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Study of Magnesite Binders' Resistance in Liquid Aggressive Environments

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Abstract. The study presents the results of operational properties of magnesia binders. The research objective is to determine the nature of aggressive liquid media impact on magnesia binders of various compositions. Comparative tests of strength properties of caustic magnesite and a combined magnesia-slag binder were carried out. Diffractometric analysis was used to study binders' composition. Binders' microstructure was studied by electron microscopy. The nature of impact of water, chloride and sulfate solutions on binders' strength was estimated by a resistance coefficient. Effectiveness of combination of caustic magnesite and metallurgical slag was proven. Activating effect of the magnesia binder allows latent compounds of metallurgical slag to be involved in the hydrate formation processes. Expediency of using a complex grout containing solutions of magnesium chloride and sulfate for magnesia binders is shown. Aggressive effect of water and salt solutions on the strength properties of magnesite binders was studied. Information on the change in strength of binders after various periods of exposure to aggressive liquids was provided. The causes of destructive processes occurring in the magnesite binders were identified. Magnesium pentahydroxychloride's decisive role in the formation of durability of magnesite stone was confirmed. The nature of impact of aggressive salt environments on durability of binders of various compositions was established. Technological methods for increasing durability of magnesite binders in the liquid environments were proposed, consisting in the rational minimization of magnesium pentahydroxychloride due to the use of a combined binder and (or) complex salt grouting fluids.

Key words: caustic magnesite, metallurgical slag, combined binders, salt aggression, water resistance.

Introduction

Concrete is a common composite material, its quality largely determines the efficiency of construction. Technological and operational properties of concrete are determined by a binder, which acts as a matrix in the composite material. Cement concretes dominance is due to their high construction and technical indicators and durability. High resource intensity of cement production, accompanied by large CO_2 emissions [1] – [2] predetermines the need for active distribution of low-energy binders with a small carbon footprint.

Caustic magnesite is a type of magnesite binder obtained by firing magnesite rock at a temperature not exceeding 800°C. Comparative characteristics of Portland cement and caustic magnesite allow you to evaluate advantages and identify problems of each binder.

Portland cement is based on calcium silicates, a binder also contains calcium aluminates and aluminoferrites. To obtain a plastic mass (dough), cement is mixed with water. When cement phases interact with water, the hydrated compounds of various compositions are formed, mainly of a weakly crystallized structure. Heat and moisture treatment is used at a temperature of 80 – 95°C to accelerate hardening of cement concrete.

Caustic magnesite consists mainly of magnesium oxide. Caustic magnesite is mixed with solutions of magnesium chloride or sulfate to ensure active magnesium oxide hydration. Preference for magnesium chloride solution is due to binder's intensive hardening and high strength. Main hydrated formations of caustic magnesite mixed with magnesium chloride solution are crystalline compounds of neutral composition – magnesium hydroxychlorides. Compared to Portland cement, the caustic magnesite does not require a humid environment for hardening, it is characterized by intensive increase in early strength, which makes it possible to exclude heat treatment for accelerated hardening of a binder. Design strength values of caustic magnesite are not inferior to similar indicators of high-grade Portland cement. Magnesia binders are favourably distinguished by increased adhesion to various types of filler; this contributes to creation of a wide range of composite materials [3] – [7]. Crystalline base of the hardened magnesite stone ensures durability of a highly porous structure of cellular concrete [8] – [10]. Due to significant amount of chemically bound water, magnesite materials are fire-resistant and suitable for biological protection. Activating ability of the mixture of magnesium oxide and chloride in relation to various substances allows even latent materials to be involved in the processes of structure formation and hardening. This principle underlies the resource-saving technology of combined (mixed) magnesite binders, in which up to 30 – 50% of caustic magnesite is replaced by natural and man-made origin materials [11] – [14].

Low water resistance of magnesite binders prevents the widespread use of caustic magnesite despite its unique properties. When exposed to a humid environment, the main hydrates are destroyed, and binder's strength is significantly reduced. A number of technological solutions were proposed to solve the urgent problem of increasing water resistance of magnesite binders. The first group of measures involves introduction of additives that help increase the resistance of magnesite hydrates by changing their structure [15] – [17]. The second group of solutions is based on the use of sulfate salt solutions instead of magnesium chloride solution, which ensures formation of magnesium hydroxy sulfates that are more resistant to water [18] – [21]. The third group of measures is associated with replacement of caustic magnesite with the combined binders containing mineral components of various compositions [22] – [25]. Increased water resistance of combined magnesite binders is achieved by forming new, water-resistant compounds based on the mineral components.

