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STRUCTURE FORMATION AND PERFORMANCE PROPERTIES OF MODIFIED GYPSUM AND PHOSPHOGYPSUM BINDERS

Tetiana Dovbenko; Leonid Dvorkin; Sviatoslav Homon

*National University of Water and Environmental Engineering,
Rivne, Ukraine*

Summary. The structure formation of hardened samples of gypsum and phosphogypsum binders (with a complex of additives) is investigated by X-ray diffraction analysis and electron microscopy. The influence of additives-modifiers on the composition of curing products and on the morphology of the hydrate formation – $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ – is determined. X-ray diffraction patterns and electron micrographs of hardened gypsum and phosphogypsum binders are shown.

Key words: gypsum binder, phosphogypsum binder, modifiers, hardening, microstructure, X-ray phase analysis, electron microscopic analysis, ultrasonic method, specific resistance.

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Statement of the problem. Modern production, both Ukrainian and global, is focused on the production of effective materials using technologies that would involve minimal costs of raw materials, energy, the use of secondary resources, and others [1–6]. At this stage of development of the construction industry, the leader among the production of such economically beneficial materials is gypsum binders and products made from them, as well as the use of secondary strategic raw materials – phosphogypsum, for the production of gypsum binders.

Analysis of the available investigations. As you know, from the history of the development of mineral binders, progressive theories in the field of gypsum and phosphogypsum were proposed by famous scientists, namely: K. Kelly, D. Suttard, M. A. Sanitsky, H.-B. Fisher, R. A., Rebinder, A. F. Polakom, Yu. G. Meshcheryakov, K. K., Southard J. C., Anderson C. T. etc. [7–9]. They studied the main properties of gypsum and phosphogypsum binders (FGB), established the peculiarities of modifications, improved the production technology of phosphogypsum binders and various types of gypsum binder [10–14].

However, the process of manufacturing effective gypsum or phosphogypsum materials requires a detailed study of the relationship between their composition, structure and properties. Therefore, to establish the qualitative and quantitative characteristics of gypsum or phosphogypsum binders, it is necessary to study the processes of structure formation. For this, methods of X-ray investigation of the phase composition of materials, electron microscopy, thermal methods, etc. are most often used.

Statement of the objectives. The objective of the investigation is to analyze the morphological and kinetic properties of gypsum and phosphogypsum binders, including those with complex modifiers, through experimental studies based on X-ray phase and electron microscopic analyses, as well as the speed of propagation of ultrasonic waves.

Methodology of experimental research. The objects of the study were G-5 building gypsum – a gypsum low-burning binder, which consists mainly of $\beta - 2\text{CaSO}_4 \cdot \text{H}_2\text{O}$ and phosphogypsum binder, obtained according to the following technology: 1st stage – introduction of a neutralizer additive (unquenched ground lime 3%) to phosphogypsum

dihydrate; 2nd stage – raw material drying process; 3rd stage – firing of phosphogypsum binder; Stage 4 – grinding of the phosphogypsum binder took place in a laboratory ball mill.

X-ray phase analysis (DRON-3 diffractometer), electron microscopic analysis was used to study the morphological changes of the hardened stone of gypsum and phosphogypsum binders under the influence of modifier additives. Features of the structure formation of binders during the hardening process were studied using optical microscopy in transmitted and reflected light.

The ultrasonic method was used to study the kinetics of binder structure formation over time. The speed of propagation of ultrasonic waves V_u determined on the UK-10P device at a frequency of 60 kHz according to the formula

$$V_u = l(f_1 - f_2),$$

where l is the sound base (25 mm); f_1 i f_2 – frequency difference, or phase shift, equal to the time of passage of ultrasound through the sample.

The kinetics of hardening of HV and FGV were studied by determining the electrical conductivity using the P-38 rheochord bridge and the speed of longitudinal ultrasonic waves using the UK-10 PMS device.

The value of specific resistance was determined by the formula [14]

$$\rho = R \frac{S}{l},$$

where ρ – resistivity of the material; R – material resistance; S – sample area; l – sample length.

