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Mechanical activation in the production of lime-sand mixtures

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Abstract. This paper presents a review of the author's research of the effect of different grinding units' on the physical and chemical profile and geometry of quartz powders. Thus, when quartz sand is being treated in a vortex layer machine for 3–5 minutes, the mean particle size decreases from 235 to 6–7 µm, and the maximum size – from 498 to 81–77 µm. In this case, the specific surface area of the quartz powder, determined by the BET method, is 1.14–1.5 times larger than that of the powder obtained in other grinding units. It was shown that only the treatment of quartz sand in the vortex layer machine allows identifying this process as mechanical activation. The same positive results were obtained when treating building lime in the machine. There is an intensification of the building lime hydration process observed. The rational treatment time of the lime-sand compounds mixture in the vortex layer machines is 5 minutes. For compounds obtained from such mixtures at the age of 28 days, an increase in compressive strength by 209 % and bending strength by 172 % is observed. A decrease in the porosity of the compounds by 24 % was also found. Increasing the time of treatment of a lime-sand mixture in the vortex layer machine for more than 5 minutes has shown a slight effect on the physical and mechanical properties of the materials obtained.

1. Introduction

The actual problem for the construction industry is to increase strength and durability of construction composites. One of key propriety area is the increase of physical and mechanical properties of lime-sand compounds, reducing the energy intensity of production. In compliance with topic of this article the subject of investigation is to study the effect of treatment of lime-sand and quartz powder on the physical and mechanical properties.

Nowadays, various types of grinding mills and disintegrators are used for grinding raw components [1]. It is known that during the grinding process the dispersion of particles, the roughness of their surface and, in some cases, the crystallinity degree of the raw components coating surface are being changed. These changes affect the interfacial intensity of physiochemical processes, which often leads to accelerated hydration and hardening of inorganic binder compounds [2−6].

Therefore, with all other things being equal (dispersion, component type), the impact of the grinding equipment type on the strength properties of the building compound seems natural. In particular, they tried to explain the different strengths of lime-sand materials by the different shape of quartz powder particles, their different structural strength and different solubility both in water and in different environments, etc. [7].

The paper [8] gives a hypothesis which associates the compounds strength with the quantity, speed and inter-beat intervals of disintegrator grinding bodies.

The papers [9, 10] present the results of a comparison of different grinding machines, including the vortex layer machine, and testify that the strength of the obtained building compounds primarily depends on the machine power rating and the treatment time. The frequency and velocity of the working bodies' impacts on the mill charge is secondary.

The paper [11, 12] highlights that the type of a grinding machine significantly affects the surface properties of the powder obtained from quartz sand. Thus, in a ball mill the surface of the specified powder is

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smooth and flawless, in a centrifugal mill – the particles are sharp-edged and have a large number of cracks. In a bowl mill the powder contains a large number of sharp-edged grains.

The main task of materials science is to search new technological methods for the increase stability and durability of lime-sand compounds, reduce energy intense technique.

The study purposes were determination of the effect of lime-sand in VLM on surface area and morphology and also determination of the influence of mixed girding with lime-sand on the physical and mechanical properties.

According to the study purposes the following tasks were appointed:

– to indicate properties of quartz sand by the dependence of the type of grinding machine;

– to determine the effect of the treatment duration of lime-sand in VLM on temperature change kinetics in the slaking time;

– to established banding and compression strength of lime-sand products by treatment of lime-sand in VLM.

2. Research methods

A natural consequence of the previously established patterns is the dependence of the reactive capacity of the quartz powder on the crystallinity degree of the quartz surface layer and the total surface of the powder in contact with the medium (that is, taking into account the surface roughness, the presence of cracks). In a certain variation range of these powder properties, an increase in the compound strength should be observed (this statement is a consequence of the general theory of composite materials). Moreover, for each compounding “hydraulic binder – quartz powder” system the variation range properties will be individual. Therefore, of particular interest is the study of the “building lime – quartz powder" system, the components of which were treated in the vortex layer machine (VLM). The design and the treatment technique of components in the VLM are presented in the paper [13].

