



Research article

UDC 666.962

DOI: 10.34910/MCE.113.11



Magnesia cement in the MgO-CO₂-H₂O system

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Keywords: magnesium oxide, water resistance, thermoanalysis, carbonation, microstructure

Abstract. A detailed study of the MgO-CO₂-H₂O system allows creating new inorganic binders. The present research is focused on the process of structure formation during hydration and curing of magnesia composition from active magnesium oxide and magnesium bicarbonate solution. Magnesium hydroxide and magnesium hydrocarbonates, basically hydromagnesite and dypingite are the products of MgO interaction with solution of magnesium bicarbonate Mg(HCO₃)₂. This was established by means of complex thermal analysis using thermogravimetry (TG), differential scanning calorimetry (DSC), mass spectrometry (MS), and electron microscopy. These methods allowed us to define the influence of the curing environment of magnesia composition. Results show that processes of hydration and curing of composition of magnesium oxide with magnesium bicarbonate solution proceed most effectively in water conditions at the course of cyclic reactions of magnesium hydrocarbonate formation. The magnesia composition transforms magnesia cement into the state of hydraulic magnesia cement.

Acknowledgements: Research was carried out using the core facilities of TPU's "Physical and chemical methods of analysis".

Citation: Mitina, N.A., Revva, I.B., Mitina A. Magnesia cement in the MgO-CO₂-H₂O system. Magazine of Civil Engineering. 2022. 113(5). Article No. 11311. DOI: 10.34910/MCE.113.11

1. Introduction

Due to the unique combination of properties, magnesia cement is one of the most sufficient materials for creating compositions for construction and technological purposes. A robust hardening structure of magnesia compositions is created by the synthesis of such crystalline hydrates as magnesium hydroxychlorides and hydroxysulfates [1, 2]. At the same time, these substances entail low water resistance due to their supersolubility in water conditions. The phase of magnesium oxychloride (oxysulfate) is unstable upon prolonged contact with water, which leads to leaching through the dissolution of magnesium chloride (sulfate) from the cement phase, leaving magnesium hydroxide as a binder that is unable to form hardening structures [1]. Scientists use various methods to solve the issue. The positive effect of chemical additives, sulfates, phosphates, and iron compounds has been established [3, 4]. Mineral additives of natural [5, 6] and technogenic origin [7–10] also increase the water resistance of the compositions.

The following methods for increasing the water resistance of magnesia cement do not exclude the formation of water-soluble substances in the products of hydration and hardening, since they are based on the use of magnesium salt solutions as a mixing liquid. A radical way to increase the water resistance of magnesia cement is the synthesis of water-insoluble compounds in the hardening products of magnesia binder, it is possible only on condition of a complete replacement of the mixing fluid.

Analysis of compounds in the MgO-CO₂-H₂O system showed their insolubility in water or low solubility. Characteristic compositions in the MgO-CO₂-H₂O system are hydrocarbonate groups of minerals with the general formula xMgCO₃·yMg(OH)₂·zH₂O and MgCO₃·xH₂O [1, 11–12], and also brucite Mg(OH)₂

and magnesite $MgCO_3$. Hydrocarbonate groups include barringtonite $MgCO_3 \cdot 2H_2O$, nesquehonite $MgCO_3 \cdot 3H_2O$, lansfordite $MgCO_3 \cdot 5H_2O$, pokrovskite $Mg_2(CO_3)(OH)_2 \cdot 0.5H_2O$, artinite $Mg_2(CO_3)(OH)_2 \cdot 3H_2O$, hydromagnesite $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$, dypingite $Mg_5(CO_3)_4(OH)_2 \cdot 5H_2O$, giorgiosite $Mg_5(CO_3)_4(OH)_2 \cdot 5-6H_2O$, shelkovite $Mg_7(CO_3)_5(OH)_4 \cdot 24H_2O$. Hydrocarbonates with general formula $xMgCO_3 \cdot yMg(OH)_2 \cdot zH_2O$ are subdivided into a light form with content of H_2O up to 4 molecules and a heavy one with content of H_2O from 4 up to 5 molecules.