In addition, the method of determining electrical conductivity using a rheochord bridge was used to study the processes of structure formation of hardening mixtures P-38. The study of the electrical conductivity of the samples was carried out with direct contact of the electrodes and a constant current frequency $f = 50$ Hz, voltage $U = 10$ V and calculated by the formula:

$$\alpha^{\circ} = \left\{ \frac{U}{I} - R_o \right\} \frac{S}{l},$$

where α° – specific electrical conductivity; U – voltage; I – amperage; R_o – resistance of the experimental site; S – area; l – section length.

Research results and their discussion. Practice shows that gypsum binders based on β -hemihydrate are characterized by increased water consumption – 0.55-0.6, and, accordingly, low strength – 5...6 MPa. As for the phosphohypos binder, experiments have established that the water consumption of this material reaches 0.88-0.95, while the strength is within 1.5...2 MPa. Therefore, a significant reduction in water consumption, and accordingly a sharp increase in strength, can be achieved by using effective superplasticizer thinners.

The authors suggested mixing hyperplasticizers based on polyacrylate or polycarboxylate polymers with lime in an optimal ratio, which allows you to sharply reduce the water consumption of gypsum and phosphogypsum binders to Water/Binder ≤ 0.35 and increase the strength at the age of 2 hours to 12...18 MPa. It is precisely such high-strength modified gypsum and phosphogypsum binders that are of particular interest when conducting physicochemical analysis of binders.

Using the ultrasonic pulse method [14], the speed of propagation through gypsum samples of the front of the ultrasonic wave was determined. To measure the propagation time of ultrasound, through sounding was used, while the sensors were installed on opposite sides of the material.

As can be seen from Fig. 1, with a decrease in W/G in the gypsum binder dough from 0.62 to 0.35 due to the use of a complex modifier additive (0.6% Melflux + 3% lime on CaO), an increase in specific resistance is observed, which becomes particularly noticeable after 40 min of hardening from 11...12 to 17...18 Ohm·m, and after 80 min the difference increases from 15...16 to 23...24 Ohm·m. Similar kinetics of growth of specific resistance is also observed for phosphogypsum binder: with a decrease in W/H from 0.98 to 0.37, due to the use of complex additive-modifier, the specific resistance increases from 6...7 to 8...9 Ohm·m for 80 min of hardening (Fig. 2). For compositions of gypsum and phosphogypsum binders with other combinations of additives, intermediate values of specific resistance are characteristic. In the early stages of hardening, the difference in the specific resistance value of binders with different types of additives is relatively small.

An increase in specific resistance caused by a decrease in the number of ions Ca^+ , SO_4^{2-} , OH^- in the liquid of the pore space during the hardening process and by the active growth of crystals, which is noticeable already after 30-40 minutes of hardening. Stabilization of the specific resistance values for the gypsum binder dough after 60 min of hardening indicates a significant slowdown of the structure formation processes (Fig. 1) at the final stage of hardening, while for the phosphogypsum binder dough with a complex modifier additive, the increase in specific resistance continues (Fig. 2), which is confirmed by data on the increase in strength of such material within 24 hours. In general, the value of the specific resistance of compositions made of phosphogypsum binder is significantly lower than that of gypsum, which can be explained by greater dispersion and increased content of ions Ca^+ , SO_4^{2-} , OH^- in the fluid of the pore space.

The nature of the curves of changes in the speed of ultrasound passage allows you to follow all the main stages of the formation of the structure of gypsum stone: the initial induction period, the period of growth and fusion of crystals, and the period of final strengthening of the crystal structure with subsequent drying. Analyzing the obtained curves, it should be noted that the end of the induction period (transition of the horizontal section into a vertical one) is clearly visible, which practically coincides with the time of the end of gypsum hardening according to Vic's device. The end of hardening of binders is related to the type of additive used during mixing. When using a complex modifier additive, the curing time increases significantly. The point of the end of hardening is the starting point - from it there is a rapid increase in the speed of the passage of ultrasound, and, accordingly, the strength to certain constant values, which depend on the type of additive and V/G.

