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Corrosion Resistance of Magnesia Binders in Aggressive Liquids

Olga MIRYUK*

Rudny Industrial University, 111500, Rudny, Kostanay Region, Kazakhstan

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***Abstract* – The article is devoted to the relevant problem of increasing the durability of magnesia materials. The objective of the work is to study the effect of mineral additives and salt grouting liquid on magnesia binders' hardening and resistance in aggressive liquid environments. The experiments involved the following starting materials such as: caustic magnesite, metallurgical slag, ash from thermal power plants, and iron ore processing waste. Solutions of magnesium chloride, magnesium sulfate, iron sulfate, and their combinations were used to close magnesia binders. The composition of magnesia binders was studied using diffractometric analysis. Materials' properties were determined by physical and mechanical testing. There are presented strength tests' results of magnesia binders kept in various environments such as air, water, and salt solutions. The strength indicators are supplemented by photographs of samples appearance subjected to corrosive effects. The reasons for changes in binders' resistance in aggressive liquids are substantiated. Preferred compositions of magnesia binders demonstrating the increased resistance to corrosion processes were proposed. The research results are aimed at developing resource-saving technologies and increasing the durability of effective building materials, expanding the scope of application of magnesite materials.**

***Keywords* – Caustic magnesite; composite binders; corrosion; expansive hydrates; strength.**

1. INTRODUCTION

Construction is an eternal sphere of activity where people embody their ideas and possibilities about reliability and comfort. Concrete is the main material of modern construction. Long-term experience in the construction and operation of facilities, dynamic development of innovative technologies, numerous advantages of products allow us to position concrete as a main structural material in the foreseeable future. Concrete is characterized by a variety of composition, structure, properties and purposes. Concrete is a common composite material. Technological and operational properties of concrete are determined by the binder, which acts as a matrix in the composite material.

Dominance of cement concretes is due to the following factors such as the state of knowledge in production technology, high construction and technical indicators, operational durability of cement stone, protective function in relation to steel reinforcement. Without diminishing the importance of cement concretes, it is necessary to highlight the main problems of using cements in the concrete; they are high material intensity and energy consumption of cement production, large CO_2 emissions during firing of cement clinker, long-term heat and moisture treatment at a temperature of 80–90 °C for accelerated hardening

* Corresponding author.
E-mail address: psm58@mail.ru

of products [1], [2]. Predominance of weakly crystallized hydrates in cement stone often creates difficulties for the technology of concretes of porous structure, increases the risk of destructive processes during operation of objects.

To solve the listed problems, it is important to develop energy-efficient technologies of low-carbon concretes based on low-clinker and cementless binders [3]–[6].

Analysis of scientific and technical literature of recent decades indicates a growing interest in magnesite materials. The facts testifying to the advantages of caustic magnesite and materials based on it are convincing. Caustic magnesite is a type of magnesite binder obtained by firing magnesite rocks at temperatures of 750–800 °C. Caustic magnesite contains MgO as an active phase. Solutions of salts ($MgCl_2$ and $MgSO_4$) are used to mix caustic magnesite [7], [8].

A binder based on MgO and solution of $MgCl_2$ is called Sorel cement in honor of Stanislav Sorel, the French engineer, who developed this material [7]. When hydrating caustic magnesite with magnesium chloride solution, mainly magnesium hydroxychlorides $3MgO \cdot MgCl_2 \cdot 8H_2O$ and $5Mg(OH)_2 \cdot MgCl_2 \cdot 8H_2O$ are formed, as well as magnesium hydroxide $Mg(OH)_2$ [7], [8].

Crystalline structure of the main hydrates of magnesite binders, mainly caustic magnesite, provides intensive hardening, high strength, and high resistance to bending loads. Activating ability of magnesite binders allows latent substances of natural and technogenic origin to be involved in the structure formation. The pronounced adhesive ability of magnesite binders to various fillers allows creating a number of effective composite materials that are resistant to abrasion, biological corrosion, fire and oils.

Despite its unique properties, the use of caustic magnesite in the construction is limited. The main reasons for the widespread use of caustic magnesite are the following.

Firstly, it's a relatively small natural raw material base; magnesite and brucite rocks, which are geographically unevenly distributed, are mainly used to obtain binders.

Secondly, dust captured in the refractory production during firing of magnesite into periclase is often used as a magnesite binder.

Thirdly, magnesite binder's manufacturer often does not control properties that affect the nature of hardening and the behavior of the material when exposed to a humid environment. This causes unpredictability of construction and technical properties and complicates the rational use of magnesite binders.

Fourthly, the use of salt sealants contributes to increase in the cost of magnesite materials.

