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## Study of corrosion stability of a cement stone in magnesia aggressive environment

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**Abstract**. One of the main tasks in the construction of oil and gas wells is to ensure the high quality of well casing. It is especially difficult to get it in wells, in sections of which there are salt-bearing strata. From the salts, the most dangerous are magnesia salts, which can lead to the destruction of the stone based on portland cement within a few months. The report presents the results of experimental studies on the corrosion of cement stone in aggressive magnesia media. The quantitative indicators reflecting the degree of damage to the stone are taken as the thickness of the damaged layer and the coefficient of stone resistance, characterized by the ratio of the ultimate strength of stone samples for compression or bending stored in an aggressive environment to the strength of control samples at the same time of hardening. In the course of the research, the corrosion resistance of the cement stone to the magnesian corrosive environment was assessed, after 8 weeks in a medium with a constant concentration of MgCl<sub>2</sub>. In addition, the effect of MgCl<sub>2</sub> concentration on the cement stone corrosion mechanism was investigated. The use of the palygorskit additive and the reduction of water cement ratio to reduce the porosity of the cement stone and reduce the rate of corrosion damage are proposed. The kinetics and the main factors affecting the corrosion process are considered, and the x-ray structural analysis of corrosion products and unaffected cement stone is carried out.

**Keywords**: magnesia corrosion, portland cement stone, corrosion stability, bischofite, depth of leaching, palygorskite

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The expansion of the drilling zone led to the development of a significant number of deposits, in the context of which there are formations represented by solid magnesian salts or solutions of these salts that reduce the integrity of the well casing (Agzamov, Izmukhambetov, 2005; Tolkachev et al., 2010; Kravets et al., 1979) The thickness of magnesian saline deposits varies from several to a thousand meters (Technology of subsalt wells drilling..., 1989; Zhuravlev, 1972). The amount and composition of salts in the reservoirs in each individual region varies widely. Usually, saline deposits are represented by bischofite (MgCl, 6H,O), carnallite (KCl·MgCl, 6H,O), kieserite (MgSO<sub>4</sub>·H<sub>2</sub>O), and epsomite (MgSO $_4$  7H<sub>2</sub>O). The temperature of salt formations in some regions reaches 160°C, which enhances the aggressiveness of salts (Mukhin et al., 1976).

In salt deposits, brine strata with abnormally high pressures, consisting of saturated saline and containing chlorides, calcium, magnesium, sodium and potassium

© 2019 The Authors. Published by Georesursy LLC This is an open access article under the CC BY 4.0 license (https://creativecommons.org/licenses/by/4.0/) sulfates and other salts, are often found. As a rule, the presence of magnesia salts in the section creates serious complications when casing of wells, manifested in an increase in the diameter of the well, the formation of gaps between the cement sheath and the wall of the well, the plastic flow of salts, the thickening of the cement slurry, the increase in pressure on the cement casing head, the destruction of the cement stone, and cross-flow and an increase in water cut, collapsing of intermediate or production strings, premature corrosion of casing strings, etc. (Mukhin et al., 1976; Ahmadeev, Danyushevskii, 1981; Danyushevskii et al., 1987).

For casing of the intervals represented by magnesiumcontaining salts, magnesia cements are used, which are mixed with aqueous solutions of magnesia salts (Tolkachev et al., 2010; Trupak, 1956), the effectiveness of which is confirmed by practice. At the same time, aqueous solutions of magnesia salts and magnesia cements have a low pH (less than 7), and therefore casing strings require additional protection under these conditions (Tolkachev et al., 2013).

If there are separate layers and layers in the section of the well containing dissolved magnesium salts, there is no practice in applying the technology of individual cementing of these zones, and cementing is usually

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carried out over the entire open hole interval with portland cement, which is the weakest link in the well support and can be destroyed from due to magnesia corrosion.

Therefore, despite the danger of magnesia corrosion in relation to portland cement, even at low concentrations of  $Mg^{2+}$  cation, a sufficiently large number of wells under these conditions are cemented by this material.