Along with the aquatic environment, during their operation concretes are exposed to aggressive effects of salt solutions, for example, sodium and magnesium sulfates in the groundwater, sodium and magnesium chloride present in the seawater.

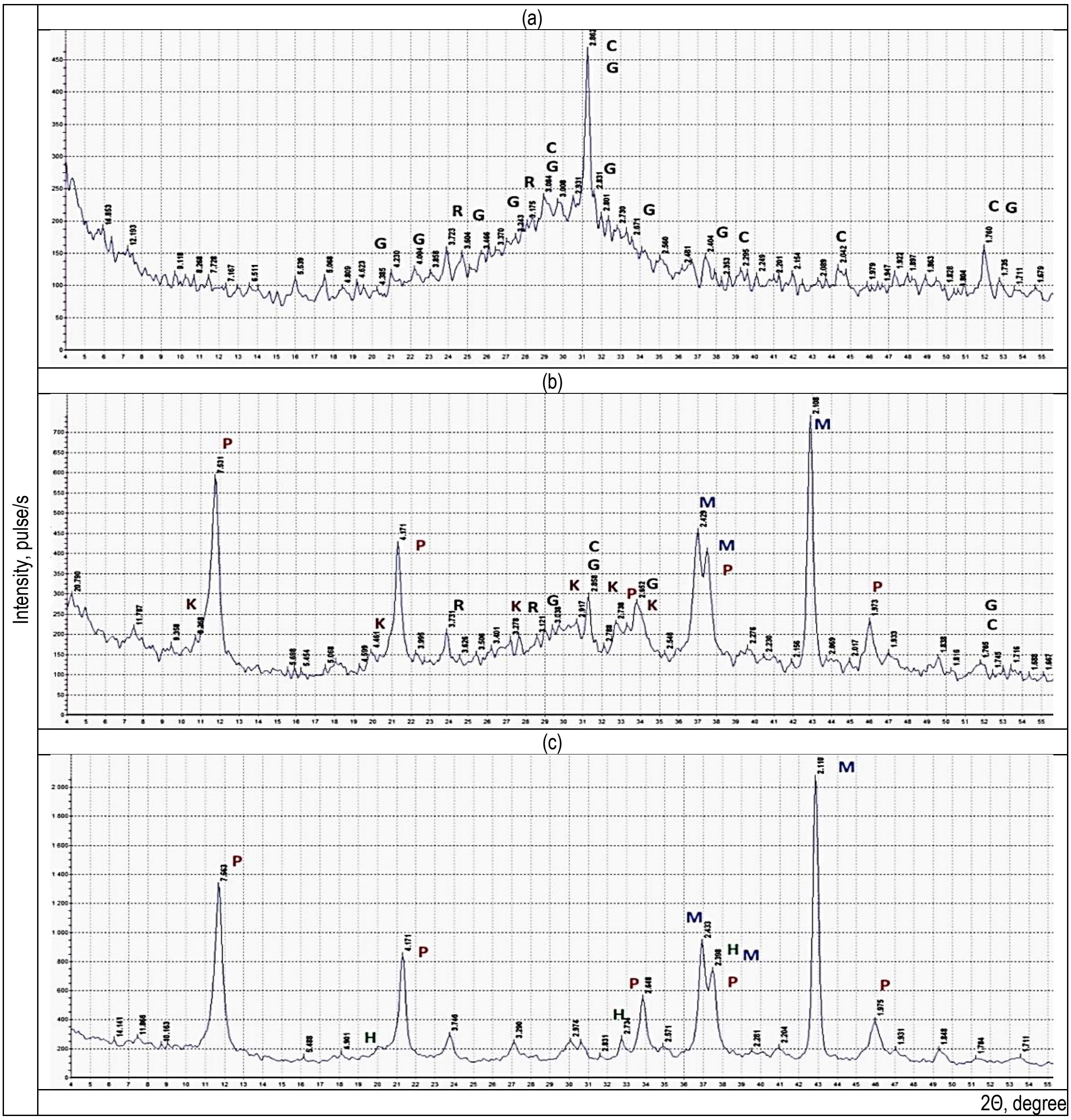
A mechanism and consequences of destructive salt effects on the cement stone are known [26].

Information on the effect of salt solutions on magnesite materials' performance properties is very limited [27] – [28].

The research objective is to study the effect of aggressive liquid environments on durability of magnesite binders of various compositions.