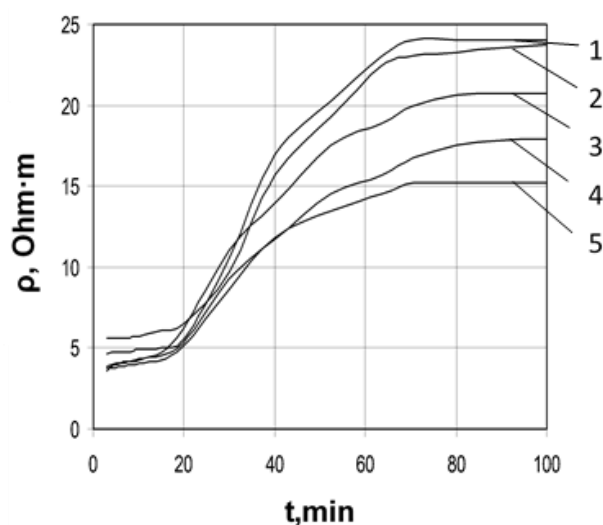


Figure 1. Dependencies of the specific resistance of the gypsum binder dough of normal density (ρ , $\Omega\cdot\text{m}$) on the hardening time (t , min): 1 – W/G=0.35 (gypsum binder + lime + Melflux); 2 – W/G=0.42 (gypsum binder + Melflux); 3 – V/G=0.58 (GV); 4 – W/G=0.48 (gypsum binder+C-3); 5 – W/G=0.62 (gypsum binder+lime)

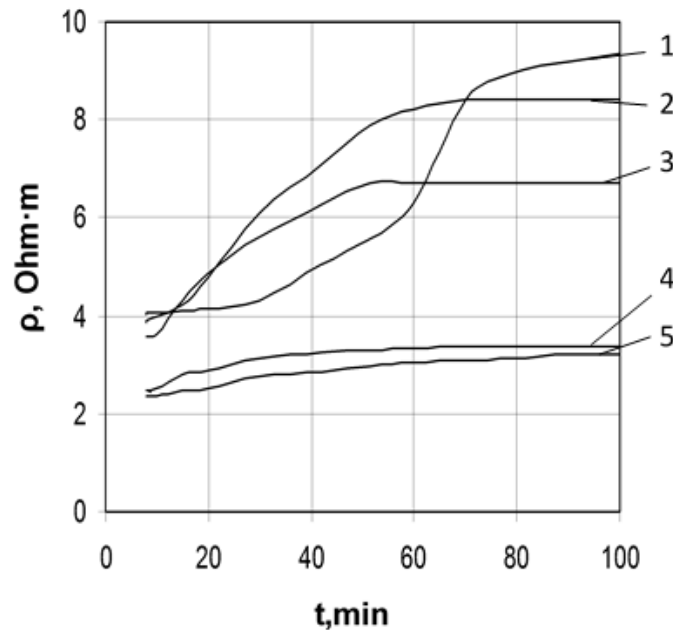


Figure 2. Dependencies of the specific resistance of the phosphogypsum binder dough of normal density (ρ , $\Omega\cdot\text{m}$) on the hardening time (t , min): 1 – $W/G=0.37$ (phosphogypsum binder + lime + Melflux); 2 – $W/G=0.45$ (phosphogypsum binder + Melflux); 3 – $W/G=0.96$ (FGV); 4 – $W/G=0.82$ (phosphogypsum binder +C-3); 5 – $W/G=0.98$ (phosphogypsum binder + lime)

Maximum speed values V_{y3x} and, accordingly, wet strength is ensured 30–40 minutes after mixing plaster with water. Further growth of strength (50...90 min) occurs due to evaporation of water and drying of the material. It was noticed that for gypsum binder, the speed of ultrasonic waves is within 1700–2500 m/s, and for phosphogypsum – 1100–1800 m/s. A decrease in W/G due to the use of the lime + polycarboxylate additive causes an increase in the intensity of the growth of the strength of the structure. The introduction of additives of other types without a significant change in W/G leads to a slower passage of the process, which can be explained by the absorption effect of their action.

Regulation of the properties of gypsum dough, due to changes in water content and the use of additives of various types, is significantly reflected in the processes of structure formation of gypsum stone: with a decrease in W/H , a more intense increase in the speed of processes is noticeable both at the initial and at the following stages of hardening. The influence of superplasticizers is more noticeable when the water content is simultaneously reduced.