For the research, the 1st grade building calcium quicklime meeting the requirements of EN 459-1:2010 "Building lime – Part 1: Definitions, specifications and conformity criteria". The quartz powder was obtained from the quartz sand of the Kama-Ustinsky field deposit, which meets the requirements of DIN EN 12620-2008 “Aggregates for concrete”. The quartz sand fineness modulus is 1.2.

A size-consist analysis of the quartz powder was carried out on a Shimadzu SALD-2300 laser light scattering particle size analyzer.

The median diameter D50 (the average diameter with respect to which half of all sample particles by mass will be larger and the other smaller) and the diameter D98 (diameter with respect to which 98 % of all sample particles by mass will be smaller) were chosen as the main size distribution index. For the original quartz sand, the D50 value is 234.6 μm, and D98 is 498.4 μm.

The indicated quartz sand is also characterized by the dependence of the grain geometry on the type of grinding machine (Table 1). To obtain a quartz powder with roughly the same specific surface area, the grinding time in mills was 45–117 minutes, and in the VLM – only 5 minutes.

Table 1. Quartz powder properties depending on the type of grinding machine.

Property	Grinding machine type			
	ball	bowl	centrifugal	VLM
Specific surface area, m^2/kg	230±20	250±20	253±20	254±20
Specific area under BET, m^2/kg	6540	7120	8670	9860
Particles size, μm:				
− D98	120	85	75	77
− D50	12	8	6	6
Roughness coefficient	28.4	28.5	34.3	38.8

The data analysis (Table 1) shows that for quartz sand treated in various types of machines, the specific surface area and the average equivalent particle diameter differ slightly. The research results obtained by the BET method indicate that the specific surface area of the quartz powder treated in the VLM is 1.14–1.51 times larger than the specific surface area of the quartz powder treated in other studied machines. This indicates the formation of a rougher surface of the quartz powder treated in the VLM. The quantitative parameter characterizing the formation of a rough surface is the roughness coefficient (Table 1), equal to:

$$k_s = \frac{S_{ud,BET}}{S_{ud,g}}, \tag{1}$$

where $S_{ud,g}$ is the specific surface area, calculated according to the size-consist using the classical geometric formula establishing the relationship between the particle diameter and its specific surface; $S_{ud,BET}$ is the specific surface area determined by the BET method.

3. Results and Discussion

A logical consequence of an increase in the phase interface area is an increase in the number of reaction products formed at the interface boundary. Here, the authors purposely do not rely on "increase in reactive capacity", "increase in activity", etc. as the indicated terms (reactive capacity, activity) for processes running at the interface phase boundary depend not only on the contact area (extensive factor according), but also on the change rate in the substance physical properties (intensive factor according to [14]).

The specified features are clearly demonstrated in Fig. 1 and 2. Fig. 1 shows optical micrographs of quartz powder grains, and Fig. 2 – images obtained using the Auger microprobe JAMP-9500F (JEOL).

