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ELEMENTS OF TECHNOLOGY FOR THE SURFACE GIPSUM COMPOSITE AND ITS PROPERTIES, AS A FACING MATERIAL

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Abstract. The paper considers the issues of obtaining a composite material based on gypsum, fly ash and sulfur with improved performance. Regularities of impregnation of a gypsum sol matrix with a sulfur melt are established, taking into account the capillary-porous structure of the gypsum sol stone and the physical and technical properties of sulfur. The conducted studies make it possible to determine the sulfur mass transfer coefficient a_{ms} and the maximum sulfur-containing U_s in impregnated products, depending on the initial values of the sulfur melt temperature, water-solid ratio and fly ash content. This allows to determine quickly and effectively the duration of impregnation to a given sulfur content for a specific composition and size of gypsum products. Technological factors influencing the hardening coefficient, water resistance and chemical resistance of gypsum ash products impregnated in sulfur melt have been studied. An analysis of the results shows that the strength of samples impregnated with sulfur increases while the degree of filling of the pore space with it is increasing. So, with increase in the relative sulfur content ($v = U_s / U_{smax}$) from 0.30 to 0.90, the compressive strength increases from 10.1 to 42 MPa. It has been established that water resistance of the impregnated samples significantly depends on the degree of impregnation and the amount of fly ash. The experiments carried out give reason to believe that the material based on gypsum, ash and sulfur refers to waterproof materials, since the softening coefficient is higher than 0.7. It has been established that gypsum and gypsum samples impregnated with sulfur melt have a chemical resistance coefficient of at least 0.7, which allows them to be classified as chemically resistant. The resulting sulfur-gypsum composite is distinguished by high strength, water and corrosion resistance to aggressive environments of livestock complexes, food and chemical industries, where it should be used in the form of special facing products.

Keywords: sulfur, gypsum, ash, water resistance, chemical resistance, sulfur melt.

Formulation of the problem. An effective way to increase water resistance and improve other construction and technical properties of capillary-porous building materials, including those based on gypsum, is their impregnation with substances that can harden in the pore space of these materials, which contributes to the compaction of the structure and prevents the penetration of moisture into them.

Sulfur is the optimal impregnating agent for the modification of gypsum concrete. It is known that sulfur is a typical inorganic thermoplastic capable of forming numerous allotropic modifications, and at the same time, in the molten state, it has a low viscosity. Sulfur is well compatible with various polymer modifiers. By introducing various additives, it is possible to control the viscosity and surface tension over a wide range. Sulfur has strong adhesive bonds to mineral fillers and aggregates. Under normal conditions, it is chemically inert. Even when heated, it is practically insoluble in water and acids. Sulfur is diamagnetic and hydrophobic, it is a poor conductor of heat and it has insecticidal properties. It has a low melting point, which makes it possible to transfer it from a solid to a liquid state at low energy costs. From a technological point of view, the process of crystallization of a sulfur melt during cooling is simpler than the polymerization of monomers in the manufacture of concrete polymers [1, 2].

From the foregoing, it follows that sulfur, as an impregnating material, has many valuable properties and can be used to impregnate various building capillary-porous materials, in particular, gypsum concrete. In addition, sulfur is less scarce and its cost is much lower than that of the monomers used to impregnate concrete. In Ukraine, there are large reserves of natural sulfur in the Rozdil and Yavoriv sulfur deposits. The increase in sulfur production is associated not only with an increase in the production of natural sulfur, but also with a sharp increase in the yield of sulfur during the purification of natural gas, flue gases and other industrial waste [1]. Therefore, the utilization of sulfur and sulfur products is in itself the most important and integral task of our time from an economic, environmental and energy point of view.

Analysis of recent research and publications. Currently, there are several technological methods for obtaining building materials and products using sulfur of various modifications. One of them is the impregnation of traditional building materials and products with molten sulfur, the other is the production of sulfur concrete.

The well-known property of the sulfur melt to form strong adhesive bonds with various mineral fillers makes it possible to use sulfur as the basis of a sulfur binder - mastic, which, in turn, is the basis of the structure of sulfur concretes [3].

