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Effect of Phosphogypsum Characteristics on the Properties of Phosphogypsum-Based Binders

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Abstract

Phosphogypsum, a byproduct of orthophosphoric acid production, is one of the large-tonnage wastes. Since phosphogypsum mainly consists of $CaSO_4$ $2H_2O$, it can be considered as an alternative gypsum-bearing raw material in the production of gypsum binders. However, its features, such as particle morphology and the presence of impurities, can negatively affect the characteristics of phosphogypsum-based binders. Identification of these factors will allow us to develop methods for their minimization and increasing the efficiency of phosphogypsum use from the required source as a raw material for the production of phosphogypsum-based binders. In this regard, the manuscript contains a comprehensive and comparative analysis of phosphogypsum and natural gypsum, which makes it possible to establish their differences in chemical composition and structural and morphological features, which subsequently affect the properties of the phosphogypsum-based binder. It has been established that the key factor negatively affecting the strength of phosphogypsum-based paste (2.58 MPa) is its high water demand (0.89), which is due to the high values of the specific surface area of the particles and the presence of a large number of conglomerates with significant porosity in phosphogypsum. It has been suggested that preliminary grinding of phosphogypsum can help reduce the amount of water required to obtain fresh phosphogypsum-based paste with a standard consistency and improve its physical and mechanical properties.

Keywords: phosphogypsum; phosphogypsum-based binder; gypsum-based binder; water demand of binder; physical and mechanical characteristics of phosphogypsum-based paste

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1. Introduction

The use of phosphogypsum as an alternative to natural gypsum in the construction industry is a relevant area of development of modern global trends in rational nature management and resource conservation. Phosphogypsum (PG) is a byproduct of the production of orthophosphoric acid and fertilizers. PG is a large-tonnage industrial waste, since the total global volumes are approximately 300 million tons per year [1–3]. At the same time, about 58% of PG is stored, 28% is discharged into coastal waters, and only 14% is recycled and reused [2]. Today, a large number of scientists around the world are looking for ways to reuse PG [1,2,4–6]. At the moment, there are a large number of studies aimed at considering the possibility of using PG as a raw material for the production of PG-based binders of β- and α-modification [7–9], as well as in multicomponent binders [10–13].

However, despite the large volume of research in this area, there are a number of key limiting factors that hinder the large-scale implementation of PG processing technologies into binders. The main ones are the presence of a large number of impurities, structural and morphological features of particles, high dispersion, etc. It should be noted that these features are predetermined by the original phosphate rock and the technological mode of orthophosphoric acid production and can vary significantly depending on the source of PG. The difference in the structural and morphological characteristics of PGs is naturally reflected in the properties of PG-based binders [6,7].

In particular, the analysis of literary sources showed that the compressive strength values of PG-based binders vary in a wide range: 1–14 MPa [7,14–16]. In addition, the authors in [7] previously studied PGs produced by three different industrial Russian enterprises and PG-based binders obtained by their calcination. The study showed that even under equal conditions of their manufacture (temperature and duration of calcination), PGs have significant differences in the values of standard consistency (0.7–0.92), initial setting time (11–17 min), final setting time (19–25 min), and compressive strength (1.72–3.38 MPa). Also, key structural and morphological features of the original PGs were identified, which have a negative impact on the characteristics of PG-based binders and, as a result, determine the differences in the properties of the binders, and ways to reduce their negative impact were proposed.

However, the identified PGs features are of a private nature. These features can differ significantly from each other depending on the PG source and, as a result, affect the performance characteristics of materials based on them in different ways.

In this regard, the object of this study was a comprehensive comparative analysis of PG and natural gypsum to identify genetic features that have a negative impact on the physical and mechanical properties of PG-based binders, which will subsequently allow us to select methods for their minimization, and, as a result, increasing the efficiency and percentage of processing and use of PG as raw materials for PG-based binder production.

2. Materials and Methods

The object of the study was PG provided by JSC URALCHEM, Voskresensk Mineral Fertilizers Branch (Voskresensk, Russia).

Natural gypsum (NG) from the Shedokskoye deposit (Krasnodar, Russia) and pure gypsum for analysis (PGA) (LLC «Crystal», St. Petersburg, Russia) were used for comparison.

XRD and XRF analyses of the PG and PG-based binder samples were carried out using an Ultima IV Rigaku high-resolution diffractometer (Rigaku, Tokyo, Japan). IR spectra were studied in the frequency range of 4000–370 cm^{-1} using a Vertex 70 IR Fourier spectrometer (Bruker, Berlin, Germany). Differential thermal analysis (DTA) was carried out using an STA 449 F1 JUPITER thermal analyzer (Netzsch, Selb, Germany), which provides synchronous thermal analysis.

Microstructures of the original PG particles, synthesized PG-based binders, and morphology of new formations in gypsum rock were carried out using scanning electron microscopy by electron microscope Mira 3 FesSem (Tescan, Brno, Czech Republic), operating in high vacuum mode (InBeam, GIFT City, India) using a high-brightness Schottky cathode. Samples were sputtered with chromium.

Measurement of the specific surface area (SSA) of PG particles by the Blaine method was carried out using a multifunctional measuring device, PSKh-12 (SP) (LLC «PSH», Moscow, Russia). Measurement of the SSA and pore size distribution was carried out using an automatic device, BELSORP miniX (MicrotracBEL Corp., Osaka, Japan).

Normal density, setting time, compressive and tensile strength for the binders were determined according to the Russian Standard GOST 23789-2018 (EN 13279-2: 2004, NEQ) "Gypsum binders. Test methods" [17]. The tensile and compressive strength was determined for prism samples of 40 × 40 × 160 mm. The pH value was determined using a pH meter pH-150M (RUE "Gomel Plant of Measuring Instruments", Gomel, Belarus). For the pH test, a solution was prepared by mixing 10 g of the solid component with 100 mL of distilled water.

The strength characteristics of the samples were determined using a laboratory press machine with a load of 10 tons.