Researches in the field of the $MgO-CO_2-H_2O$ system are focused on studying natural and artificial magnesium hydrocarbonate phases [11, 13–21]. Study of natural magnesium hydrocarbonates [14, 19–21] allows to trace history of genesis and metamorphism of carbonate and magnesium silicate rocks. Purposeful synthesis of magnesium hydrocarbonates allows to obtain pure products, without impurity, for different fields of application [13, 18, 22–23]. The researches dedicated to CO_2 removal from the atmosphere (sequestration) are also actual nowadays. These processes perform well involving the components of the $MgO-CO_2-H_2O$ system, for instance, original elements as well as compound substances [24–26].

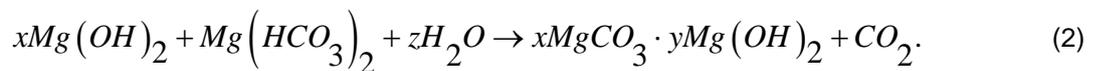
The $MgO-CO_2-H_2O$ system has great interest as the curable system which products are capable to create strong curing structures and to be used to produce new types of binders. Some scientists have already used synthesized magnesium hydrocarbonates for the purpose of hardening and creation of binding compositions [25–30]. The process of hardening is also performed with the help of external carbonization both natural one using CO_2 of environment air, and artificial one at the elevated pressure of CO_2 gas in the special chamber [11–18, 29–30]. Binding compositions have been obtained by gaging of caustic magnesite with special solutions [31–32] which essentially differ from classical magnesia Sorel cement. The compositions have high water resistance.

In this research the binding composition has been presented by mixture of caustic magnesium oxide MgO with water solution of magnesium bicarbonate (MBC) [31]. The interaction of active powder of magnesium oxide with MBC solution takes place in accordance with the following reactions (1, 2) [33]. This is consistent with studies of the carbonization magnesium compounds [22, 29–30, 32].

At first the hydration reaction proceeds:



Further formed magnesium hydroxide interacts with MBC in accordance with the reaction:



With formation of magnesium hydrocarbonate and carbon dioxide which entering the interaction with excess of magnesium hydroxide forms secondary MBC:



Secondary magnesium bicarbonate interacts with magnesium hydroxide again in accordance with the reaction (2) creating new portion of magnesium hydrocarbonate. Thus, three main crystal phases – magnesium hydroxide, magnesium carbonate and magnesium hydrocarbonates, which quantitative ratio is predetermined by content of magnesium bicarbonate in gauging liquid, are formed in cement stone as the result of proceeding consequent and parallel reactions (1, 2, 3). At that, composition and quantity of magnesium hydrocarbonate phase changes permanently and depends on the time of interaction and humidity of environment. Hydrocarbonate phases in the composition of hardening products make it possible for magnesia compositions to harden in water.

The goal of the research is determination of conditions for structuration and specification of types for new crystal formations of binding compositions using MBC solution when hardening in air-dry conditions and in water.

In connection with this goal, the tasks of studying the formation and change of hydrocarbonate phases depending on the hardening medium are solved using combined thermal analysis, differential scanning calorimetry (TG/DSC) and mass-spectrometric analysis of the interaction products.

2. Materials and Methods

2.1. Materials

The research has been performed using factory caustic magnesite of LLC "Sibirskiye poroshki" (Irkutsk) produced by roasting of magnesite rocks of the Savinsky deposit (Irkutsk region) in the rotating furnaces at a temperature of 800 °C. The chemical composition of initial and ignited rock is presented in Table 1.

Table 1. The chemical composition of caustic magnesite and initial magnesite rock

Material	Content, wt. %							Total
	MgO	SiO ₂	Al ₂ O ₃	CaO	Fe ₂ O ₃	MnO	Loss on ignition	
Initial magnesite	47,21	1.61	0.59	0.86	0.81	0.29	48.63	100.00
Caustic magnesite	75.64	3.18	0.62	4.24	0.83	0.32	15,17*	100.00

*- losses of caustic magnesite burned at 800 °C are connected with existence of undecomposed MgCO₃ with defect structure.

MBC solution has been obtained with artificial carbonization of caustic MgO suspension in the 5 l autoclave with a stirrer. The ratio of caustic powder and water was 10 g of MgO for 1 l of H₂O. Carbonization has been performed under CO₂ gas pressure of 0.9 MPa within 20 min. Concentration of MBC solution on bicarbonate ions was 6224.0 mg/l [33].