1. Methods and materials

The object of the study was a combined magnesite-slag binder consisting of caustic magnesite grade PMK-75 and metallurgical granulated slag. The main phase of caustic magnesite is magnesium oxide. The phase composition of metallurgical slag includes calcium and magnesium silicates (rankinite $3CaO{\cdot}2SiO_2$, okermanite $2CaO{\cdot}MgO{\cdot}2SiO_2$), calcium aluminosilicates (gehlenite $2CaO{\cdot}Al_2O_3{\cdot}SiO_2$), amorphous phases (Figure 1a). Chemical composition of the binder components is given in Table 1. Properties of caustic magnesite, studied during experiments were adopted for comparison of binders with different compositions.



C – okermanite, G – gehlenite, R – rankinite, M – magnesium oxide, H – brucite, P – magnesium pentahydroxychloride, K – hydrates based on slag compounds

Fig.1. – Diffraction patterns of metallurgical slag (a), hardened magnesia-slag binder (b) and hardened caustic magnesite (c)

Table 1. Chemical components of magnesite binder

Material name	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Others	Loss on ignition
Caustic magnesite	1.1	0.5	2.3	2.7	87.5	0.1	0.2	5.6
Metallurgical slag	44.0	14.1	0.7	32.7	5.1	2.1	1.3	–

Magnesia-slag binder was obtained by joint grinding of components in the «E-max» vibration mill. Magnesia binders were mixed with magnesium chloride and magnesium sulfate solutions and their combinations. Density of salt solutions was determined with a hydrometer.

Strength properties of a binder were determined on 20x20x20 mm samples by testing on PGM-1000MG4 hydraulic press after a specified curing period. Resistance of the hardened binder to aggressive liquids was estimated by the resistance coefficient taking into account the samples strength after exposure to an aggressive environment for a specified period and samples strength before immersion into a liquid medium. Phase composition of the binders was determined using DRON-3M diffractometer with BSV-24 X – ray tube with CuK α-radiation. Microstructure of the hardened stone was examined using a JSM-649OLV scanning electron microscope.

2. Results and discussion

Addition of 10 – 50% metallurgical slag to the caustic magnesite results in a 5 – 25% decrease in consumption of salt solution to obtain plastic dough (Figure 2). Magnesia-slag binders are characterized by slow hardening during the first three days; with increase in the slag content, the early strength of binders decreases by 4 – 22% (Figure 2). With subsequent hardening, strength properties of the combined binders increase. At the age of 28 days, strength of the binders increases as the technogenic component’s content increases. Strength of the combined binder containing 50% slag is 46% higher than the strength of caustic magnesite. The increased strength properties of the magnesia-slag binder are due to changes in the hydrates composition. Features of hydrate formation of the combined magnesia binder are: increased activity of MgO, minimization of brucite $Mg(OH)_2$ content due to its binding into the magnesium pentahydroxychloride $5Mg(OH)_2 \cdot MgCl_2 \cdot 8H_2O$ (5 – form) and the hydrates based on slag compounds (Figure 1b). According to diffractometric analysis, a phase composition basis of magnesia-slag binder’s stone is represented by magnesium pentahydroxychloride, characteristic of caustic magnesite. Moreover, the amount of 5 – form is proportional to the share of caustic magnesite in the binder (Figure 1b). The presence of free unhydrated magnesium oxide indicates incomplete implementation of the binding properties of caustic magnesite (Figure 1c).

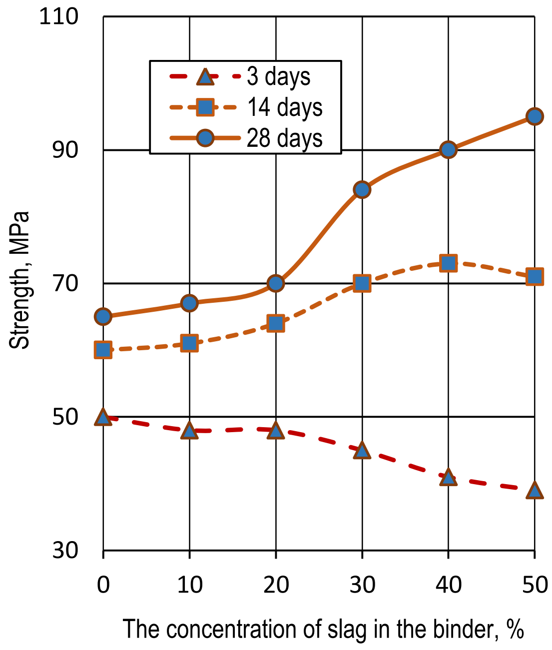


Fig. 2. – Effect of metallurgical slag content on the strength of air-cured magnesia binders

