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CURING OF MAGNESIUM COMPOSITIONS

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ABSTRACT

The results of investigations of magnesia compositions containing various additives. Factors which impact activity of magnesium oxide in compositions of different structure are investigated. The effect of semi-aquatic calcium sulfate on a hardening of the mixed magnesia binder. Defined effect of concentration calcium sulfate hemihydrate to the hardening of caustic magnesite. Here are proposed structures of sulphomagnesium compositions containing techno genetic components. It revealed a beneficial effect on the hardening of ferrous component of the mixed magnesia binder. Influence of the method of preparation on properties of magnesium compositions of iron ore wastes is investigated during study.

KEYWORDS: Caustic Magnesite, Magnesium Compositions, Hydration, Hardening

INTRODUCTION

Production of cement is large user of material and energetic recourses. It requires development mixed cement less binding substances. Technology of mixed binders assumes the maximum involvement in the production of industrial wastes.

Magnesium binders reveal activate capacity in relation to numerous number of materials. This became background for getting mixed binders from caustic magnesite and mineral component [1 – 7]. The combination of caustic magnesite of natural and techno genetic materials expands the range and increased the volume of magnesia binders. Magnesium compositions characterized by low energy intensity of production, intensive hardening and high strength. Containing of magnesium component in the composition of mixed binders is 50 – 70 %. Recourses of magnesium rocks are limited. It is necessary to reduce deficit caustic magnesite in the composition with mixed binders. The advantages of mixed binders are to improve the physical and mechanical properties while saving caustic magnesite and rational use of natural and technogenic raw materials.

Purpose of the Work: Is synthesis and investigation magnesium compositions of different structures.

For achieving purpose were identified following objectives:

1. Investigation hydration activity of magnesium oxide in magnesium compositions;
2. Development structure of sulphomagnesium compositions;
3. Research of mixed magnesium binder with various ferriferous components;
4. Study the effect of preparation method on the properties of magnesia-based composition.

Objective of research – to study of the influence of technological factors on the hydration and hardening of magnesia compositions

METHODOLOGY

For experiments were used: caustic magnesite mark PMK – 75, building plaster mark G – 5AII, technogenic materials (wastes of concentration magnetite ore, cullet, concrete crow, limestone and silica sand sifting).

Binding compositions were prepared by thoroughly mixing fine components. Strength properties were determined on samples with size 20x20x20 mm, made with dough of normal density. Phase composition of hardened materials evaluated by X – ray method of analysis. Stone structure of the materials was investigated by the means of electronic microscopy.

RESULTS AND DISCUSSIONS

Activity of Magnesium Oxide in Magnesium Compositions

Structure of magnesium binders is unique by presence of magnesium oxide, which in comparison with calcium oxide is slightly soluble and slowly reacts with water. This is [8] due to skinning effect $Mg(OH)_2$, which is prevent from water penetration into deep into the grain binder. Hardenings of magnesia materials, mixing with water, do not have much practical value.

With the introduction of salt (chloride or magnesium sulfate), into hardener significantly increased solubility of magnesium oxide, increases degree of super saturation and accelerates the crystallization of brucite, forms hydro oxide salt.

Magnesium binders, mixing a solution of magnesium chloride hardens rapidly, are highly durable. Caustic magnesite, coupled with a solution of magnesium chloride activates siliceous, aluminosilicate and other substances. This capability is implemented in the magnesium binder compositions using natural and anthropogenic materials. Activity of MgO in compositions depends on many factors. The nature of these factors requires clarification.

Increasing density of solution $MgCl_2$ in the limits 1150 – 1400 kg/m^3 for caustic magnesite (Figure 1) limits or fully exclude formation of $Mg(OH)_2$; provides growth of containing magnesium pentahydrate oxychloride $5Mg(OH)_2 \cdot MgCl_2 \cdot 8H_2O$ («5 – form»), which is prevail in the composition of crystalline hydrate using solution with density 1250 –1400 kg/m^3.

A large proportion of the «5 – form» is formed in the early stages, providing a high rate of hardening stone. In the structure of binder

As part of a binder, mixing solutions of high density (1350 and 1400 kg/m^3), identified hydrated complex $3Mg(OH)_2 \cdot MgCl_2 \cdot 8H_2O$ («3 – form») – magnesium trihydrate oxychloride.

Hydrate «3 – form» is formed by recrystallization of a small part of «5 – form». There is no clear relationship between the concentration of $MgCl_2$ solution, and the degree of hydration of MgO. This indicates the dependence of activity MgO from content and structure of hydrates.