The PG-based binder was obtained by keeping 2 kg of PG in a laboratory drying oven at 175 °C for 3 h. To achieve more uniform calcination, before placing it in the drying oven, PG was passed through a sieve with a mesh size of 2.5, which allowed breaking up large conglomerates. After calcination of PG, the resulting PG-based binder was removed from the laboratory oven and kept for 24 h in ambient laboratory conditions to average the composition.

For comparison, the study used a gypsum binder produced from natural gypsum (NG-based binder), which was precrushed to a particle size of no more than 2.5 mm and calcined according to the above-mentioned regime in a drying oven. After calcination, the binder was ground to a SSA corresponding to commercial building gypsum, which is ≈430 m^2/kg.

3. Results and Discussions

3.1. Analysis of Gypsum-Bearing Raw

Analysis of the chemical composition of FG (Table 1) showed the presence of a wider range of oxides than in NG (Table 2). The main oxides of FG are SO_3 and CaO. Phosphorus oxide is also present in the amount of 1.33%. According to the results of some studies [18–21], fluorine oxide can stick to the surface of gypsum crystals or even be embedded in the crystal lattice, negatively affecting the structure formation.

Table 1. PG oxides content (%).

SO_3	CaO	SrO	P_2O_5	SiO_2	CeO_2	Na_2O	Al_2O_3	TiO_2	Nd_2O_3	PuO_2	Fe_2O_3	MgO	Others
47.89	46.38	2.01	1.33	0.51	0.42	0.35	0.17	0.15	0.13	0.13	0.13	0.13	0.27

Table 2. NG oxides content (%).

SO_3	CaO	MgO	SrO	SiO_2	Al_2O_3	Others
49.83	49.62	0.135	0.123	0.064	0.037	0.191

XRD analysis of PG and NG showed the presence of all reflections corresponding to $CaSO_4$ $2H_2O$. However, comparison of the main peaks of $CaSO_4$ $2H_2O$ allowed us to establish that the values of intensity and half-width within one peak differ significantly from each other (Figure 1), which indicates differences in the crystal lattice parameters and the sizes of the crystallites in the studied gypsum-bearing components.

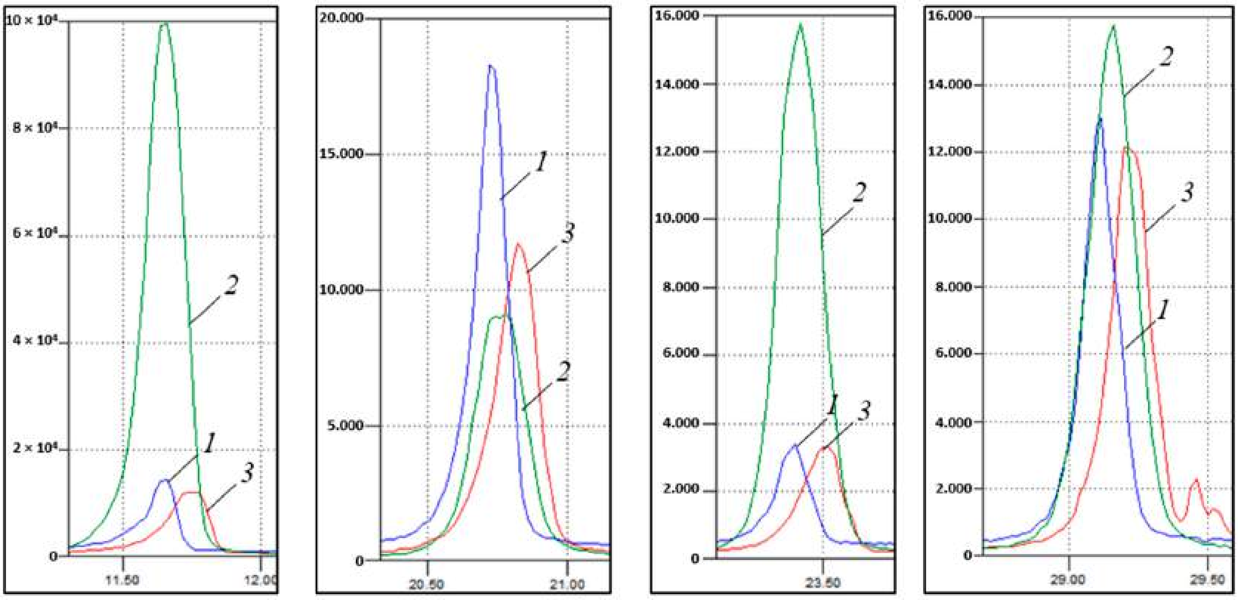

Figure 1. Reflections, corresponding $CaSO_4 \cdot 2H_2O$ phase in 1—PG; 2—NG; and 3—PGA.

The analysis of the IR spectrum of PG showed the presence of absorption bands corresponding to $CaSO_4$ $2H_2O$ (Figure 2). The peaks, corresponding to $SO_4{}^{2-}$ were detected at 602, 669, 1014–1140, and 2117–2244 cm^{-1}. The presence of H_2O is confirmed by the appearance of peaks at 1621, 1687, and 3200–3550 cm^{-1}. The band at 838 cm^{-1} indicates the presence of $HPO_4{}^{2-}$ ions and the formation of solid solutions with the following chemical composition: $Ca(SO_4)_{1-x}(HPO_4)_x$ $2H_2O$, where $x \approx 0.5$, is not clearly identified for the studied PG. DTA analysis showed that for the studied PG, the temperatures corresponding to certain phase transformations differ from NG (Figure 3). In particular, the removal of moisture (double endothermic effect) in PG occurs at lower temperatures than in NG. The restructuring of the crystal lattice with the formation of insoluble anhydrite in PG occurs at a higher temperature (444.1 °C) than in NG (353.6 °C) but is characterized by a less pronounced exothermic effect.