The main aggressive component in the corrosion of cement stone in contact with magnesium salts or their aqueous solutions is magnesium cation. As a result of chemical reactions of components of an aggressive environment with cement hydration products, and primarily with calcium hydroxide located in the pore fluid, reaction products are formed that are either removed from the cement stone as a result of diffusion, or precipitate remain in its pores. The accumulation and crystallization of poorly soluble reaction products, creating internal stresses, can lead to damage to the structure of cement stone.

In the works of builders who have achieved the greatest success in the study of corrosion of concrete and cement, the processes of corrosion of portland cement are classified into several types (Kind, 1955; Moskvin et al., 1980; Babushkin, Ratinova, 1968; Rakhimbaev et al., 2012; Rakhimbaev, Tolypina, 2015; Rakhimbaev, 2012).

Corrosion of the first type is associated with the gradual hydrolysis of cement hydration products and the leaching of calcium hydroxide. A decrease in calcium hydroxide violates the stability of cement stone hardening products, leading to their subsequent hydrolysis and dissolution. In borehole conditions this type of corrosion is characteristic of injection wells.

Corrosion of the second type is different in that in the surface layers of cement stone which are in contact with the aggressive environment, there is an intensive destruction of the structural elements of hydrated cement stone. In this case, the process of destruction of the surface layers can be complete while maintaining intact stone in the adjacent layers. This type is characteristic of the interaction of cement stone with acidic environments. In borehole conditions, this is observed when dissolved hydrogen sulfide or carbon dioxide acts on the well casing (Kind, 1955; Babushkin, Ratinova, 1968; Agzamov et al., 2011).

The third type of corrosion is characterized by the fact that crystalline products accumulate in the pores and capillaries of the cement stone, which are the result of chemical reactions of an aggressive environment with the components of the cement stone. The growth and accumulation of corrosion products can lead to the development of tensile stresses on the pore walls and destroy the structural elements of the stone. One of the signs of this type of corrosion is the destruction of stone with an increase in volume, which is preceded by an accelerated, in comparison with control samples, increase in strength. In borehole conditions, this type of corrosion can be observed when sulfate condition or gaseous hydrogen sulfide acts on a cement stone (Babushkin, Ratinova, 1968; Agzamov et al., 2011).

The destruction of cement stone caused by  $MgSO_4$ , according to (Kind, 1955), at low salt concentrations in water is caused by sulfate corrosion, which is dangerous only for portland cement. At high concentrations of  $MgSO_4$ , magnesia-gypsum corrosion is observed, which is also dangerous for other types of cements. This means that with an increase in the  $Mg^{+2}$  cation in the solution, it is magnesia corrosion that prevails, the mechanism of which differs from the types of corrosion described above.

There are several opinions regarding the corrosion mechanism of portland cement in magnesia environments.

Upon contact of the cement stone with an aggressive magnesia, chemical reactions occur between the dissolved salt and calcium hydroxide located in the pores of the cement stone and near its surface according to the equation:  $Ca(OH)_2 + MgCl_2 \rightarrow Mg(OH)_2 \downarrow + CaCl_2$ .

Calcium chloride, being a readily soluble product, dissociates into ions, and the  $Ca^{2+}$  cation is carried into the environment. Chlorine ion remains in the pores of the cement stone and can diffuse further into the cement stone.

We believe that, depending on the concentration of the aggressive substance, the composition of the hardening products of cement stone, its structural characteristics during magnesia corrosion, the meeting of flows of aggressive ions with calcium hydroxide and their subsequent neutralization can occur in different places. It can be outside the cement stone, either inside the stone near its surface or at a considerable distance from its surface.

Accordingly, a different mechanism of magnesia corrosion can be observed.

A decrease in calcium hydroxide in the pores of the cement stone upsets the balance between the hardening products and dissolved  $Ca(OH)_2$ , leading to the dissolution and hydrolysis of the hardening products and leaching of new portions of calcium hydroxide. In this case, leaching corrosion is observed, leading to the destruction of cement stone due to leaching of calcium hydroxide from it. The corrosion rate will depend on the diffusion rate of calcium cations from the cement stone, i.e. on the porosity of the formed leached layer, which, in turn, depends on the initial porosity of the cement stone.