Along with salt solutions, magnesium binders are recommended to temper with acids solution HCl [7, 8]. To obtain comparative characteristics was used $MgCl_2$ solution with density of 1250 kg/m^3 and complex solution (solution of MgCl : HCl : H_2O – 2:1:1, volumetric parts).

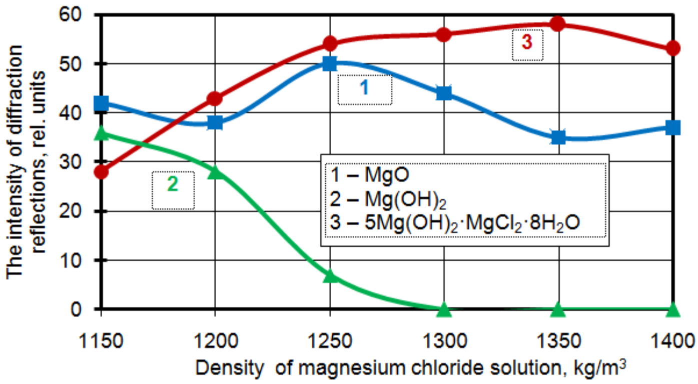


Figure 1: Influence of Sealer Density on Phase Composition and Strength of Caustic Magnesite Stone (7 Days)

When tempering caustic magnesite with complex solution hydration level of MgO is increased on 6%. In the cured binder is formed Mg (OH) $_2$. A content of magnium pentahydrate oxychloride reduces on 28%, which is lead to decreasing strength of the stone. Staying in the water samples twice reduces the proportion of the initial MgO; provides a significant increase quantity of Mg $(OH)_2$. There is a part of hydrate 5Mg $(OH)_2 \cdot MgCl_2 \cdot 8H_2O$ in the water. According to the X – ray analysis, in the stone, which was hardened 28 days, save up to 30% uncombined MgO.

Hardening sulfomagnesium binders is occur in initial hydration of magnesium oxide, which activity rises with increasing portion of building plaster (Table 1). The highest degree of hydration of MgO is typical for the first 7 days, later reactive ability of phase reduced. Effect of temper concentration on the activity of MgO sulfo magnesium binders is inexpressively. In the structure of new formations is prevail 5Mg (OH) $_2 \cdot MgCl_2 \cdot 8H_2O$. In the low magnesia binders id formed also magnesium hydrate oxychloride carbonate Mg (OH) $_2 \cdot MgCl_2 \cdot 2MgCO_3 \cdot 6H_2O$. Intensive formation of magnesium hydrate complexes provides high strong characteristics of sulfomagnesium binder.

The tendency of growth activity of MgO and increasing the degree of its transformation into hydrate oxychloride complexes safes when added to the caustic magnesite mineral component. Character of hydrate formation with participation MgO also depends on the composition of the mineral component. These approve results of investigations binders of caustic magnesite (35%) and minerals – silicates (65%), tempered with solution of magnesium chloride with density 1220 kg/m^3 (Table 2). The highest degree of conversion of magnesium oxide is characteristic for compositions containing silicates insular structure (andradite and epidote), which are more prone to hydrolytic decomposition in a solution of magnesium chloride.

Another indication of the influence of material composition on hydration activity of magnesium oxide and the composition of hydrates is the nature of phases changes in mixtures MgO with Al_2O_3 and SiO_2 (additive content 45%). Ratio of diffraction intensity reflections of magnesium oxide for binders consisting of MgO, «MgO + Al_2O_3», «MgO + SiO_2», , respectively, equal to 1 : 0,5 : 0,3;, the diffraction reflections of magnesium pentahydrate oxychloride – 1 : 3,6 : 4,3.