The most important component of the structure of the mastic is the filler, the introduction of which reduces the consumption of sulfur and contributes to a change in the structure and all properties of the sulfur mastic. One of the most valuable properties of sulfur-based composite materials is their high corrosion resistance. In a number of works, it is noted that sulfur concrete is resistant to the effects of various acids [3-6].

However, in these works, issues related to the physicochemical phenomena occurring during the interaction of a gypsum or gypsum sol matrix and sulfur, the resistance of impregnated samples in aggressive environments have not been investigated, and the processes of drying and impregnation have not been sufficiently considered from the point of view of their optimization.

The purpose of the work is to develop a composite material based on gypsum and sulfur with improved construction and technical properties, to establish the patterns of impregnation of the gypsum-sol matrix with sulfur melt, taking into account the capillary-porous structure, and to determine the physical and technical properties of the modified material.

Materials and research methodology. When conducting research, building gypsum grade G-4 was used as a binder for the manufacture of prototypes, which meets the requirements of DSTU B V.2.7-82:2010. The choice of gypsum grade G-4 is due to the fact that lower grades after molding, hardening of gypsum stone and its further heating to the temperature of the sulfur melt (120 ... 150 ^{0}C) give insufficient strength for further technological operations. The use of gypsum of higher grades is not economically feasible, since the strength of the impregnated material is determined mainly by the strength of the sulfur framework.

As a filler, fly ash from the Ladyzhinskaya TPP was used, corresponding to Ukrainian national standardization system (UNSS) B V.2.7-205:2009. The choice of fly ash from the Ladyzhinskaya TPP is due to the fact that aluminosilicate acid fly ash has good compatibility with gypsum.

For the impregnation of gypsum sol samples, technical sulfur from the Razdolsky Production Association "Sera" was used, which corresponds to UNSS 2181-93, since chemically pure sulfur has a higher cost. The impregnation of gypsum sol samples in a sulfur melt was carried out in a special chamber at atmospheric pressure [7, 8].

The main experimental method in studying transfer processes is the method of measuring mass transfer coefficients from the kinetics of capillary impregnation.

Physical and technical parameters of the samples were determined in accordance with standard methods.

Research results. In order to optimize the sulfur melt preparation technology, experiments were carried out to study the influence of the temperature history and temperature of the sulfur melt on the mass transfer coefficient of sulfur. The kinetics of capillary impregnation was recorded at sulfur melt temperatures of 125, 140 and 155 ^{0}C, then the sulfur melt was heated to a temperature of 170 ^{0}C, while the viscosity of the sulfur melt increased significantly due to the rupture of S_8 ring molecules and their combination into long chains. When the sulfur melt was cooled, the kinetics of capillary impregnation were recorded at the same fixed temperatures of the melt (125, 140 and 155 ^{0}C). The results of the experiment are shown in Fig. 1. Analysis of the results showed that with an increase in the temperature of the sulfur melt to 155 ^{0}C, the sulfur mass transfer coefficient a_{ms} increases. This is due to allotropic transformations of sulfur and a decrease in its viscosity.

The graph (Fig. 1) shows the presence of a temperature hysteresis in the sulfur mass transfer coefficient a_{ms}. The presence of a temperature hysteresis of the a_{ms} coefficient makes it possible to optimize the process of preparing the sulfur melt, since it is possible to apply intense heating of sulfur without fear of melt overheating above 160 ^{0}C, and to obtain a sulfur melt with effective impregnating properties.