Preference for magnesium chloride solution for mixing magnesite binders is due to significant hardening rate and high strength of the hardened stone. To increase binder's resistance to water, it is recommended to use the solutions of sulfate salts [18] – [21]. However, replacing a magnesium chloride solution with magnesium sulfate or iron sulfate solutions is accompanied by a decrease in strength of caustic magnesite [18] – [21]. Possibility of using combined salt solutions consisting of magnesium chloride solution (density is 1250 kg/m^3) and magnesium sulfate (density is 1210 kg/m^3) for mixing caustic magnesite was studied (Figure 3). Samples of hardened binder aged 7 days were immersed into aggressive environments (water, magnesium chloride and sodium sulfate solutions with a density of 1050 kg/m^3). Binders were tested at the age of 28 days from the moment of mixing.

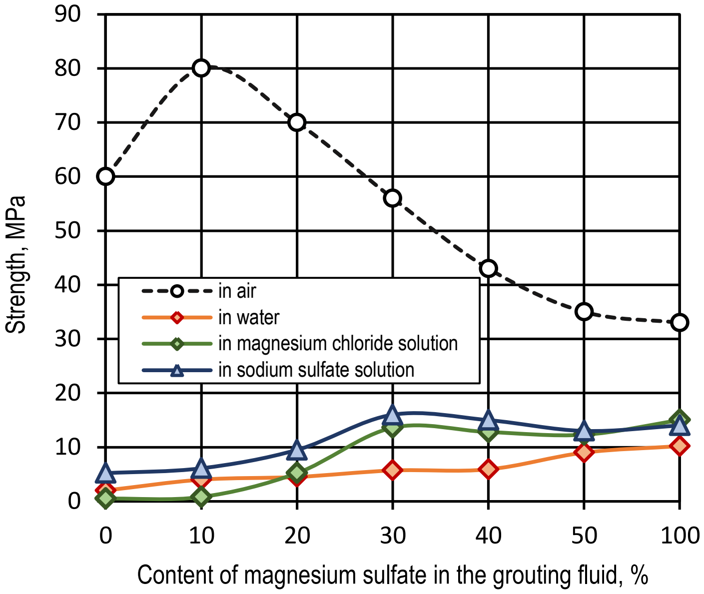


Fig. 3. – Effect of combined grouting fluid on strength of caustic magnesite under various conditions

