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ASSESSMENT OF LONG-TERM WATER RESISTANCE OF MODIFIED PRESSED GYPSUM COMPOSITES

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Abstract
Introduction. The most important indicator of the durability of gypsum materials is their water resistance, which is most often assessed by the degree of strength reduction after short-term water saturation. In our view, the durability of composite building materials based on gypsum and other air binders can be more accurately predicted based on the results of longer laboratory tests, which include prolonged immersion of control samples in water, as well as an assessment of their ability to restore their original strength after re-drying. The **aim of this study** is to assess the effect of a complex modifier, consisting of a monosubstituted salt of orthophosphoric acid and a fine carbonate filler, on the water resistance properties of pressed gypsum composites after prolonged immersion in water. **Methods.** The technical characteristics of the studied composites were determined using standard methods with control samples manufactured by pressing. The long-term water resistance of the material was assessed based on the change in the values of the softening and water resistance coefficients, calculated based on the results of testing control samples stored for 1, 7, 28 and 90 days in water. **Results.** We show that the high-strength fine-crystalline structure of pressed gypsum binder without modifying additives is characterized by extremely low strength, both during short-term and long-term immersion of control samples in water, as well as reduced ability to restore its original strength after drying. This is revealed in the comparison of the water resistance indicators of the material, even with similar characteristics of cast samples made from pure gypsum binder, and especially with pressed samples from composite binders containing the proposed complex modifier. We established that gypsum-modified pressed composites are characterized by fairly high durability during prolonged immersion in water. After 90 days of testing, the softening coefficient of the studied compositions of composite binders ranged from 0.62 to 0.64, and the water resistance coefficient ranged from 0.90 to 0.95. This indicates the possibility of using products based on them in building enclosures, as well as in rooms with humidity above 75 %, provided that the water resistance of the studied pressed composites is maintained for a long time only if complete hydration of the gypsum binder is ensured.

Keywords*:* gypsum binders; chemical water treatment sludge from thermal power plants; monoammonium phosphate; pressed composites; water resistance.

Introduction

Various types of building products and structures are subject to temperature and humidity effects during operation, which involve prolonged and alternating moisture exposure, as well as repeated freezing and thawing. The ability of the material from which the products are made to resist these impacts primarily determines their durability (Ferronskaya, 1984, 2004). According to many authors, an important feature of the influence of various operational factors on gypsum materials is that the impact of alternating stresses is also accompanied by the dissolution of crystallization contacts of hardened calcium sulfate dihydrate, leading to an irreversible decrease in strength (Khalil et al., 2018; Petropavlovskaya et al., 2019; Safonova et al., 2018). Many researchers in Russia and other countries around the world are currently engaged in the search for methods and the development of technologies to enhance the durability of construction products based on gypsum binders (Domanskaya et al., 2018; Pervyshin et al., 2017; Petropavlovskaya et al., 2021; Zhukov et al., 2021). The research results allow a significant expansion of their application area, in particular making it possible to use them in building enclosures, as well as in rooms with indoor air humidity exceeding 75 %. The most well-known works in this field are related to the joint introduction of 15–25 % Portland cement and 10–25 % pozzolans of natural or technogenic origin into the composition of gypsum molding mixtures. The durability of products based on such mixed binders is ensured by the formation of sparingly soluble calcium hydrosilicates and hydroaluminates during hardening, as well as calcium hydrosulfoaluminate, which crystallizes in the monosulfate form (Barkovskaya and Terehova, 2023; Koroviakov and Bur'yanov, 2015; Lesovik et al., 2019).

Another method for increasing the strength and durability of construction materials and products based on low-fired gypsum binders is to reduce their porosity, primarily open porosity, by using intensive methods of compacting molding mixtures (Ferronskaya, 1984;

Pervyshin et al., 2017; Petropavlovskaya et al., 2019). In this case, the highest strength is achieved when the water content in the gypsum mixture is close to the amount theoretically required to ensure the hydration of the binder (water-to-gypsum ratio of 18.6 %). This creates the conditions for the formation of a fine-crystalline structure of the resulting calcium sulfate dihydrate with maximum strength (up to 60 MPa and above) (Kaklyugin et al., 2020c). However, obtaining gypsum products from such semi-dry mixtures becomes possible only when using the method of high-pressure compaction. The possibility of using this method for the production of gypsum facing slabs, wall and partition stones, hollow-core interlocking blocks, and other small gypsum products has been attracting domestic and foreign researchers since the mid-20th century. Nonetheless, the production of pressed gypsum materials has not gained wide practical application, which, in our opinion, is due to the increased consumption of gypsum binder compared to products made using casting technology, as well as their insufficient water resistance for the intended use (Kaklyugin et al., 2020c).