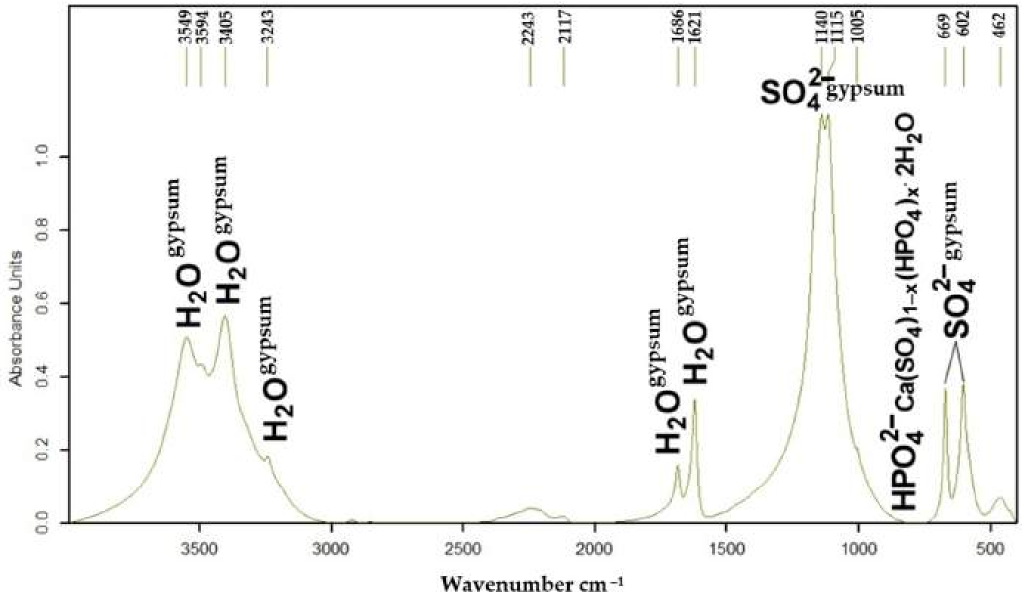

Figure 2. IR spectrum of PG.

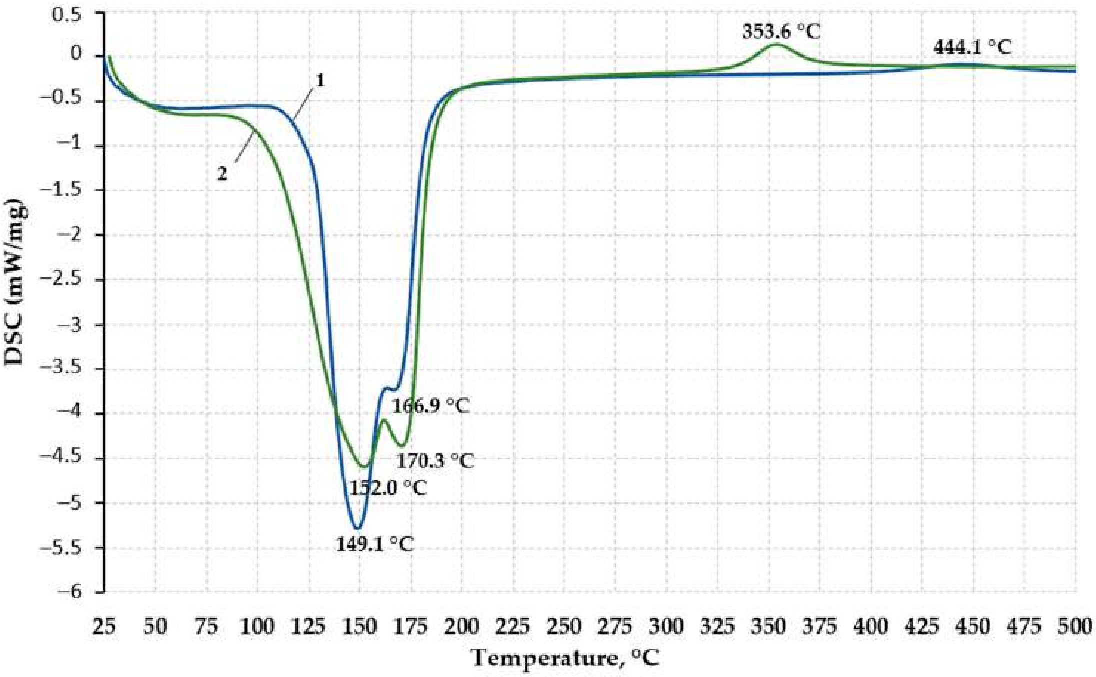

Figure 3. DTA diagrams of 1—PG and 2—NG.

The differences in the area of exo- and endothermic effects (peaks), temperatures, and main phase transitions are most likely due to the difference in the crystal lattice structure of NG and PG, as well as the presence of impurities in the PG. Analysis of the particle morphology revealed that PG is represented by fine rhombic particles ranging in size from 5 to 40 µm and their conglomerates ranging in size from 50 to 300 µm (Figure 4). It should be noted that the size of the conglomerates varies widely, and the largest ones are formed from smaller ones. It should be assumed that large conglomerates will be characterized by high internal porosity, which will consist of the porosity of individual particles, pores between particles in the structure of small conglomerates, and pores formed by small conglomerates in the structure of large ones. Since the structure of large FG conglomerates is similar to a sponge, it is likely that the PG-based binder will have high water demand.

The morphology of the studied raw materials (Figure 4) and the available morphological groups [6] demonstrate PG consists of particles that belong to the 2nd group (fine rhombic type) and their conglomerates. The fine rhombic type (2nd group) is characterized by a texture of small agglomerated needles 5–30 µm in size and a crystallite size of 70 to 300 nm. A more detailed description of all types of particles characteristic of PGs is presented in [6]. Visually, studied PG is a powdery material with an SSA of 133 m^2/kg (according to the Blaine method). When determining the SSA using the BET and BJH methods, it was found that PG has higher SSA (Table 3) than NG, which is due to the presence of a large number of small particles and their conglomerates in PG, which are characterized by a developed surface (Figure 4).