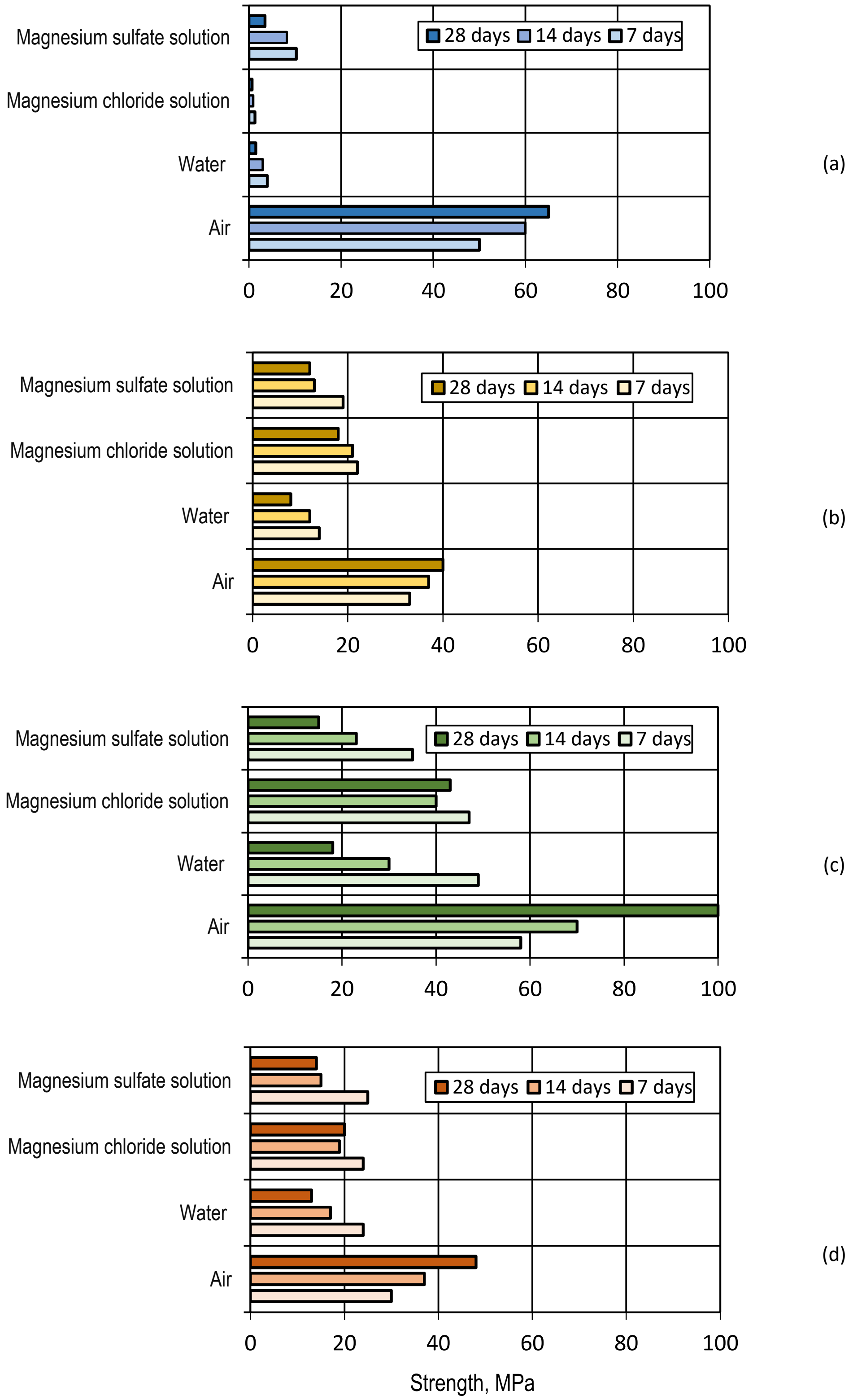
Presence of 10 – 20% magnesium sulfate in the setting solution increases the strength of the air-hardening binder by 17 – 33%, while with a content of 30 – 50% magnesium sulfate, the strength of the binder decreases by 7 – 42%. With a content of 50% or more magnesium sulfate in the setting solution, the effect of this salt on hardening of caustic magnesite dominates. Presence of samples in liquid media is accompanied by a decrease in strength compared to samples hardened in air. The greatest decrease in strength is observed for binders for which combined solutions containing 0 – 20% magnesium sulfate were used for mixing. Analysis of the test results shows that when using a combined sulfate-chloride sealer containing 20 to 40% magnesium sulfate, it is possible to achieve increased resistance to aggressive environments while ensuring the strength of the air-hardening binder comparable to the strength of caustic magnesite based on magnesium chloride.

Comparative studies of the performance properties of caustic magnesite and magnesia-slag binder containing 50% of the technogenic component were conducted. A magnesium chloride solution (density 1250 kg/m^3) and a sulfate-chloride solution containing 50% magnesium chloride (density 1250 kg/m^3) and 50% magnesium sulfate (density 1210 kg/m^3) were used to mix the binders. For easy description of binders, the following abbreviations were introduced: MX – caustic magnesite mixed with magnesium chloride solution; MC – caustic magnesite mixed with sulfate-chloride solution; SX – magnesia-slag binder mixed with magnesium chloride solution; SC – magnesia-slag binder mixed with sulfate-chloride solution. After 7 days of air hardening, binder samples were placed into the liquids of various compositions (water, magnesium chloride and magnesium sulfate solutions with a density of 1100 kg/m^3). The choice of composition of aggressive salt solutions is determined not only by t distribution of the salts in the operational environment of concrete, but also by participation of salts in the structure formation of the binders under study.

Figure 4 shows the data on the change in strength of binder samples under different conditions. The age of samples is indicated from the moment of mixing, therefore duration of the samples' stay in liquid media is 7 days less than the indicated age. To compare binders’ strength, the indicators of caustic magnesite mixed with magnesium chloride solution and hardened in air are taken as a standard. Along with the assessment of the strength indicators of binders in liquid media, it is very important to compare the values of a resistance coefficient of these substances at the final stage of testing (Figure 5).

