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Hydrotalcite synthesis via co-precipitation reactions using MgO and $Al(OH)_3$ precursors

Rafael Salomão [a,*], L.M. Milena [a], M.H. Wakamatsu [a], Victor C. Pandolfelli [b]

[a] *Federal University of the ABC Region, Rua Santa Adélia, 166 ZIP 09210-170, Santo André, SP, Brazil*
[b] *Federal University of São Carlos – Materials Engineering Department, Rodovia Washington Luís, km 235, São Carlos, SP, Brazil*

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Abstract

Hydrotalcite ($Mg_6Al_2(OH)_{16}(CO_3)\cdot 4H_2O$), also known as aluminum–magnesium layered double hydroxide (LDH) or anionic clay, is a synthetic compound that was broadly investigated in the past decade due to its many potential applications, such as clinic anti-acid, catalyst support, adsorptive flotation, flame retardant, acid scavengers in polymer composites and as a raw material for high temperature insulating porous ceramics. This compound is usually produced by controlled chemical equilibrium shifting processes (such as co-precipitation) that requires various other purification steps (centrifugation, for example) and careful drying (freeze drying or ultrafiltration). In this paper, a novel route to synthesize hydrotalcite is presented, based on the hydration, dissolution and co-precipitation reactions carried out almost simultaneously in aqueous suspension containing reactive magnesium oxide and aluminum hydroxide. Compared to other methods (the regular co-precipitation, particularly), it presents various technological advantages such as low time-energy consumption, no further purification step requirement, high output and competitive production costs.

Keywords: Hydrotalcite; Magnesium oxide; Aluminum hydroxide; Hydration

1. Introduction

The term hydrotalcite refers to a family of synthetic compounds with a structure based on the magnesium hydroxide ($Mg(OH)_2$)-like layers in which some of the divalent cations have been replaced by trivalent ones. This substitution generates positively charged sheets that are stacked together and kept electrically neutral by intercalating anions among them, as seen in Fig. 1 [1,2]. Their general formula can be expressed as $[M_x^{II}M_y^{III}(OH)_{(2x+2y)}](A^{n-})_{(y/2)}\cdot mH_2O$, where, the divalent and trivalent cations (M^{II} and M^{III}, respectively), the stoichiometric coefficients values (x and y, $2 \leq x/y < 4$), the interlayer anions (A^{n-}, $(CO_3)^{2-}$, $(SO_4)^{2-}$, OH^-, Cl^-, $(NO_3)^-$, for example) and the water content (m) can be varied over a wide range, giving rise to a class of isostructural materials also known as layered double hydroxides (LDHs) or anionic clays (contrary to the regular clays that present positive charges in the interlayer sites) [1–5].

Among the various types of hydrotalcites, the magnesium–aluminum one (MAH, $Mg_6Al_2(OH)_{16}(CO_3)\cdot 4H_2O$) has been broadly investigated over the past decade due to its various technological applications, which involves its use as a clinic anti-acid, catalyst support, adsorptive flotation carrier, flame retardant and acid scavengers in polymer composites [6]. MAH was also recently described as a raw material for high temperature insulating porous ceramics [7] by combining two important aspects: when added to a dense calcined alumina matrix: (i) the great weight loss during its thermal decomposition results in a large amount of pores [8] and, (ii) it induces the aluminum–magnesium spinel ($MgAl_2O_4$) formation, whose intrinsic difficulties for sintering inhibits the densification of the porous structure formed [9].

There are various possible methods to synthesize MAH, with different time-energy profiles, purity requirements for the reactants and for the final compound attained [10–15]. The most common route is based on the co-precipitation process (Fig. 2), which starts with the preparation of an aqueous solution of magnesium and aluminum salts ($MgCl_2$ and $AlNO_3$,

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* Corresponding author. Present address: University of São Paulo – Materials Engineering Department, Avenida Trabalhador São-carlense, 400 ZIP 13566-590, São Carlos, SP, Brazil. Tel.: +55 16 33738672; fax: +55 16 33739590.
E-mail addresses: rsalomao@sc.usp.br (R. Salomão), vicpando@power.ufscar.br (V.C. Pandolfelli).