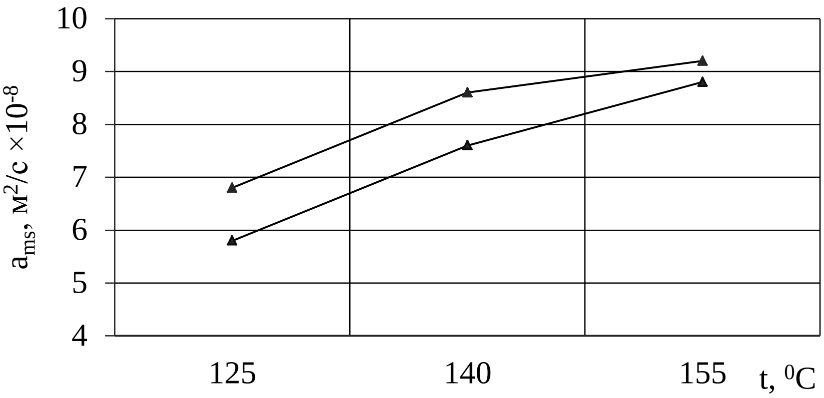

Fig. 1. Dependence of the mass transfer coefficient ams
on the temperature of the sulfur melt

The results of studies of the influence of the filler concentration and the water-solid ratio on the mass transfer coefficient a_{ms} and the maximum sulfur content U_s are shown in Fig. 2. Quantitative analysis of the experimental results showed that with an increase in the filler concentration, the a_{ms} and U_s coefficients decrease, and with an increase in the water-to-solid ratio, they increase. This is due to the fact that with an increase in the filler concentration, the porosity and average pore radius decrease, and with an increase in the water-to-solid ratio, these values increase accordingly.

Thus, the conducted studies make it possible to determine the sulfur mass transfer coefficient a_{ms} and the maximum sulfur-containing U_s in impregnated products, depending on the initial values of the sulfur melt temperature, water-solid ratio and fly ash content. This allows for a specific composition and size of gypsum products to determine the duration of impregnation to a given sulfur content in a quick and effective way.

Evaluation of the effectiveness of the impregnation of the samples was carried out by the coefficient of hardening (K_h), which was determined as the ratio of the strength of the sample after impregnation to the initial strength.

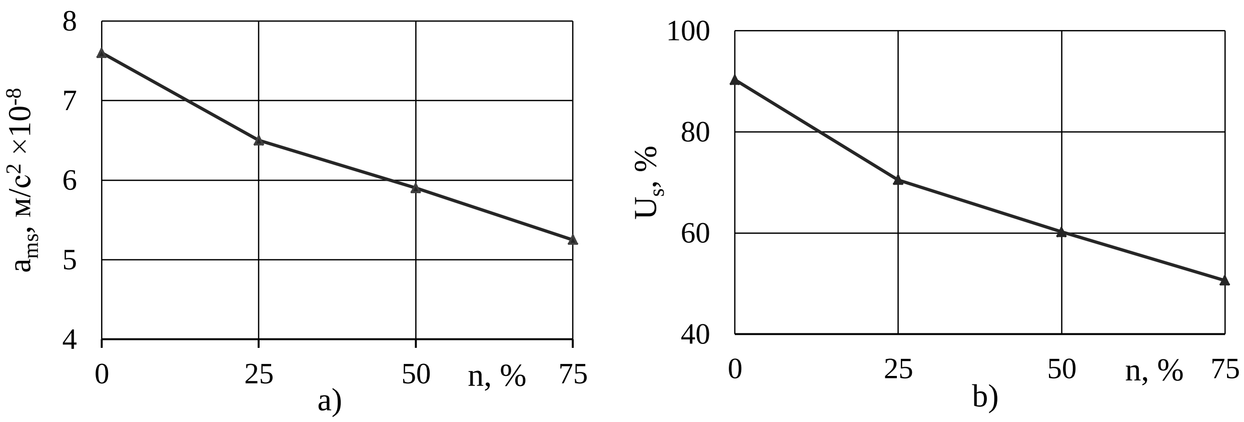

Fig. 2. Mass transfer coefficient a_{ms} (a) and sulfur content U_s (b) in gypsum sol samples versus fly ash content

The results of the study of the influence of the water-gypsum ratio and the relative sulfur content in impregnated gypsum samples on the strength and hardening coefficient are shown in Fig. 3 and in Table 1.