Analysis of air-curing binders strength indices. Replacement of 50% caustic magnesite with metallurgical slag promotes an increase in the strength of magnesia-slag binder, mixed with magnesium chloride solution, by 16% in the early stages and by 54% at the age of 28 days. When using a sulfate-chloride solution, the strength indices of caustic magnesite MC and magnesia-slag binder SC are comparable, and compared to the standard, they decrease by 1.3 – 1.6 times. It is noteworthy that the strength at the early stages of hardening is more sensitive to a decrease in magnesium chloride in the salt solution. A stable increase in the strength of all binders indicates ongoing structure formation processes over time. A comparative analysis of the strength properties of air-hardening binders revealed the highest strength values for magnesia-slag binder SX, mixed with magnesium chloride solution. This is due to the favorable combination of crystalline magnesium pentahydroxychloride and weakly crystallized hydrates based on slag phases (Figure 1b).

Analysis of the influence of the aqueous environment on the strength of binders. The effect of water on all magnesia binders is accompanied by a decrease in strength. This fact is explained [29] – [30] by decomposition of magnesium pentahydroxychloride with the formation of magnesium hydroxide. The nature of change in the strength of the binders under study corresponds to known scientific concepts.



(a) – MX; (b) – MC; (c) – SX; (d) – SC

Fig. 4. – Influence of the hardening environment on strength of magnesia binders of various compositions

The most intense rates of strength decline were noted for caustic magnesite of oxychloride hardening MX, which has the highest content of unstable magnesium pentahydroxychloride. MX samples differ from samples of other binders by the presence of a crack network (Figures 6a, 6c, 6e). Cracks, along with decomposition of the structure-forming phase $5Mg(OH)_2 \cdot MgCl_2 \cdot 8H_2O$, contributed to a decrease in the strength of the MX binder.

The crack network in the structure of magnesia stone is a consequence of the formation of $Mg(OH)_2$ both due to decomposition of magnesium pentahydroxychloride and due to hydration of free magnesium oxide. Magnesia slag binder of oxychloride hardening SX is characterized by a water resistance coefficient of 0.85 after 3 days of continuous exposure to water (Figure 4c). As time passes, the water resistance coefficient decreases on SX.

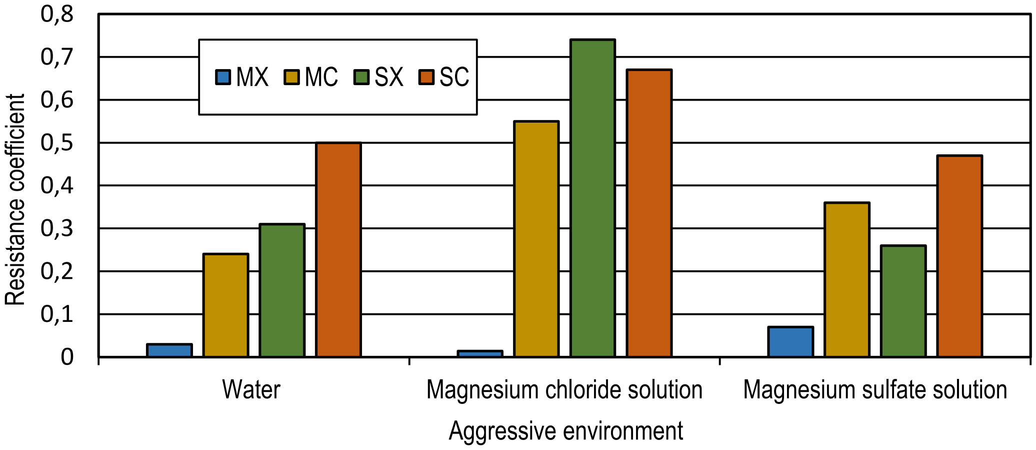
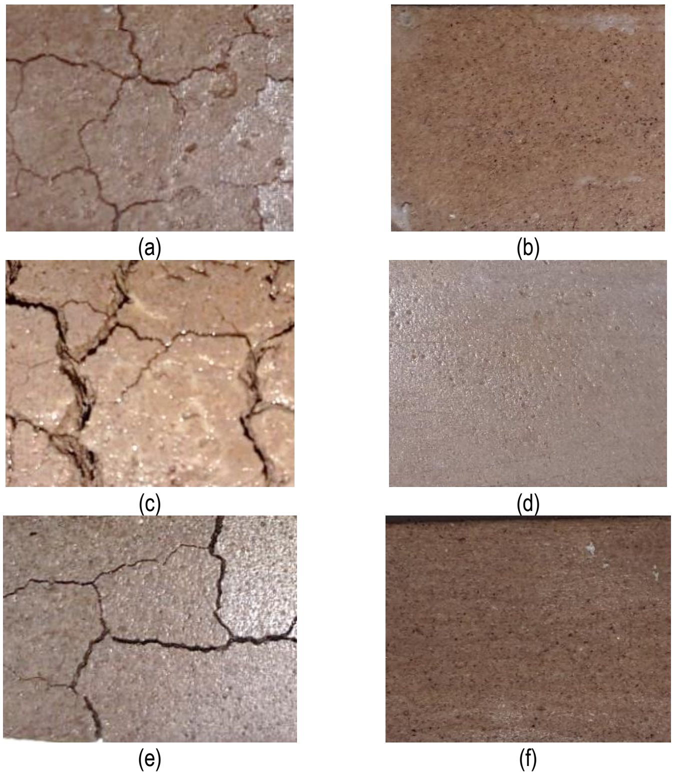