2.2. Methods

To study processes of hydration and curing of magnesia composition with the curing time up to 28 days we prepared mixtures of caustic magnesia powder and MBC solution with ratio of L/S = 0.5, and formed samples. After air curing within 24 hours these samples were located in different environments. Curing environments of the samples differed on humidity: 1) air humid – humidity was about 70 %, air temperature was 22–25 °C; 2) air moist – humidity was 95 ± 5 %, air temperature was 22–25 °C; 3) water (the samples were dipped into water, water layer over the sample was 2 cm). The samples cured within 3, 7, 14, and 28 days. After certain curing term the samples were taken from environment, dried up to the constant weight at temperature not more than 60 °C and tested.

To carry out thermal analysis we used the device for synchronous thermal analysis STA 449 F3 *Jupiter*® NETZSCH (Germany). The samples were heated in the air from 25 to 1000 °C with speed of 10 °C/min. Analysis of evolved gaseous products was performed with the QMS-403D quadrupole mass spectrometer of NETZSCH firm (Germany) production.

Microstructure of the samples was observed by means of the scanning electron microscope JEOL JSM 6000.

3. Results and Discussion

3.1. Microstructure

The curing environment of magnesia composition exercises considerable influence on composition and microstructure of products. After 28 days of curing in various kinds of environments different degrees of crystallinity, new crystal formation size and character of microstructure are registered during the experiment (Fig. 1).

The storage in humid and water conditions causes a big number of aggregations of foliated crystal formations: dypingite [25] and hydromagnesite [26]. They are formed at the edges of lamellar crystals of magnesium hydroxide Mg(OH)₂ that confirms theoretical assumptions about primary formation of magnesium hydroxide, and then its interaction with bicarbonate ions creating hydrocarbonates. There are no such processes in the samples, which were exposed to air with low humidity. Most new formations are light crystallized particles of magnesium hydroxide and magnesium hydrocarbonates, and also residues of unreacted magnesium oxide. This difference is caused by beneficial effect of water environment where magnesium oxide is constantly and actively hydrated and conditions for its interaction with bicarbonate ions are created. There is no supply of reacting components, in particular H₂O, during air curing after mixing of magnesium oxide with MBC solution. Thus, intensity of magnesia composition curing with MBC solution depends on the degree of MgO hydration.