However, all of the listed problems can be solved mainly by organizational and technological measures. Magnesia binders harden and retain their strength only in air environment. Low water resistance of the hardened stone is the main obstacle to the widespread use of magnesia binders. Numerous studies and developments are devoted to solving this urgent scientific problem. All recommendations aimed at increasing the water resistance of magnesia binders can be presented in the form of three groups.

The first group is based on the introduction of chemical additives that help increase the resistance of magnesia hydrates by changing their structure [9]–[13].

The second group of measures is aimed at preferential use of sulfate salt solutions instead of magnesium chloride solution. These actions ensure the formation of magnesium hydroxysulfates, which are more stable in the water [14]–[19].

The third group of recommendations provides for a combination of caustic magnesite with mineral components of various compositions [20]–[24].

Increased water resistance of composite magnesite binders is achieved by forming hydrated compounds with the participation of mineral components activated in a magnesite medium. Creation of composite magnesite binders allows not only to increase the water resistance of

materials, but also to reduce energy costs by reducing the proportion of the firing component and to ensure resource conservation by involving man-made waste.

During operation, composite materials are exposed to the aggressive effects of salt solutions present in industrial, ground and sea water. Information on the effect of salt solutions on the performance properties of magnesite materials is very limited and ambiguous [25]–[27].

The purpose of the research is to study the effect of mineral additives and salt sealers on the hardening and resistance of magnesite binders in aggressive liquid environments.

2. MATERIALS AND METHODS

The object of the study was caustic magnesite and composite magnesia binders based on it. Caustic magnesite of PMK-75 brand (caustic magnesite powder containing at least 75 % magnesium oxide) with a specific surface area of 290 m^2/kg was used in the experiments. To obtain composite binders, technogenic materials of various compositions and origins were added to the caustic magnesite (see Table 1), they are metallurgical slag, ash from thermal power plants, and waste from iron ore enrichment. Composite binders were obtained by thoroughly mixing caustic magnesite with pre-crushed technogenic components. The specific surface area of the composite binders was 290–310 m^2/kg. Solutions of magnesium chloride, magnesium sulfate, and iron sulfate of various densities were used to mix the magnesia binders. Strength properties of magnesite binders were determined by testing 20 mm cube samples on a PGM-1000MG4 hydraulic press. Conditions for testing the operational durability of magnesite binders are as follows: the samples were immersed into the aggressive liquid media after 7 days of hardening in air (water, magnesium chloride solution with a density of 1100 kg/m^3, magnesium sulfate solution with a density of 1100 kg/m^3). The concentration of magnesium chloride in the solution is 23.7 %. The concentration of magnesium sulfate in the solution is 22.5 %. Drinking water was used for experiments (total mineralization is 350 mg/L; hardness 3.7 mg-eq/L). The nature of impact of aggressive liquids on the magnesite binders was assessed by the resistance coefficient: the ratio of samples strength after constant exposure to aggressive liquid for a specified period and the strength of air-cured samples.

The chemical composition of technogenic materials was determined by X-ray fluorescence (XRF**)** on an ED 2000 X-ray fluorescence spectrometer (Oxford Instruments Analytical). The method for determinations of mass loss ignition is based on calcining materials at a temperature of 1000±50 °C to a constant mass and determining the mass loss of materials by gravimetric method. The specific surface area of the materials was evaluated using a PSH-11M device by recording the passage time of the volume of air through the powder layer. Strength properties of caustic magnesite mixed with magnesium chloride solution with a density of 1250 kg/m^3 (let us denote M) were adopted as reference when comparing properties of binders of different compositions. Phase composition of binders was determined using a DRON-3M diffractometer with a BSV-24 X-ray tube with CuK α-radiation. The microstructure of the hardened binder was studied using a JSM-649OLV scanning electron microscope.

TABLE 1. CHEMICAL COMPOSITION OF TECHNOGENIC COMPONENTS OF THE BINDER

Material name	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Other	Loss on ignition
Metallurgical slag	44.0	14.1	0.9	32.5	5.2	2.1	1.2	–
Ash from thermal power plants	48.3	17.8	1.4	14.9	3.4	1.3	2.5	10.4
Iron ore beneficiation waste	40.8	11.9	16.9	12.5	5.2	4.3	5.0	3.4

3. Results and Discussion

The studied caustic magnesite, mixed with magnesium chloride solution with a density of 1250 kg/m^3, demonstrated intensive hardening in the early stages and high design strength at the age of 28 days when hardening in air, as seen in Fig. 1. After three days of hardening, the strength of the magnesite stone was 65 % of the design value. Setting time of caustic magnesite: start is 45 min, end is 2 hours 15 minutes. Along with the indicators characterizing properties of the air-hardening binder, caustic magnesite was tested for uniformity of volume change using the method proposed in the research [28]. Samples-cakes made from the plastic caustic magnesite dough were immersed into the water after 3 days of hardening in air. After one day in the water, the cake samples retained their integrity, but cracks appeared along the edges of the samples, amounting to 10–12.5 % of the diameter of the cake samples (Fig. 1). The formation of fine cracks on the caustic magnesite cake samples indicates increased activity of magnesium oxide. The use of caustic magnesite, which exhibits a tendency to crack formation, made it possible to simulate the most common situation and evaluate the effectiveness of measures to improve the operational durability of binders.