Fig. 5. – Resistance coefficient of binders after 21 days of testing in aggressive environments



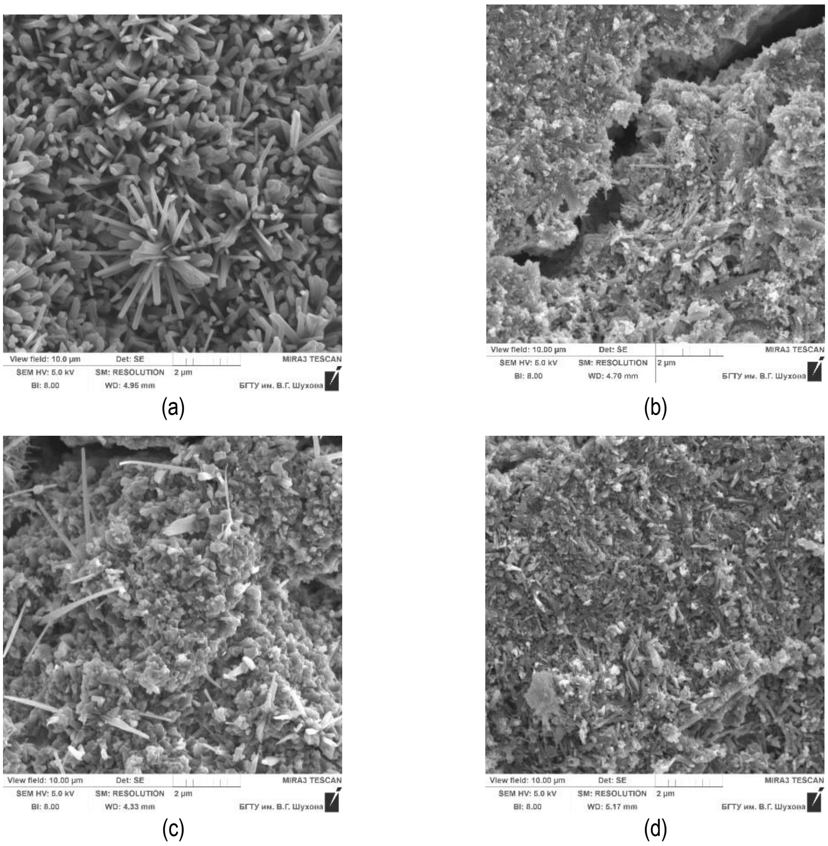
a) – MX (water); b) – SX (water); c) – MX (magnesium chloride solution); d) – SX (magnesium chloride solution); e) – MX (magnesium sulfate solution); f) – SX (magnesium sulfate solution)

Fig. 6. – Appearance of samples after exposure to aggressive environments:

Binders MC and SC, mixed with a sulfate-chloride solution, demonstrated increased water resistance compared to caustic magnesite of oxychloride hardening. After the first 3 days of exposure to water, the water resistance coefficient of binder MC was 0.42 (Figure 4b), and that of binder SC was 0.80 (Figure 4d). During subsequent testing, binders mixed with a sulfate-chloride solution demonstrated decreased sensitivity to the effect of water, as evidenced by a tendency to stabilize strength indicators.

The highest water resistance coefficient was achieved for binder SC (Figure 5). Increased water resistance of magnesia-slag binder is provided by the features of the microstructure (Figures 7c, 7d), the basis of which is made up of lamellar crystals of magnesium pentahydroxychloride formed in the environment of slag compounds that form weakly crystalline hydrates. Needle-shaped crystals of magnesium pentahydroxychloride, forming the structure of the MX binder, are less resistant to the effects of a liquid medium (Figures 7a, 7b).