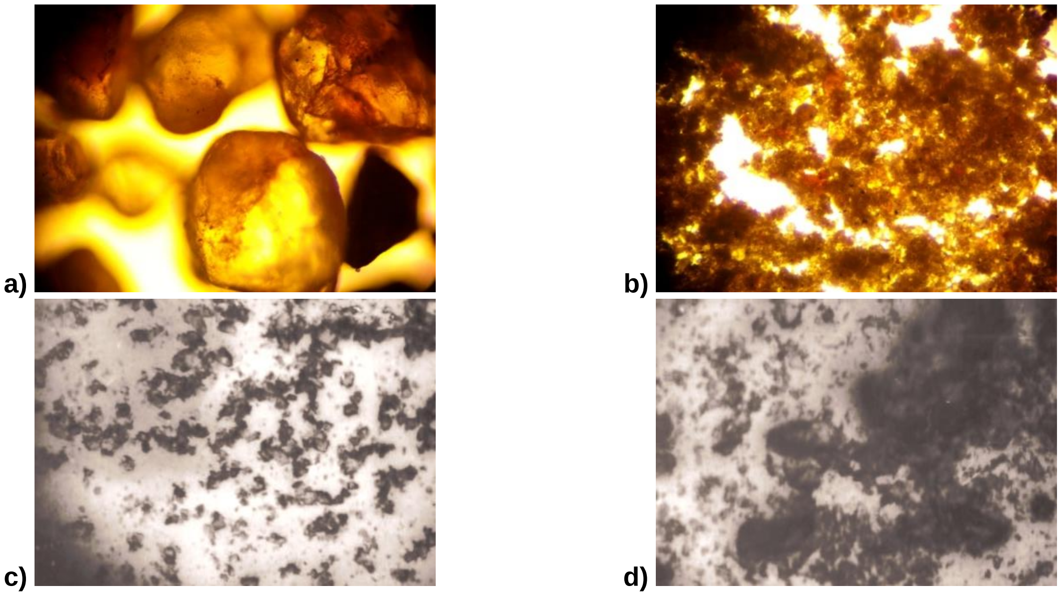

Figure 1. Micrographs of quartz powder grains (magnification 100$^{\times}$):
quartz sand grains; b) quartz powder after grinding for 5 minutes in the VLM;
c) the same in a centrifugal mill; d) the same in a ball mill

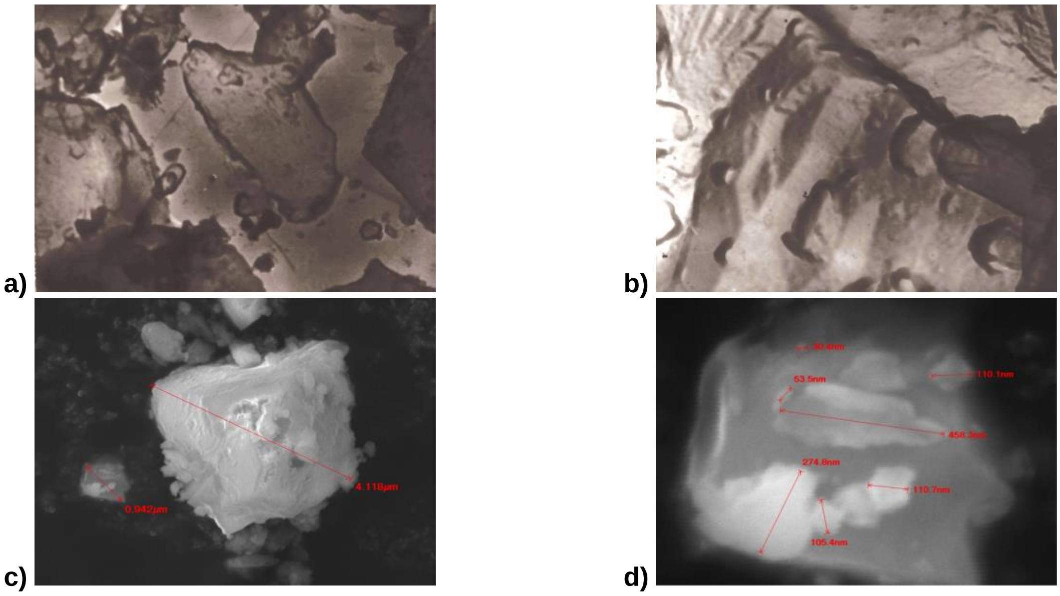

Figure 2. Electronic images of quartz powder grains:
a) treated in a ball mill (4000$^{\times}$); b) the same in a centrifugal mill (20000$^{\times}$);
c) the same in the VLM (10000$^{\times}$); d) the same in the VLM (75000$^{\times}$).

The particles of quartz powder treated in a ball mill have a smooth, flawless surface, which proves the grinding effect. The surface of the powder milled in a centrifugal mill is shard-edged with a fracturing pattern indicating the splitting action of the grinding machine. There are inclusions in the particles of quartz powder, indicating the impact effect during treatments in a centrifugal mill.