Varying concentration of the grout affects the rate of interaction of magnesium oxide with magnesium chloride, the composition of hydrates and the properties of the hardened binder. The effect of density of magnesium chloride solution on the strength and resistance to aggressive environments of caustic magnesite was studied (Fig. 2).

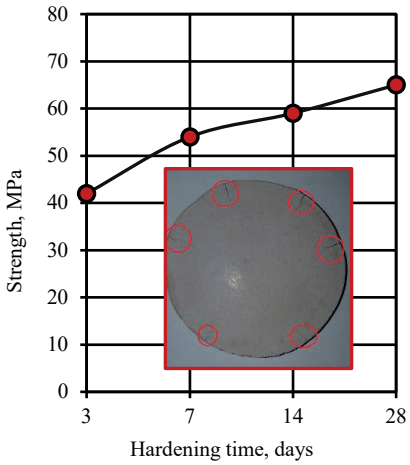


Fig. 1. Strength indices of caustic magnesite at different hardening timeframes and appearance of sample-cake.

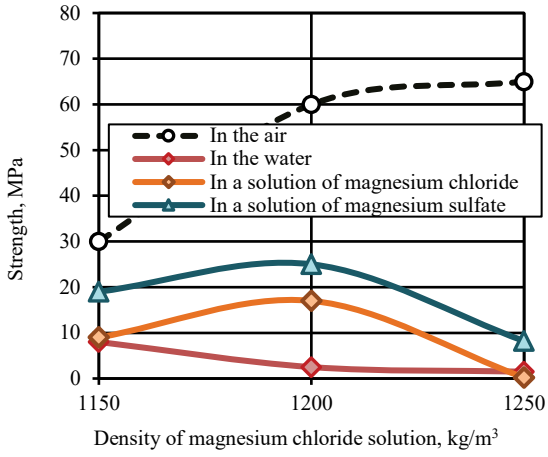


Fig. 2. Effect of magnesium chloride solution's density on the strength of caustic magnesite under different conditions (age 28 days; exposure to liquids 21 days).

When using a low-density magnesium chloride solution (1150 kg/m^3), the strength of caustic magnesite is reduced by almost half. The basis of the hardened binder is magnesium hydroxide $Mg(OH)_2$, the content of magnesium pentahydroxychloride $5Mg(OH)_2 \cdot MgCl_2 \cdot 8H_2O$ (5 – phase) is insignificant [29]. With increase in concentration of magnesium chloride in the solution, the content of the 5 – phase increases, which is accompanied by increase in the strength of caustic magnesite. After exposure to water, the highest strength was demonstrated by the samples of caustic magnesite mixed with

magnesium chloride solution with a density of 1150 kg/m^3 (Fig. 2). With increase in density of the grouting agent, the strength of the samples decreases, on which cracks are formed.

Exposure to aggressive chloride solution led to severe destruction of the samples obtained using a grouting agent with a density of 1250 kg/m^3; gaping cracks formed on the samples (Fig. 3). The number of cracks is smaller on the samples obtained with a grouting agent of lower density.

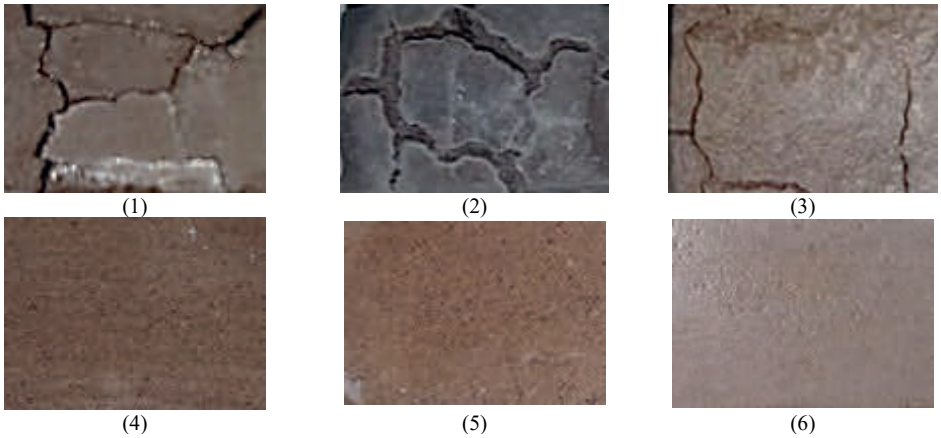


Fig. 3. Appearance of samples after exposure to aggressive liquids: 1 – caustic magnesite + magnesium chloride solution with a density of 1250 kg/m^3 (in the water); 2 – as above (in aggressive chloride solution); 3 – as above (in aggressive sulfate solution); 4 – caustic magnesite + magnesium chloride solution with a density of 1150 kg/m^3 (in aggressive chloride solution); 5 – caustic magnesite + complex solution of magnesium chloride (50 %) and magnesium sulfate (50 %) (in aggressive chloride solution); 6 – caustic magnesite + metallurgical slag (50 %) + magnesium chloride solution with a density of 1250 kg/m^3 (in aggressive chloride solution).