At the same time, despite the higher SSA, the average pore diameter of PG is more than 2 times smaller, and the total pore volume is 3 or more times smaller than for NG. The curve of pore volume corresponding to a specific diameter is polymodal for PG and unimodal for NG. FG is characterized by two peaks, at the maximum corresponding to a pore size of 4.24 nm (predominant) and 2.43 nm. NG has one peak, at the maximum corresponding to a pore size of 2.21 nm (Figure 5).

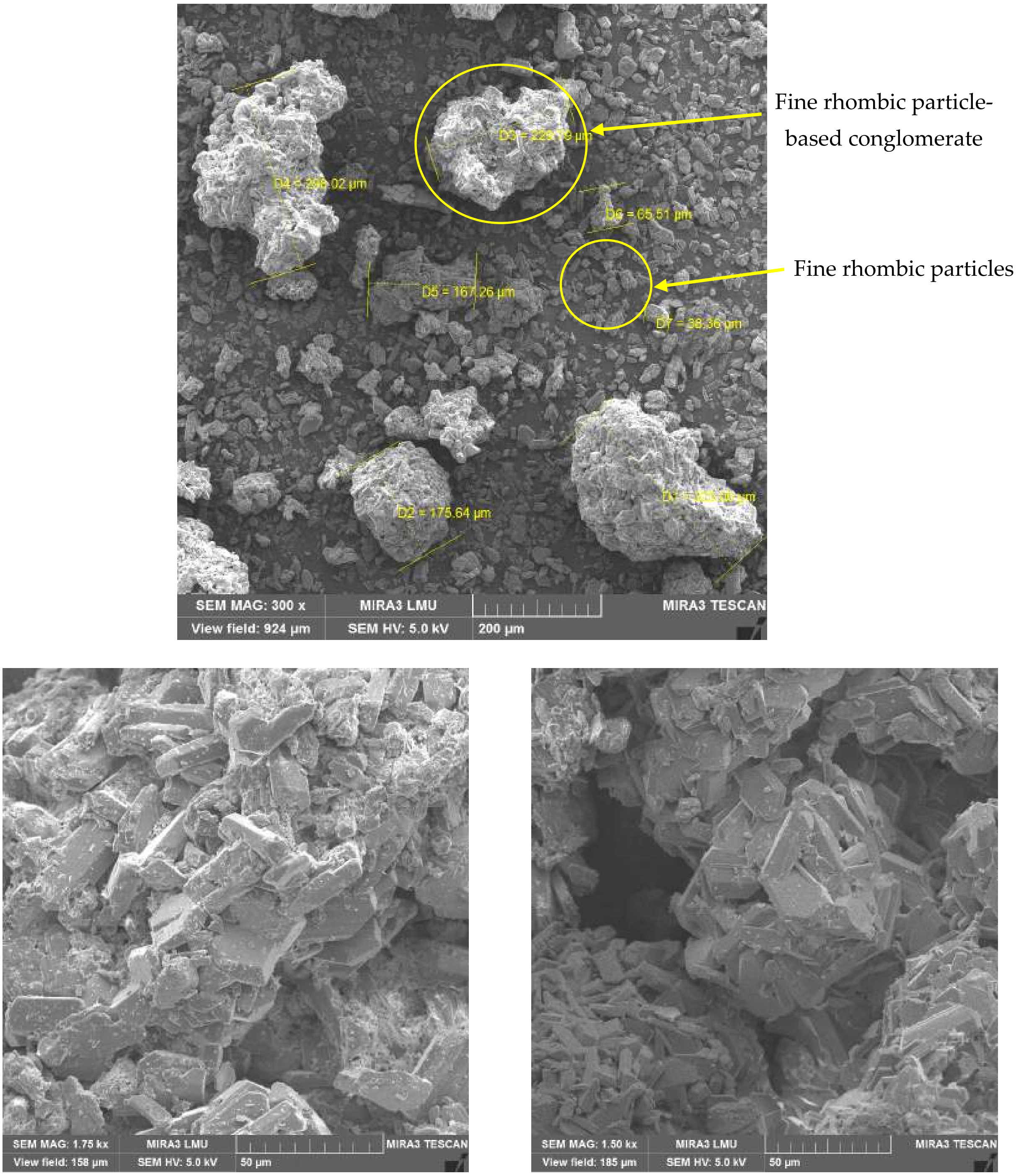

Figure 4. Morphology of PG particles.

The morphology of PG particles and, in particular, the presence of a large number of conglomerates, as well as high SSA, allow us to assume PG-based binders will have a high water demand, which, in turn, can negatively affect the physical and mechanical properties of the PG-based material. As is known, the pH of gypsum-containing raw materials has a great influence on the hydration of binders and, as a consequence, the physical and mechanical properties of the final products [22]. To establish the relationship between the pH value of the gypsum-containing raw materials and binders based on it, the pH for PG and NG was determined, and pH changes over time were studied (Figure 6).

Table 3. SSA and pore size distribution of PG and NG.

Parameter	FG	NG [7]
Blaine method		
SSA, m^2/kg	133.5	–
BET method		
SSA, m^2/kg	18,608	14,413
Total pore volume (p/p0 = 0.9900), cm^3/g	0.0282	0.053
Average pore diameter, nm	6.061	14.76
BJH method		
SSA, m^2/kg	18,793	14,912
Total pore volume, cm^3/g	0.027	0.072
Average pore diameter, nm	5.75	19.29