In order to eliminate the above-mentioned shortcomings, we developed a complex modifier of gypsum binder and the structure of the resulting pressed composites, consisting of a carbonate-containing filler, sludge from chemical water treatment of thermal power plants, and a salt of orthophosphoric acid, monoammonium phosphate ($NH_4H_2PO_4$). The chemical interaction of the chemical additive with calcium sulfate of the gypsum binder and calcium carbonate of the modifying filler begins during the preparation of the molding mixture. At the same time, the chemical interaction between $NH_4H_2PO_4$ and $CaCO_3$ is accompanied by a short-term release of CO_2, which must be completed before the start of product molding. We described the mechanism of the proposed complex modifier in detail, with chemical reaction equations provided, in (Kaklyugin et al., 2020a, 2020b). The same studies show that the interaction of the chemical additive with the calcium sulfate of the binder and the calcium carbonate of the filler results in formation of screening phase films of sparingly soluble dicalcium phosphate dihydrate ($CaHPO_4 \cdot 2H_2O$) on the surface of the particles of the hydrated neoplasms and sludge grains. This compound is isomorphic with dihydrate gypsum, has 10 times lower solubility compared to it, and alters the crystallization structure of the pressed material. Films made of sparingly soluble calcium phosphate on the elements of the crystallization structure of the pressed material add to the cementing effect and contribute to an increase in its strength and water resistance (Kaklyugin et al., 2020a, 2020b).

One of the main technical characteristics determining the durability of gypsum products is considered to be their water resistance, which is assessed using the softening coefficient. The conditions for determining this coefficient for gypsum building products are currently not standardized. As a rule, it is calculated as the ratio of the compressive strength of the material after short-term (24–48 hours) water saturation to its strength in a dry state (Drebezgova et al., 2018; Cao et al., 2019). In our view, the durability of composite building materials based on gypsum and other air binders can be more accurately predicted based on the results of longer laboratory tests, which include prolonged immersion of control samples in water, as well as an assessment of their ability to restore their original strength after re-drying. The *aim of this study* is to assess the effect of a complex modifier, consisting of monoammonium phosphate and carbonate sludge from the chemical water treatment of a thermal power plant, on the water resistance indicators of pressed gypsum composites after prolonged immersion in water.

Methods

In experimental studies, we used G-5 grade gypsum binder according to GOST 125-2018 “Gypsum binders. Specifications”. With respect to the setting time, the binder is of normal setting type, and based on the degree of grinding, it is of medium grind. The water resistance of pressed gypsum composites was enhanced by introducing a complex modifier into the molding mixtures, consisting of a fine carbonate filler, chemical water treatment sludge from a thermal power plant, and a chemical additive, monoammonium phosphate. For these purposes, part of the gypsum binder in the studied compositions was replaced with powdered sludge. Monoammonium phosphate was dissolved in mixing water and introduced into the composition of the semi-dry molding mixture by sprinkling during its mixing in a laboratory slider mixer. The assessment of the long-term water resistance of gypsum-modified composites was carried out using control cylinder samples with a height and diameter of 50.5 mm, manufactured by pressing under 40 MPa. The choice of this pressing pressure is due to the fact that, as our previous studies have shown, lower values fail to provide the best combination of physical and mechanical properties of pressed gypsum composites, and in cases of higher values, we often observed water separation during the compaction of samples (Kaklyugin et al., 2022a, 2022b). Along with these tests, we also tested control samples made from gypsum mixtures without additives and molded under the same pressure, as well as the ones manufactured by casting from a paste of normal consistency (water-to-gypsum ratio of 0.52). The compositions of the studied molding mixtures, adopted based on the results of preliminary tests, as well as the physical and mechanical characteristics of the samples molded from them, are presented in the table.