Aggressive sulfate solution had the least destructive effect on the samples of caustic magnesite obtained on the basis of a grouting agent of different densities (Fig. 2).

In aggressive salt environments, the highest strength is demonstrated by the samples of caustic magnesite mixed with magnesium chloride solution with a density of 1200 kg/m^3.

It is known [15], [18], [30], [31], that the use of sulfate salt sealants increases the water resistance of magnesia binders. Properties of caustic magnesite sealed with solutions of magnesium sulfate (let us denote as MM) and iron sulfate (let us denote as MF) with a density of 1200 kg/m^3 were studied (Fig. 4 and Fig. 5). The strength of MM and MF samples hardened in air is half the strength of caustic magnesite sealed with magnesium chloride (let us denote as M). At the same time, the strength of MM and MF samples kept in liquid media exceeds similar indicators of M samples by 3–30 times. The conflict situation predetermined the combination of solutions of different salts. Solutions of complex chlorine-sulfate composition containing $MgCl_2$ and $MgSO_4$, $MgCl_2$ and $FeSO_4$ in various ratios were used for sealing caustic magnesite as depicted in Fig. 4 and Fig. 5. Solutions containing up to 30 % $MgSO_4$ and $FeSO_4$ provide materials comparable in strength to the reference composition M. Caustic magnesite in combination with solutions containing 20–50 % $MgSO_4$ and $FeSO_4$, after exposure to aggressive environments, has strength values close to those of MM and MF samples.

Creation of caustic magnesite compositions with mineral components is a promising direction in the technology of magnesite materials. Compositions of caustic magnesite with technogenic components of various origins were studied as seen in Fig. 6. Combinations of caustic magnesite with metallurgical slag and iron ore processing waste provide increase in

the strength of the composite binder, therefore they are accepted for further research. Using the example of magnesite-slag binder, the effect of content of the technogenic component on the strength of the composite material under various curing conditions was studied (Fig. 7).

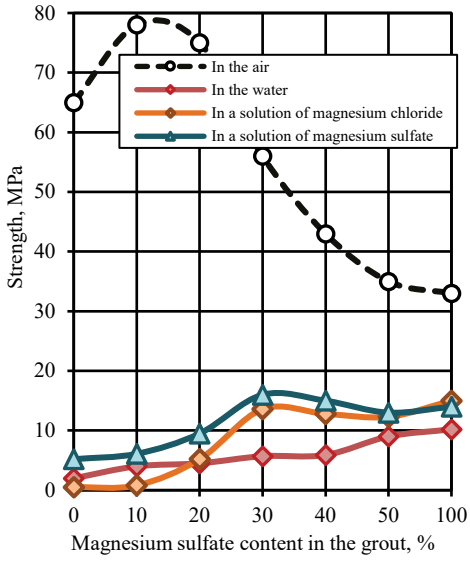


Fig. 4. Effect of magnesium sulfate content on the strength of caustic magnesite under different conditions (age 28 days; exposure to liquids 21 days).

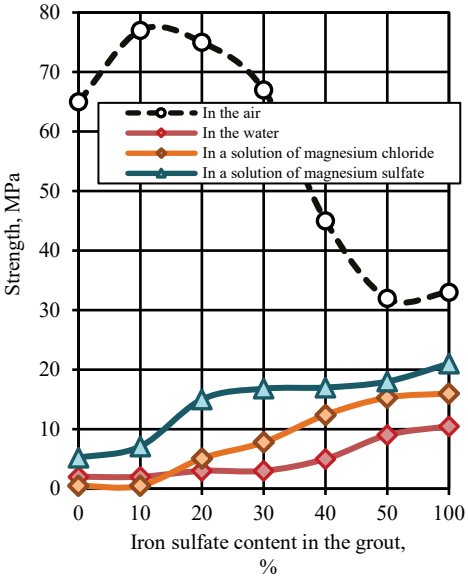


Fig. 5. Effect of iron sulfate content on the strength of caustic magnesite under different conditions (age 28 days; exposure to liquids 21 days).