The composition and structure of neoplasms of gypsum and phosphogypsum binders were studied using X-ray phase analysis (Fig. 3, Fig. 4) and electron microscopy (Fig. 5, Fig. 6). The photomicrographs show images of connective tissue structures at magnifications of 40, 800, and 6,000 times. Different compositions based on gypsum and phosphogypsum were used for comparative analysis of structure formation (Table 1).

X-ray phase analysis of samples of hardened gypsum and phosphogypsum binders (Fig. 3, Fig. 4) show that the main reflexes of two-water gypsum remain unchanged for all versions of the compositions $\text{CaSO}_4\cdot 2\text{H}_2\text{O}$ ($d = 7,60; 4,27; 3,06 \times 10^{-10}\text{m}$ etc.), as well as the ratio of the intensities of these reflexes. The intensity of reflexes of gypsum and phosphogypsum binders are practically the same. The presence or absence of modifier additives does not affect the composition of the hardening products and the morphology of the hydrated neoplasm – $\text{CaSO}_4\cdot 2\text{H}_2\text{O}$.

Table 1

Physico-mechanical properties of composite systems based on gypsum and phosphogypsum binders

| Plasticizer type | Amount of plasticizer, wt. %. | Amount of slaked lime, wt. %. | Water/binder | Time of setting, min | | Strength at 2 h, MPa | |
|---|-------------------------------|-------------------------------|--------------|----------------------|--------|----------------------|-------------|
| | | | | Start | Finish | Bend | Compression |
| Materials based on gypsum binder | | | | | | | |
| - | - | - | 0.60 | 5 | 10 | 2.5 | 4.80 |
| - | - | 3 | 0.62 | 5 | 25 | 3.85 | 5.30 |
| C-3 | 1.5 | 3 | 0.52 | 7 | 13 | 2.9 | 5.40 |
| Melflux 1641F | 0.6 | - | 0.42 | 18 | 80 | 5.69 | 6.77 |
| Melflux 1641F | 0.6 | 3 | 0.35 | 24 | 26 | 5.53 | 12.4 |
| Materials based on phosphogypsum binder | | | | | | | |
| - | - | - | 0.96 | 3 | 8 | 1.66 | 2.6 |
| - | - | 3 | 0.98 | 4 | 10 | 0.57 | 2.16 |
| C-3 | 1.5 | 3 | 0.82 | 5 | 12 | 1.65 | 2.28 |
| Melflux 1641F | 0.6 | - | 0.45 | 15 | 60 | 4.99 | 7.12 |
| Melflux 1641F | 0.6 | 3 | 0.35 | 18 | 20 | 5.25 | 11.77 |

Electron microscopic studies (Fig. 5, Fig. 6) show that the morphology of the crystals $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ does not depend on the type and amount of additives-modifiers: crystals in all microphotographs $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ have the form of prisms, elongated plates and growths, which is generally characteristic of this compound. At the same time, the crystal morphology of hardened gypsum and phosphogypsum binders is practically the same, but the crystals $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ hardened phosphogypsum binder without additives look significantly smaller than in the case of gypsum, which was noted in the works. When lime additives are added to phosphogypsum, C-3 or Melflux (without lime) crystals $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ increase in size, but the intercrystalline hollowness remains high. For gypsum binder with the use of modifier additives of optimal composition (0,6% Melflux + 2% lime in terms of CaO) and phosphogypsum binder (also, but with 3% of CaO), which provides lower values of W/G (W/FGV) to obtain a dough of normal thickness, a more dense arrangement of crystals is noticeable $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ and, accordingly, significantly smaller intercrystalline hollowness than in composites of other compositions. This, obviously, explains the increase in the strength of modified binders.

The microstructure of gypsum or phosphogypsum stone with a complex additive is formed from plate-shaped crystals, in contrast to needle-like crystals of ordinary gypsum stone. It is shown that the phenomenon of adsorption modification [10–13] of crystals is manifested in the conditions of an alkaline environment: the lamellar shape of crystals develops.

