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Scientific paper

Formation and Evolution of the Hydrotalcite-Like Phase During Ageing of Dolomite-Cement Mortars under Various Conditions

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Received: 11-30-2022

Abstract

This work considers the reactions of dolomite powder mixed with Portland cement during ageing under accelerating conditions (60 °C, water or alkaline medium). The phase composition and microstructure of cement-dolomite mortars were studied using X-ray diffraction (XRD), thermogravimetric analysis (TGA), and scanning electron microscopy with X-ray microanalysis (SEM-EDS). The results showed that the alkalinity of the medium increased the dolomite reaction degree but did not affect the reaction products' composition. The formation of the hydrotalcite-like phase with the general formula $Mg_4Al_2(CO_3)_{1-x}(OH)_{12+2x}$. nH_2O proved to be the predominant route of dolomite consumption in the presence of available aluminium in the hardened cement paste. Then, after three months of ageing, an interlayer anions replacement became noticeable: carbonate anions in the structure of hydrotalcite are gradually replaced by hydroxide ones.

Keywords: dolomite-cement mortars, accelerating ageing conditions, dedolomitisation, hydrotalcite-like phase, interlayer anions replacement

1. Introduction

Concrete is the most widely used building material and is characterised by high compressive strength, availability, durability, low price, easy preparation and possibility of casting in desired shapes and sizes.¹ It is a composite material consisting essentially of cement paste in which particles or fragments of aggregates are embedded. Due to their abundance, dolomitic aggregates are the most commonly used for concrete preparation in the Alpine region.²

Dolomitic aggregates in concrete can undergo chemical reactions when exposed to the alkaline environment of the cement paste: this is the so-called dedolomitisation reaction with the formation of calcite and poorly soluble brucite: ^{3–5}

$$CaMg(CO_3)_2 + 2OH^- \Rightarrow CaCO_3 + + Mg(OH)_2 + CO_3^{2-}$$
(1)

Dolomite powder, which releases carbonate ions, may be involved in the hydration reaction of cement, forming calcite and carbonate-AFm (calcium aluminate) phases, thereby affecting the mechanical strength of concrete. It has also been shown that dolomite can react with alumina sources and portlandite to form hydrotalcite (HT) and calcite: ^{3,5,6} $\begin{aligned} & 6\text{CaMg}(\text{CO}_{3})_{2} + 2\text{Al}(\text{OH})_{4}^{-} + 8\text{OH}^{-} + 5\text{H}_{2}\text{O} \\ & \rightarrow \text{Mg}_{6}\text{Al}_{2}(\text{CO}_{3})(\text{OH})_{16} \cdot 5\text{H}_{2}\text{O} + 6\text{CaCO}_{3} + \\ & + 5\text{CO}_{3}^{2^{-}} \end{aligned} \tag{2}$

Aluminate ions $Al(OH)_4^-$ originate from soluble nature of alumina in cement minerals (tricalcium aluminate $3CaO\cdotAl_2O_3$ and tetracalcium aluminoferrite $4CaO\cdotAl_2O_3\cdotFe_2O_3$) under alkaline conditions. Subsequently, they migrate to the aggregate-cement paste interface. At the interface, dolomite dissolves, liberating Ca^{2+} and CO_3^{2-} ions into the cement paste and absorbing aluminate ions from the cement paste in accordance to equation (2). This reaction was proposed to be a typical metasomatism caused by the replacement of mobile components between dolomite and cement paste, keeping the texture of the original outlines of dolomite aggregate.³ As a result, a narrow rim composed of hydrotalcite is formed on the dolomite aggregate.

It has been suggested that this route is preferable for Mg^{2+} released from dolomite because the resulting hydrotalcite is thermodynamically more stable than equivalent amounts of brucite, boehmite (AlOOH), magnesite (Mg-CO₃), and water. According to the thermodynamic data,⁷ the ΔG°_{f} of hydrotalcite is about 10 kJ/mol lower than that of the separated phases listed above.