Table 1: Containing Phases in the Hardened Sulfomagnesium Binder

Content Semi-Aquatic Calcium Sulfate, %	Density of Solution $MgCl_2$, kg/m^3	Age, Days	The Intensity of the Reflection Phase in the Diffraction Pattern, Relation of Units	
			MgO (0,148 nm)	$5Mg(OH)_2 \cdot MgCl_2 \cdot 8H_2O$ (0,196 nm)
0	1240	28	100	100
20			94	140
40			92	225
60			83	250
40	1240	1	102	190
40		7	96	210
40		28	92	225
40		90	90	185
40	1200	7	98	165
40	1240		94	210
40	1280		97	260

Table 2: Influence of Minerals Structure on Phase Content in Hardened Mixed Magnesium Binders

Mineral	The Intensity of the Diffraction Reflections of Phases, Rel. Units					
	MgO (0,148 nm)		$Mg(OH)_2$ (0,157 nm)		$5Mg(OH)_2 \cdot MgCl_2 \cdot 8H_2O$ (0,196 nm)	
	7 Days	80 Days	7 Days	80 Days	7 Days	80 Days
No	100	87	100	93	100	96
Andradite	43	35	29	30	93	100
Epidote	53	44	33	33	116	114
Albite	66	47	no	no	139	161
Diopside	64	52	23	25	91	99

Important indicator of phase activity is hydration with prolong hardening. Here are investigated mixed binders, tempered with solution $MgCl_2$ with density 1250 kg/m^3 and which were hardened in various mediums during 5 years. By the data of X – ray analysis, stone crystal foundation of air hardening are form hydrate oxychloride complexes, formed on initial stages of hydration. Presence of 20 % free MgO indicates on not full realization binding properties of component. This can serve as recourse for further hydro formation. In the stone of water hardening saves up to 5 – 10 % of initial MgO; contains Mg $(OH)_2$, formed by the decay of a significant amount of hydrates.

Replacement of magnesium chloride by complex temper (solution $MgCl_2$: HCl: H_2O – 2:1:1, volumetric parts) for the mixed binder is accompanied by a slight slowing of the hydration MgO in the initial period of hardening; formation of Mg $(OH)_2$ in the stone of binder; formation hydrate oxychloride complexes in amounts comparable with the content of these phases in the composition, tempered with $MgCl_2$ solution. Staying samples of hardened binder in water accelerates the hydration of MgO and helps to complete the process to 28 days of hardening. As part of the stone increases number of Mg $(OH)_2$ and reduced the proportion of $5Mg(OH)_2 \cdot MgCl_2 \cdot 8H_2O$.

With complete elimination of magnesium chloride from the structure of temper and use of hydrochloric acid solution with density 1050 kg/m^3 hydration activity of magnesium oxide is changed slightly. In this case MgO in the mixed binders also differs by high speed of hydration. Strength values of samples based on HCl solution are reduced nearly threefold compared with salt temper. However, staying samples in water within 60 days provided the increase of strength on 40% compared with similar samples of air hardening. Hardening of the stone in the water promoted complete hydration and participation of MgO in the formation of hydro magnesium. This confirms the reduction intensity of the diffraction reflections of minerals and invariability of reflexes Mg $(OH)_2$ on diffraction patterns of binder with various degrees of hydration of MgO.

Therefore, hydration activity of magnesium oxide of magnesium binders in varying degrees depends on the temper composition, conditions of hardening, types and content of mineral addition.

Investigation of Sulfomagnesium Compositions

The advantage of adding calcium sulfate to magnesia binder is mentioned in work [9]. For optimization the concentration of gypsum component investigated series of compositions «caustic magnesite – semi-aquatic calcium sulfate», tempered by solution of $MgCl_2$.

With the introduction semi-aquatic calcium sulfate reduced water demand, prolonged time of setting binder. Replacement to 60% of caustic magnesite semi-calcium sulfate does not reduce the strength of the compositions, which in some cases exceed the benchmark on 25% (Figure 2). Hardening of sulfomagnesium binder takes place at the initial hydration of MgO. Activity MgO rises with increasing of gypsum component. In the structure of hydrates dominated 5Mg $(OH)_2 \cdot MgCl_2 \cdot 8H_2O$. In low magnesium binders also formed Mg $(OH)_2 \cdot MgCl_2 \cdot 2MgCO_3 \cdot 6H_2O$.

High values of hydration activity and strength of sulfomagnesium binder let use it as an activator of hardening. Were investigated compositions based on sulfomagnesium binder and anthropogenic materials in the component ratio 50: 50%. As temper were used magnesium chloride and sulphate solutions with density 1250 kg/m^3. Caustic magnesite was a control composition.

The introduction of technogenic component reduces the volume of solution on 32 – 37% compared with the caustic magnesite. The test results (Figure 3) confirm the possibility of synthesis of ternary binders: the strength characteristics of majority compositions are comparable to caustic magnesite indicators.

The greatest influence on the hardening mixed binders has composition of salt solution. The use of $MgCl_2$ solution provides increased hydration activity MgO, contributes to the intense stone hardening and increases its strength on 12 – 70%.