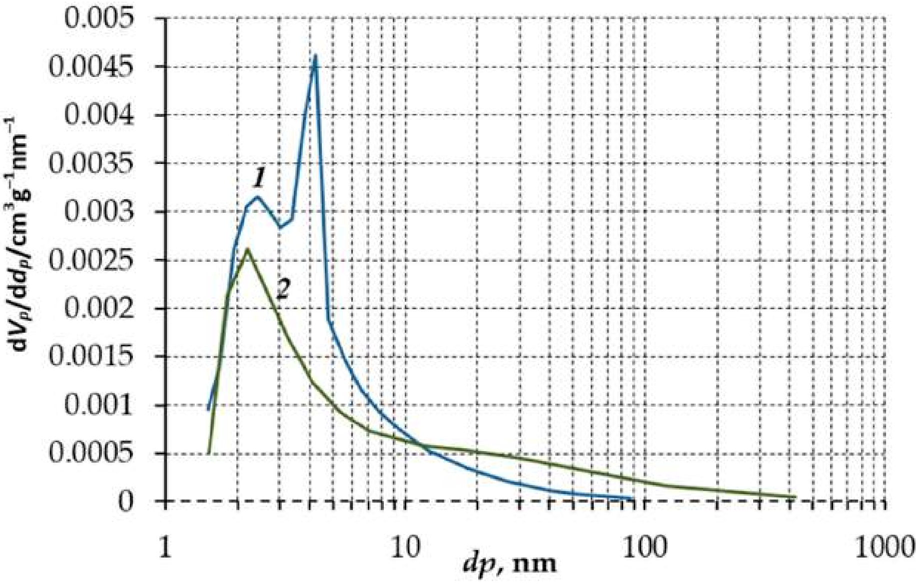

Figure 5. The pore volume corresponding to a specific diameter for 1—PG and 2—NG.

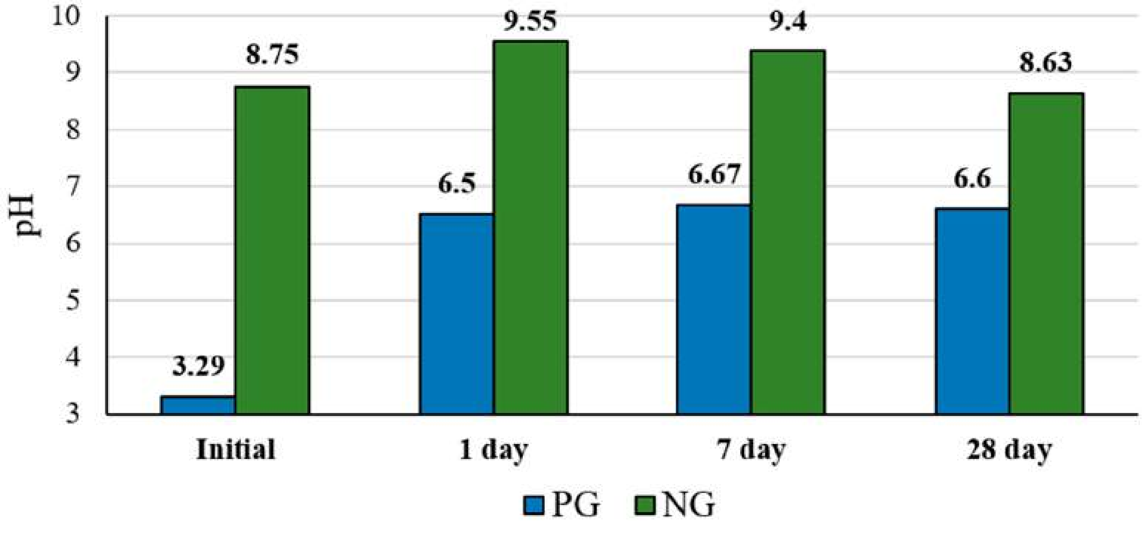

Figure 6. pH of gypsum-bearing raw materials.

The analysis of the obtained results showed that the initial pH for FG is 3.29, which can be explained by the presence of orthophosphoric acid residues in it. However, it should be noted that after an hour the pH for PG increases to 6.5 and then changes insignificantly. Such an extremal change is probably due to the presence of water-soluble impurities in FG, which either react with the orthophosphoric acid residues and lead to their partial neutralization or add water to form a compound with a higher pH value. At the same time, NG is naturally distinguished by a higher pH of 8.75, which also changes over time, but not as significantly as in PG.

3.2. Analysis of Binders Based on Gypsum Bearing Components

The main phases that are formed during the calcination of gypsum-bearing raw materials are calcium sulfate hemihydrate and anhydrite, which is transformed into $CaSO_4$ $0.5H_2O$ under open air. However, the presence of unreacted $CaSO_4$ $2H_2O$ particles in the binders is also possible. XRD analysis showed that, like the initial PG (Figure 1), the PG-based binders exhibit a shift in interplanar distances, as well as differences in the intensity and half-width of the main diffraction peaks typical for $CaSO_4$ $0.5H_2O$, compared to the PGA-based binder and NG-based binder under the same temperature regime (Figure 7).

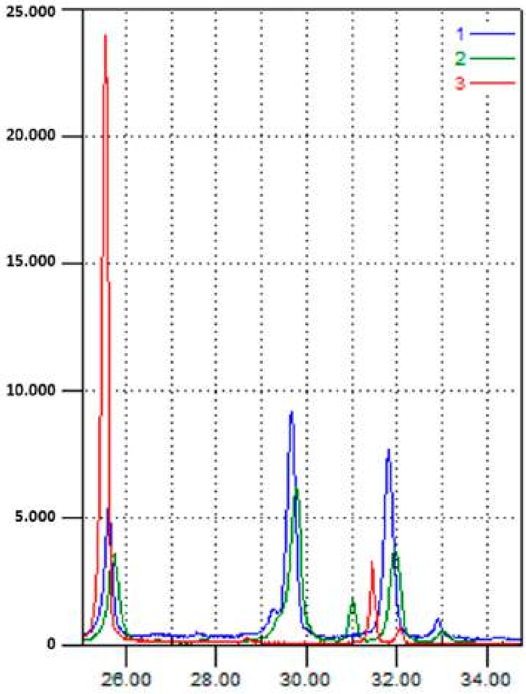

Figure 7. The main peaks corresponding to $CaSO_4{\cdot}0.5H_2O$: 1—PG-based binder; 2—NG-based binder; 3—PGA-based binder.

Analysis of the morphology of the PG-based binder particles showed that high-temperature exposure did not affect the particle shape. The particles retained the morphology of the original PG. However, the surface of the particles became looser and more developed due to the detachment of $1.5H_2O$ molecules (Figure 8).