If cement stone hardening products are easily soluble, for example, highly basic calcium hydrosilicates, then their hydrolysis occurs quickly, and the zone of penetration of aggressive ions narrows. In this case, the corrosion of cement stone can take place according to the acid mechanism, i.e., the destruction of the stone proceeds in layers, and the destruction can reach full development while maintaining the cement stone in nearby intact layers without almost changing the structure and composition (Moskvin et al., 1980; Kravtsov et al., 1987). The corrosion rate in this case is determined by the diffusion of aggressive fluids, i.e. the process has diffusion control. The higher the concentration of the magnesia salt solution, the more dense the structure of the membrane formed on the surface of the stone, and the slowing effect depends on its density, strength and permeability.

Another mechanism of damage is associated with the formation of magnesium hydroxide in the pores of cement stone by replacing the calcium cation with magnesium cation. According to (Trupak, 1956), this reaction is accompanied by an increase in the volume of corrosion products, leading to volumetric destruction of the stone. The possibility of volumetric destruction of portland cement stone upon contact with a magnesia environment was also indicated in the works of V. Daniushevsky (Akhmadeev, Danyushevskii, 1981; Danyushevskii et al., 1987). He noted that osmotic pressure, due to the presence of a semi permeable shell in the surface layers of cement stone, and leading to the development of high pressures inside the stone, contributing to its destruction, is a reinforcing factor of magnesia corrosion. In his opinion, magnesium hydroxide (solubility 18.2 mg/l) can accumulate at the border of a cement stone or inside a stone near the surface, forming a semi-permeable septum, leading to osmotic effects.

According to data (Kind, 1955), cement stone corrosion proceeds more slowly in MgCl<sub>2</sub> solutions than in MgSO<sub>4</sub> solutions having a similar concentration of Mg<sup>2+</sup> ions. This is due to the effect of the clogging of the pores of the cement stone with magnesium hydroxide deposited on the surface of the cement stone and in pores adjacent to the surface. With a high porosity of the cement stone, the effect of the formation of a mud layer decreases, and the active corrosion of the stone under the influence of MgCl<sub>2</sub> begins to appear when the salt content in the solution is about 2 %, which corresponds to about 5000 mg /l of MgCl<sub>2</sub> ions (Kind, 1955).

If the flow of calcium hydroxide is insufficient due to the low rate of hydrolysis of the hardening products and the low concentration of calcium hydroxide, for example, in slag cement, and the amount of aggressive ions is large, then there is a possibility of deeper penetration of magnesium cations into the stone and the formation of poorly soluble precipitate of magnesium hydroxide at a larger thickness inside cement stone.

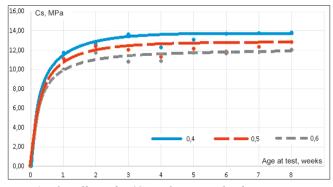
Thus, when exposed to aggressive environments containing magnesium cations, several types of corrosion can be observed. In particular, it can be corrosion of the first type associated with the leaching of calcium hydroxide from cement stone, corrosion of the second type (acid), as well as corrosion of the third type, accompanied by the accumulation of corrosion products in the pores of the cement stone.

During the experimental studies, an aqueous solution of  $MgCl_2$  (bischofite) with a concentration of 10 %, updated weekly, was used as an aggressive condition. To exclude the influence of changes in the concentration of aggressive condition, the ratio of the volume of aggressive liquid and the volume of cement stone was 10:1. The tested cement samples were prepared from portland cement slurries with a water-cement ratio (W/C) of 0.4, 0.5, 0.6, which hardened both in water and in air. Weekly, 3 samples were taken from the solution, which were tested for flexural and compression strength. The depth of corrosion was determined at the fracture of the samples, and the phase composition of the hardening and corrosion products was determined in various layers of cement stone.