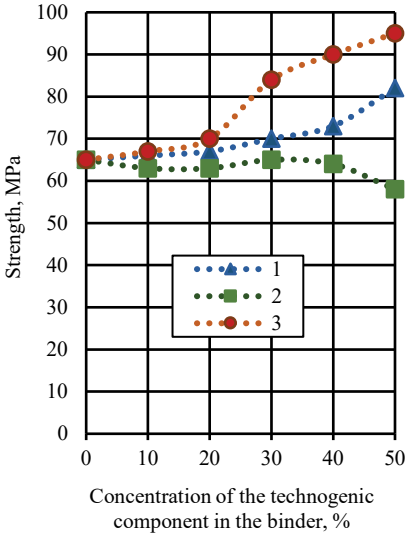


Fig. 6. Influence of the technogenic component content on the strength of binder at the age of 28 days under air hardening: 1 – iron ore processing waste; 2 – thermal power plant ash; 3 – metallurgical slag.

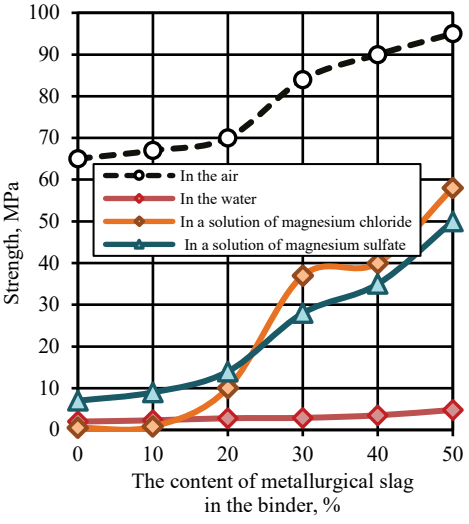


Fig. 7. Effect of metallurgical slag content on the strength of composite binder under different conditions (age 28 days; exposure to liquids 21 days).

Magnesite-slag binders containing 30–50 % of the technogenic component exhibit increased resistance in aggressive environments. It is noteworthy that the highest strength indicators were achieved by binders kept in salt solutions. The strength of magnesia-slag binder with increased slag content after aging in salt solutions is comparable to the strength of air-hardening magnesia binders. Samples of magnesia-slag binders aged in water retained the integrity of the structure, but reduced strength. Discrepancy between the obtained research results and the known information [32], [33] on the increased water resistance of composite magnesia binders is due to the harsh conditions of the described experiments, which provide for a long stay of the samples in water.

To study the combined effect of solutions of complex composition and mineral components on the corrosion resistance of magnesia materials, composite binders with 50 % of technogenic component, mixed with solutions with the maximum content of sulfate salts, were used (see Figs. 8–9).

Mixing magnesia-slag binder with complex salt solution helps to increase water resistance, but under other conditions the strength of the binder decreases compared to samples based on magnesium chloride solution as observed in Fig. 8. Composite binder containing 50 % of iron ore enrichment waste, when replacing the magnesium chloride solution with a complex solution ($MgCl_2$: $FeSO_4$ = 50:50, %) reduces the strength under all conditions of sample curing.

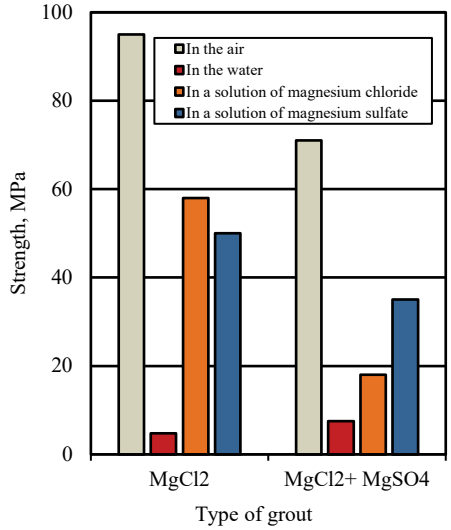


Fig. 8. Effect of the grouting fluid composition on the strength of a binder containing 50 % of metallurgical slag, under various conditions (age 28 days; exposure to liquids 21 days).

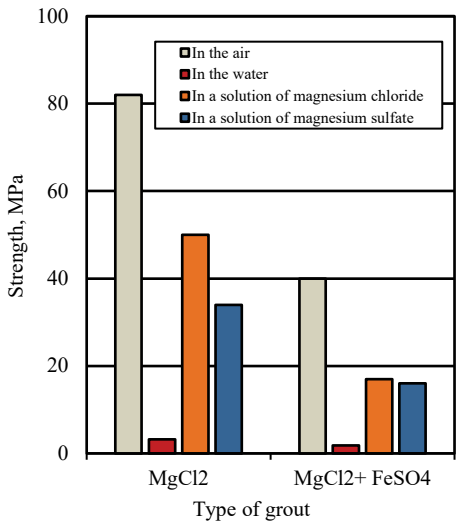


Fig. 9. Effect of the grouting fluid composition on the strength of a binder containing 50 % of iron ore processing waste under various conditions (age 28 days; exposure to liquids 21 days).