Analysis of SSA values (according to the Blaine method) showed (Table 4) that high-temperature exposure did not affect the SSA of PG-based binder. This is due to the structural and morphological features of PG and, in particular, the presence of a large number of conglomerates in PG, which have sufficiently large cohesive forces between particles. According to the results obtained by the BET method, calcination of gypsum-bearing raw materials contributed to a decrease in pore volume and an increase in the average pore diameter, which, in turn, contributed to a decrease in the SSA of the binder in relation to the original raw materials (Table 3).

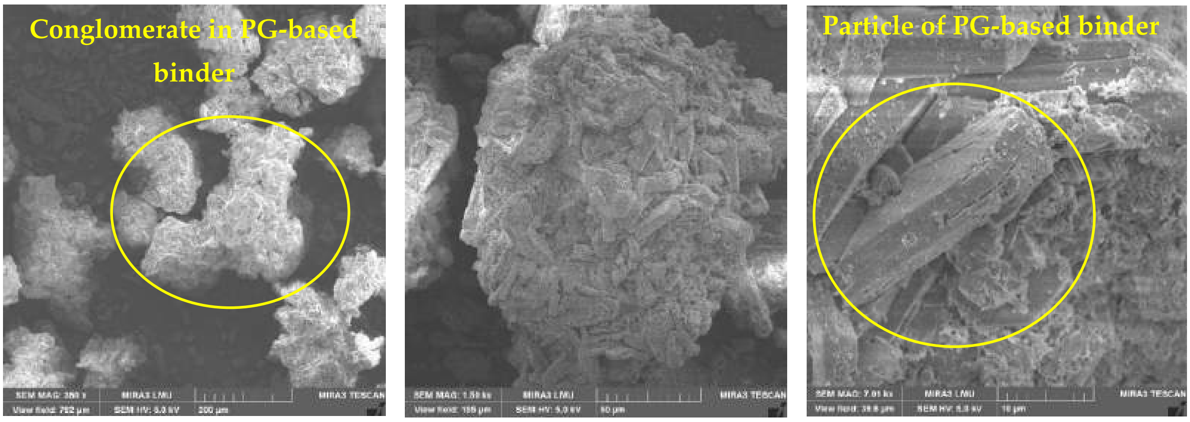

Figure 8. Morphology of particles surface in PG-based binder.

Table 4. SSA and pore size distribution in PG-based binder and NG-based binder.

Parameter	FG-Based Binder	NG-Based Binder
Blaine method		
SSA, m^2/kg	135.8	425.6
BET method		
SSA, m^2/kg	7162	7594.2
Total pore volume ($p/p0 = 0.9900$), cm^3/g	0.015	0.065
Average pore diameter, nm	8.11	34.10
BJH method		
SSA, m^2/kg	7013.4	10,963
Total pore volume, cm^3/g	0.014	0.081
Average pore diameter, nm	78.30	29.42

Calcination also had a significant effect on the distribution of pores by volume, which is confirmed by a decrease in intensity and a change in the character of the curve reflecting the volume of pores corresponding to a specific diameter (Figure 9). In the case of the NG-based binder, a shift in peaks is observed towards pores of larger diameter in relation to the original NG (see Figure 5). For the PG-based binder, peaks appear corresponding to pores whose size lies in a smaller size range than that of the original PG (Figure 5).

As noted earlier, the pH value has a strong influence on the properties of gypsum binders due to its effect on the solubility of calcium sulfate crystal hydrates. Optimal conditions for hydration are those that provide a slightly acidic environment with a subsequent transition to a neutral environment [22]. In this regard, a study was carried out to determine the pH value of PG-based binders and NG-based binders, as well as its changes over time (Figure 10).

Analysis of the obtained data (Figure 10) allowed us to establish the calcination of NG contributed to a decrease in the pH value from 8.75 (see Figure 6) to 6.6 (Figure 10), which subsequently increased over time and reached a value of 7.83 at the age of 28 days, which is consistent with the previously obtained results of V.G. Klimenko [22]. At the same time, calcination did not affect the pH value of PG-based binders; after mixing with water, it was 3.29 and did not change significantly over time. This gives reason to believe the calcination at 175 °C for 3 h does not ensure the removal of residual phosphoric acid, which may subsequently affect the setting time and structure formation of the PG-based binder [23,24].

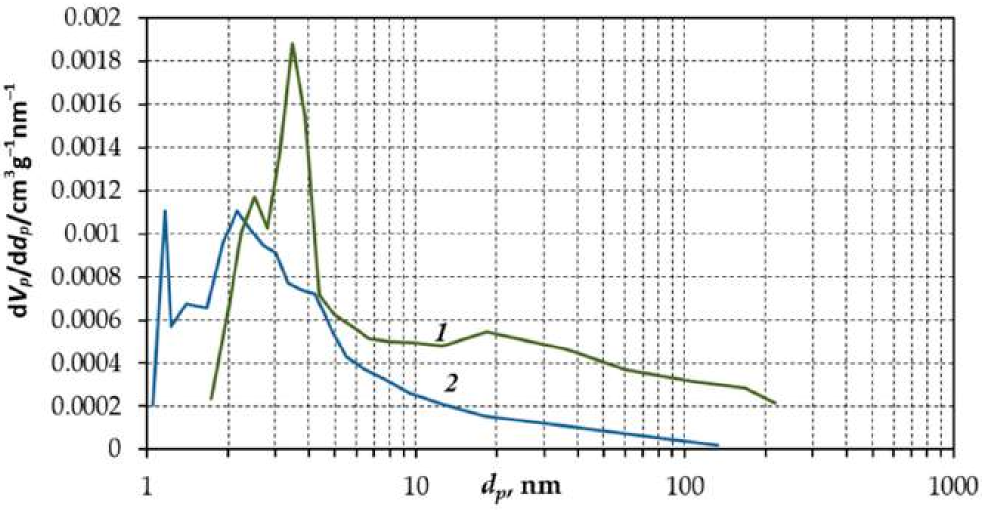

Figure 9. The volume of pores corresponding to a specific diameter in 1—PG-based binder and 2—NG-based binder.