The high strength characteristics of three-component binders – is the result of a joint magnesium – chloride and sulfate activation of hardening anthropogenic material. Activity of anthropogenic component depends on the structure and chemical properties of minerals.

Gypsum and magnesium binders harden only in the air. Strength of material decreases when long storage in damp conditions and after immersion in water.

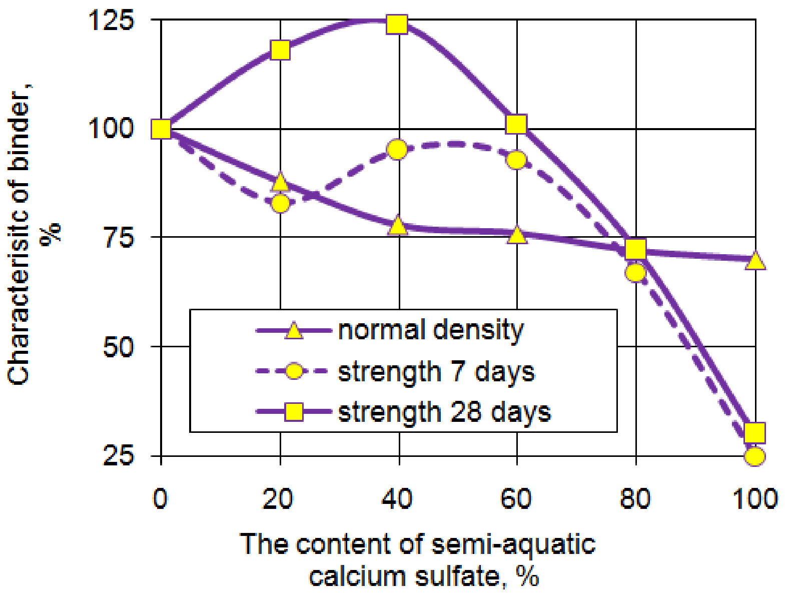


Figure 2: Influence of Semiaquatic Calcium Sulfate on Properties Sulfomagnesium Composition