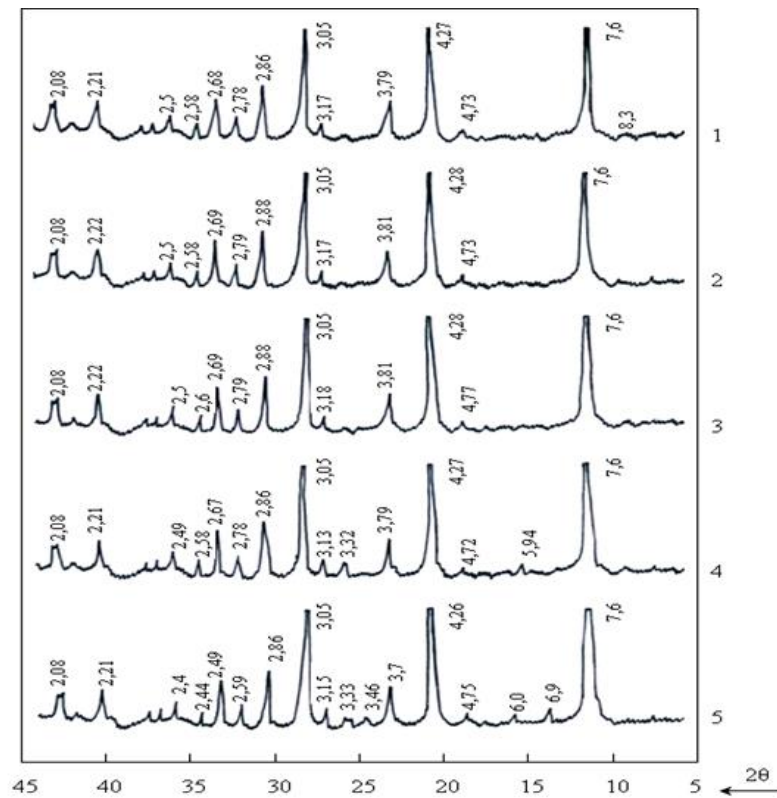


Figure 3. Radiographs of hardened gypsum binder: 1 – gypsum binder; 2 – gypsum binder + slaked lime; 3 – gypsum binder +C-3; 4 – gypsum binder +Melflux; 5 – gypsum binder +lime+ Melflux

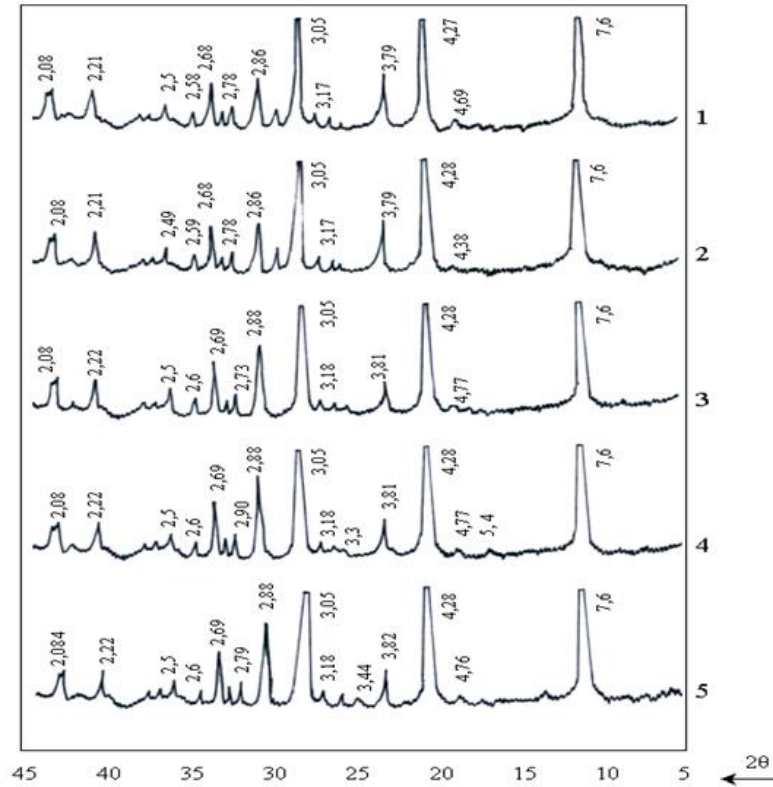


Figure 4. Radiographs of hardened phosphogypsum binder: 1 – phosphogypsum binder; 2 – phosphogypsum binder + slaked lime; 3 – phosphogypsum binder +C-3; 4 – phosphogypsum binder +Melflux; 5 – phosphogypsum binder + lime + Melflux