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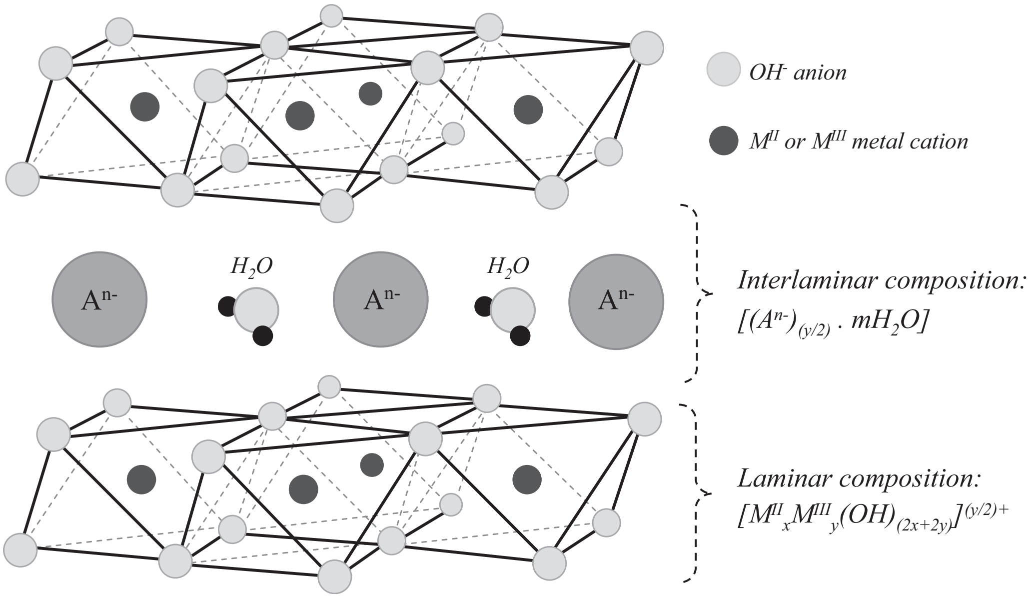

Fig. 1. Schematic representation of the hydrotalcite structure.

for example, Fig. 2a), keeping the molar ratio between them close to 3:1 or 2:1 (this ratio will define the degree of substitution of the trivalent cations in the $Mg(OH)_2$ layers). In neutral-acid pH, the solution is stable and the Mg^{2+} and Al^{3+} cations remain dissolved (Fig. 2b). Afterwards, the pH of the solution is carefully increased in order to induce the precipitation of the double hydroxide (usually, $NaHCO_3$ or NH_4HCO_3 are frequently used to shift the pH as they also behave as $(CO_3)^{2-}$ anions source, Fig. 2c and d [14,15]). By changing the pH, the temperature or the stirring intensity, the nucleation–precipitation mechanism can be controlled, resulting in the required particles' sizes and shapes (Fig. 2e) [15]. After finishing the reaction, the counter-cation used to increase the pH (Na^+ or NH_4^+, in this case) still remains in the obtained hydrotalcite suspension and must be withdrawn usually by centrifugation, dialysis or ultra-filtration (Fig. 2f). Further hydrothermal treatments can be used to enhance the particles' crystallinity (Fig. 2g and h) and to adjust the particle size distribution [16]. In order to attain a dried powder, spray-drier or liofilization must be applied to prevent particle agglomeration (Fig. 2i) [12].

Considering the applications of MAH as a raw material for high temperature insulating porous ceramics, two main drawbacks can be pointed out in these co-precipitation based processes: (i) the presence of alkali-based impurities (Na, K and Li) in MAH can induce the formation of low refractoriness compounds and, (ii) the excessively time-energy consuming process compared to the production rate of other raw materials used in the refractory industry. Therefore, based on these aspects and the good potential performance of MAH for this application, it is reasonable for pointing out that alternative methods to synthesize this material would have significant technological interest. Reports in the literature suggest that the soluble salts could be replaced by reactive oxides as cation sources [17–19]. In these cases, hydration/hydrolysis reactions would provide the desired ions, excluding the presence of residual ones at the end of the process [17–19].

Authors' previous work has reported on the formation of hydrotalcite in some systems comprised by a blend of alumina and magnesia reactive sources (respectively, hydratable alumina or calcium aluminate cement and caustic magnesia), simply by combining these raw materials in an aqueous suspension, with no need of additional reactants or purification steps [20]. This paper presents and discusses this process route in order to produce hydrotalcite. A mechanism based on the hydration, dissolution and co-precipitation reactions carried out in aqueous suspension of reactive magnesium oxide and aluminum hydroxide is presented and the fine counter-cations-free hydrotalcite particles attained were characterized.

2. Materials and techniques

Four raw materials with different chemical reactivities were used in this work: (1) caustic magnesia, (2) magnesium hydroxide (caustic magnesia previously hydrated in autoclave for 3 h, at 150 °C), (3) calcined alumina and (4) aluminum hydroxide. These powders were initially characterized (Table 1) by means of: (a) particle size distribution (ASTM C1070-01 *Standard Test Method for Determining Particle Size Distribution of Alumina or Quartz by Laser Scattering,* Zetasizer NanoZS, Malvern); (b) density (Helium picnometry, Ultrapicnometer, DP Union); (c) specific surface area (BET method, ASTM C1069-09 *Standard Test Method for Specific Surface Area of Alumina or Quartz by Nitrogen Adsorption*, Gemini 2370, Micromeritics) measurements; (d) X-ray diffraction (XRD, Kα = Cu, Simens D 5000, Kristalloflex 710, Ni filter) for phase identification and mean crystallite size measurements (using Scherer's equation); (e) free moisture (C324-01(2007) *Standard Test Method for Free Moisture in Ceramic Whiteware Clays*) and loss of ignition (at 1000 °C, adapted after C324-01); (f) scanning electron microscopy (SEM, Jeol 6460LV, 25 K).