The applicability of this method to assess the effect of the aggressiveness of magnesium salts on cement stone is due to the fact that during the reaction of the sample with the environment, hydrolysis and leaching of cement hardening products occur, thereby increasing the pore space and decreasing the strength of the stone. The depth of corrosion is equal to the penetration depth of magnesium ions and was measured as the boundary of the resulting white products.

The effect of W/C on the tensile strength of cement stone installed in an aggressive environment, when compressed, is shown in Fig. 1, from which it can be seen that with a decrease in the initial water content of the solution, the strength of the portland cement stone increases with time.

At the same time, the effect of hardening conditions and the composition of cement stone on the change in the strength of the stone was clearly manifested only due to the change in W/C. This is due to the fact that in the cement stone in the first weeks of hardening, hydration processes actively continue, which compete with destructive corrosion processes. Since the structural processes associated with hydration and hardening of



*Fig. 1. The effect of W/C on the strength of cement stone in MgCl, environment* 

portland cement during this period proceed faster than corrosion processes, it is not possible to single out the role of the latter during this period. This is quite obvious and does not contradict the basic provisions of hydration and hardening of cements (Danyushevskii et al., 1987; Agzamov et al., 2011; Kravtsov et al., 1987).

The decrease in W/C always reduces the porosity of the cement stone and increases its corrosion resistance, which confirms the assumption of diffuse control of the process.

At the fracture of the samples (Fig. 2), zones of different color are clearly visible. The depth of the corroded zone increases with time, and with increasing W/C the depth of the corrosion zone increases (Fig. 3, 4).

At the fracture of the samples (Fig. 2, 3), layer-bylayer destruction of the stone, characteristic of the second type of corrosion is visible. At the same time, a white loose layer about 1 mm thick formed on the surface of the cement stone, the composition of which according to x-ray phase analysis showed the predominance of brucite Mg(OH)<sub>2</sub>, which is a reaction product of MgCl<sub>2</sub> and Ca (OH)<sub>2</sub>. The middle zone of the sample (uncorroded) corresponds to the phase composition of the control sample of cement stone. Between these layers inside the stone, a damaged (corroded) layer with a partially

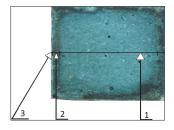


Fig. 2. Cement stone sample after 14 days in the magnesium chloride environment. 1 - non-corroded part; 2 - part of corrosion; 3 - precipitate of magnesium hydroxide on the surface of the sample.

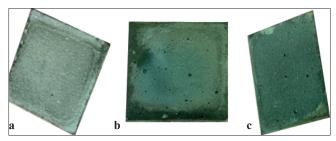
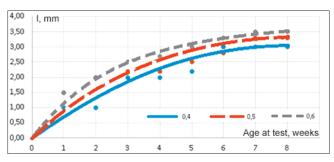


Fig. 3. Samples of cement stone after being within 28 days in an aggressive environment. a - W/C = 0.6; b - W/C = 0.5; c - W/C = 0.4.



*Fig. 4. The effect of W/C on the depth of corrosion of cement stone in MgCl, environment* 

changed phase composition is distinguished (Table 1). Quantitative analysis of the results of x-ray phase analysis (XRD) was carried out in the Topas Diffrac licensed program for the number of minerals of the initial cement ( $C_3S$  – three-calcium silicate;  $C_2S$  – two-calcium silicate;  $C_4AF$  – four-calcium aluminoferrite;  $C_3A$  – three-calcium aluminate), cement hardening products ( $Ca(OH)_2$  – portlandite;  $C_3S_3H$  – xonotlite;  $C_5S_6H_5$  – tobermorite;  $C_3S_3H$  – rosenhanite) and corrosion products (Mg(OH)<sub>2</sub>-bristite; MgO – periclase, aluminum, silicon and iron oxides).

The table shows that in the central non-corroded part of the samples there are unhydrated cement minerals and their hydration products. The presence of corrosion products is not observed.