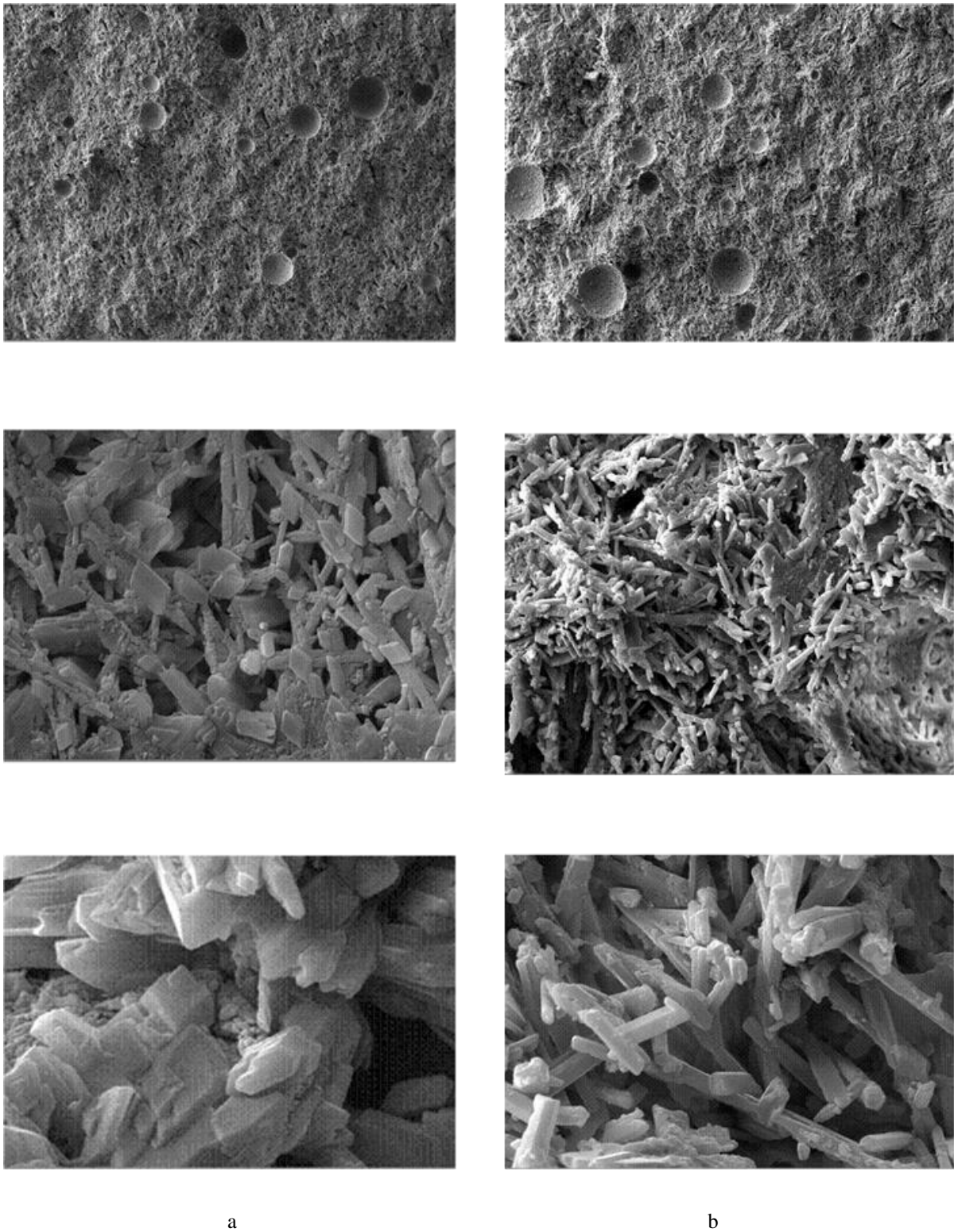