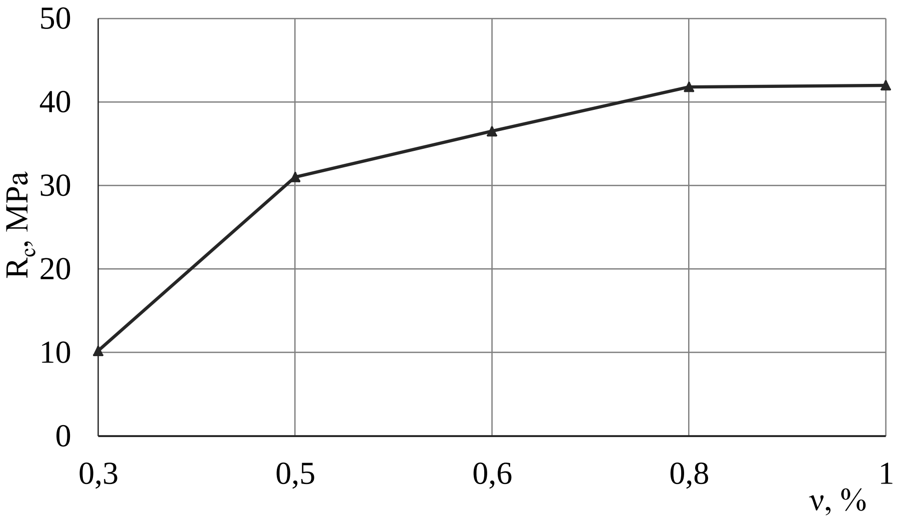

Fig. 3. Dependence of the compressive strength of the impregnated gypsum stone on the relative sulfur content $\nu=U_s/U_{smax}$

Table 1 – Influence of water-gypsum ratio on the strength of gypsum stone impregnated with sulfur melt

W/G	Porosity, %	U_s, %	R_c, MPa	R_b, MPa	$K_h(R_c)$	$K_h(R_b)$
0,45	50,0	71,2	34,1	10,5	4,7	5,0
0,50	53,2	83,5	38,0	10,8	6,5	6,7
0,55	55,8	90,8	42,4	12,5	8,3	8,8
0,60	58,4	97,1	42,8	12,1	9,4	9,8
0,65	60,7	100,0	40,2	11,8	10,2	11,2
0,70	63,1	100,2	36,5	10,2	10,6	12,5

An analysis of the results shows that the strength of samples impregnated with sulfur increases with an increase in the degree of filling of the pore space with it. Thus, with an increase in the relative sulfur content ($\nu=U_s/U_{smax}$) from 0.30 to 0.90, the compressive strength increases from 10.1 to 42 MPa.

A significant increase in strength as a result of impregnation with sulfur is due to the fact that the high porosity of the gypsum stone, which also increases due to the removal of part of the crystalline water, makes it possible to create a sulfur framework during impregnation. The formation of a sulfur framework with a fine-grained sulfur structure determines the high strength of the resulting composite material.

Analysis of the data given in Table 1 shows that the hardening factor of impregnated gypsum specimens increases as the water/gypsum ratio increases. Thus, with an increase in W/G from 0.45 to 0.70, the coefficient of hardening in compression increases from 4.7 to 10.6, and in bending, from 5 to 12.1. This dependence is explained by the fact that with an increase in the water-gypsum ratio, the porosity of the gypsum stone increases, which makes it possible to obtain a more "powerful" sulfur framework during impregnation. As a result, the strength of the impregnated samples increases. However, with an increase in the water-gypsum ratio, the number of pores larger than 0.2 mm increases, which are partially clogged with sulfur, which leads to a decrease in the strength of the sulfur framework. This can explain a slight decrease (by 1.17 times) in the strength of the impregnated samples with an increase in W/G from 0.60 to 0.70, but the strength of the original gypsum sample decreases by 1.3 times, which causes an increase in the strengthening coefficient.

After impregnation with sulfur, the gypsum matrix largely loses its properties and the role of the framework, since during dehydration it significantly reduces the already low strength.