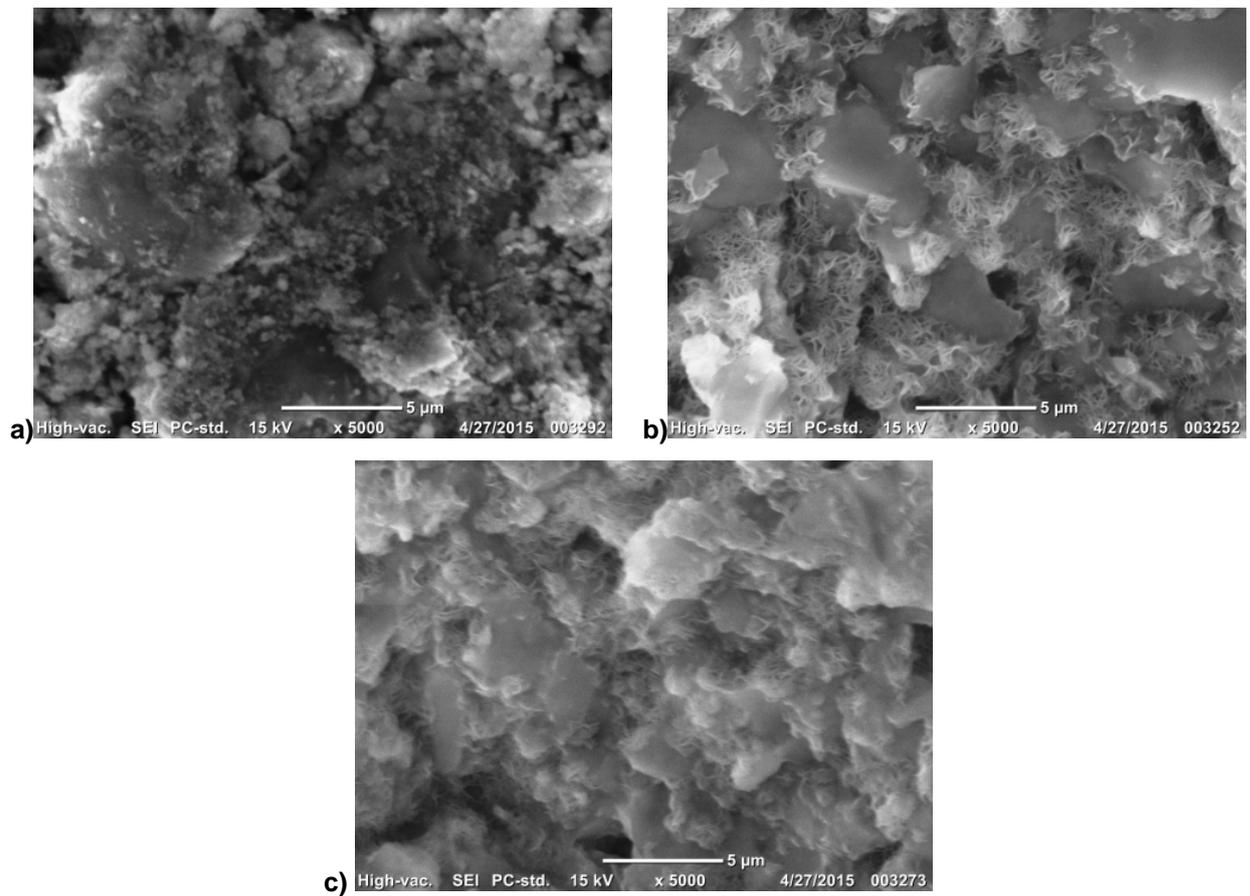
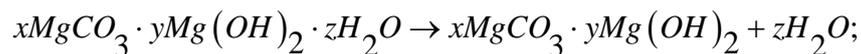


Figure 1. Microstructure of magnesia compositions after curing within 28 days in: a) air humid environment; b) air moist environment; c) in water.

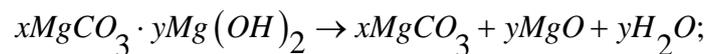
3.2. Simultaneous thermal analysis

The main processes of heating hydration products and curing the magnesia composition [12, 29, 32] are:

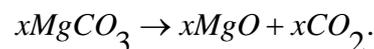
- dehydration, that is removal of crystallization water



- dehydroxylation, that is removal of hydroxyl group with decomposition of $Mg(OH)_2$ on MgO and H_2O



- decarbonization



Influence of curing environment of magnesia compositions is well traced by results of thermal analyses. TG and DSC curves of magnesia compositions cured on air and in water within 7 days (Fig. 2a) and 28 days (to Fig. 2b) are presented in Fig. 2. Results of thermogravimetric analysis of the magnesia composition samples at the age of 3, 7, 14, and 28 days when curing on air and in water are shown in Table 2. It is clear that weight losses at curing magnesia compositions in water exceed these values while curing them on air. Endothermic effects of DSC curves at average temperatures of 240, 400, and 590 °C correspond to processes of dehydration, dehydroxylation, and decarbonization of the magnesia composition samples at heating [12]. The endothermic effect at the temperature of 720–740 °C is well showed in Fig. 2 (b) and corresponds to the decarbonization of magnesium carbonate $MgCO_3$. The presence of magnesium carbonate is explained by underburned in the original caustic magnesite.