After molding, all control samples were setting for three days in air-dry conditions. Five samples from each series were dried at a temperature of (50±2) °C until constant mass; then we determined their compressive strength. The remaining samples were immersed in containers with water, where they were kept for 1, 7, 28, and 90 days at a temperature of (25±3) °C. At the specified times, 10 control samples were removed from the water, 5 of which were immediately tested for compression, while the others were tested after being re-dried at the aforementioned temperature. Based on the calculated average strength values of the control samples, the softening (k_s) and water resistance (k_{wr}) coefficients of the material were determined, and water absorption by mass was calculated from the change in the mass of the samples before and after immersion in water. The softening coefficient was calculated as the ratio of the compressive strength of the material in a water-saturated state to that in a dried-to-constant-mass state, while the water resistance coefficient was determined as the quotient of the strength of dried-to-constant-mass samples, stored in water for a corresponding time, to the strength of dry samples not subjected to water saturation.

Results

Our laboratory studies revealed the effectiveness of the proposed method for modifying the composition and structure of pressed gypsum composites to ensure their long-term water resistance. The change in compressive strength of dried and water-saturated samples as the duration of their immersion in water increases is shown in Fig. 1. The analysis of research results shows that the strength of dried and water-saturated samples, molded from all studied compositions, decreases with an increase in the duration of their exposure to water. However, this trend is observed to varying degrees for the compositions under consideration. Thus, the greatest decrease in strength is observed in cast and pressed samples made from pure gypsum binder without a complex modifier (compositions 1 and 2). The strength of samples molded from gypsum paste of standard consistency after 90 days of storage in water in a dried state was 8.2 MPa, which is 24 % less than their strength before immersion in water, and in a water-saturated state, it was 4.0 MPa, which is almost 30 % less than the similar indicator after 1 day of water saturation. Composition No. 2 (pressed gypsum binder without additives) can be called the least water-resistant. It is characterized by the lowest softening coefficient: after just one day of water saturation, the samples had k_s of only 0.15, and with increased duration of sample storage in water, there is a further decrease in strength. After 90 days, the strength decreased by 40 % in a water-saturated state, and by 22.3 % in a dried state. This is explained by the excessive vulnerability of the crystallization structure of pressed gypsum binder to the wedging action of water films and indicates the extremely low durability of such material in humid conditions. As seen in Fig. 1, the reduction in the strength of hardened composite binders (compositions 3, 4, 5, 6) in a water-saturated state occurs to a lesser extent compared to compositions 1 and 2, and after drying, the material, within the accepted testing periods, mostly restores its initial strength. For example, after 24 hours of water saturation, the strength of the control samples molded from mixture composition No. 4 was 61.7 MPa in a dry state and 44.0 MPa in a water-saturated state. After 90 days of storing the samples in water, the strength of the pressed material decreased by 13.2 % and amounted to 38.2 MPa, while the strength of the samples after drying was 60.0 MPa, which is only 2.8 % less than the same indicator before the samples were immersed in water. This is explained by the change in the structure of the pressed stone and the appearance of newly formed sparingly soluble calcium phosphates among the products, which hinder the dissolution of the crystallization contacts of calcium sulfate dihydrate.

Compositions of molding mixtures and physical and mechanical characteristics of control samples

Number of composition	Content of components, % by mass		Monoammonium phosphate, over 100 %	Water-solid ratio	Compressive strength of samples, MPa		Average density, kg/m^3	Water absorption by mass, %	Open porosity, %
	gypsum binder	sludge from chemical water treatment of thermal power plants			dry	water-saturated			
1	100	0	0	0.520	10.8	5.7	1200	28.4	34.0
2	100	0	0	0.190	32.0	5.3	1800	11.0	19.80
3	80	20	2	0.170	59.5	38.2	1930	7.3	14.00
4	80	20	2	0.185	61.7	44.0	1950	5.8	11.30
5	80	20	2	0.200	54.5	36.5	1940	8.0	15.50
6	60	40	2	0.170	46.8	29.5	1860	8.5	15.80