Experiments have shown (Fig. 4) that the strength of the impregnated samples decreases with an increase in the content of fly ash. This is due to the fact that with an increase in the amount of fly ash, the porosity decreases and, as a result, the maximum sulfur content in the impregnated samples decreases. However, as the fly ash content increases, the hardening coefficient increases. This is due to the fact that with an increase in the amount of fly ash, the strength of the original gypsum sol sample decreases faster than that of the impregnated one.

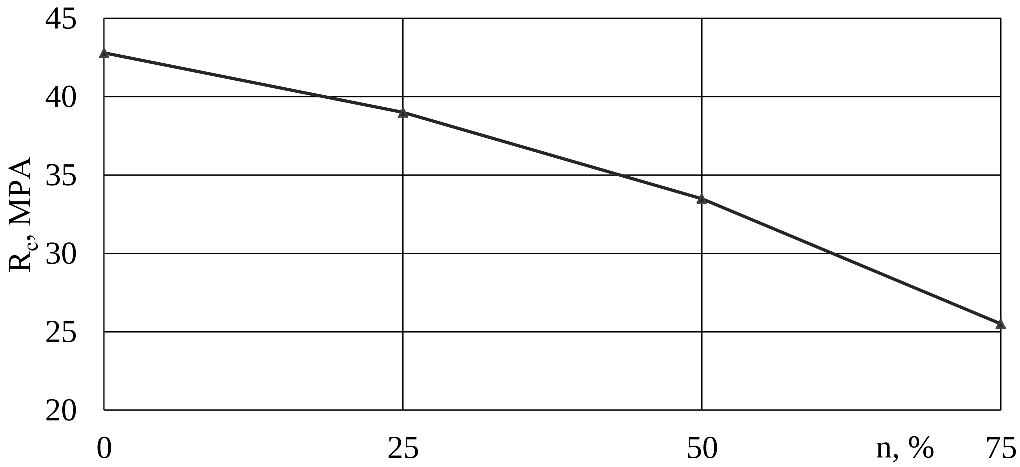

Fig. 4. Dependence of the compressive strength of the impregnated gypsum stone from fly ash content

An increase in the content of polymeric sulfur in the impregnated samples contributes to an increase in their strength by reducing internal stresses and forming stronger adhesive bonds between sulfur and gypsum stone.

In order to determine the area of rational use in the construction of products from a material based on gypsum and sulfur, studies of their water resistance and chemical resistance were carried out.

The results of the study of water resistance impregnated with sulfur samples with varying the content of fly ash and relative sulfur content are given in Table. 2.

Table 2 – Dependence of the softening coefficient on the composition of the impregnated samples

Sample composition			Relative sulfur content	Softening coefficient ($K_{w.r.}$)
Gypsum, %	Fly ash, %	W/A		
100	0	0,55	0,75	0,50
			1,00	0,72
75	25	0,48	0,75	0,58
			1,00	0,78
50	50	0,41	0,75	0,64
			1,00	0,85
25	75	0,34	0,75	0,68
			1,00	0,88

Analysis of the data shows that the water resistance of the impregnated samples significantly depends on the degree of impregnation and the amount of fly ash. So, with partial impregnation of gypsum samples, the softening coefficient is 0.50, while with full impregnation it is 0.72. A more significant decrease in water resistance in the case of partial impregnation is due to the following reasons. Firstly, due to the diffusion of moisture through the outer impregnated layer in the inner region of the sample, hemihydrate gypsum is hydrated due to the topochemical addition of water molecules with the formation of highly dispersed calcium sulfate dehydrate. The resulting internal stresses lead to the appearance of microcracks in the outer impregnated layer. Secondly, due to the subsequent topochemical hydration of capillary condensation, significant amounts of water arise, leading to intensive dissolution of the contact zones between gypsum crystals and the appearance of a wedging effect. It is quite obvious that with a decrease in the depth of impregnation of samples, the intensity of destructive processes will increase and their water resistance will decrease. It has been established that the coefficient of water resistance of impregnated gypsum sol samples increases with an increase in the concentration of fly ash and is 0.80...0.88. This is due to the denser structure of the impregnated gypsum stone, which reduces its permeability. With an increase in the concentration of fly ash, the relative content of water-soluble intercrystalline contacts of gypsum decreases [9]. Thirdly, when impregnating gypsum ash samples (especially with a high content of fly ash), the sulfur melt, filling the intergranular space of fly ash, acts as a binder, since sufficiently strong adhesive bonds arise between sulfur and fly ash. As a result, a matrix of water-resistant material is formed, similar to sulfur mastic, which, as is known [3], has high water resistance (0.92).