	Steps	Remarks
a)	Solution of divalent cation salt (Ex.: $6MgCl_2$) + trivalent cation salt (Ex.: $2AlCl_3$) ↓	Mg:Al = (3-2):1 (Excess of Mg^{2+} or Al^{3+} results hydrotalcite plus $Mg(OH)_2$ or $Al(OH)_3$ respectively)
b)	$6Mg^{2+}_{(Aq.)} + 2Al^{3+}_{(Aq.)} + 18Cl^{-}_{(Aq.)} + nH_2O$ ↓	pH ≤ 7 (Stable solution)
c)	pH increase (Ex.: $NaHCO_3$ addition) ↓	$(CO_3)^{2-}$ can be replaced by other anions (Ex.: F^-,Cl^- or carboxilic organic anions)
d)	$6Mg^{2+}_{(Aq.)} + 2Al^{3+}_{(Aq.)} + 18Cl^{-}_{(Aq.)} + mNa^{+}_{(Aq.)} + m(CO_3^{2-})_{(Aq.)} + mOH^-$ ↓	pH ~ 10-12 (Metastable solution)
e)	$Mg_6Al_2(OH)_{16}(CO_3)\cdot 4H_2O_{(Precipitated)} + 18NaCl_{(Dissolved)}$ ↓	pH ~ 10-12 (Hydrotalcite suspension)
f)	Purification (Ex.: Centrifugation) ↓	NaCl withdraw
g)	$Mg_6Al_2(OH)_{16}(CO_3)\cdot 4H_2O_{\text{(Aqueous suspension)}}$ ↓	Low cristallinity particles
h)	Hydrothermal treatment (Ex.: Autoclave) and controlled drying (Ex.: liofilization or freeze drying) ↓	Improves cristallinity, narrows particle size distribution, prevents particles agglomeration
i)	$Mg_6Al_2(OH)_{16}(CO_3)\cdot 4H_2O_{(Dried)}$	Dried hydrotalcite particles

Fig. 2. General route for MAH co-precipitation.

The candidates for Mg^{2+} (MgO and $Mg(OH)_2$) and Al^{3+} (calcined alumina and $Al(OH)_3$) sources were combined at different ratios (10–90 wt.%) in order to find out the best condition for hydrotalcite formation. The total amount of MgO, $Mg(OH)_2$, Al_2O_3 and $Al(OH)_3$ was defined considering the density, the free moisture content and their chemical composition in the raw materials.

The raw materials were initially dry blended and were mixed afterwards with a large excess of water (two times greater than the theoretical content required to attain full hydration) in a paddler mixer (T 50 Ultra-Turrax, IKA, Germany) for 300 s, at a constant speed of 5000 rpm. No additives (such as dispersants and plasticizers) were added to the compositions. This intensive mixing step assures that all particles were individualized even in the absence of dispersants.

Afterwards, the suspensions were cast into polymeric cylindrical non-adherent molds (30 mm × 30 mm) sealed in individual flasks (50 mm × 50 mm) to prevent drying, and kept at different temperatures (10, 30, 50 and 80 °C) for 7 days. After this period, samples were dried overnight at 110 °C, deagglomerated using a high speed mill (2800 rpm, 10 s, A11 Analytical mill, IKA, Germany) and the mass loss evaluated after drying at 150 °C in a vacuum furnace during 72 h (M_{Dried}) and after calcination (5 °C/min) at 1000 °C for 5 h

Table 1
Characteristics of the raw materials tested.

Raw materials	Mg^{2+} ions sources		Al^{3+} ions sources	
	Magnesium oxide[a]	Magnesium hydroxide[a]	Calcined alumina[b]	Aluminum hydroxide[c]
Composition (wt.%, typical)	MgO: 98.90; Fe_2O_3: 0.19; SiO_2: 0.17; CaO: 0.74	Hydrated magnesium hydroxide (autoclaved during 3 h, at 150 °C)	α-Al_2O_3: 99.78; Na_2O: 0.08; Fe_2O_3: 0.02; SiO_2: 0.03; CaO: 0.02; MgO: 0.07	$Al(OH)_3$: 99.7; Na_2O: 0.256; Fe_2O_3: 0.007; SiO_2: 0.004
Particle size (D_{50}/D_{90}, μm)	12/25	4.1/8.6	0.5/1.2	0.9/2.5
Density (g cm^{-3})	3.55	2.48	3.90	2.43
Surface area (m^2 cm^{-3})	6.7	5.8	7.2	4.3
JCPDS identification and mean crystallite size (C_{50}, μm)	Periclase (45–946); 12.8	Brucite (7–239); 9.2	Corindun (46–1212); 0.9	Gibbsite (7–324); 2.1
Free moisture (wt.%, after 24 h at 110 °C)	1.2	0.5	0.4	0.4
Loss of ignition (wt.%, 110–1100 °C, 5 °C/min, 5 h)	0.9	30.8	0.8	35.5