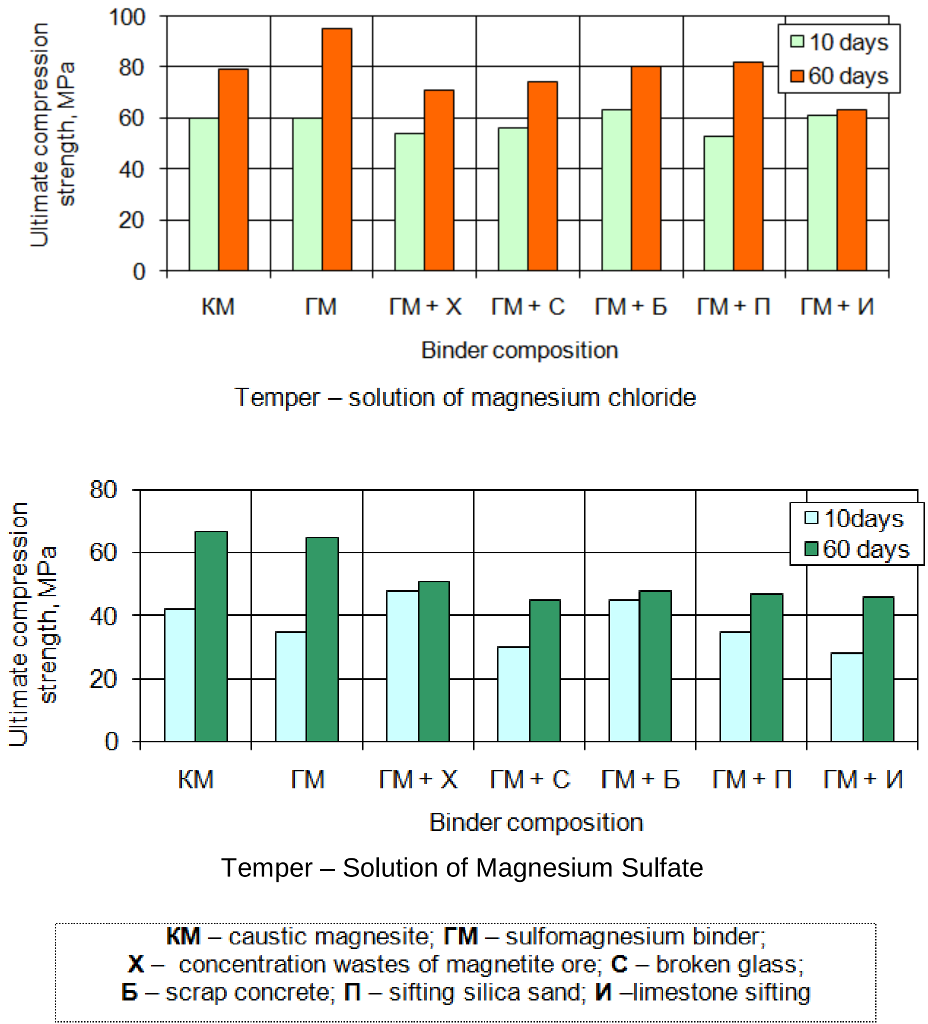


Figure 3: Influence of Material Composition on the Strength Characteristics of Binders

Investigated influence conditions of hardening on the stone strength of binders with different compositions. For this experiment used methods: after preliminary hardening in the air for 3 days, the samples were divided into three groups, which were placed in different conditions (water, air – humid environment with humidity 80 – 85%; air). The samples were stored in the selected media for different times (2 – 60 days) to study characteristic of changes in the composition and structure of stone. Material testing was performed in the age of 10, 28 and 60 days. Was measured resistance coefficient - the ratio of stone strength after being in water (air – humid) environment to the stone strength of air hardening.

Analysis of results revealed increased resistance of sulfomagnesium binder to the action of environment with high humidity. Caustic magnesite samples cracked after 7 days of storage in water. Samples sulfomagnesium compositions saved structural integrity.

It is noteworthy that tempering by solution of $MgSO_4$ promoted increasing stability of caustic magnesite and stone sulfomagnesium binder to the impact of a wet environment. All binders based on magnesium chloride showed stability of strength in the air – humid environment during test period. The highest strength values of most three-component binders subjected to the action of air – humid environment, marked in tempering $MgCl_2$. Slow hydration of $CaSO_4 \cdot 0{,}5H_2O$ is due to increased concentration of MgO and $MgCl_2$. This provides a smooth character of crystallization of $CaSO_4 \cdot 2H_2O$ and promotes compaction and increasing durability of the stone. Three-hardening binders of oxychloride hardening dramatically reduce the strength to 28 days being in the water. Subsequently, these compositions strengthen the structure in terms of high humidity. This indicates the involvement of anthropogenic component, activated magnesium chloride, in the formation of additional hydrates. Improved durability of sulfomagnesium compositions is specified by sealing stone structure as a result of the continued hydration and strengthening the cohesion of hydrates. As part of the hardened sulfomagnesium binders fixed phases, typical for hydration of each component. It is possible formation of hydrates in the form of double or triple salts. The greatest role in strengthening and moisture resistance of sulfomagnesium stone belongs to the processes of structure formation.

Hardening of Magnesia Compositions Containing Ferrous Component

The range of the magnesia cement extends at the expense of compositions from the caustic magnesite and filler. Preference of ferriferous by-product additives in the magnesia cement is noted [7, 10].

For receiving the binding materials used the caustic magnesite, chemical reactants, and natural minerals. Binding materials added water to a mix of magnesium chloride. Phase structure of the hardened compositions was estimated by X – ray and differential thermal methods. Stone structure of the binding materials was investigated by the means of electronic microscopy.

Nature of curing of binding materials depends on the maintenance of MgO (Figure 4). The analysis of diffraction patterns revealed that with increase in a share of Fe_2O_3 MgO hydration is accelerated. In comparison with binding materials, which containing SiO_2 and Al_2O_3, in composition the Fe_2O_3 hyperactivity of MgO. The extent of transformation of Fe_2O_3 in hydrates 5 – 18%. Basis of the hardened binding materials forms $5Mg(OH)_2 \cdot MgCl_2 \cdot 8H_2O$.