Table 2. Weight losses and TG of magnesia compositions

Peak	Age of 3 days		Age of 7 days		Age of 14 days		Age of 28 days	
	Temperature peak, °C	Weight losses, %						
1	86.6/113.3	5.1/5.9*	149.8/97.6	4.5/4.6	122.9/107.1	5.4/6.3	92.9/92.9	4.6/5.9
	240.3/240.0		252.7/245.3		242.2/245.3		246.3/245.0	
2	388.2/414.3	10.3/15.5	406.2/400.1	10.8/16.9	396.8/403.2	11.3/16.6	397.5/405.2	11.3/17.4
3	586.8/614.2	14.1/14.9	617.7/584.5	14.1/13.2	593.1/587.1	16.3/14.9	588.6/585.9	14.1/13.0

*- in numerator data for air environment, in denominator data for water environment

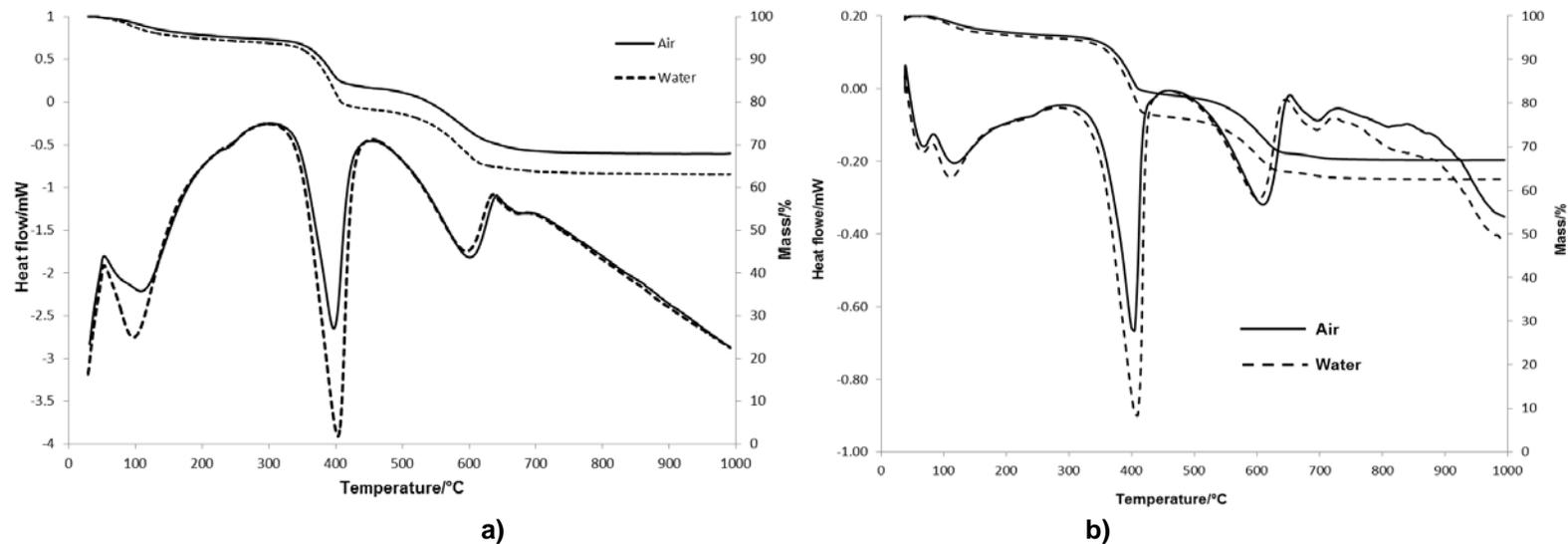


Figure 2. Thermograms of magnesia compositions at the age of 7 (a) and 28 (b) days.

The greatest difference in weight loss of magnesia compositions while curing in different conditions is registered during dehydroxylation process at the temperature of 400 °C. There is decomposition of $Mg(OH)_2$ both as component of magnesium hydrocarbonates crystallohydrates, and as initial magnesium hydroxide, which is formed in the reaction (1). Therefore, this thermal effect has been chosen to assess intensity of hydrocarbonate product formation at curing magnesia compositions with MBC solution (Fig. 3).

Data of Fig. 3 testify the different level of weight loss size at heating magnesia compositions in accordance with curing environment. The weight losses are increased on 50–70 % in the process of curing composition in water conditions. It implies that the quantity of magnesium hydrocarbonates is bigger in comparison with their quantity during formation in air environment. In addition, it is noted that if curing time of magnesia binding samples in water increases, the quantity of magnesium hydrocarbonates crystallohydrates increases, so that causes continuous increase of weight losses at dehydration and dehydroxylation. Such phenomenon is not observed during air curing because of lack of liquid component and the practical cessation of formation of magnesium hydroxide and, consequently, of magnesium hydrocarbonates.