[a] Minérios Ouro Branco, Brazil.
[b] CT3000SG, Almatis, US.
[c] Hydral 710, Almatis, US.

(M_{Calcined}). Samples' hydration degree (W_{HExp}, %) were calculated using the following expression:

$$W_{\text{HExp}} = 100\% \times \frac{M_{\text{Dried}} - M_{\text{Calcined}}}{M_{\text{Calcined}}} \quad (1)$$

The values obtained were compared to the theoretical ones (W_{HT}), calculated based on the mass ratio of each compound assuming that the reaction occurred stoichiometrically.

The samples dried at 150 °C were also investigated using XRD and SEM.

3. Results and discussion

3.1. Aqueous mixing of different sources of Mg^{2+} and Al^{3+} ions

Fig. 3 presents the experimental hydration degree (W_{HExp}) after mixing and reacting MgO or $Mg(OH)_2$ with calcined alumina or $Al(OH)_3$ after 7 days at 50 °C, and the theoretical values (W_{HT}) for $Mg(OH)_2$ and $Al(OH)_3$ and $Mg(OH)_2$ and Al_2O_3 mixtures (dotted lines). Initially, nor the 100 wt.% $Mg(OH)_2$, neither the 100 wt.%. $Al(OH)_3$ samples presented any weight gain for the conditions tested, as expected. The 100 wt.% MgO containing suspension one became fully hydrated after the seventh day of testing as pointed out by the hydration degree values ($W_{\text{HT}} = 44.68\%$ and $W_{\text{HExp}} = 43.06 \pm 1.97\%$) and the $Mg(OH)_2$ XRD pattern presented in Fig. 4a. The MgO reactivity is related to its high surface area and small mean crystal size [21], as also observed in similar studies in the literature [21–23]. The 100 wt.% calcined alumina suspension also did not present any significant change due to the high chemical stability of α-alumina. The small W_{HExp} observed ($W_{\text{HExp}} = 0.37 \pm 0.12\%$) can be related to the residual presence of $Al(OH)_3$. Considering the compositions with mixed compounds, it can be observed that for the calcined alumina and MgO (Fig. 3a) or with $Mg(OH)_2$ (Fig. 3b) and $Al(OH)_3$ and with $Mg(OH)_2$ (Fig. 3c), similar values of W_{HExp} and W_{HT} were observed.

For a theoretical and stoichiometric reaction between $Mg(OH)_2$ and $Al(OH)_3$, as described in expression (2),

$$6Mg(OH)_2 + 2Al(OH)_3 + 3H_2O + CO_2 \rightarrow Mg_6Al_2(OH)_{16}(CO_3)4H_2O \quad (2)$$

the ratio between these compounds in order to result 100% hydrotalcite would be 69 wt.% $Mg(OH)_2$/31 wt.% $Al(OH)_3$ and the MAH W_{HT} would be 78.3 wt.%. By mixing $Mg(OH)_2$ and $Al(OH)_3$ in the same proportion but without any reaction, the W_{HT} value will be 47.18 wt.%. Based on these parameters and the results presented in Fig. 3c ($W_{\text{HExp}} = 46.5 \pm 2.45\%$) and Fig. 4a, it can be pointed out that, for these samples, no significant chemical reaction between the components occurred, resulting only in a physical mixture of $Mg(OH)_2$ and $Al(OH)_3$ or $Mg(OH)_2$ and calcined alumina.