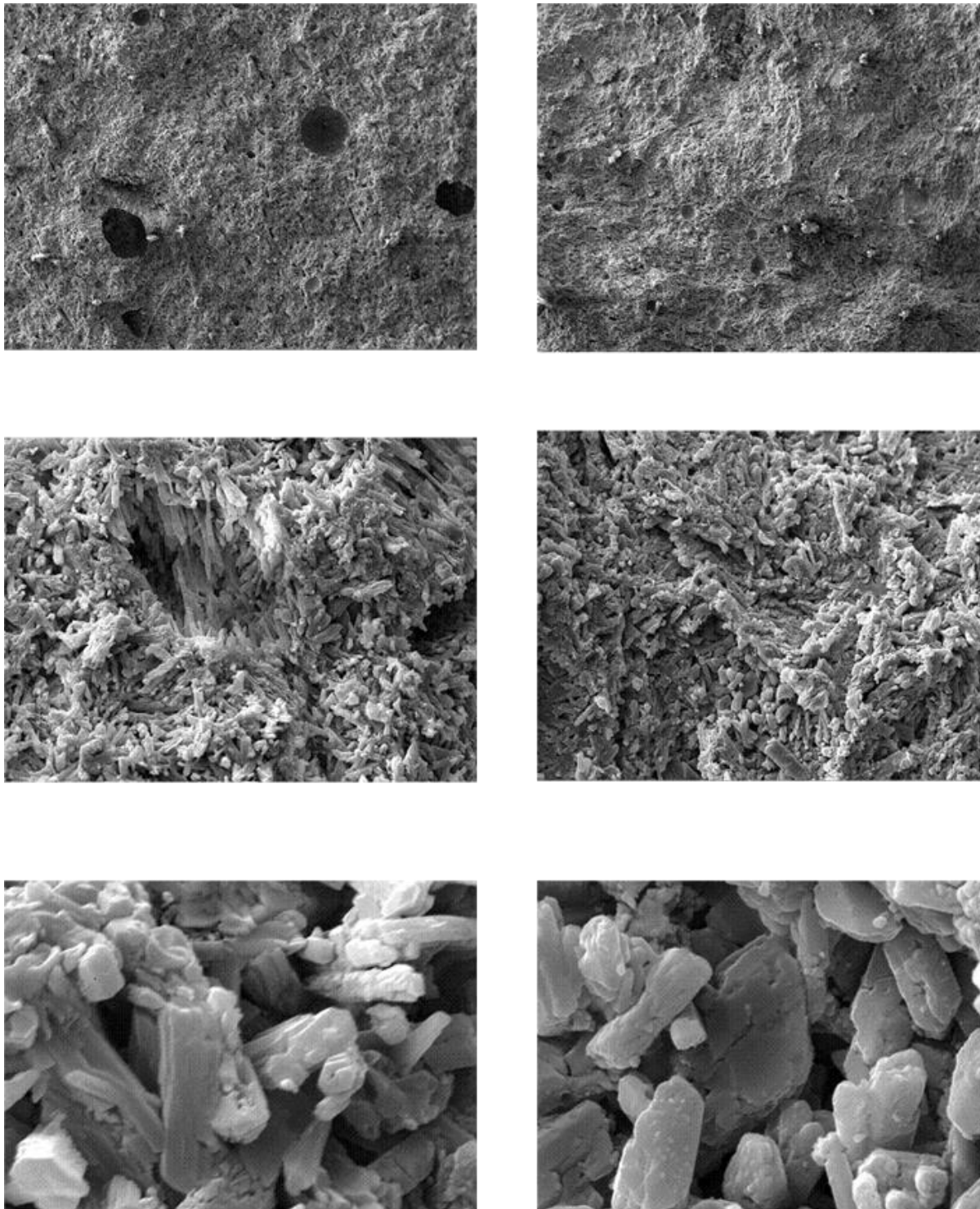