Analysis of the strength indices of binders exposed to magnesium chloride solution. For oxychloride-hardened caustic magnesite MX, exposure to $MgCl_2$·solution turned out to be the most destructive both in comparison with other test conditions and in comparison with other binders (Figures 4 and 5). Cracks appeared on the MX samples, indicating an intense effect of expansive phases (Figure 6c).



(a) – MX (after 7 days of air hardening); (b) – MX (after 3 days of exposure to water);
(c) –SX (after 7 days of air hardening); (d) – SX (after 3 days of exposure to water)

Fig.7. – Microstructure of magnesite binder stone:

The dominant expansive effect is exerted by secondary magnesium pentahydroxychloride, which is formed during the interaction of an aggressive $MgCl_2$ solution with magnesium oxide and hydroxide, including that formed during the decomposition of the 5 – form. The resistance coefficient of caustic magnesite MC, mixed with a sulfate-chloride solution, equal to 0.55 – 0.67, exceeds the similar indicator of this binder in other liquid media. The integrity of the structure of the MC samples is due to the limited content of unstable magnesium pentahydroxychloride and the presence of magnesium hydroxysulphates.

The aggressive effect of the $MgCl_2$ solution on the oxychloride magnesia-slag binder SX is insignificant: the resistance coefficient at different test periods was 0.74 – 0.81. The strength values of the SX binder exposed to the $MgCl_2$ solution are comparable to or exceed the strength indicators of other binders that hardened in air (Figure 4). Under the influence of magnesium chloride solution, the hydraulic activity of latent slag compounds is activated, which contributes to the formation of an additional amount of hydrates that compact the structure of the SX stone.

Analysis of strength indices of samples exposed to magnesium sulfate solution. Compared with other liquid media, magnesium sulfate solution has a less aggressive effect on oxychloride-hardened caustic magnesite MX: the resistance coefficient at different test times is 0.07 – 0.20. Cracks in the MX stone structure are caused by magnesium hydroxide formed both during MgO hydration and during the decomposition of the 5 – form (Figure 6e). When mixing caustic magnesite with a sulfate-chloride solution, the resistance coefficient of the MC binder increases and is 0.36 – 0.61. Oxychloride magnesite-slag binder SX in a magnesium sulfate solution is characterized by a resistance coefficient of 0.26 – 0.60. For magnesia-slag binder SC, mixed with sulfate-chloride solution, the resistance at different test times is estimated by a coefficient of 0.18 – 0.68. Binder SC is characterized by the highest coefficient of resistance to the aggressive impact of magnesium sulfate solution (Figure 5).

Summarizing the results of studies, it should be noted that a significant content of magnesium pentahydroxychloride under absence or limited amount of weakly crystallized and amorphous hydrate formations increases the vulnerability of crystalline structure of magnesite stone to the effect of aggressive liquids.

Conclusions

There was studied the influence of aggressive liquid environments on durability of magnesite binders of various compositions. The study results expand understanding of causes in destructive processes and contribute to technological methods development for increasing magnesite materials’ operational durability.

It is shown that a destructive effect of water is resisted by measures that ensure minimization of unstable magnesium pentahydroxychloride while maintaining the specified strength indices of magnesite binders. This is achieved by reducing the proportion of magnesium oxide in the binder by replacing caustic magnesite with metallurgical slag, as well as by using a sulfate-chloride solution as grouting fluid.

The effect of salt solutions on the durability of magnesia binders of various material compositions was studied for the first time. It was established that an aggressive salt environment creates conditions for hydrate formation and participates in hydrate phase's formation.

Formation of a significant amount of secondary magnesium pentahydroxychloride in a stone surrounded by aggressive $MgCl_2$ solution is facilitated by the presence of $Mg(OH)_2$ in the binder. Expansion of a newly formed crystalline 5 – form destroys the binder stone.

To prevent the expansive action of secondary hydrates, combined binders, such as magnesia-slag binders are preferable. The salt environment initiates hydrate formation with the participation of latent slag compounds, which helps to maintain integrity and strength of a stone structure. Reducing the proportion of magnesium chloride in the composition of the grout also prevents the formation of dangerous quantities of secondary hydrates.

The revealed behavioral features of magnesite binders of various compositions in aggressive salt environments can serve as a basis for substantiating the area of application of magnesite materials in construction.

The research results indicate the possibility of using magnesia-slag binder in composite materials operated in conditions of high humidity and exposure to solutions of chloride and magnesium sulfate.

Caustic magnesite of hydroxychloride hardening should not be exposed to water and salt solutions.

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