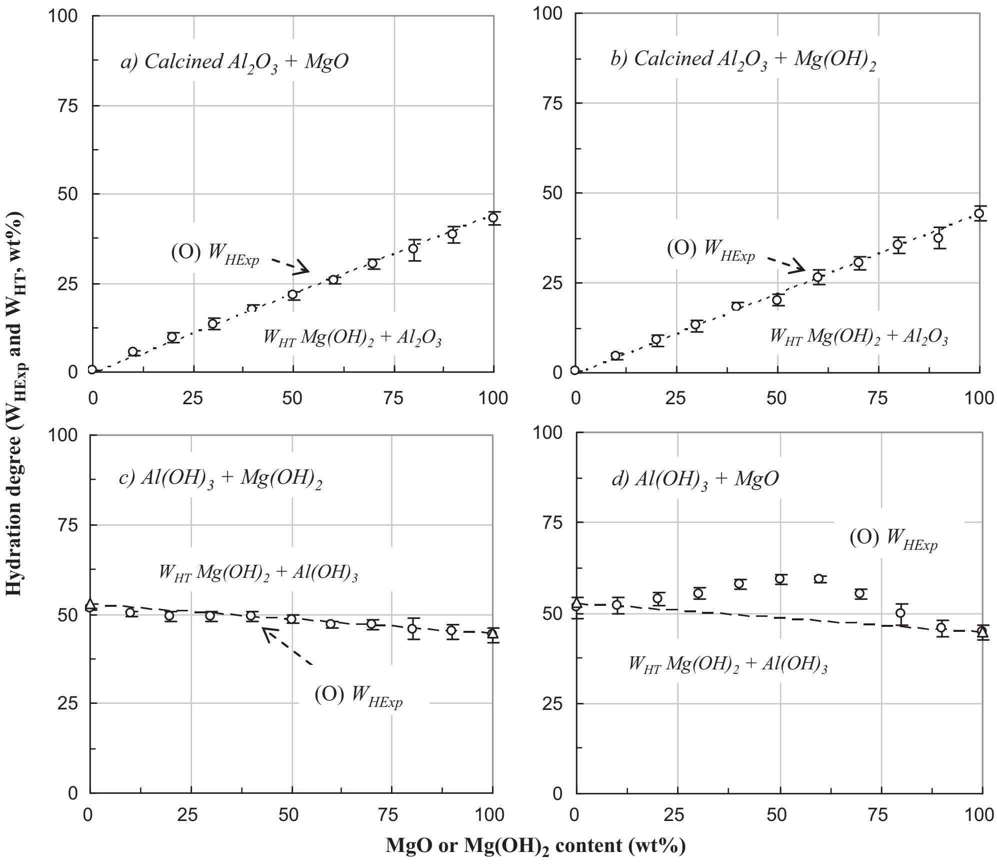

Fig. 3. Hydration degree results for mixing the tested raw materials, after 7 days at 50 °C.

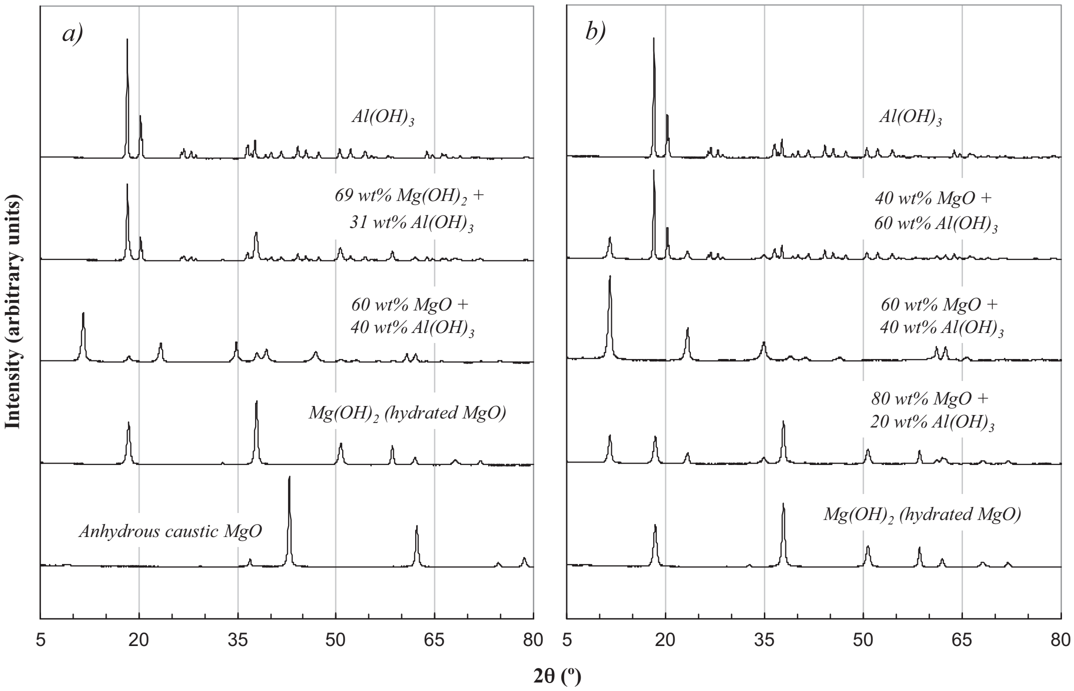

Fig. 4. X-ray diffraction (XRD) profiles for the raw materials tested, after 7 days at (a) 50 °C and (b) 80 °C.