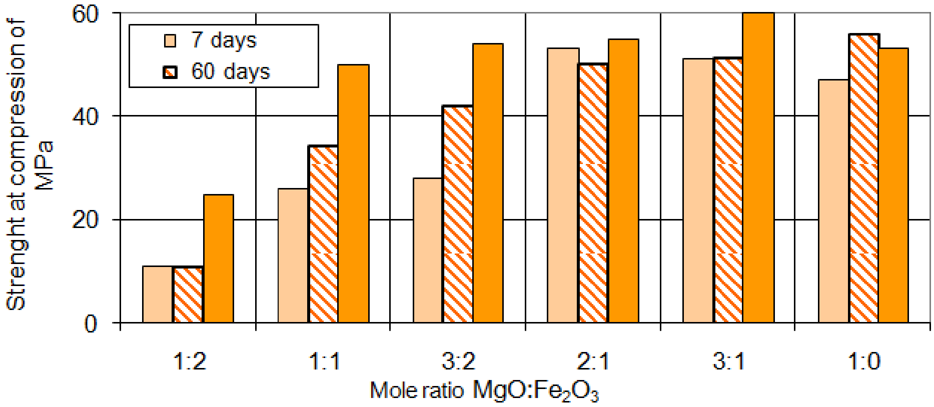
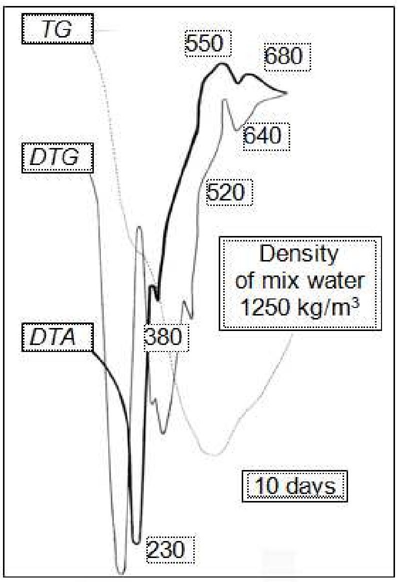


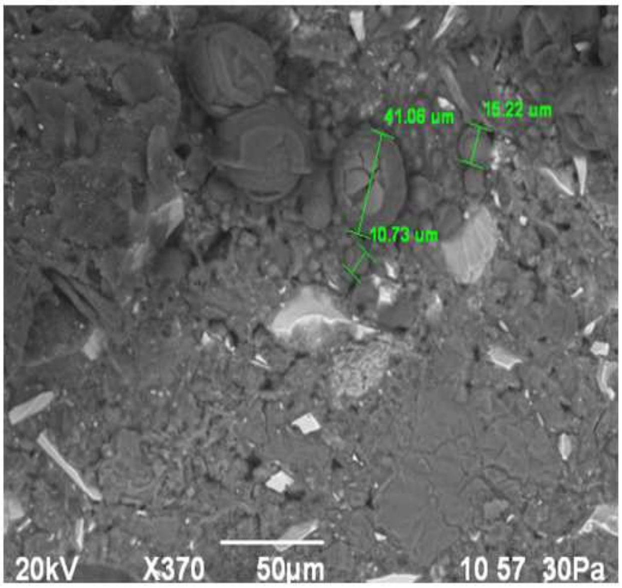
Figure 4: Influence of the Content of Ferric Oxide on Curing of the Binding Materials

Pyrites Fe [S_2] – the widespread sulfide of crust. Possibility of introduction 70% of pyrites in magnesium knitting [7] is proved. As a part crystalline hydrates prevails $5Mg(OH)_2 \cdot MgCl_2 \cdot 8H_2O$. 40 – 60% of pyrites are involved in hydrate formation of the binding materials. Processes of hydrolysis and the oxidations of pyrites accelerated in $MgCl_2$ solution are presented by the scheme:

$$(Fe^{2+} + S_2^{2-}) + 3(H^+ + OH^-) + 4O_2 \longrightarrow Fe(OH)_3 + 2(SO_4^{2-}) + 3H^+. \quad (1)$$

Exothermic effect at 550°C characterizes oxidation of pyrites (Figure 5). Pentahydroxichloride of magnesium remains in composition of long-term curing. The microconglomerate structure of a stone from particles of various morphology promotes consolidation and increase of stability of structure to destructive processes.





a) Thermo Gram **b) Stone Microstructure After 10 Years Curing**

Figure 5: Characteristics of Magnesium Composition Stone With 70% of Pyrites

Andradite Ca_3Fe_2 [SiO_4]$_3$ – the widespread scans mineral, provides stable curing of the binding materials. Compositions from 30 – 60% of andradite are investigated. The main hydrate phase is $5Mg(OH)_2 \cdot MgCl_2 \cdot 8H_2O$.