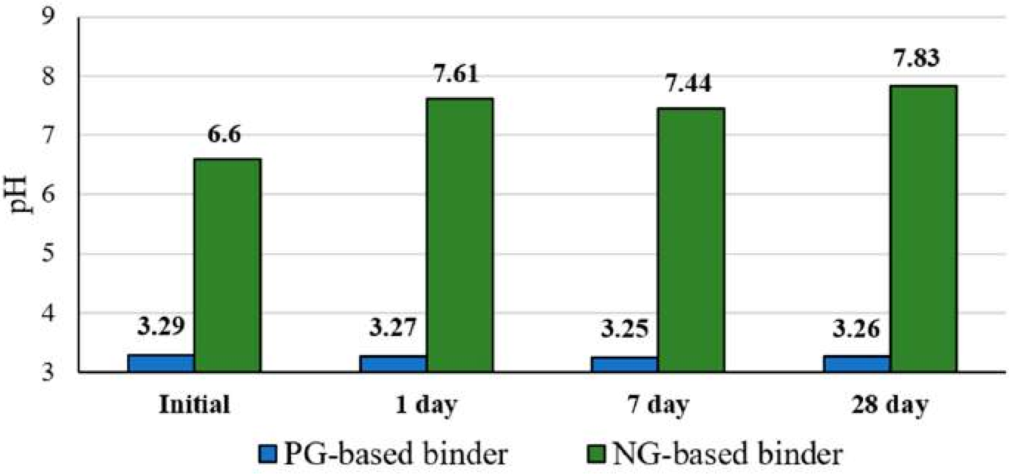

Figure 10. pH values of PG-based binder and NG-based binder in different time periods.

At the next stage of the research, the physical and mechanical characteristics of PG-based binders were studied according to Russian Standard GOST 23789-2018. It should be noted that when mixing the PG-based binder with water until homogeneity, a large number of small and large air bubbles were formed on the surface of the binder (Figure 11a). This is not a typical phenomenon for NG-based binders (Figure 11b) and indicates the presence of impurities in the PG-based binder that have surfactant properties. Their presence can have a negative effect on hydration, structure formation, and hardening of the PG-based binder. In turn, increased air entrainment can also negatively affect the average density and, as a consequence, water absorption and strength of the PG-based binder.

Setting time, tensile, and compressive strength at the age of 2 h and at an absolutely dry state were determined on prisms of 40 × 40 × 160 mm, prepared from binders of standard consistency (according to Russian Standard GOST 23789-2018 and EN 13279-2: 2004, NEQ). Analysis of the obtained results (Table 5) show that PG-based binder, compared with NG-based binder, differs in substantially greater amounts of water necessary to ensure standard consistency.

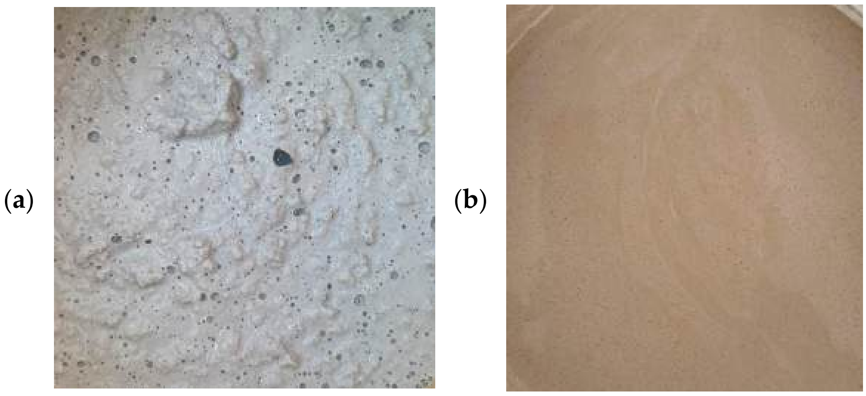

Figure 11. Appearance of fresh (**a**) PG-based paste; (**b**) NG-based paste.

Table 5. Standard consistency, setting times, and physical and mechanical characteristics of PG-based binder and NG-based binder.

Binder	W/S Ratio	Setting Times, min		Average Density, kg/m^3		Tensile Strength, MPa		Compressive Strength, MPa	
		Initial	Final	at 2-h Age	at Dry State	at 2-h Age	at Dry State	at 2-h Age	at Dry State
PG-based	0.89	24	30	1561	1120	1.37	2.32	2.58	6.64
NG-based	0.70	18	25	1570	1120	3.11	5.74	4.76	11.96

However, it should be noted that the selected amount of water for the PG-based binder provides the required cone flow diameter according to the Suttard viscometer (180 mm). But this is probably unnecessary, as evidenced by the increased water separation during the setting time test (Figure 12). Increased water separation is due to the high porosity of the particles in the binder (Table 4) and the features of their morphology, namely the presence of conglomerates (see Figure 8), capable of absorbing and retaining water during mixing water with the binder, which begins to separate over time (Figure 12). Apparently, for the same reason, the PG-based binder is characterized by significantly longer initial and final setting times compared to the NG-based binder (Table 5).

Figure 12. Effect of water separation during setting time test for PG-based binder.

The average density and strength characteristics of PG-based binders are significantly lower than for NG-based binders, both at the age of 2 h and in a dry state.

To study the effect of water content in the binder on the physical and mechanical properties of gypsum paste, cube samples of 30 × 30 × 30 mm in size were molded at W/S ratios of 0.5, 0.6, 0.7, 0.8, and 0.9 (Tables 6 and 7).

Table 6. Effect of W/S ratio and binder type on average density of consolidate paste.

Binder Type	**Average Density in a Dry State (kg/m^3) for Different W/S Ratio**				
	0.5	**0.6**	**0.7**	**0.8**	**0.9**
PG-based	1094	1170	1075	1021	1005
NG-based (according to [7])	1384	1255	1170	1054	961

Table 7. Effect of W/S ratio and binder type on compressive strength of consolidate paste.

