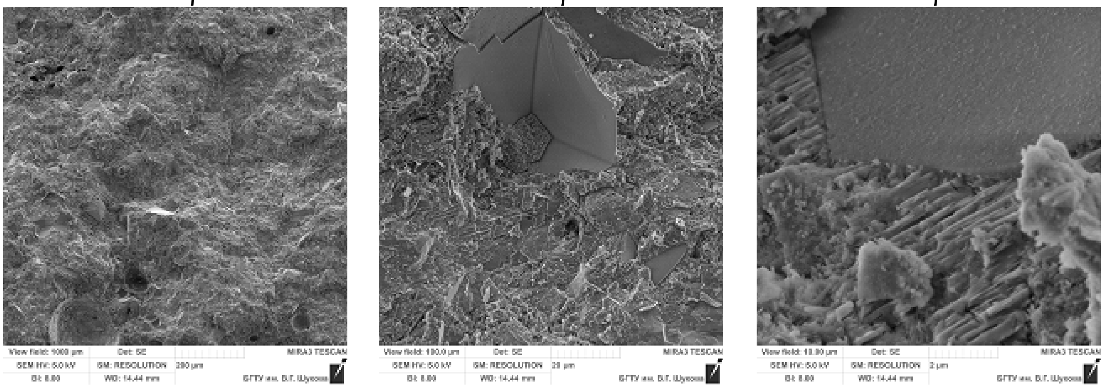

Figure 7. Cement stone microstructure from binding composition 6 cured for 28 days

1000 µm 100 µm 10 µm

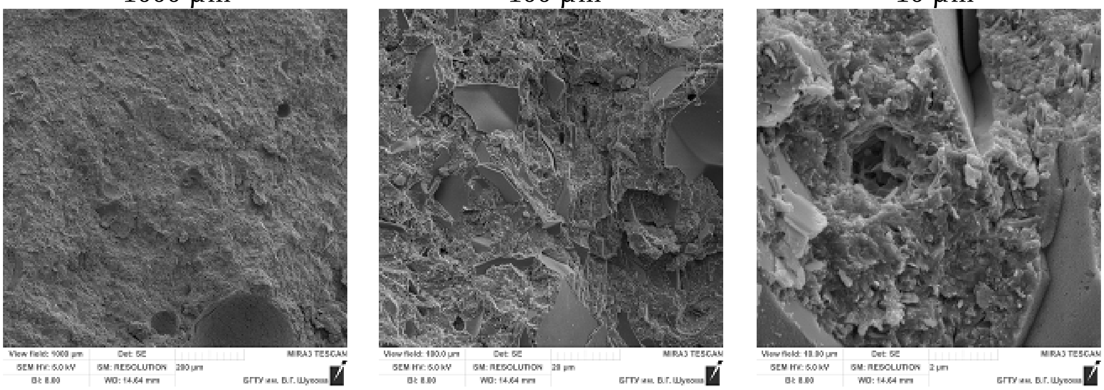

Figure 8. Cement stone microstructure from binding composition 15 cured for 28 days

By the 28th day of stone curing, the decreased concentration in the liquid phase results in spatial recrystallization of prismatic crystals of calcium hydroxide into hexagonal plates occupying entire free space between initial cement grains. Hexagonal plates first form pores in the center surrounded by acicular portlandite crystals. This testifies high supersaturation of a liquid phase at the pore's base and low supersaturation in its center. Further recrystallization and growth of hexagonal crystals of portlandite obey the laws of collective crystal growth and carry on metasomatically. This results in blocks of twining laminar portlandite crystals spatially oriented by boundary planes of internal rhythm structures, corresponding to the contours of initial cement grains.

The structure of internal rhythm is formed inward from the initial cement grain surface and consists of crystal seeds — scales precipitating from highly supersaturated solutions in the volume of the dissolved compound. The rhytmicity of the hydration process determines the laminar arrangement of scales in the structure. The structure of the external rhythm is formed outward from the initial cement grain surface and consists of crystals obeying the laws of collective growth in constrained environment. This structure with the curing time of cement stone is subjected to genesis. The portlandite crystals initially crystallize from highly supersaturated solutions as hexagonal prismatic crystals; then, at reduced concentration of liquid phase, they recrystallize into hexagonal plates. This recrystallization begins already inside a pore and is directed towards the surface.

This very complex process results in the formation of a block-rhythmic microstructure of the dense fraction of the cement stone, which in combination with pores and non-hydrated cement grains, represents the structure of the cement stone. The formed structure constantly changes in time, which is conditioned by the aspiration of a heterogeneous material to equilibrium. The structure of the cement stone is known to determine its properties, primarily strength. Strength and porosity are in direct dependence. The porosity is classified into open (capillary) and closed (vacuolar). It was established that with similar porosity, the presence of the second type of pores improves the technological properties of the cement stone. Pores of the first type are initially formed by the gaps between cement grains trapped in the constrained volume. In normal conditions of cement curing, they are always filled with water; their number and size with the curing time decrease. Pores of second type are filled with air and remain practically unaltered with curing time.

According to electron microscopy of cements and binding compositions prepared using perlite fillers, open pores are always filled with newgrowths at different stages of collective growth: single crystals or their druses; separate crystal growth, completely overgrown pores and as blocks in the stone structure. A specific role in the formation of theses newgrowths is played by mineral fillers—perlite production waste.

The analysis of the binding composition microstructure testifies the creation of a densely intergrown structure due to a rationally proportioned composition, effective mineral filler — perlite waste — that creates additional substrates for internal composite microstructure formation, additional activation of raw mixture, which allows obtaining composites with required properties.

Thus, the study of the microstructure of Portland cement and binding compositions activated in the vortex jet mill has convincingly proven previously obtained physicomechanical characteristics of these binders.

4. Conclusions

The results allow concluding that the binding compositions obtained in the vortex jet mill with different variants of mixing of initial materials have demonstrated that the obtained composite haspeculiar features conditioned by the shape and size of particles and various technological and physicomechanical properties. The analysis of the binding composition microstructure testifies the creation of a densely intergrown structure due to rationally proportioned composition, an effective mineral filler — perlite waste — that creates additional substrates for internal composite microstructure formation, additional activation of raw mixture, which allows obtaining composites with required properties.

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