Comparison of absolute strength indices of magnesite materials (Fig. 2, Figs. 4–9) allows us to identify binder compositions with maximum and minimum strength and determine their area of application. To assess the effect of operating conditions on the strength of specific binders, it is advisable to use the resistance coefficient (Fig. 10). Comparative analysis of the resistance coefficient values shows that the aqueous medium has the strongest corrosive effect on the magnesite binders. The highest water resistance coefficient is found in the caustic magnesite samples mixed with sulfate salt solutions. Increased resistance coefficient values are achieved in the aggressive chloride environment, when mixing caustic magnesite with

iron sulfate solutions, as well as for composite binders mixed with magnesium chloride solution (Fig. 10). All samples kept in the aggressive sulfate environment are characterized by comparatively high resistance coefficient values. In this case, the greatest resistance was demonstrated by samples of caustic magnesite mixed with solution of magnesium chloride with a density of 1150 kg/m^3; samples of caustic magnesite mixed with solutions containing iron sulfate; samples of magnesite-slag binder mixed with solution of magnesium chloride and chlorine-sulfate solution, as validated in Fig. 10.

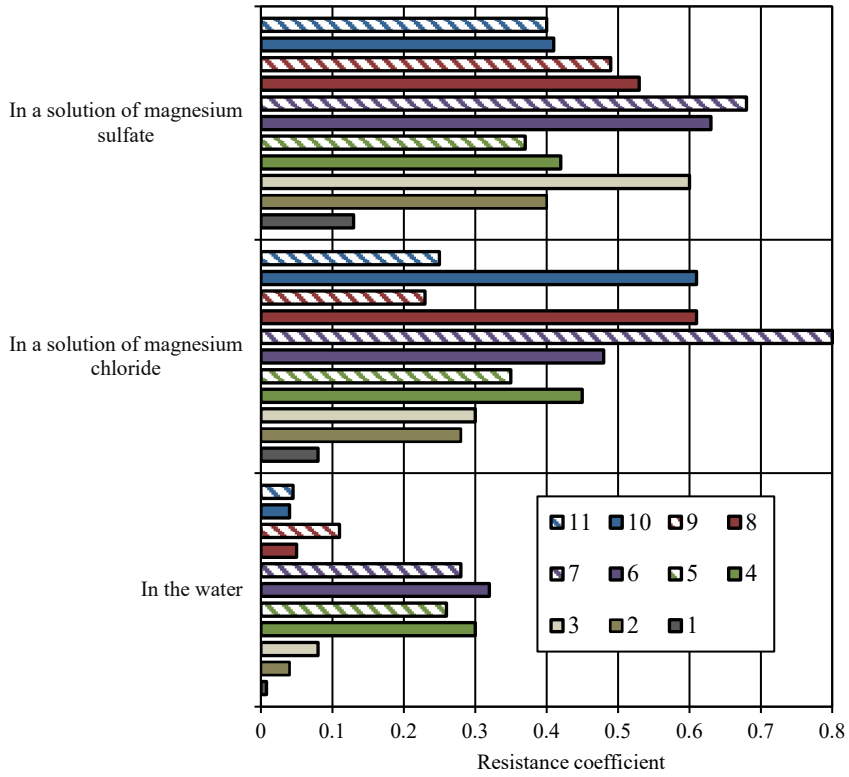


Fig. 10. Resistance coefficient of magnesite binders of various compositions in the aggressive liquids. 1 – caustic magnesite + magnesium chloride solution with a density of 1250 kg/m^3; 2 – caustic magnesite + magnesium chloride solution with a density of 1200 kg/m^3; 3 – caustic magnesite + magnesium chloride solution with a density of 1150 kg/m^3; 4 – caustic magnesite + magnesium sulfate solution with a density of 1200 kg/m^3; 5 – caustic magnesite + complex solution of magnesium chloride (50 %) and magnesium sulfate (50 %); 6 – caustic magnesite + iron sulfate solution with a density of 1200 kg/m^3; 7 – caustic magnesite + complex solution of magnesium chloride (50 %) and iron sulfate (50 %); 8 – caustic magnesite + metallurgical slag (50 %) + magnesium chloride solution with a density of 1250 kg/m^3; 9 – caustic magnesite + metallurgical slag (50 %) + complex magnesium chloride solution (50 %) and magnesium sulfate (50 %); 10 – caustic magnesite + iron ore processing waste (50 %) + magnesium chloride solution with a density of 1250 kg/m^3; 11 – caustic magnesite + iron ore processing waste (50 %) + complex magnesium chloride solution (50 %) and iron sulfate (50 %).