The hydrotalcite structure can be described as brucite-like layers in which some of the Mg^{2+} cations are replaced by Al^{3+} cations.⁸ Between the structural layers there are weakly bound charge-balancing anions (CO_3^{2-} , SO_4^{2-} , Cl^- , OH^- , etc.), which can enter into anion-exchange reactions,⁹ and water molecules. Natural hydrotalcite has the formula $Mg_6Al_2(CO_3)(OH)_{16}$ · $4H_2O$ with an Mg/Al ratio of 3. However, studies of synthetic HT show that this ratio can vary in the range of 0.5–3.2 depending on the pH of the medium.¹⁰ For cement-dolomite systems, the Mg/Alratio of hydrotalcite has been reported to be 1.9–3.2 in various works.^{6,11,12} It was found that the formation of hydrotalcite in the dolomite reaction was strongly limited by the alkalinity of the pore solution and the portlandite content of the cement paste.

The formation of Mg-Al hydrotalcite in dolomite-cement mortars has been shown to contribute to strength development and durability properties (resistance to leaching and carbonation), as well as an efficient binding capacity of chloride ions.¹³ However, there is insufficient information on the stability of hydrotalcite and the kinetics of its formation under various ageing conditions. The anionic composition of the hydrotalcite-like phase and its possible evolution during ageing has yet to be studied.

The present work aims to study the reaction products of dolomite during the ageing of dolomite-cement mortars, in particular, the hydrotalcite phase formed during the reaction of dolomite with Al-containing compounds from the cement paste, its composition, the kinetics of its occurrence and possible transformation depending on the ageing conditions. Various techniques (XRD, TGA, SEM-EDS) were used to study the phase composition and microstructure of the mortars. Since the dedolomitisation reaction is rather slow at room temperature, the accelerated conditions (de-ionised water or 1M NaOH at 60 °C), are suggested. In this way, the influence of the alkalinity of the medium on the dolomite reaction degree and the properties of the resulting products could also be determined. Dolomite powder with fine particles was used to increase the surface-to-volume ratio, since the reactions occur mainly on or near the interface of the dolomitic aggregates and the cement paste binder.

2. Materials and Methods

2. 1. Raw Materials and Samples Preparation

A dolomite-cement mortar mixture was prepared from dolomite aggregate from the southern part of Slove-

nia and Portland cement CEMI 42.5 N supplied by Salonit Anhovo. A crushed sand (fraction 0-2 mm) was used, characterised by water absorption of 0.14% and density of 1470 kg/m³. The chemical compositions of the cement and dolomite aggregate determined by ICP-OES analysis (Agilent 5100) are given in Table 1. The results of quantitative X-ray analysis (QXRD) show that dolomite accounts for 93.6 % of the mineral content of the aggregate, while the minor phases are calcite (4.5%), lime, periclase and quartz.

A total of 1093 g of Portland cement was dry mixed with 3216 g of dolomite aggregate using a RILEM-CEN mixer for 3 min at a speed of 300 rpm, providing the aggregate-to-cement mass ratio 3:1. Then 500 ml of de-ionised water was added and mixed for 1.5 min at 300 rpm to ensure good homogenisation of the pastes. To improve the rheology of concrete suspensions during mixing, 17.7 ml of superplasticizer SP-481 was added. The water-to-binder ratio of the mortars was 0.46.

Mortar tablets (25 mm in diameter and 10 mm high) were prepared from dolomite-cement mortar mixture to study phase composition and microstructure. After hardening at room temperature for 24 hours, the mortar tablets were demoulded and cured in an environment with a relative humidity of 90% and a temperature of 20 °C for 28 days. Then, the tablets were divided into two groups and placed in plastic containers filled with (a) 500 ml of de-ionized water with pH = 7.9 or (b) 500 ml of 1 M aqueous NaOH solution with pH = 14. The amount of liquid significantly exceeded the volume of cement tablets, so they were all submerged in liquid. Plastic containers, tightly closed to avoid evaporation of the solutions, were placed in a drying oven at a temperature 60 ± 2 °C for 1–12 months. The samples were submitted to analyses as prepared and after 1, 3, 6, 12 months of ageing, named M0 and M1-M12, respectively.