Binder Type	**Compressive Strength at a Dry State (kg/m^3) for Different W/S Ratio**				
	0.5	**0.6**	**0.7**	**0.8**	**0.9**
PG-based	7.37	11.75	9.28	7.26	5.50
NG-based (according to [7])	16.86	13.96	11.90	9.34	7.50

Analysis of the obtained results showed that at an equal W/S ratio, the average density of PG-based samples in an absolutely dry state at W/S of 0.5–0.7 is lower than the values for NG-based samples at W/S = 0.5 by an average of 18.5%. At W/S = 0.8, the average density of PG-based paste is slightly lower than the average density of NG-based paste, and at W/S = 0.9, it begins to exceed it. This is due to the separation of fresh gypsum paste at a high water content. It should also be noted that if in the case of NG-based paste the average density naturally increases with decreasing water content, then in the case of PG-based paste the average density increases with decreasing W/S ratio from 0.9 to 0.6, and then the average density decreases. This is probably due to insufficient compaction of PG-based paste due to the high porosity of particles in PG-based binder. The compressive strength of PG-based paste and NG-based paste (Table 7) are directly dependent on the average density (Table 6). The maximum compressive strength of NG-based paste is 16.86 MPa with a minimum W/S = 0.5; for PG-based paste it is 11.75 MPa with W/S = 0.6.

SEM analysis of the samples molded at an equal W/S (0.6) showed a difference in the size, morphology, and density of new formations. The size of new formations in PG-based paste is smaller than in NG-based paste (Figure 13a). This is due to the fact that the PG-based binder contains smaller particles, which causes their rapid dissolution, providing supersaturation of the solution, where small crystals are formed. The microstructure of NG-based paste and PG-based paste (Figure 13b) is represented by needle-shaped elongated crystals and their intergrowths. However, their size is significantly larger, which predetermines the higher compressive strength of NG-based paste in relation to PG-based paste.

Comparison of microstructures showed that, in contrast to the new formations in NG-based paste, the new formations in PG-based paste are less ordered, which predetermines the formation of a larger number of pores and, as a consequence, lower average density (Tables 6 and 7).

Also, the morphology of new formations can be affected by the residues of orthophosphoric acid present in PG, which, as noted earlier, negatively affects the structure formation and, as a consequence, the physical and mechanical characteristics of the paste.

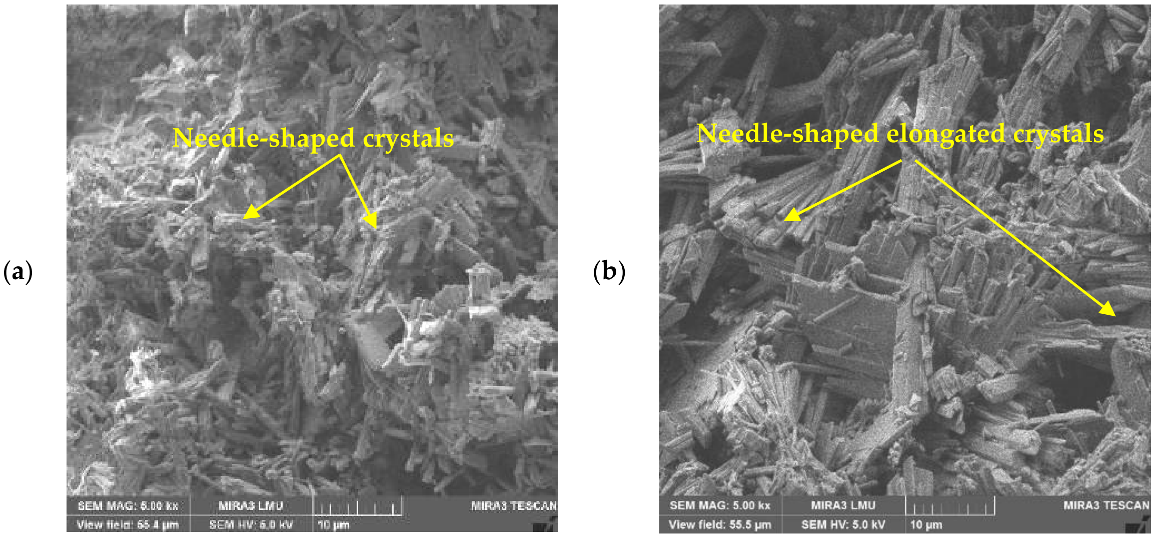

Figure 13. Microstructure of (**a**) PG-based paste and (**b**) NG-based paste.

4. Conclusions

A comprehensive comparative analysis of PG and NG revealed that they have significant differences in both chemical composition and structural and morphological features, which subsequently affect the properties of materials based on them.

PG-based binder consists of particles with developed SSA and their conglomerates, which predetermines the higher water content required to ensure standard consistency (0.89). This, as well as the presence of impurities in the FG, contributes to increased air involvement and predetermines a decrease in the average density and, as a consequence, a decrease in the strength of PG-based paste (2.58 MPa) in relation to NG-based paste (4.76 MPa).

It was found that lower pH values for PG-based binders indicate the presence of orthophosphoric acid residues, which, together with the morphological features of PG-based binder particles, negatively affects the structure formation and the final physical and mechanical characteristics of the paste.

Comparison of the physical and mechanical characteristics of PG-based paste and NG-based paste, produced with an equal W/S ratio, gives reason to believe that to increase the efficiency of PG use as a raw material for binders, it is necessary to develop methods aimed at reducing its water consumption. Since in this case, the determining factor influencing the water requirement is the characteristics of the particles in the PG-based binder and, in particular, their high SSA and a large number of conglomerates with high porosity, it is likely that the destruction of porous conglomerates by preliminary PG grinding could contribute to a decrease in the water requirement for obtaining fresh PG-based paste with a standard consistency and an increase in the physical and mechanical characteristics of hardened PG-based paste.

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