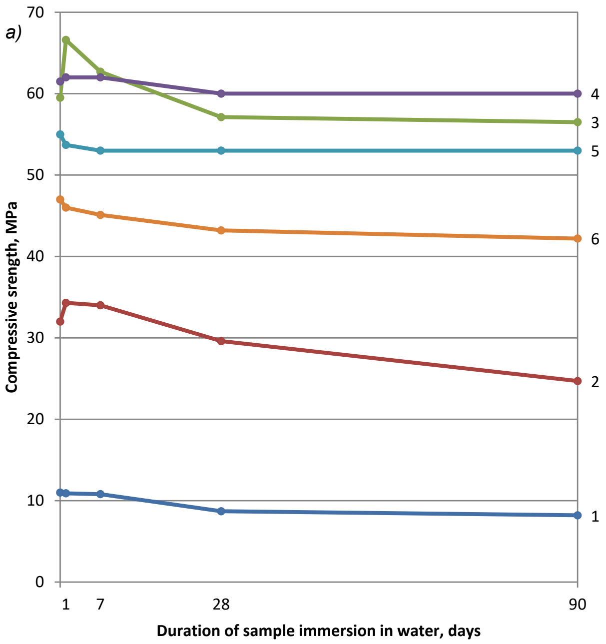

Fig. 1. The effect of the duration of keeping control samples in water on their strength in a dried (a) and water-saturated (b) state: 1–6 — composition numbers

The analysis of the change in the strength of samples made from compositions with the same content of a complex modifier, but with different water-solid ratios, shows that in humid conditions, the physical and mechanical characteristics of composition No. 3 deteriorate most noticeably, i.e., at a water-solid ratio of 0.17. Although samples of this composition, tested after 1 and 7 days of storage in water, exhibit strength in a dry state that is even slightly greater than before immersion in water, ultimately, after 28 and 90 days of water exposure, they show a decrease in strength, which is more noticeable than in samples made from compositions 4 and 5. Samples of composition No. 3, after 90 days of water immersion, exhibit a compressive strength of 31.1 MPa in a water-saturated state, which is 18.5 % less than the same indicator after 1 day of water saturation. Meanwhile, the strength of the samples of compositions 4 and 5 in a water-saturated state decreases by only 13 % and 9 %, respectively, and amounts to 38.2 and 33.2 MPa. A more noticeable decrease in the physical and mechanical properties of samples molded from mixture composition No. 3 after prolonged storage in water is explained by the fact that at a water-solid ratio of 0.17, complete hydration of the gypsum binder is not achieved during the hardening stage. When the samples are immersed in water, the unreacted calcium sulfate hemihydrate hydrates causing volumetric expansion of the stone and, consequently, weakening its crystalline structure. It should be noted that the decrease in the strength of samples made from all the studied compositions, when subjected to prolonged immersion in water, was accompanied by a slight increase in their water absorption by mass, as shown in Fig. 2.

This is due to the fact that with prolonged water saturation, water penetrates into increasingly smaller pores of the material, resulting in an intensified wedging effect on the crystallization structure of the gypsum stone. Fig. 3 shows how the softening and water resistance coefficients of the studied compositions change with the increase in time of the control samples immersion in water. As can be seen from the graphs presented in Fig. 3, the softening coefficient of gypsum-modified pressed composites of compositions 4, 5, and 6 exceeds 0.6 in all the tests. These compositions are also characterized by the highest water resistance coefficient k_{wr}, which was 0.90–0.97 after 90 days. This once again indicates a slight decrease in their strength when moistened and the ability to restore it upon drying. Fig. 3 also shows that with an increase in the duration of storage in water, the softening coefficient of samples of composition No. 3 decreases and after 90 days of testing is only 0.55, while its water resistance coefficient remains quite high (k_{wr} = 0.95).

As previously noted, control samples made from unmodified gypsum binder using casting (composition No. 1) and pressing (composition No. 2) methods are characterized by the greatest reduction in strength when saturated with water. After just one day of being in water, the softening coefficient of these compositions was 0.53 and 0.17, respectively, and with longer storage in water, it decreases even further, reaching 0.49 and 0.13 after 90 days. The extremely low water resistance

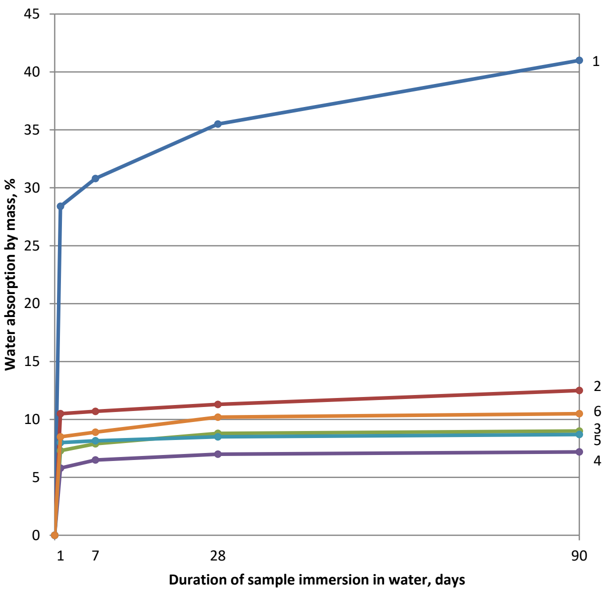

Fig. 2. Change in water absorption by mass of control samples with increasing duration of their immersion in water: 1–6 — composition numbers

of these compositions is further indicated by the obtained values of the water resistance coefficient. After 90 days of water storage, it decreased to 0.76 for samples made from paste of normal consistency and to 0.77 for pressed samples.