2. 2. Methods

X-ray powder diffraction (XRD) data of the samples were collected at room temperature with a PANalytical X'Pert PRO high-resolution diffractometer using Cu-K_{a1} radiation ($\lambda = 1.5406$ Å) in the reflection geometry. Data were collected in the 2θ range from 5 to 80° in steps of 0.033° using a 128-channel linear multi-strip detector to achieve a total integration time of 500 s per step. The peak positions and relative heights of the peaks were determined based on the experimental patterns. The crystalline phases present in the samples studied were identified using the PDF-4 database (release 2021). Quantitative phase analysis was performed using the Rietveld method, which is a re-

 Table 1. Chemical composition of cement and dolomite aggregates.

| Oxide (wt%) | CaO | MgO | SiO ₂ | Al_2O_3 | Fe ₂ O ₃ | K ₂ O | Na ₂ O | SO3 | LOI |
|-------------|------|------|------------------|-----------|--------------------------------|------------------|-------------------|-----|-----|
| cement | 60.4 | 2.5 | 22.2 | 5.5 | 3.3 | 0.8 | 0.5 | 3.0 | 1.3 |
| aggregate | 33.2 | 18.9 | 1.4 | 0.8 | 0.3 | 0.1 | < 0.1 | - | 0.1 |

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liable analytical approach for quantifying the amounts of different cementitious phases in multiphase mixtures containing phases with significant peak overlap and various peak widths. Topas Academic V4.1 software (Burker-AXS) and the ICSD database were used.

A scanning electron microscope, FE-SEM Zeiss Ultra Plus, equipped with EDS (Oxford X-Max SDD 50 mm² 106 detector and INCA 4.14 X-ray microanalysis software) at 20 kV accelerating voltage, a vacuum environment of $1-2\cdot10^{-6}$ mbar, and a beam current of about 20 nA was used on polished sections to detect microstructural changes during the dedolomitisation process. The EDS spectra were recorded on flat areas of the C-coated samples with a process time of 5 s, a lifetime of 120 s and an accelerating voltage of 20 kV. Qualitative analysis of the X-ray spectra was performed according to the standard procedure provided by the software manufacturer. Different fields of view were analysed in different areas of interest to obtain statistically reliable data.

Thermogravimetric analysis (TG/DTG) of samples was performed using a Netzsch STA 449 F3 Jupiter instrument in conjunction with a QMS 403 C Aëolos mass spectrometer. Typically, approximately 100 mg of sample was used for each run. The sample was heated in a 30 ml/min O_2 + Ar flow from 30 to 1200 °C at a heating rate of 10 °C/min under oxidative conditions (synthetic air containing 20 vol.% O_2 and 80 vol.% Ar). QMS analysis focused on monitoring the signals characteristic of H₂O and CO₂ (m/z = 18 and m/z = 44, respectively) to describe accurately the reactions associated with H₂O and CO₂ release from the different phases. Microcal Origin 6.0 was used for deconvolution and calculating overlapping peak areas.

3. Results and discussions

3.1. Phase Composition

XRD analysis of the initial dolomite-cement mortar before ageing (sample M0) indicates the presence of a pre-

dominant phase of dolomite and cement hydration products such as portlandite, ettringite, hemi-/monocarbonate, hydrogarnet, as well as unreacted ferrite and belite. The main difference in the phase composition of the samples in the XRD patterns during ageing is seen at low angles, where the main non-overlapping reflections of the minor phases are located.