The particles of quartz powder treated in the VLM have a layer-like structure, a high degree of surface roughness, a large extended network of cracks, as well as impact-resulted inclusions. A feature of VLM-treatment is that when grinding quartz sand particles, separation occurs along individual conglomerates (Fig. 2 c), and not along cleavage surfaces, as it is in the case of ball and centrifugal mills (Fig. 2 a, b), which illustrates the VLM power rating.

The physical change of the silica in the powders obtained can be estimated by the change in its solubility. The silica content by dissolution in alkalis was defined. The results are presented in Table 2.

Table 2. The content of alkali-soluble soluble silica in the obtained powders treated in various grinding machines.

Grinding machine type	Alkali-soluble soluble silica content			
	Time after grinding*, days			
	0	10	20	30
Ball	0.0110 / $1.68 \cdot 10^{-6}$	0.0093 / $1.42 \cdot 10^{-6}$	0.0093 / $1.42 \cdot 10^{-6}$	0.0093 / $1.42 \cdot 10^{-6}$
Bowl	0.0130 / $1.83 \cdot 10^{-6}$	0.0110 / $1.54 \cdot 10^{-6}$	0.0110 / $1.54 \cdot 10^{-6}$	0.0110 / $1.54 \cdot 10^{-6}$
Centrifugal	0.0180 / $2.08 \cdot 10^{-6}$	0.0167 / $1.93 \cdot 10^{-6}$	0.0167 / $1.93 \cdot 10^{-6}$	0.0167 / $1.93 \cdot 10^{-6}$
VLM	0.0320 / $3.24 \cdot 10^{-6}$	0.0290 / $2.94 \cdot 10^{-6}$	0.0290 / $2.94 \cdot 10^{-6}$	0.0290 / $2.94 \cdot 10^{-6}$

Notes: the numerator presents the content of alkali-soluble soluble silica in wt. %; and the denominator – the same in % / m^2; * – the powders were stored under natural conditions.

The presented data (Table 2) clearly point to an increase in solubility of alkali-soluble silica for powders treated in the VLM. Given the natural dependence of the solubility of the substance on the contact area [15–20], the criterion confirming this conclusion is written as:

$$k_p = \left(\frac{S_{ud,n}}{S_{ud,i}} - 1 \right) \cdot \left(\frac{C_i}{C_n} - 1 \right), \tag{2}$$

where the index “*n*” indicates the parameters of the base powder (in this case, the powder treated in a ball mill); C standing for the content of alkali-soluble silica.

The values of k_p are presented in Table 3.

Table 3. Values of the k_p criterion.

Property	Grinding machine type			
	ball	bowl	centrifugal	VLM
k_p criterion	-	0.96	0.72	1.83

As can be seen from Table 3, an increase in solubility is observed only for quartz powder treated in the VLM more intensively as compared with an increase in the powder total surface (Table 1).

After storage under natural conditions, in all cases there is a decrease in the alkali-soluble silica content. The most intense decrease in alkali-soluble silica is observed for powders treated in ball and bowl mills, 15.5 and 15.4 % respectively. The lowest value of this parameter is observed for a centrifugal mill – 7.2 %, for the VLM – 9.4 %. These changes are observed only after 10 days of storage of the powders, and then stabilization is observed.

Summarizing the data presented, we can conclude that VLM quartz powder treatment is a very effective way leading to the formation of a quartz powder with a highly rough surface. The VLM treatment conditions have a natural effect on the particle size distribution of quartz powder: an increase in the VLM treatment time leads to a shift in the distribution maximum to the area of smaller sizes (Fig. 3). It should be noted that the maximum is diffused with almost uniform distribution of various diameters particles after treating quartz powder in the VLM during 3 minutes.