Nevertheless, a distinct behavior was observed for the samples containing $Al(OH)_3$ and MgO (Fig. 3d). In this case, the W_{HExp} values obtained were significantly higher than the expected (W_{HT}) for the simple mixture of $Al(OH)_3$ and $Mg(OH)_2$, indicating that a different compound was formed. The XRD analysis (Fig. 4a) was carried out for the 60 wt.% of MgO sample, which is the closest to the MAHs theoretical composition (61.3 wt.% of MgO). The main diffraction peaks of MgO (43°), $Mg(OH)_2$ (38°) and $Al(OH)_3$ (18°) almost disappeared and intense MAH peaks were observed (12° and 23.5°, JCPDS card 22-0700). Other studies where aqueous suspensions of MgO were investigated using this technique reported that the lack of the MgO main diffraction peaks is usually related to its conversion to $Mg(OH)_2$, whose peaks become more intense (as observed for the hydrated 100 wt.% MgO sample) [20]. For the referred composition, conversely, the simultaneous reduction of the main peaks of MgO, $Mg(OH)_2$ and $Al(OH)_3$ indicates that these compounds were most likely dissolved and re-precipitated as a MAH. In order to understand this aspect, the chemical reactivity and hydration behavior of each raw material must be considered.

MgO hydration is a dissolution/precipitation mechanism, expected by the following reactions [21,23]:

$$\mathrm{MgO}_{(\mathrm{Solid})} + \mathrm{H_2O}_{(\mathrm{Liq.})} \rightarrow \mathrm{MgOH}_{(\mathrm{Surface})}{}^{+} + \mathrm{OH}_{(\mathrm{Aqueous})}{}^{-} \rightarrow \mathrm{MgOH^{+}OH}_{(\mathrm{Surface})}{}^{-} \qquad (3)$$

$$\mathrm{MgOH^{+}OH}_{(\mathrm{Surface})}{}^{-} \rightarrow \mathrm{Mg}_{(\mathrm{Aqueous})}{}^{2+} + 2\mathrm{OH}_{(\mathrm{Aqueous})}{}^{-} \rightarrow \mathrm{Mg(OH)}_{2(\mathrm{Solid})} \qquad (4)$$

During the MgO hydration two main effects can be observed: (i) a high concentration of Mg^{2+} cations dissolved and (ii) a significant increase in the pH of the aqueous suspension (above 10–11), due to the massive dissolution of OH^- ions. If this alkalinization is carried out in the presence of $Al(OH)_3$, its hydroxylation and further dissolution are favored [17–20], as described by expression (5), releasing Al^{3+} cations:

$$\mathrm{Al(OH)}_{3(\mathrm{Solid})} + \mathrm{OH}_{(\mathrm{Aqueous})}{}^{-} \rightarrow \mathrm{Al(OH)}_{4(\mathrm{Aqueous})}{}^{-} \rightarrow \mathrm{Al}^{3+} + n(\mathrm{OH})^{-} \qquad (5)$$

Therefore, the simultaneous presence of Al^{3+}, Mg^{2+}, OH^- and $(CO_3)^{2-}$ (dissolved in the water during the mixing) ions in an alkaline medium are the suitable conditions for MAH precipitation. It is worth mentioning the similarity of the co-precipitation methods described in the first section of this paper (Fig. 2). The main difference lies on the fact that, for this novel process, the sources of Mg^{2+} and Al^{3+} ions are also the equilibrium shifting agents that cause the hydrotalcite co-precipitation. On the other hand, for the samples containing calcined alumina and $Mg(OH)_2$, MAH could not be formed under this condition as their high chemical stability prevented their dissolution and no significant amount of cations was available.

3.2. The MgO–$Al(OH)_3$ system

Because the MgO–$Al(OH)_3$ system presented the most promising results in order to better understand the mechanism by which MAH was formed, hydration/dehydration tests were conducted combining these raw materials at different temperatures (10–80 °C, Figs. 4b and 5). Various works that investigated the synthesis of MAH pointed out that the ratio