Analysis of the strength indices of magnesite binders of various compositions and comparison of the appearance of samples kept in aggressive liquids allows us to imagine the nature of destructive processes. Conventionally, two types of destructive processes are distinguished as follows: the processes accompanied by a decrease in the strength of the material while maintaining integrity of the samples; processes leading to cracking and a sharp drop in the strength of the samples. Both types of processes are caused by decomposition of

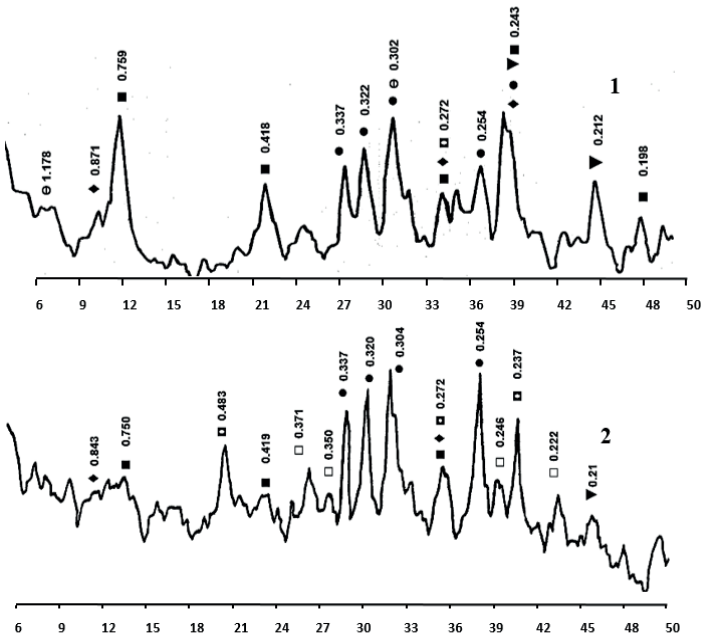
magnesium pentahydroxychloride, the main carrier of the strength of magnesite binders [32], [34], [35].

The first type of processes is typical for binder samples in which the content of magnesium pentahydroxychloride is limited (when using a low-density magnesium chloride solution, when using chlorine-sulfate solutions, in the presence of mineral components).

The second type of processes is typical for binder samples in which expansive phases (secondary magnesium hydroxide, secondary magnesium pentahydroxide) are formed under the influence of aggressive liquid. If the resistance of the magnesite rock structure is less than the stress created by expansive hydrates, cracks are formed in the samples. Fractures of caustic magnesite samples sealed with magnesium chloride solution (composition M) are observed when kept in the aggressive chloride solution. This is apparently due to the fact that the original rock, containing a significant amount of magnesium pentahydroxychloride, weakens after decomposition of this hydrate. The formation of secondary magnesium pentahydroxychloride is accompanied by a fracture of the weakened structure.

Increased values of the resistance coefficient of magnesite binders kept in aggressive salt solutions are, on the one hand, due to a decrease in the rate of decomposition of magnesium pentahydroxychloride, and on the other hand, to the formation of magnesium hydroxide chlorides and magnesium hydroxide sulfates based on magnesium oxide (including magnesium oxide formed during the decomposition of the 5 – phase) with the participation of aggressive salt solutions.

High corrosion resistance of composite magnesia binders is also ensured by the formation of hydrates based on compounds of technogenic components. This is confirmed by the results of diffractometric analysis of magnesia-slag binder (Fig. 11).



Θ, degree; ▼ – MgO; ◘ – $Mg(OH)_2$; ■ – $5Mg(OH)_2 \cdot MgCl_2 \cdot 8H_2O$; ♦ – $3Mg(OH)_2 \cdot MgCl_2 \cdot 8H_2O$; **ϴ** – $Mg(OH)_2 \cdot MgCl_2 \cdot 2MgCO_3 \cdot 6H_2O$; ● – metallurgical slag; □ – magnesium hydrosilicates.

Fig. 11. Diffraction patterns of magnesia-slag binder: 1 – air hardening, 28 days; 2 – aging in an aggressive chloride solution 21 days.

The crystalline basis of magnesia-slag binder is formed by hydroxychloride complexes. Magnesia-slag binder, kept in aggressive chloride solution, contains magnesium hydroxide formed during decomposition of hydrate complexes. Appearance of diffraction reflections, for example, $d = 0.222$; 0.246; 0.350; 0.371 nm, indicates the presence of magnesium hydrosilicates. Formation of magnesium hydrosilicates is a result of the activating effect of aggressive salt solution on the hydration capacity of metallurgical slag. Increased corrosion resistance of magnesia-slag binder is provided by the features of the microstructure (Fig. 12).