Discussion

The results of the conducted studies indicate that the high-strength fine-crystalline structure of pressed gypsum binder without modifying additives is characterized by extremely low durability, both during short-term and long-term immersion of control samples in water, as well as a reduced ability to restore its original strength after drying. This is revealed in the comparison of water resistance indicators of pressed samples even with similar characteristics of cast samples made from pure gypsum binder and, moreover, pressed samples from composite binders containing the proposed complex modifier. We established that gypsum-modified pressed composites are characterized by fairly high durability during prolonged immersion in water. After 90 days of testing, the softening coefficient of the studied compositions of composite binders ranged from 0.62 to 0.64, and the water resistance coefficient ranged from 0.90 to 0.95.

The increase in the long-term water resistance of the studied pressed gypsum composites is due to the synergistic effect of modifying additives: chemical water treatment sludge from a thermal power plant and monoammonium phosphate. Complex modification of gypsum binder ensures the formation of a monolithic fine-crystalline structure of artificial pressed stone-like material. The formation of an additional sparingly soluble framework of dicalcium phosphate dihydrate (brushite), which crystallizes isomorphously with dihydrate gypsum, enhances the resistance of the structure of modified composites to the wedging action of water films even during prolonged exposure to humid conditions. The presented kinetic dependencies of changes in the compressive strength of the material, as well as the softening and water resistance coefficients on the duration of storing control samples in water indicate the high water resistance of pressed modified gypsum composites and suggest the possibility

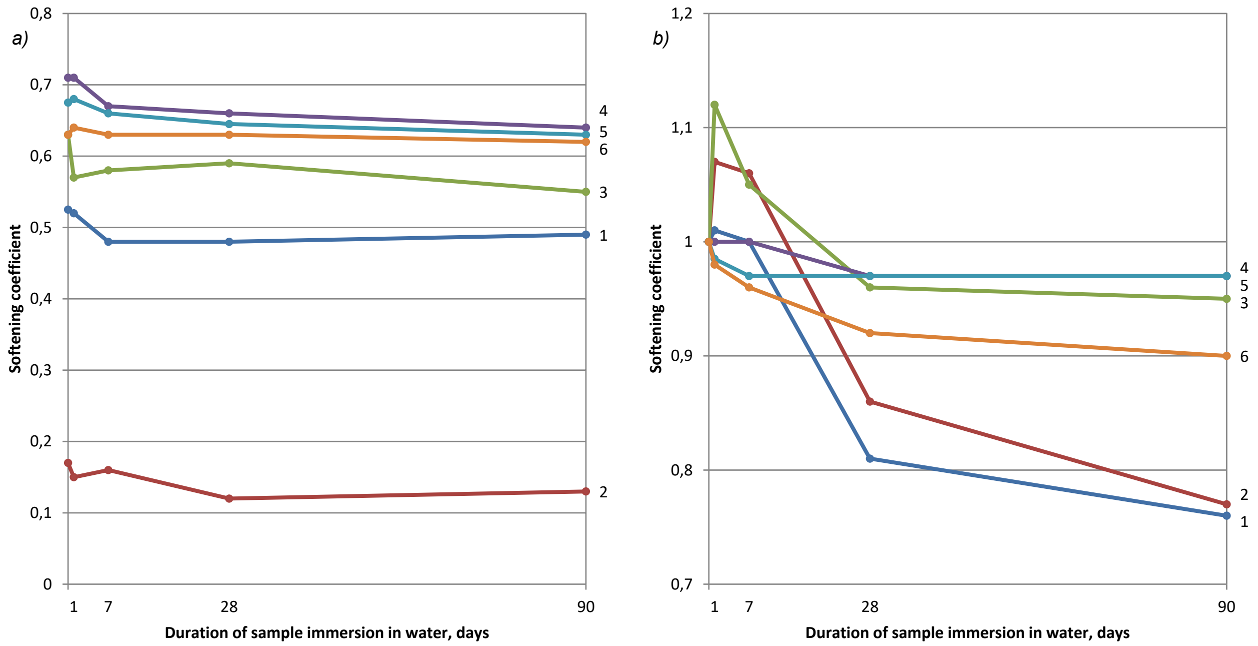

Fig. 3. Change in softening (a) and water resistance (b) coefficients with increasing duration of control samples immersion in water: 1–6 — composition numbers