Fig.1a–b show the XRD patterns in the range of $10-25^{\circ} 2\theta$ for samples aged for 0-12 months at 60 °C in water and 1M NaOH. Under accelerated ageing conditions, the process of dedolomitisation begins in the dolomite grains: the dolomite content decreases significantly, and calcite and brucite are formed according to the reaction (1). Traces of brucite appear after 1 month of ageing in NaOH, while the reaction in water is much slower and shows noticeable brucite formation only after 6 months of ageing.

It is also seen that dolomite readily reacts with Al-containing compounds from cement paste to form a hydrotalcite-like phase (HT) and calcite (reaction 2). At the same time, it was observed that the monocarbonate, ferrite and ettringite peaks become smaller and almost disappear with time in NaOH. This is consistent with the results of J. Xu et al., who suggested that the formation of hydrotalcite promotes the decomposition of these Al-containing compounds due to its lower solubility.¹⁰ Meanwhile, hydrogarnet Ca₃(Al,Fe)₂(SiO₄)(OH)₈, which is also formed during the hydration of cement paste at elevated temperatures, remains stable in the long run, as previously reported.14 It should be mentioned that the hydrotalcite-like phase appears earlier than brucite (especially during ageing in water, where the dedolomitisation is slower), confirming the assumption that the Mg²⁺ cations released from the dolomite prefer to form hydrotalcite rather than brucite.

The intensity of the hydrotalcite peak at $11.40^{\circ} 2\theta$ began to increase sharply with increasing ageing time and reached the maximum after 3 months for the sample aged in NaOH. During this period, most of the available



Figure 1. XRD patterns for the samples aged for 0-12 months at 60° C (a) in water and (b) in NaOH. Here and below in the figures: D – dolomite, P – portlandite, C – calcite, B – brucite, Ht –hydrotalcite, F – ferrite, Et – ettringite, Mc – monocarbonate, G – hydrogarnet.



Figure 2. Phase composition of dolomite-cement mortars aged for 0–12 months at 60 °C in (a) water and (b) NaOH, according to QXRD data (crystalline phases only).

aluminium from the cement paste was likely bound in the form of hydrotalcite. Thereafter, the reflections in the XRD patterns indicate the changes in the hydrotalcite-like phase: the peak broadened, and a shoulder at 11.28° 2θ appeared in the samples aged for 6–12 months both in water and NaOH. The detailed study of hydrotalcite-like compounds with different interlayer anions (NO₃⁻, Cl⁻, SO₄^{2–}, OH⁻ and CO₃^{2–}) showed that the basal spacing for hydrotalcite increased when CO₃^{2–} ions were replaced by OH⁻ ions in the interlayer region.¹⁵ Thus, the occurrence of the shoulder could be related to the formation of the hydrotalcite-like phase in which carbonate anions are replaced with hydroxide ions (HT-OH).

QXRD was used to quantify the amount of unreacted dolomite and the products of the dedolomitisation process in the cement-containing samples. The quantitative XRD

results of the main reaction products found in the samples aged in water and NaOH are presented in Fig.2a-b. Thus far, the calculations have been performed without considering the amorphous phases (the amount of which can be determined by adding an external standard). The conversion of dolomite during 12 months of ageing in water is rather low, less than 40%, and ettringite also disappears slowly during ageing. The concentration of HT increases slightly with time and reaches 10% after 12 months of ageing, while the content of brucite is rather low and is 2.5%. For the sample aged in NaOH, OXRD showed that a significant amount of dolomite reacted after 12 months of ageing: the conversion was about 75%. The fraction of the hydrotalcite-like phase is 10-11% after 3 months of ageing and then remains almost constant, while the fraction of brucite increases steadily to 5.8%.



Figure 3. DTG curves for the samples aged for 0-12 months at 60 °C in (a) water and (b) NaOH. C-S-H - calcium-silicate hydrate.



Figure 4. $H_2O(m/z = 18)$ and $CO_2(m/z = 44)$ release in the temperature range 350–500 °C for the samples aged for 0–12 months at 60 °C in (a) water and (b) NaOH.