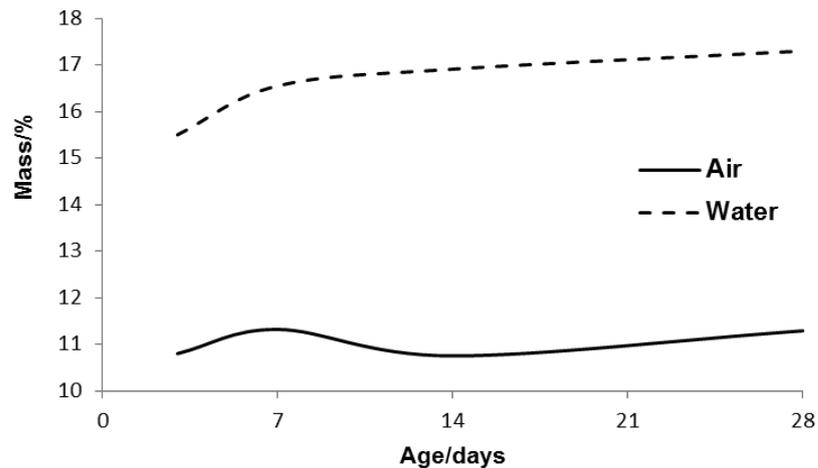


Figure 3. Dependence of weight losses during magnesia compositions' heating on curing time in different conditions.

In case of continuous presence of liquid phase while dipping and exposure in water of "the caustic magnesium oxide – MBC solution" system, continuous hydration of MgO takes place and cyclic process of magnesium hydrocarbonate formation in accordance with the reactions (1-3) occurs with the maximum intensity.

To confirm data of TG/DSC interaction products of caustic magnesite with MBC solution (Fig. 2b) we carried out analysis of gaseous products of the cyclic reactions 1-2 (Fig. 4) evolving during heating of the samples that were cured within 28 days in different conditions. Thermogravimetric and differential thermal analysis shows that total weight loss in the case of long presence of the reacting system on air is 33.01 %, during long presence in water – 37.5 %.

Ion current curves show that water vapors with the charge-mass ratio of 18 are removed at temperatures of 112, 242 (small amount) and at 405 °C [14]. The OH ion current curve (charge –mass ratio of 17) has the characteristic peaks corresponding to exudation of water. The beginning of CO₂ emission (charge-mass ratio of 12) is observed already at the temperature of 405 °C, the main process of magnesium hydrocarbonates decarbonization takes place at 606 °C. Emission of CO₂ at 696 °C occurs due to magnesium carbonate as an underburnt in the original caustic magnesia powder [34, 35].

Ion current curves with charge-mass ratio of 18 and 12 characterize the processes of thermal decomposition of magnesium hydrocarbonates (Table 3). Comparing curing conditions of magnesia compositions with MBC solution on the area of DSC thermograms peaks and ion current curves can indirectly inform about quantity of new formations. For this purpose, we chose characteristic peaks at maximum temperature of 405 °C, that are responsible for removal of water, hydroxyl ion and partial decarbonization and at 600 °C, according to decarbonization.