In the middle (corroded) layer, the amount of clinker minerals decreased with a simultaneous increase in hydration products. In particular, the amount of minerals that make up the original cement decreased from 76.7 % to 27.2 %. An increase in the proportion of hardening products may indicate an increase in cement hydration processes with a decrease in pH. This effect was also noted in other works where it was proposed to obtain hardening of cement stone when mixing it, for example, with water with dissolved carbon dioxide or dissolved hydrogen sulfide. This is due to the fact that calcium hydroxide released during hydration of clinker minerals is more quickly neutralized by an acidic medium and, as a result, causes accelerated hydration of cement minerals to maintain the necessary concentration of calcium hydroxide in the slurry (Agzamov et al., 2011).

The amount of portlandite (calcium hydroxide) in these layers differs slightly. In the non-corroded layer this is due to the onset of equilibrium between the solid phase and the pore liquid, and in the corroded layer it is associated with the hydrolysis of the hardening products due to the constant binding of calcium hydroxide from the pore liquid due to the reaction with magnesium chloride.

In the surface layer of cement stone, calcium hydroxide (oxide) completely went into the slurry and bound with aggressive ions, due to which only insoluble or poorly soluble compounds remained in this layer which play a significant role in inhibiting the corrosion process. These include aluminum and magnesium hydroxide, silicon oxide and a small amount of carbonates. Aluminum hydroxide, which has a larger size than aluminum oxide, reducing pore size acts as an additional agent that reduces the permeability of the cement stone layer near the border with the aggressive environment. Magnesium hydroxide in this case acts as a semipermeable membrane and a binding agent for silicon oxide.

Analysis of the results allows us to clarify the idea of the mechanism of corrosion of cement stone in a magnesia environment. The great importance of the content of brucite in the surface sediment (Table 1) indicates a second type of corrosion, during which exchange reactions occur between the components of the cement stone and slurry, and the resulting reaction products are deposited on the cement surface and in the pores (Moskvin et al., 1980; Rakhimbaev et al., 2012; Rakhimbaev, 2012). Corrosion in this case can come with an inhibition due to the partial compaction of cement stone with corrosion products.

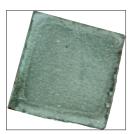
At the same time, no signs of volumetric destruction due to the accumulation of corrosion products inside the cement stone were noted, which may indicate a reaction between calcium hydroxide and magnesium chloride at the surface of the cement stone. This may be evidenced by a small amount of corrosion products in the middle (damaged) layer.

The experiments confirmed that the factors determining the kinetics of the corrosion process are the diffusion rate, the porosity of the stone and the compaction of the cement stone with reaction products.

To reduce the porosity of the stone, an addition of salt-resistant clay – palygorskite in an amount of 3 % was used. In this case, the W/C of the slurry was kept equal to 0.5. The experiments showed (Fig. 5) that the corrosion is layer-by-layer, but the boundaries of the layers are less noticeable, and the corrosion process slows down.

Zone	Formula	Component name	%
Not corroded	C <sub>3</sub> S	monoclinic (NISHI)	47,21
	$C_2S$ (total $\alpha$ , $\beta$ , $\gamma$ )	dicalcium silicate	14,59
	C <sub>4</sub> AF	tetracalcium alumino ferrite	12,67
	C <sub>3</sub> A cubic	tricalcium aluminate	2,22
	Ca(OH) <sub>2</sub>	portlandite	2,18
	C <sub>3</sub> S <sub>3</sub> H	ksonolite	7,66
	C <sub>5</sub> S <sub>6</sub> H <sub>5</sub>	тоbermorite	7,4
	MgCO <sub>3</sub>	маgnesite	6,02
Corroded	C <sub>3</sub> S	monoclinic (NISHI)	7,17
	$C_2S$ (total $\alpha, \beta, \gamma$ )	dicalcium silicate	11,46
	C <sub>4</sub> AF	tetracalcium alumino ferrite	7,32
	C <sub>3</sub> A cubic	tricalcium aluminate	1,2
	C <sub>12</sub> A <sub>7</sub>	мауепіtе	2,69
	Ca(OH) <sub>2</sub>	portlandite	2,01
	C <sub>5</sub> S <sub>6</sub> H <sub>5</sub>	тоbermorite	19,54
	C <sub>3</sub> S <sub>3</sub> H	rosenhahnite	17,75
	C <sub>6</sub> S <sub>3</sub> H	gamma Dellaite	12,65
	$(Mg,Fe)_2Al_4Si_5O_{18}$	cordierite	9,49
	C <sub>2</sub> AS	gehlenite	4,57
	SiO <sub>2</sub>	quartz	0,44
	MgCO <sub>3</sub>	magnesite	3,05
	CaCO <sub>3</sub>	vaterite	1,1
Precipitate	$C_3S$	monoclinic (NISHI)	1,24
	C3A cubic	tricalcium aluminate	0,29
	C <sub>4</sub> AF	tetracalcium alumino ferrite	0,84
	C <sub>3</sub> ACs <sub>3</sub> H <sub>31</sub>	ettringite	1,71
	Mg(OH) <sub>2</sub>	brucite	54,70
	SiO <sub>2</sub>	silica LeBail	29,01
	Al(OH) <sub>3</sub>	nordstrandite	8,63
	CaCO <sub>3</sub>	calcite	3,58
	MgO	periclase	0,19