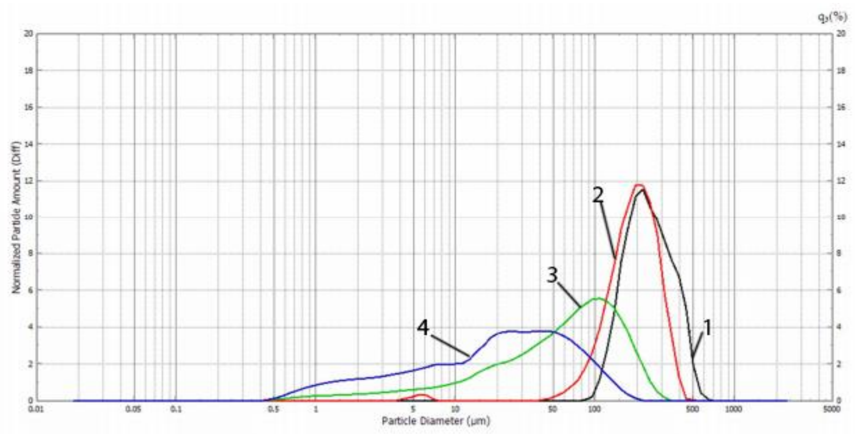

Figure 3. Quartz powder particles distribution: 1 – not VLM-treated; 2 – VLM-treated for 20 sec.; 3 – the same during 1 min; 4 – the same during 3 min.

In addition, it is significant that there is a fairly rapid decrease in both the average and maximum particle sizes in the VLM. Thus, when quartz powder is treated for 3 minutes, the average particle size D50 decreases from 235 to 7 μm, and D98 – from 498 to 81 μm.

Such influence on quartz powder should affect the properties of the structural formation of the “building lime – quartz powder” system. Such properties at the first stage were the temperature and the lime slaking time. The determination methods were in accordance with EN 459-2:2010 "Building lime – Part 2: Test methods".

Additionally, the effect of both VLM-treatments and its duration (3 and 5 min) were studied. The temperature change kinetics of the lime test is presented in Fig. 4, showing an increase in the temperature and a reduction in the slaking time with an increase in the VLM-treatment time. The change in the qualitative-quantitative characteristics/the rate of reaching the maximum temperature:

$$\vartheta = T_{\max} / t\left(T_{\max}\right), \quad (3)$$

(here $T_{\max}$ – is the maximum temperature; $t(T_{\max})$ is the time to reach the maximum temperature) demonstrates an increase in the chemical reaction rate of $Ca(OH)_2$. Thus, for the original building lime $\acute{\upsilon}_0$ = 29.8° C/min, for building lime treated in the VLM, $\acute{\upsilon}_3$ = 38.0 и $\acute{\upsilon}_5$ = 52.7 °C/min respectively, 3 and 5 min.