3. 2. Thermogravimetric Analysis

Thermal analysis was used for a more detailed study of the reaction products in aged mortars, especially for the composition of the hydrotalcite-like phase. Fig. 3a-b show the DTG curves of the samples aged at 60 °C in water and 1M NaOH for 0-12 months. Six main peaks of mass loss were found in the DTG curves. The first peak at about 120 °C corresponds to the dehydration of C-S-H and ettringite/monocarbonate.¹⁶ The decomposition peaks of monocarbonate were found mainly in the initial unaged sample, which is consistent with the XRD observations. The second peak at about 230 °C, which appeared in aged samples, was attributed to the dehydration of hydrotalcite.¹⁷ The third peak is due to two parallel processes: a) further decomposition of bound hydroxyl groups and removal of carbonate ions in the interlayers of hydrotalcite and b) decomposition of brucite. This peak increases with increasing duration of ageing. The fourth peak at 475-485 °C corresponds to the decomposition of portlandite. Two

pronounced peaks at high-temperature ranges (550–780 °C and up to 880 °C) are due to the decomposition of dolomite and calcite, respectively.¹⁸ The broad peaks that occurred at lower temperatures could be related to the formation of amorphous and finely divided particles of calcite (probably secondary calcite) or dolomite.

It can be seen that the general trends in phase composition established by XRD are also confirmed by thermal analysis. The reaction proceeds more slowly in water: the peaks corresponding to hydrotalcite are less intense, and brucite is practically undetectable, while the peaks of dolomite and portlandite decrease only slightly. Another difference is the absence of peaks corresponding to fine calcite/dolomite particles in the 515–725 °C temperature range, after ageing in water at 60 °C for 3–12 months. This could be due to the dissolution of calcite in water, especially in the presence of CO_2 (open system), while the dissolution of calcite in an alkaline medium is much lower.¹⁹



Figure 5. Comparison of H_2O release and CO_2 release due to decomposition of HT in the range 350–500 °C, and H_2O : CO_2 ratio for the samples aged for 0–12 months at 60 °C in (a) water and (b) NaOH.

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Figure 6. SEM image and elemental maps of Mg, Al, Ca and Si for the sample aged in (a) water at 60 $^{\circ}$ C for 12 months and in NaOH at 60 $^{\circ}$ C for (b) 3 months, (c) 6 months and (d) 12 months.

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For further analysis of the hydrotalcite composition, the weight losses of the samples and the releases of water and CO₂ were compared in the temperature range from 300 to 550 °C (Fig.4). The peaks at about 420 °C, where the simultaneous release of H₂O and CO₂ is observed, correspond to the decomposition of hydrotalcite. The additional release of CO₂ at other temperatures could be due to the decomposition of monocarbonate or other carbonate-containing phases. The peak areas associated with the decomposition of hydrotalcite were calculated and compared with the total mass loss in this temperature range. In this way, the percentage ratio of H_2O and CO_2 released was determined, which is about 3.27 for natural hydrotalcite. In our case, the estimated H₂O : CO₂ mass ratio was 2.23 ± 0.10 for the unaged sample (M0) and the samples at an early stage of ageing (M1/M3), which is close to the ratio in hydrotalcite-like structure with lower Mg/Al ratio, e.g., quintenite Mg₄Al₂(CO₃)(OH)₁₂·3H₂O, whose thermal decomposition is characterised by slightly higher reaction temperatures compared to hydrotalcite.²⁰

The CO₂ and H₂O releases corresponding to the decomposition of hydrotalcite increase up to three months of ageing in both water and in NaOH. Thereafter, the amount of water released continues to increase for samples aged 6 and 12 months, consistent with XRD data on hydrotalcite phase content, while the amount of CO₂ released decreases. As a result, the H₂O : CO₂ ratio changes to 5.3 when aged in water and 7.5 when aged in NaOH for samples aged for 6 and 12 months. These data suggest a possible substitution of carbonate anions in the HT-CO₃ structure by hydroxide anions or the formation of a predominantly HT-OH phase, confirming the conclusions of the XRD study.