The conducted experiments give grounds to believe that the material based on gypsum, ash and sulfur refers to waterproof materials, since the softening coefficient is higher than 0.8. Therefore, it can be predicted that facing products made of such material will have high durability.

It is known that the chemical resistance of a material depends mainly on its permeability and the reactivity of the material components to aggressive media. It has been established that the impregnation of gypsum sol samples with sulfur significantly reduces their total porosity, which can significantly reduce the permeability of the gypsum sol matrix, and therefore a decrease in the potential aggressiveness of the medium can be predicted [10, 11]. The chemical stability of impregnated samples was determined in solutions of magnesium sulphate, sulfuric, acetic and oxalic acids, which are the most characteristic components of aggressive environments in livestock buildings, chemical and food industries. The results of testing samples after six months of exposure to these environments are given in Table. 3.

It has been established that gypsum and gypsum sol samples impregnated with sulfur melt have a chemical resistance coefficient of at least 0.7. This allows them to be classified as chemically resistant in these environments.

Table 3 – Results of testing impregnated samples for chemical resistance

Sample composition		U_s, %	Chemical resistance coefficient ($K_{c.r.}$)			
Gypsum, %	Fly ash, %		Acetic acid	Oxalic acid	H_2SO_4	$MgSO_4$
100	0	92.0	0.71	0.71	0.70	0.70
75	25	74.2	0.76	0.78	0.75	0.75
50	50	61.4	0.84	0.84	0.83	0.84
25	75	54.0	0.88	0.88	0.88	0.88

Thus, it was found that the value of the hardening coefficient increases with an increase in the amount of absorbed sulfur, fly ash content and water-solid ratio. The positive role of fly ash in increasing the water resistance ($K_{w.r.}$>0.8) and chemical resistance ($K_{c.r.}$>0.8) of the impregnated gypsum ash stone is shown by reducing the relative content of water-soluble gypsum contacts and the formation of a matrix of serosol material like sulfur mastic.

The conducted studies have established that products made of composite material based on gypsum, ash and sulfur do not include compounds potentially dangerous for the human body and the environment; are chemically stable and do not emit unstable inorganic compounds into air, water and acid environments. Based on the conclusion of the Chief Sanitary Doctor of Ukraine, a composite material based on gypsum, ash and sulfur is recommended for the construction of buildings of groups "B" and "C" (industrial and public buildings) [12].

Conclusions. On the basis of the studies carried out, the patterns of impregnation of a gypsum sol matrix with a sulfur melt have been established, which make it possible to implement a composite formed by two adhesively bonded continuous substructures - an optimized porous gypsum ash stone and amorphous-crystalline chamois.

An analysis of the main construction and technical characteristics of a composite based on gypsum, ash and sulfur shows that facing products made from such a material have high performance characteristics and it is advisable to use them for lining fertilizer storage facilities, drainage systems, floors and walls of livestock complexes, chemical and Food Industry.

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ЕЛЕМЕНТИ ТЕХНОЛОГІЇ ОТРИМАННЯ СІРКОГІПСОВОГО КОМПОЗИТУ І ЙОГО ВЛАСТИВОСТІ, ЯК ОБЛИЦЮВАЛЬНОГО МАТЕРІАЛУ