of using products based on them in building enclosures, as well as in rooms with humidity above 75 %. At the same time, it is necessary to consider that the water resistance of the studied pressed composites is maintained for a long time only if the complete hydration of the gypsum binder is ensured.

The proposed method for increasing the water resistance of gypsum binders can only be used in the technology of manufacturing pressed products from semi-dry mixtures. This is due to the fact that during the mixing process of plastic consistency mixtures, as well as after they are poured into the mold, they will be significantly porous due to carbon dioxide released as a result of the chemical reaction between the calcium carbonate filler and the chemical additive. In the technology of pressed products, the formation of CO_2 occurs during the preparation of the molding mixture and does not negatively affect the structure formation of pressed composites.

In conclusion, it should be noted once again that prolonged laboratory observation of the changes in the physical and mechanical characteristics of artificial composites based on air binders, in our opinion, allows a more accurate and comprehensive prediction of their behavior in humid conditions. This is in contrast to the usual assessment of the water resistance of similar building materials, which is typically conducted by determining the softening coefficient after briefly soaking control samples in water. Therefore, the proposed method for assessing long-term water resistance is recommended for use in assessing the effectiveness of other methods of modifying gypsum binders and products based on them to enhance their durability.

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ОЦЕНКА ДЛИТЕЛЬНОЙ ВОДОСТОЙКОСТИ ГИПСОВЫХ МОДИФИЦИРОВАННЫХ ПРЕССОВАННЫХ КОМПОЗИТОВ

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Аннотация
Введение. Важнейшим показателем долговечности гипсовых материалов является их водостойкость, которую чаще всего оценивают по величине снижения прочности после кратковременного насыщения водой. На наш взгляд, более точно прогнозировать долговечность композиционных строительных материалов на основе гипсовых и других воздушных вяжущих, можно на основании результатов более длительных лабораторных испытаний, предусматривающих продолжительное выдерживание контрольных образцов в воде, а также оценку их способности восстанавливать первоначальную прочность в результате повторного высыхания. **Целью настоящей работы** является оценка влияния комплексного модификатора, состоящего из однозамещенной соли ортофосфорной кислоты и тонкодисперсного карбонатного наполнителя, на показатели водостойкости прессованных гипсовых композитов после их длительного выдерживания в воде. **Методы.** Технические характеристики исследуемых композитов определяли по стандартным методикам с использованием контрольных образцов, изготовленных методом прессования. Длительную водостойкость материала оценивали по изменению значений коэффициентов размягчения и водостойкости, рассчитываемых по результатам испытаний контрольных образцов хранившихся в течение 1, 7, 28 и 90 сут в воде. **Результаты.** Показано, что высокопрочная мелкокристаллическая структура прессованного гипсового вяжущего без модифицирующих добавок характеризуется крайне низкой стойкостью, как при кратковременном, так и при длительном выдерживании контрольных образцов в воде, а также пониженной способностью восстанавливать первоначальную прочность после высыхания. Это выявлено в сравнении показателей водостойкости материала даже с аналогичными характеристиками литых образцов из чистого гипсового вяжущего и, тем более, прессованных образцов из композиционных вяжущих, содержащих предлагаемый комплексный модификатор. При этом установлено, что гипсовые модифицированные прессованные композиты характеризуются достаточно высокой стойкостью при длительном хранении в воде. Через 90 сут испытаний коэффициент размягчения исследованных составов композиционных вяжущих составил от 0,62 до 0,64, а коэффициент водостойкости от 0,90 до 0,95. Это указывает на возможность использования изделий на их основе в ограждающих конструкциях зданий, а также в помещениях с влажностью более 75 %, учитывая, что водостойкость исследованных прессованных композитов сохраняется длительное время, только если обеспечивается полная гидратация гипсового вяжущего вещества.

Ключевые слова*:* гипсовые вяжущие вещества; шлам химводоподготовки теплоэлектростанций; однозамещенный фосфат аммония; прессованные композиты; водостойкость.