Figure 5. Photomicrographs of gypsum stone
(from top to bottom: x 40; 800; 6000):
a – gypsum binder (W/G = 0.60);
b) – phosphogypsum binder (W/ PHG = 0.90)



c)

d)

Figure 6. Photomicrographs of gypsum stone
(from top to bottom: x 40; 800; 6000):
c) – gypsum binder + Melflux (V/G = 0.50);
d) – phosphogypsum binder + Melflux (B/PHG = 0.80)

Conclusions. By means of ultrasonic research, it was determined that the process of hardening of gypsum binders consists of two periods: the first is the formation of a coagulation structure and the second is the formation of a crystallization structure and, accordingly, an increase in strength.

It is defined that with the change in specific electrical resistance and speed of propagation of ultrasonic waves, higher values are characteristic for gypsum and phosphogypsum binders with the addition of a modifier of optimal composition (0.6% Melflux +3% lime on CaO) than for binders without additives or with other combinations of modifiers, which indicates more intense structure formation and increased strength.

Due to X-ray phase analysis and electron microscopy, it was found that the presence or absence of modifier additives does not affect the composition of the hardening products and the morphology of the hydrated neoplasm – $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$. At the same time, a denser arrangement of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ crystals is noticeable for gypsum and phosphogypsum binders with the additive modifier of the optimal composition, which indicates a significantly smaller intercrystalline hollowness than in composites of other compositions.

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СТРУКТУРОУТВОРЕННЯ ТА ЕКСПЛУАТАЦІЙНІ ВЛАСТИВОСТІ МОДИФІКОВАНИХ ГІПСОВИХ І ФОСФОГІПСОВИХ В'ЯЖУЧИХ

Тетяна Довбенко; Леонід Дворкін; Святослав Гомон

*Національний університет водного господарства та
природокористування, Рівне, Україна*

Резюме. Проведено детальний аналіз утворення структури гіпсових та фосфогіпсових в'язучих у чистому вигляді, а також і з додаванням оптимального складу добавок-модифікаторів. Розроблено добавку-модифікатор гіпсових в'язучих матеріалів, основу якої складають гіперпластифікатори (поліакрилатні або полікарбоксілатні полімери) в комплексі з будівельним вапном в оптимальному співвідношенні, що дозволяє різко знизити водопотребу гіпсового й фосфогіпсового в'язучих 0,35 та підвищити міцність у віці 2 годин до 12...18 МПа. Саме такі високоміцні модифіковані гіпсові та фосфогіпсові в'язучі особливо цікавлять при проведенні фізико-хімічного аналізу в'язучих речовин. Проведено експериментальні дослідження гіпсових та

фосфогіпсових в'язучих за допомогою ультразвукового імпульсного методу (УК-10П), рентгенофазового (дифрактометр ДРОН-3) та електронно-мікроскопічного аналізу. Для порівняльного аналізу структуроутворення використано різні композиції на основі гіпсу та фосфогіпсу. Ультразвукове імпульсне дослідження побудоване на визначенні швидкості поширення через гіпсові зразки переднього фронту ультразвукової хвилі. Використано наскрізне прозвучення матеріалу. За допомогою рентгенофазового аналізу і електронної мікроскопії вивчено новоутворення гіпсового й фосфогіпсового в'язучих. Збільшення зображень структуроутворення за електронної мікроскопії проведено в 40, 800 і 6000 раз. Встановлено, що процес твердіння гіпсових в'язучих відбувається в 2 етапи – утворення коагуляційної структури та формування кристалізаційної будови, що супроводжується збільшенням міцнісних характеристик. Виявлено, що зміна питомого електричного опору й швидкість поширення ультразвукових хвиль для гіпсового та фосфогіпсового в'язучих з комплексною добавкою на основі полімерів характеризується вищими значеннями в порівнянні з в'язучими без добавок або з іншими модифікаторами. За допомогою методів рентгенофазового та електронно-мікроскопічного досліджень розкрито, що на процес структуроутворення гіпсових та фосфогіпсових в'язучих наявність або відсутність добавок-модифікаторів не впливає. Проте при введенні в структуру добавки-модифікатора на основі суперпластифікатора Melflux у гіпсовому та фосфогіпсовому в'язучих помітне цільніше розташування кристалів $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, що є ознакою значно меншої міжкристалічної порожнистості, ніж у композитів інших складів.

Ключові слова: гіпсове в'язуче, фосфогіпсове в'язуче, модифікатори, твердіння, мікроструктура, рентгенофазовий аналіз, електронно-мікроскопічний аналіз, ультразвуковий метод, питомий опір.

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