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Анотація. В роботі розглянуті питання отримання композиційного матеріалу на основі гіпсу, золи-виносу та сірки з підвищеними експлуатаційними характеристиками. Встановлено закономірності просочення гіпсозольної матриці розплавом сірки з урахуванням капілярно-пористої структури гіпсозольного каменю та фізико-технічних властивостей сірки. Проведені дослідження дозволяють визначати коефіцієнт масопереносу сірки a_{ms} та максимального сірковмісту U_s у просочених виробах, залежно від вихідних значень температури розплаву сірки, водотвердого відношення та вмісту золи-виносу. Це дозволяє ефективно та оперативно визначати тривалість просочення до заданого вмісту сірки для конкретного складу та розміру гіпсозольних виробів. Досліджено технологічні фактори, що впливають на коефіцієнт зміцнення, водостійкість та хімічну стійкість гіпсозольних виробів, просочених у розплаві сірки. Аналіз результатів показує, що міцність просочених сіркою зразків підвищується зі збільшенням рівня заповнення нею порового простору. Так, при збільшенні відносного вмісту сірки ($v= U_s/U_{smax}$) з 0,30 до 0,90 межа міцності при стисканні підвищується з 10,1 до 42 МПа. Встановлено, що водостійкість просочених зразків істотно залежить від ступеня просочення та кількості золи-винесення. Проведені експерименти дають підставу вважати, що матеріал на основі гіпсу, золи та сірки відноситься до водостійких матеріалів, оскільки коефіцієнт розм'якшення вище 0,7. Встановлено, що гіпсові та гіпсозольні зразки, просочені розплавом сірки, мають коефіцієнт хімічної стійкості не менше 0,7, це дозволяє віднести їх до хімічно стійких. Отриманий сіркогіпсовий композит відрізняється високою міцністю, водо- та корозійною стійкістю до агресивних середовищ тваринницьких комплексів, підприємств харчової та хімічної промисловості, де його слід використовувати у вигляді спеціальних облицювальних виробів.

Ключові слова: сірка, гіпс, зола, водостійкість, хімічна стійкість, розплав сірки.

ЭЛЕМЕНТЫ ТЕХНОЛОГИИ ПОЛУЧЕНИЯ СЕРОГИПСОВОГО КОМПОЗИТА И ЕГО СВОЙСТВА, КАК ОБЛИЦОВОЧНОГО МАТЕРИАЛА

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Аннотация. В работе рассмотрены вопросы получения композиционного материала на основе гипса, золы-унос и серы с повышенными эксплуатационными характеристиками. Установлены закономерности пропитки гипсозольной матрицы расплавом серы с учетом капиллярно-пористой структуры гипсозольного камня и физико-технических свойств серы. Проведенные исследования позволяют определять коэффициент массопереноса серы a_{ms} и максимальное серосодержащее U_s в пропитанных изделиях, в зависимости от исходных значений температуры расплава серы, водотвердого отношения и содержания золы-уноса. Это позволяет для конкретного состава и размера гипсозольных изделий эффективно и оперативно определять продолжительность пропитки до заданного содержания серы. Исследованы технологические факторы, влияющие на коэффициент упрочнения, водостойкость и химическую стойкость гипсозольных изделий, пропитанных в расплаве серы. Анализ результатов показывает, что прочность пропитанных серой образцов повышается с увеличением степени заполнения ею порового пространства. Так, при увеличении относительного серосодержания ($v= U_s/U_{smax}$) с 0,30 до 0,90 предел прочности при сжатии повышается с 10,1 до 42 МПа. Установлено, что водостойкость пропитанных образцов существенно зависит от степени пропитки и количества золы-унос. Проведенные эксперименты дают основание считать, что материал на основе гипса, золы и серы, относится к водостойким материалам, поскольку коэффициент размягчения выше 0,7. Установлено, что гипсовые и гипсозольные образцы, пропитанные расплавом серы, имеют коэффициент химической стойкости не менее 0,7, что позволяет отнести их к химически стойким. Полученный серогипсовый композит отличается высокой прочностью, водо- и коррозионной стойкостью к агрессивным средам животноводческих комплексов, предприятий пищевой и химической промышленности, где его следует использовать в виде специальных облицовочных изделий.

Ключевые слова: сера, гипс, зола, водостойкость, химическая стойкость, расплав серы.

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