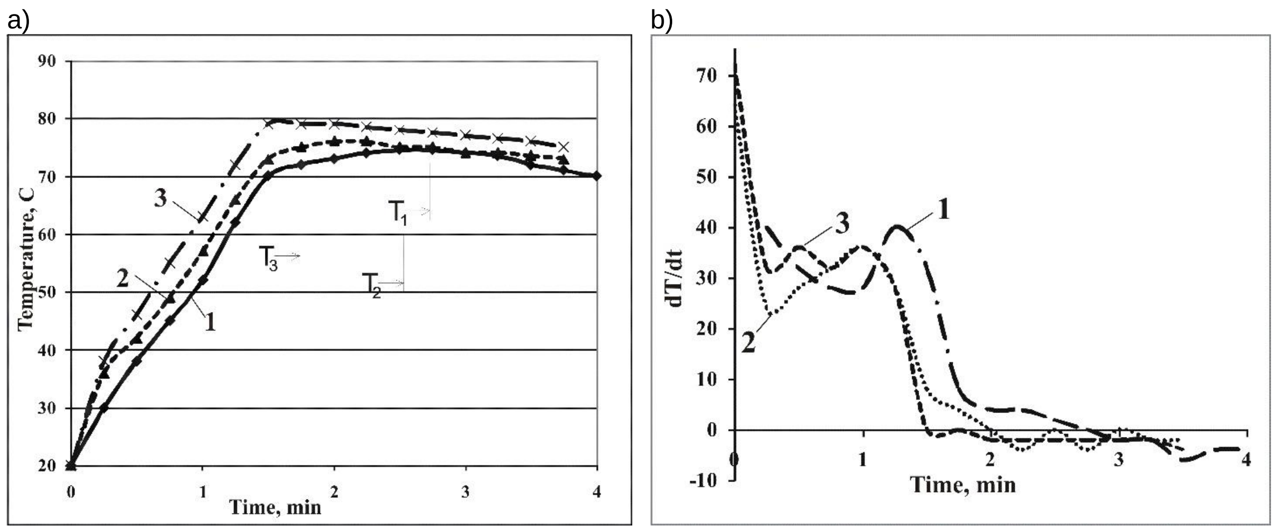

Figure 4. Kinetics of heat generation during a lime slaking treatment (a) and the dependence of dT/dt on the hydration time (b): 1 (T_1) – original building lime; 2 (T_2) – treated for 3 min in a VLM; 3 (T_3) – the same for 5 min (T_i – lime slaking time).

Another important kinetics feature of Fig. 4 is the presence of sections at $T = f(t)$, characterized by different changing rates (Table 4).

Table 4. Parameters of kinetic dependences of heat generation during the slaking of lime treated in a VLM.

Type of lime	Sections characteristics											
	Section I			Section II			Section III			Section IV		
	k_I	T_{max}, °C	$t(T_{max})$, min	k_{II}	T_{max}, °C	$t(T_{max})$, min	k_{III}	T_{max}, °C	$t(T_{max})$, min	k_{IV}	T_{max}, °C	$t(T_{max})$, min
Original	32	52	1	36	70	1.5	3.6	74.5	2.75	–	–	–
VLM-treated for 3 min	64	36	0.25	26	49	0.75	32	73	1.5	1.6	75	2.75
VLM-treated for 5 min	72	38	0.25	32.8	79	1.5	-1.2	77.5	2.75	–	–	–

The presented data on kinetics of the heat generation during the slacking of lime lead to the conclusion that the lime treated in a VLM is characterized by a more intense interaction with water in the initial hydration period: in the first section, the k_1 coefficient is greater than the same indicator for the original lime. Moreover, very valuable information is contained in the dependence $dT/dt = f(t)$ presented in Fig. 4 b, which demonstrates that the processes of VLM-treated lime interacting with water are significantly more intense. The above follows not only from the value of dT/dt, but also from the amplitude and frequency of oscillations dT/dt in the time interval 0–2 min. Such temperature fluctuations are typical for the building lime hydration kinetics. The peculiarity of the building lime treated in the VLM is the shift of the velocity of the temperature peak to the origin of coordinates (for VLM treatments during 3 minutes) and an increase in the number of peaks for VLM treatments during 5 min should be noted that for the building lime treated in a VLM, a shift in time of reaching $dT/dt = 0$ is also observed. In consequence, for the original building lime, this condition is satisfied at $t_0 = 2.75$ min, for VLM-treated lime – 2.25 and 1.75 minutes for treatments during 3 and 5 minutes respectively.

Y. Khint proved the possibility to obtain silicalcite in different disintegrators. These devices are technically complex. Moreover, the rapid wear of the titanium alloy pins has led to the loss of the initial leading position of these machines in mechanical activation.

In this regard, we have obtained data on the effect of joint VLM treatment of building lime and quartz sand on the kinetics of changes in the strength of lime-sand products, hardened in natural conditions (Table 5) for the first time. The ratio of quartz sand to lime was taken equal 3:1. The strength of lime-sand products, the content of unhydrated grains were determined in accordance with EN 459-2:2010 "Building lime – Part 2: Test methods".

Table 5. Banding and compression strength of lime-sand products.