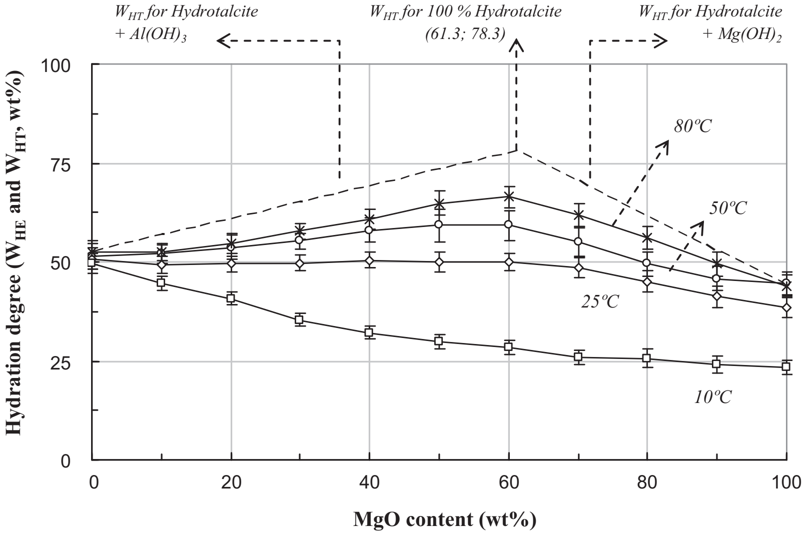

Fig. 5. Hydration degree results for combining MgO and $Al(OH)_3$, after 7 days at different temperatures.

between Mg^{2+} or Al^{3+} ions in the reactant medium must be around 3–2:1 in order to attain stoichiometric hydrotalcite, otherwise, the excess of cations precipitates as ordinary hydroxides generating combinations of MAH and $Mg(OH)_2$ or MAH and $Al(OH)_3$, depending on which cation is in excess [5,12,14,16]. Based on this behavior, in Fig. 5, besides the W_{HExp} results (continuous line), a dotted line highlights the W_{HT} values for a theoretical combination of MAH and these hydroxides (the highest value corresponds to 100 wt.% hydrotalcite when 61.3 wt.% of MgO and 38.7 wt.% of $Al(OH))_3$ are present).

Except for the samples kept at 10 °C, it can be observed that for the compositions where the MgO content is distinct from 60 wt.%, the excess of Mg^{2+} or Al^{3+} (or the correspondent amount of MgO and $Al(OH)_3$) resulted in the presence of MAH and hydroxides. This hypothesis is attested by the XRD results presented in Fig. 4b, where the diffraction peaks of MAH and hydroxides can be seen for the samples containing 80 wt.% and 40 wt.% of MgO, respectively. Therefore, there is an ideal ratio between MgO and $Al(OH)_3$ to maximize the content of MAH obtained (~60 wt.% MgO). It also can be noted that this process is strongly temperature dependent, because as the testing temperature increases, the W_{HExp} values are closer to the W_{HT} ones for MAH or a mixture of MAH and the correspondent hydroxide. Considering this, for the MAH formation, the hydration/dissolution of MgO and $Al(OH)_3$ is required, it is reasonable to expect that the temperature increase would enhance the reactions' rates [16]. For low temperatures (10 °C, in this case), the driving force for MgO hydration decreases, slowing down its hydration rate, as well as the pH increase and, consequently, the $Al(OH)_3$ dissolution [22,23].

The 60 wt.% MgO/40 wt.% $Al(OH)_3$ sample prepared at 80 °C for 7 days was characterized regarding its particle size distribution (Fig. 6) and morphology (Fig. 7). Compared to the initial raw materials, it can be observed that the MAH mean particle diameter (D_{50}) is significantly smaller ($D_{50\,MgO} = 12$ μm; $D_{50\,Al(OH)_3} = 0.91$ μm; $D_{50\,MAH} = 0.37$ μm). For certain oxides, such as MgO and CaO, the hydroxylation causes intense mechanical damage due to the stresses associated to the density mismatch [22]. SEM

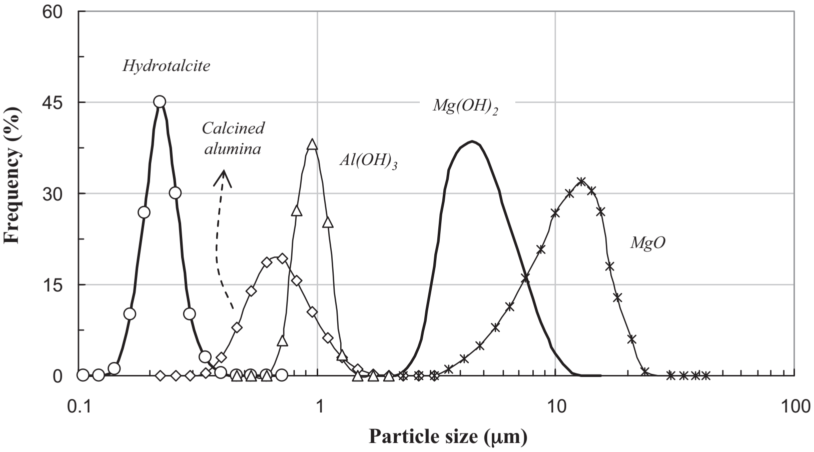

Fig. 6. Particle size distribution of the raw materials tested and of hydrotalcite attained (60 wt.% MgO/40 wt.% $Al(OH)_3$ sample prepared at 80 °C for 7 days).