General Scheme of Probable Transformation of Andradite

$$Ca_3Fe_2(SiO_4)_3 + nMg^{2+} + (4x + 1)\ H_2O \longrightarrow$$

$$\longrightarrow Ca_3Fe_2(SiO_4)_{3-x}(OH)_{4x} + nMgO \cdot xSiO_2 \cdot H_2O. \quad (2)$$

Hydrate of Mg $(OH)_2 \cdot MgCl_2 \cdot 2MgCO_3 \cdot 6H_2O$ is formed after 1 days of curing. Intensive formation of hydro-oxchloride carbonate magnesium is promoted the increase in a ratio of $MgCl_2$: MgO in the binding materials. The extent of andradite transformation in the hydrates, defined by X – ray method, was made 20 – 23% for 7 days.

Effect of Preparation Method on the Properties of Magnesia Composition of Iron Ore Wastes

Multicomponent structure of investigated materials includes various options for combining the components in the preparation of molding compounds, characterized by a sequence of contacts.

The object of research – the composition of the mixed magnesia binder and granular ore waste. As part of the mixed magnesia astringent 30% is caustic magnesite and 70% is from fine iron ore wastes. The influence of the method of preparation was investigated at various levels: the formation of the microstructure of the mixed binder and the formation of a macrostructure composition with granular ore wastes.

In the first stage were studied three methods of preparation mixed magnesia binder:

1. Mixing a mixture of components (traditional technology);
2. Mixing caustic magnesite with magnesium chloride solution and subsequent addition of a mineral component;
3. Mixing the mineral component with magnesium chloride solution and subsequent addition of magnesite.

Influence of the preparation on the properties of the test results confirm the compositions of binder (Table 3). Increase strength characteristics of the binder is achieved by pre-treatment of wastes a mixing conducive hydrolysis minerals. Primary contact of caustic magnesite with a large volume contributes to a mixing transformation of the MgO and $MgCl_2$ in magnesium pentagidrooksihlorid. As a result of weakening magnesia component activating effect on the mineral component. Proof of this can serve as the data on the effect of partial treatment of caustic magnesite. A limited number of magnesite (15% of total consumption), introduced in the preparation of salt solution without compromising the rheological properties of a mixing enrich it with magnesium ions.

Table 3: Effect of Preparation Method on the Strength of the Binder

Way	Compressive Strength with Compression in %, Aged, Days				
	1	3	7	14	28
I	75	82	92	95	100
II	73	85	91	93	98
III	87	92	97	103	110

Granular Component: The inert component of concrete mixtures, but the role of primary contact of this ingredient in the preparation of molding compounds is often quite substantial. This is due to the influence of the nature of the granular preparation of concrete mixes on the formation and the state contact layer between the aggregate particles and the binder stone. Structural features of the contact layer determine the strength properties of composite materials and affect the durability of items.

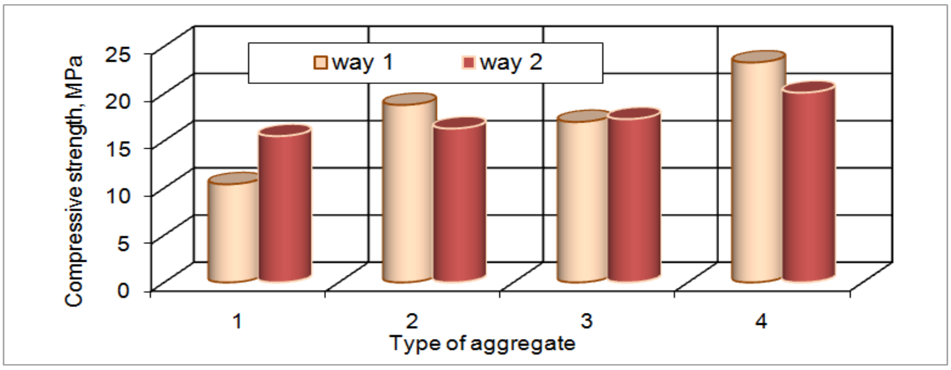
In the second phase investigated the following methods for the preparation of magnesium-based compositions of granular aggregates:

1. Joint mixing all components of the mixture;
2. Initial contact with a mixing aggregate for 2 to 3 minutes, stirring and the subsequent introduction of the mixed binder.

Comparative characteristics of the properties of granular compositions obtained in different ways (Figure 6) shows a tendency sealing and hardening of the composition while maintaining the primary contact of the particles ore wastes with a solution of magnesium chloride. This is confirmed by the results of studies granular compositions by electron microscopy (Figure 7), showing a decrease of defects in the contact zones in the microstructure of the composites, increasing the proportion of crystalline hydrates in the border area.