Time of VLM activation, min	Average strength values, MPa, in days								Average density, kg/m³	Porosity, %	Unhydrated lime grains content, %
	2		3		7		28				
	R_{bt}	R_b	R_{bt}	R_b	R_{bt}	R_b	R_{bt}	R_b			
0	0.31 100 %	1.5 100 %	0.51 100 %	2.4 100 %	0.67 100 %	3.2 100 %	1.2 100 %	5.3 100 %	1530±31	16.4±0.3	3.8
1	0.35 113 %	1.77 118 %	0.56 110 %	2.7 112 %	0.73 109 %	3.6 112 %	1.3 108 %	5.8 109 %	1545±31	15.8±0.3	3.3
2	0.38 123 %	2.08 138 %	0.64 125 %	3.26 136 %	0.83 124 %	4.32 135 %	1.46 122 %	7.0 132 %	1555±31	15.2±0.3	2.1
3	0.42 135 %	2.4 160 %	0.68 133 %	3.72 155 %	0.91 136 %	4.99 156 %	1.65 138 %	8.2 155 %	1560±31	14.3±0.3	0.9
4	0.48 155 %	2.74 182 %	0.77 151 %	4.32 180 %	1.04 155 %	5.71 178 %	1.79 149 %	9.5 179 %	1560±31	13.5±0.3	0.3
5	0.56 181 %	3.2 213 %	0.91 178 %	5.25 219 %	1.19 177 %	7.01 219 %	2.07 172 %	11.1 209 %	1570±31	12.8±0.3	0
8	0.56 181 %	3.28 218 %	0.92 180 %	5.28 220 %	1.20 179 %	7.03 220 %	2.07 172 %	11.5 217 %	1570±31	12.5±0.3	0

Notes: R_{bt} – is the ultimate bending strength, R_b – is the ultimate compression strength showing the absolute value of the indicator in the numerator and the relative one in % of the control in the denominator.

Table 5 shows that the maximum strength values of lime-sand compounds are observed after 5 minutes of a VLM-treatment of dry mixtures. For these compounds at the age of 28 days, the compressive strength increases by 209 %, and bending – by 172 % compared with the control composition. A further increase in the VLM treatment time almost never leads to significant changes.

A typical feature of all compounds obtained is a relatively lower rate of increase in the bending strength than in compression. With an increase in VLM-treatment time, a decrease in the porosity of lime-sand compounds up to – 24 % relative to the control composition is observed. When a mixture is treated in a VLM for more than 4 minutes, the presence of unhydrated lime grains is not observed, which is consistent with the results of the authors [14].

4. Conclusions

1. It was established that when quartz sand is treated in the VLM for 3–5 minutes, the average particle size decreases from D50 = 235 to 6–7 µm, and the maximum – from D98 = 498 to 81–77 µm. A similar quartz powder particles distribution can be obtained by means of treatments in spring and centrifugal mills for 45–117 minutes. That being said, the specific surface area of the quartz powder, determined by the BET method, increases significantly (by 1.14–1.5 times).

2. The peculiarity of VLM treatments is that when quartz sand particles are being ground, separation occurs along individual conglomerates, and not along cleavage planes, as compared to ball and centrifugal mills.

3. It was found that only for quartz powder treated in a VLM, an increase in solubility is observed more intensively as compared with an increase in the total powder surface. This allows us to conclude that the quartz sand treatment in a VLM leads to its mechanical activation, i.e., a nonlinear increase in the physiochemical property (in this case, solubility) with an increase in the interface phase is observed.

4. The building lime VLM treatment leads to a reduction of the lime slaking time from 2.75 minutes to 1.75 minutes, which naturally decreases with increasing treatment time in the VLM. In this case, there is a significant increase in the maximum hydration temperature, which illustrates an increase in the reactive capacity of building lime.

5. The kinetics of the lime-sand mixtures strength gain depending on the VLM treatment was established. The rational treatment time in the VLM was determined. It was found that when a VLM treatment lasts more than 5 minutes, the compressive strength of the lime-sand compound at the age of 28 days increases by 209 %, and the bending strength – by 172 %, as compared with the control composition. Moreover, there is a decrease in the porosity of the compound by 24 %. An increase in the VLM treatment time for more than 5 minutes does not lead to significant changes in the physiotechnical parameters of the studied compounds.

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