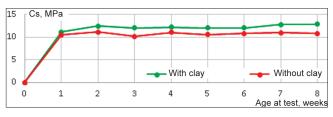
Table 1. The results of quantitative x-ray phase analysis



*Fig. 5. Corrosion of portland cement stone with the addition of palygorskite* 

A layer of brucite  $Mg(OH)_2$  with a thickness of less than 1 mm was formed on the surface of the cement stone.

The results of determining the tensile strength of portland cement stone and the depth of corrosion of the samples are presented on the Fig. 6 and 7. With the same porosity, the corrosion rate of the stone obtained from cement with added palygorskite is 17 % lower compared to control samples. We believe that this is due to a slowdown in the diffusion of aggressive ions into the stone due to the clogging of its pores with swollen, salt-resistant clay.



*Fig. 6. The effect of the addition of palygorskite on the strength of portland cement stone in MgCl*<sub>2</sub> *environment* 

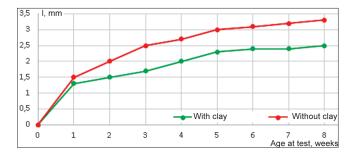


Fig. 7. The effect of palygorskite additives on the depth of corrosion of portland cement stone in MgCl, environment

A study of the behavior of cement stone in environments with the presence of magnesium chloride concentrations showed that a concentration of 10 % is most interesting for considering corrosion processes, since the corrosion occurs on the boundary line between cement stone and cement slurry, and in this case we can observe the second type of corrosion.

When the concentration of the aggressive environment is more than 10%, in the experiment with 20 %, the corrosion front moves towards the stone and the corrosion passes to the third type and, in addition, MgCl<sub>2</sub> begins to crystallize on the surface of the samples (Fig. 8).



Fig. 8. Crystal formation of magnesium chloride on the stone surface

At concentrations of aggressive medium less than 10 %, the corrosion front extends beyond the cement stone, and corrosion occurs only due to the leaching of  $Ca(OH)_{2}$ .

## Conclusion

This scientific work shows the relationship of the kinetics of corrosion damage to Portland cement stone with its structural properties, which makes it possible to control the corrosion rate by reducing the water-cement ratio of the cement slurry.

The possibility of reducing the rate of corrosion of cement stone in a magnesia environment with the addition of palygorskite which acts as a pore colmatant, is shown.

Theoretically substantiated and experimentally shown a change in the mechanism of damage to cement stone in magnesia environment depending on the concentration of Mg cation.

Corrosion damage to the cement stone has a layered character, from an intact sample to a corroded layer which differ in phase composition, and the kinetics of damage is characterized by a slowdown in the corrosion rate.

It was shown in this article that on the open surface of the stone from Portland cement at high concentrations of  $MgCl_2$  crystallization occurs with the formation of a layer that reduces the corrosion rate.

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