The basis of the microstructure is made up of plate crystals 5 – phase formed in the environment of weakly crystallized hydrates based on slag phases. Needle crystals of magnesium pentahydroxychloride, characteristic of hydration of caustic magnesite, are less resistant to the effects of a liquid medium (as seen in Fig. 12).

Dominance of magnesium pentahydroxychloride in the microstructure in the absence of shock-absorbing amorphous hydrate formations causes increased vulnerability of the binder to the effects of aggressive liquid media.

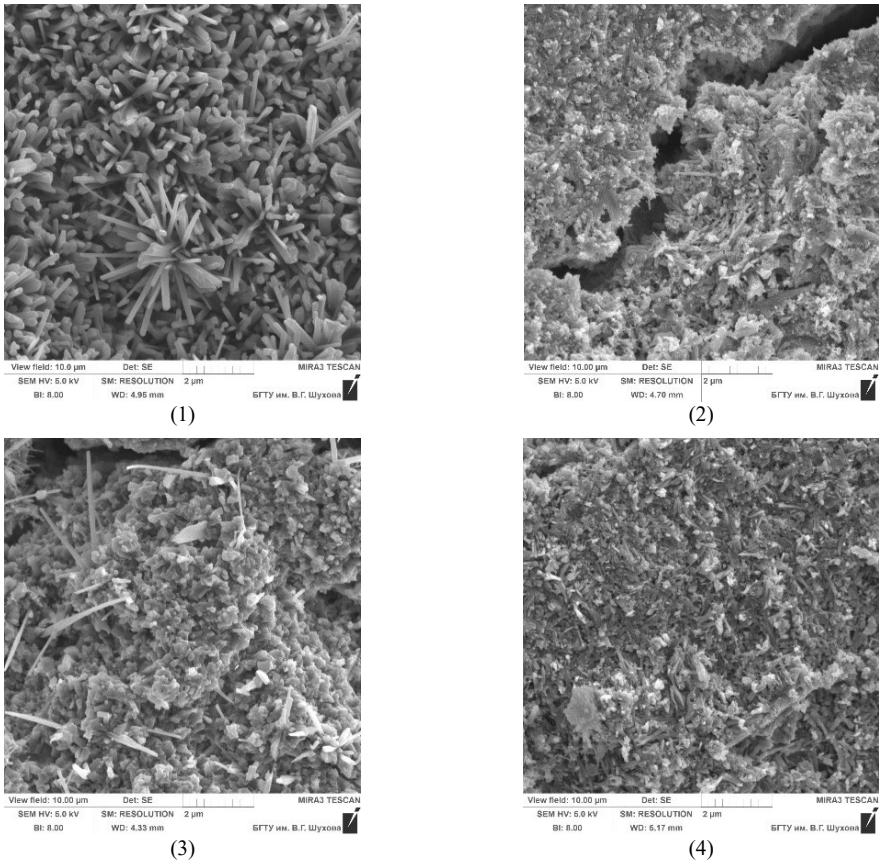


Fig. 12. Microstructure of magnesium binder stone: 1 – caustic magnesite + magnesium chloride solution with a density of 1250 kg/m^3 (air hardening); 2 – as above (ageing in water); 3 – magnesia slag binder + magnesium chloride solution with a density of 1250 kg/m^3 (air hardening); 4 – as above (ageing in water).

4. CONCLUSION

The concepts of the impact of aggressive environments on the strength and corrosion resistance of magnesia binders have been developed.

Varying the material composition of the magnesia binder and salt sealant affects the hardening rate and strength indicators, and is also accompanied by a change in the resistance of the hardened binder in aggressive liquids.

It was revealed that the destructive effect of water is reduced by minimizing unstable magnesium pentahydroxychloride and maintaining the strength of the stone structure of magnesia binders. This is achieved by using complex chlorine-sulfate solutions for sealing; using composite magnesia binders containing a technogenic component. It was established that aggressive salt environment is involved in the formation of hydrate phases in the hardened magnesite stone, which favors the corrosion resistance of magnesite binders and ensures the formation of a stable structure of composite magnesite binders.

Formation of secondary magnesium pentahydroxychloride in stone kept in aggressive chloride solution has expansive effect on the stone structure. The absence or limited amount of weakly crystallized and amorphous hydrates increases the vulnerability of crystalline structure of the stone and leads to cracking and destruction of the stone. To prevent the expansive action of secondary hydrates, composite binders are preferable. Formation of dangerous quantities of secondary hydrates is prevented by reducing the proportion of magnesium chloride in the composition of the grout.

A targeted change in the material composition of the magnesite binder will allow more complete use of the unique properties of caustic magnesite, expand the list and improve the technology of effective composite building materials, and promote the development of modern building technologies. Increasing the operational durability of magnesite products is aimed at resource conservation at all stages of the life cycle of construction projects and at creating an ecologically safe living environment.

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