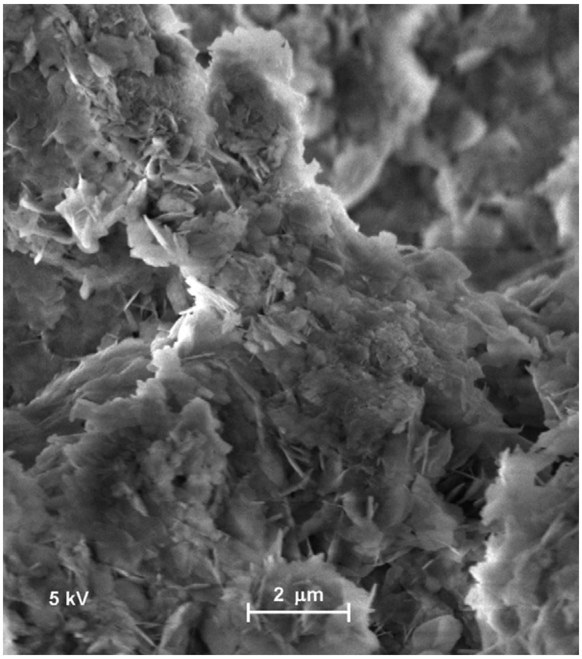

Fig. 7. SEM of the magnesium-aluminum hydrotalcite obtained in this work (sample containing 60 wt.% MgO/40 wt.% $Al(OH)_3$, after 7 days at 80 °C).

observation of these hydroxylated particles reveals a cracked and angular aspect due to the stresses involved [21]. In the present study, however, the regular and highly asymmetric plate-like shape of the MAH particles suggests that there was a co-precipitation [15]. In these systems, this sort of morphology is very common due to the preferential crystal growth in certain crystallographic planes of lower atomic adsorption energy [5,10,14]. These observations corroborate the hydration/dissolution/co-precipitation mechanism suggested for MgO and $Al(OH)_3$.

Considering the production of MAH aiming at its potential use combined with other ceramic raw materials (especially calcined or fused alumina), the methodology described in this paper presents three other important technological aspects: (1) the MgO hydration can be very exothermic, depending on the particles' surface area, the amount of MgO in the reactor and on the heat exchange conditions [23]. Therefore, this effect could be used to keep the MAH reactions at a proper temperature, without external heating. (2) Because the reaction batch comprises only the aqueous suspension of the reactants, the magnitude of the process' output can be designed according to different needs (from a few grams up to several tons). (3) As the MAH is obtained as an aqueous suspension, it could be directly incorporated into the ceramic formulation of refractory castables or insulating linings, for example. These three features are currently under investigation.

4. Remarks

In the present work, a novel method for synthesize magnesium–aluminum hydrotalcite (MAH, $Mg_6Al_2(OH)_{16}(CO_3)\cdot 4H_2O$) from aqueous suspensions of magnesium oxide (MgO) and aluminum hydroxide ($Al(OH)_3$) was presented. The mechanism investigated is based on the hydration/dissolution/co-precipitation reactions that take place simultaneously. Starting from an aqueous suspension of MgO and $Al(OH)_3$, the highly reactive source of MgO (caustic magnesia) initially reacts with water, dissolving MgO and releasing Mg^{2+} ions. It also enhances the suspensions' pH, favoring the hydroxylation and further dissolution of the $Al(OH)_3$ in Al^{3+} ions. The combination of Mg^{2+}, Al^{3+} and $(CO_3)^{2-}$ (dissolved in water during the mixing) in this alkaline environment results the precipitation of the hydrotalcite above 50 °C. This process results in a high loaded cation-free aqueous suspension of very fine ($D_{50} = 0.37$ μm) MAH particles. It was observed that three main aspects strongly affected the efficiency of the process: (1) the raw materials used must be able to hydrate/dissolved in order to release Mg^{2+}, Al^{3+} and OH^- ions. Therefore, the low reactivity ones (calcined alumina and $Mg(OH)_2$) did not yield good performance as precursors, whereas MgO and $Al(OH)_3$ allowed the generation of a compound with 84 wt.% of the mass loss expected for the theoretical hydrotalcite and with high crystallinity. (2) Considering that these two species were consumed in the reaction, compositions close to the hydrotalcite stoichiometry (~61 wt.% of MgO) presented higher quantities of this compound (for other composition with more or less MgO, a mixture of MAH and $Mg(OH)_2$ or MAH and $Al(OH)_3$ were obtained). (3) The higher the testing temperature, the greater the driving force for hydration/dissolution reactions and, therefore, the greater the efficiency of the process. Finally, it is also important to highlight that the presented route resulted in MAH particles with suitable characteristics as a raw material for high temperatures insulating porous ceramics and most likely for the various other applications mentioned in the text.

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