The role of primary contact of the granular component with a solution of magnesium chloride is determined to the process the surface of the particles of crushed material, and to free it from dust fractions, preventing contact with the stone binder. When crushed rock and ore wastes, along with the desired fraction by fine dust-like particles content in the concrete mix are trying to limit.

To confirm this hypothesis, was prepared concrete mixture on technogenic aggregate of initial state and on the basis of ore wastes, washed from dust particles. Washing were subjected to separate fractions of waste ore dressing. Separation of dust particles from the aggregate grains can improve the strength of concrete (Table 4).



1– Fraction «0, 63 – 0,315» mm; **2** – fraction «1, 25 – 0, 63» mm;
3 – Fraction «2, 5–0,315» mm; **4** – fraction «2, 5 – 0, 63» mm

Figure 6: Effect of Preparation Method on the Properties of Granular Compositions

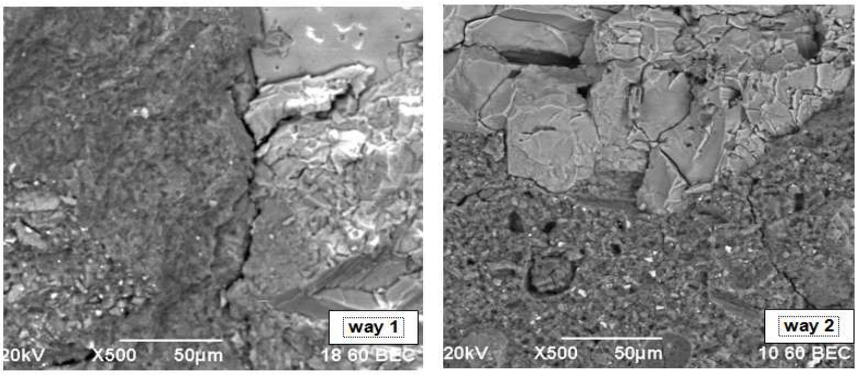


Figure 7: The Structure of the Composition by Different Preparation Method

Table 4: The Effect of Treatment on the Strength of the Filler Composition

Aggregate State	Containing Fraction (mm), %					The Proportion of Magnesium Chloride Solution	Tensile Strength, MPa, Aged, Days	
	0,14 – 0,315	0,315 – 0,63	0,63 – 1,25	1,25 – 2,5	2,5 – 5,0		3	28
Washed	–	–	–	–	100	0,33	13,5	37,3
Original	–	–	–	–	100	0,33	11,3	34,7
Washed	50	50	–	–	–	0,60	3,2	10,4
Original	50	50	–	–	–	0,64	1,5	6,5
Washed	–	–	50	50	–	0,40	7,6	21,5
Original	–	–	50	50	–	0,40	7,8	21,2

The results show that with decreasing grain size fraction increases the proportion of fines. This can be explained by the fact that smaller grains have greater force of attraction of the surface, so these grains most densely covered with fines. For a fraction of «0,140 – 0,315» mm mass loss account for nearly half, it may be due to the very small grain size fractions: small grain fractions can be carried out with water with the dust. In addition, during the classification in this fraction could concentrate a large amount of dust particles. The nature of the influence on aggregate treatment depends on the proportion of fines.

CONCLUSIONS

Maximum speed of hydration magnesium oxide is typical for the initial period. In the stone of long hardening remains unconnected magnesium oxide, which participation in the hydrate is able to provide hardening of the material structure. Staying binder stone in water environment is foster to more complete hydration of magnesium oxide.

Most activity of magnesium oxide is obtained in the mixed binders. In magnesium compositions formed a significant amount of hydrate oxychloride complexes, formed hydrates based on other components.

In the article was established activating influence of sulfomagnesium binder on the hardening anthropogenic materials of different composition.

It is revealed that intensive hardening in air and air-humidity environment, preferably-tempering the mixed binders with solution of magnesium chloride. To increase the resistance of stone to long-term action of water should be used as temper solution of magnesium sulfate.

Efficiency of sulfo magnesium compositions determined by the possibility of replacing part of caustic magnesite by plasters binder and technogenic component, with saving high technical characteristics of material.

Reactionary ability of a ferriferous component provides high activity of magnesium composition of oxychloride curing, variety of crystal and gel hydrates.

Pretreatment of technogenic component can improve the properties of magnesia compositions.

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