3. 3. Morphology

The changes in the microstructure of the mortars due to the dedolomitisation reaction and formation of new

phases in the vicinity of dolomite grains were followed using SEM microscopy. Fig. 6a–d show typical SEM images of dolomitic grains and elemental maps (Mg, Al, Ca, and Si) in the samples aged at 60 °C in water for 12 months and in NaOH for 3–12 months.

It is noteworthy that during the first six months of ageing in the water environment, no significant changes were observed in the microstructure: the dolomitic aggregate grains remained almost intact. A new, well-defined Mg-Al phase has appeared as a narrow rim within a decaying dolomitic grain only after 12 months of ageing, indicating that Mg²⁺ does not move outside the grain boundary of the dolomite due to its limited mobility in water and alkali environment. The thickness of the hydrotalcite rim reaches only $3.4 \pm 1.1 \,\mu\text{m}$ after ageing in water and does not exhibit a unique thickness that is likely sensitive to aggregate composition. When aged in NaOH, the Al-containing rim appears already after only 3 months of ageing, and its thickness increases with time from $3.3 \pm 0.8 \ \mu m$ to about 8.9 \pm 2.8 µm after 12 months of ageing. At the same time, the small dolomite grains look completely dedolomitised. Thus, the SEM data show that ageing in NaOH for 3 months results in the same changes in the dolomite grains as ageing in water for 12 months. This is also confirmed by XRD and TGA data on the amount of brucite and hydrotalcite-like phase formed.

The composition of the hydrotalcite-like phase in the samples was determined by plotting the Mg/Ca ratio *vs* the Al/Ca ratio from EDS analyses (Fig.7). A good correlation between Mg/Ca and Al/Ca indicates the presence of a hydrotalcite-like phase with a molar ratio of Mg/Al of about 2 for both water-aged and NaOH-aged samples. A similar ratio was reported for dolomite-cement systems.¹¹ Fig.7a indicates that the Mg/Al ratio in this phase does not change with time of ageing. Machner et al. have also shown that the Mg/Al ratio in dolomite/metakaolin-cement mortars was stable and did not change during leaching, carbonation or chloride exposure.¹³



Figure 7. Results of SEM-EDS chemical analysis of hydrotalcite phase in the samples aged for 3-12 months at 60 °C in (a) water and (b) NaOH.

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3. 4. Final Composition of HT-like Phase

According to XRD and TGA data, the formation of the hydrotalcite-like phase in dolomite-cement mortars starts from the very beginning of the ageing process of the samples at 60 °C both in water and in NaOH. The reaction can be written as following:

$$4\text{CaMg}(\text{CO}_3)_2 + 2\text{Al}(\text{OH})_4^- + 4\text{OH}^- + n\text{H}_2\text{O} \Rightarrow$$

$$\Rightarrow \text{Mg}_4\text{Al}_2(\text{CO}_3)(\text{OH})_{12} \cdot n\text{H}_2\text{O} + 4\text{CaCO}_3 + (3)$$

$$+ 3\text{CO}_3^{2-}$$

since the data from SEM give the Mg/Al ratio of 2 and, in our case, it does not depend on the alkalinity of the medium. The reaction proceeds more slowly in water, as expected from the literature data: phase composition and microstructure show that ageing in water for 12 months leads to the same changes in dolomite grains as ageing in 1M NaOH for 3 months. The amount of HT apparently increases with time as long as available aluminum cations remain in the cement paste.