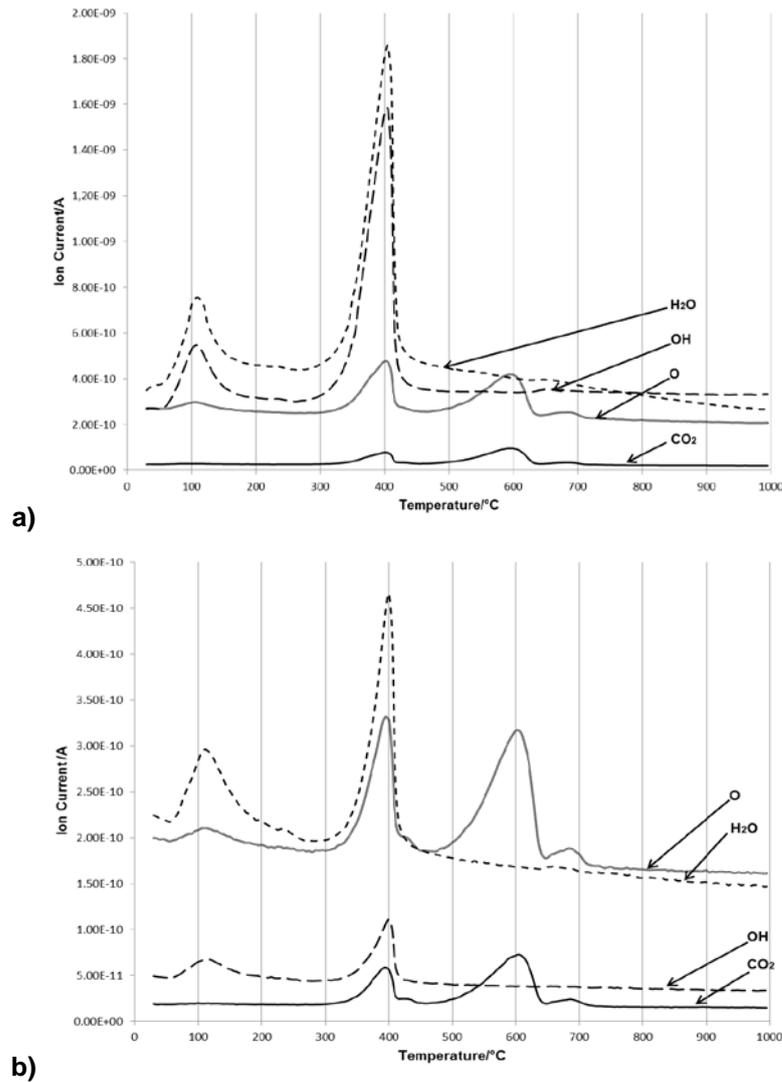


Figure 4. Ion current curves of gas emission at heating of magnesia compositions cured in air conditions (a) and in water conditions (b).

Table 3. Area of characteristic peaks of curves of thermal analysis and ion current

Peak temperature, °C	Processes	Area of peaks of DSC, J/g		Area of peaks of ion current curves, A	
		Air environment	Water	Air environment	Water
405	Removal of H ₂ O Removal of OH Removal of CO ₂	344.8	559.2	$1.35 \cdot 10^{-7}$	$8.08 \cdot 10^{-7}$
606	Decarbonization	331.9	337.3	$0.07 \cdot 10^{-7}$	$0.16 \cdot 10^{-7}$

Data of DSC and ion current curves show that the water environment provides formation of larger quantity of magnesium hydrocarbonates. The area under the integral sign of peak characterizes quantitatively the processes of thermal decomposition of the studied products and emission of gases. At the temperature of 405 °C, which causes the removal of water and hydroxyl ion, the peak area on thermogram is practically twice as much at curing magnesia composition in water, in comparison with curing one in air conditions. It is confirmed by data of mass spectroscopy on H₂O. During the decarbonization process at 606 °C by thermogram there was no difference in number of evolved CO₂, the area of peaks in air and water environment is almost identical – 331.9 and 337.3 J/g. However, according to mass spectrometry data the area of peak at the given temperature in water conditions is much bigger than one in air conditions that implies higher efficiency of magnesium hydrocarbonate formation during curing magnesia composition in water.

4. Conclusions

The results obtained confirm that:

1. In the interaction between magnesia caustic powder and solution of magnesium bicarbonate magnesium hydroxide and hydrocarbonate phases, dypengite and hydromagnesite, are formed.
2. The hardening of the magnesia composition under moist conditions after preliminary exposure to air contributes to a more intense structure formation, which is confirmed by thermal analysis methods. The formation of an increased amount of magnesium hydroxide is observed, since the hydration process in water conditions is continuous. $Mg(OH)_2$ in a highly dispersed state is the basis for new phases of magnesium bicarbonates. Thus, the bicarbonate magnesia binder is capable of hardening in water.
3. Structures generated from magnesium bicarbonates are insoluble in water. Therefore, magnesia binders relating to the $MgO-CO_2-H_2O$ system may be classified as hydraulic magnesia binders.

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Received 25.02.2021. Approved after reviewing 13.10.2021. Accepted 15.10.2021.