The XRD and TGA data indicate that carbonate anions are replaced with hydroxide anions in the hydrotalcite-like phase during ageing:

$$Mg_4Al_2(CO_3)(OH)_{12} \cdot nH_2O + 2xOH^- \rightarrow \rightarrow Mg_4Al_2(CO_3)_{1-x}(OH)_{12+2x} \cdot nH_2O + xCO_3^{2-}$$

$$(4)$$

The formation of hydrotalcite containing mainly OH⁻ in the interlayer in carbonate-free hydrated Portland cement and of hydrotalcite containing both CO_3^{2-} and OH⁻ in Portland cement in the presence of carbonates was predicted.¹⁵ Moreover, thermodynamic data showed that HT-OH is more stable than HT-CO₃ under conditions typical for Portland cement.²¹ The released carbonate ions can migrate into the surrounding hydrated cement paste and assist in portlandite dissolution. This process takes place more easily when ageing occurs in water, since CO_3^{2-} ions readily migrate into solution under these conditions.

The final reaction products can therefore be identified as the hydrotalcite-like phase $Mg_4Al_2(CO_3)_{1-x}$ - $(OH)_{12+2x} nH_2O$, where *x* is the substitution degree of carbonates for hydroxides. This value was calculated from TGA data to be 0.53 and 0.64 for the hydrotalcite-like phase formed after 12 months of exposure in water and in an alkaline medium, respectively. Rietveld refinement of the HT peaks in the diffraction patterns yields close values of the substitution degrees (0.52 and 0.66 for samples aged in water and alkaline medium, respectively). The substitution degree will likely increase during the subsequent ageing of the samples.

4. Conclusion

In this work, we investigated the phase composition and microstructure of dolomite-cement mortars after ageing for 0–12 months under accelerated conditions (water or 1M NaOH, 60 °C). It was found that dolomite aggregates interact with cement paste and form the same reaction products (calcite, brucite and hydrotalcite) in alkaline environment and in water. However, the reaction in the water is much slower than in NaOH: the changes that occurred in dolomite grains during 12 months of ageing in water correspond to the changes that occurred during 3 months in 1M NaOH. The formation of the hydrotalcite-like phase rather than brucite is the predominant pathway for dolomite transformation in the presence of a large amount of available aluminium sources.

The hydrotalcite formed has the formula $Mg_4Al_2(CO_3)_{1-x}(OH)_{12+2x} \cdot nH_2O$, and the Mg/Al ratio does not change with ageing. No effect of the alkalinity of the medium on the composition of the hydrotalcite phase (Mg/Al ratio) was found. However, the carbonate anions in the structure of hydrotalcite are gradually replaced by hydroxide anions after three months of ageing, resulting in the formation of hydrotalcite containing mainly OH⁻ in the interlayer. The substitution degree *x* was estimated to be 0.53 or 0.64 after 12 months of ageing in water or NaOH, respectively.

Acknowledgements

The authors thank Petra Štukovnik (University of Ljubljana, Faculty of Civil and Geodetic Engineering) for her help with sample preparation and Edi Kranjc (National Institute of Chemistry, Ljubljana) for performing XRD measurements. We also thank the Slovenian Research Agency for financial support through grant P1-0175.

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Povzetek

To delo obravnava reakcije dolomitnega prahu, zmešanega s portlandskim cementom, med staranjem v pogojih pospeševanja (60 °C, voda ali alkalni medij). Fazno sestavo in mikrostrukturo cementno-dolomitnih malt smo preučevali z rentgensko difrakcijo (XRD), termogravimetrično analizo (TGA) in vrstično elektronsko mikroskopijo z rentgensko mikroanalizo (SEM-EDS). Rezultati so pokazali, da je alkalnost medija povečala stopnjo reakcije dolomita, vendar ni vplivala na sestavo reakcijskih produktov. Izkazalo se je, da je tvorba hidrotalcitu podobne faze s splošno formulo $Mg_4Al_2(CO_3)_{1-x}(OH)_{12+2x}\cdot nH_2O$ prevladujoča pot porabe dolomita ob prisotnosti razpoložljivega aluminija v strjeni cementni pasti. Nato je po treh mesecih staranja postala opazna zamenjava vmesnih anionov (or anionov med plastmi): karbonatne anione v strukturi hidrotalcita postopoma zamenjajo hidroksidni.



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