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Novel fire-protecting mortars formulated with magnesium by-products

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ABSTRACT

Several kinds of sprayable mortars are commonly used as passive fire protection of building structures. Several authors have reported the effect of different kinds of aggregates (e.g. vermiculite, fly ashes) in the thermal behaviour of fire-protecting mortars. In this study, the use of magnesium by-products as aggregates in fire-protecting mortars has been evaluated. These by-products were obtained during the calcination process of natural magnesite. Endothermic decompositions of the different aggregates have been determined and analysed by means of thermal techniques. Mortars with different mixtures of these by-products have been prepared. Mechanical properties and temperature behaviour tests have been performed to evaluate the suitability of these substances as aggregates in fire-protecting mortars. During the endothermic decomposition of the studied aggregates the advance of temperature inside the mortar is delayed. Mortar with a mixture of 50% of both magnesium by-products shows a good agreement between mechanical properties and temperature behaviour.

1. Introduction

Fire protection is a topic of increasing interest due to the enforcement of the existing standards as well as to the society awareness of fire risks [1]. Different approaches can be taken in order to protect a building structure depending, among others, on the risk level, the required resistance time and material involved. Intumescent paints and fire-protecting mortars formulated with lightweight aggregates are a common solution to protect reinforced concrete, wood, or metal structures. Nevertheless, some differences should be made between these two products: i) intumescent paints provide a smooth finish, while mortars leave rough surfaces and ii) high resistance times cannot be reached with an intumescent paint.

Conventional fire-proof mortars formulated with vermiculite and/or perlite as lightweight aggregates take benefit of the low thermal conductivity and weight of these minerals as well as their capacity to retain water to obtain a porous insulating mortar easy to spray [2–5]. The initial critical temperature for steel structures is considered to be 400 °C [5–7]. In the case of concrete formulated with Portland cement, it undergoes important changes with temperature: release of free water (around 100 °C) and chemically bonded water (in a range of 100–300 °C), and portlandite decomposition (at approximately 450 °C). During the portlandite decomposition, volume changes can cause cracks in concrete which would contribute both to the decrease in mechanical properties as well as to the increase of thermal flow to the steel in the case of reinforced concrete [8]. Hence, developing new mortar formulations that increase the time needed to reach these temperatures keeping or improving the rest of the aspects would be a great challenge.

Another aspect of interest in the development of new mortars suitable as passive fire protection is the use of industrial wastes or by-products [9–11]. Fire resistance of mortars containing different kinds and contents of fly ashes has been tested successfully. The tests show that the addition of fly ashes increases the mortar insulating capacity and tendency to retain water [12]. This is due to the high porosity and specific surface of the added fly ashes.

This work has been focussed on the development of fire-protecting Portland mortars formulated with magnesium by-products obtained during the calcination of natural magnesite [13]. The possibility to develop new formulations to obtain mortars with enhanced thermal and fire behaviour from industrial by-products would lead to a combination of an economical and sustainable feasible solution for passive fire protection. Two magnesium by-products have been used: a low-grade magnesium hydroxide (LG-MH) and a low-grade magnesium carbonate (LG-MC). Both products undergo endothermic decompositions with the release of water and carbon dioxide in the range of 300–750 °C and 500–800 °C, respectively. The combined effect of heat absorption and water release from LG-MH has been used in the field of flame retardancy of polymers with promising results [14]. It is expected that the mortars formulated with these endothermic magnesium by-products will exhibit longer times to reach the external temperature. The aim of the present study is to evaluate the thermal and mechanical properties of the resulting

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mortars and to establish the optimal formulation to develop a fire-protecting mortar based on LG-MH and LG-MC by-products.

2. Experimental procedure

2.1. Materials

The magnesium by-products were supplied by Magnesitas Navarras S.A. located in Navarra (Spain). The low-grade magnesium hydroxide (LG-MH) is obtained as a result of the weathering with water in an open air storage of the cyclone dust generated during the calcination of natural magnesite ($MgCO_3$), whereas the low-grade magnesium carbonate (LG-MC) is a by-product containing mainly $MgCO_3$ and dolomite ($CaMg(CO_3)_2$) produced during the flotation of the natural magnesite before calcination process [13]. Around 40–50 kg of LG-MH and LG-MC were taken from various stockpiles. The samples were previously homogenized, quartered to a 1/16 split and dried in the oven at 105 °C for 24 h in order to obtain representative sub-samples of about 500 g for physical and chemical characterization. A common Portland cement, CEM I 52,5R (according to EN-197-2000), supplied by Ciments Molins, (Barcelona, Spain), was used as binder.

2.2. Magnesium by-products characterization

2.2.1. Physico-chemical characterization

Magnesium by-products were analysed by X-ray fluorescence (XRF) using a Philips PW2400 X-ray sequential spectrophotometer to elucidate major and minor elements. X-ray diffraction was performed in a Bragg–Brentano Siemens D-500 powder diffractometer with CuKα radiation in order to obtain information about the crystalline phases. Particle size distribution was determined with a laser analyser Beckman Coulter LS 13 320. Density was measured with a helium pycnometer and the specific surface area by the BET single point method with a Micromeritics porosimeter. Environmental Scanning electron microscopy (ESEM) images of the magnesium by-products were obtained with a FEI Quanta 200 microscope.

2.2.2. Thermal analysis

The thermal stability of the magnesium by-products was evaluated by means of thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). TGA and DSC were simultaneously performed in air atmosphere using a TA Instruments SDT Q600 at a heating rate of $10\ °C\ min^{-1}$ from 30 to 1000 °C. For each experiment a mass of $30\ mg \pm 0.5\ mg$ was used and the flow rate of gas was $100\ mL\ min^{-1}$.

2.3. Fire-protecting mortars

2.3.1. Mortar formulations and preparation

Mortars were prepared with Portland cement as binder and magnesium by-products as aggregates. In all experimental series the ratio cement:aggregate (C/A) was fixed at 1:4. Formulations with different combinations of the two magnesium by-products were considered. In all cases, consistency was measured by the flow table test [15], and fixed in the range of 180–200 mm. This range ensures an appropriate consistency and workability for possible future applications such as sprayable or wall coating mortars. Because of the different physicochemical characteristics of both by-products, different water:cement (W/C) ratios were needed for each formulation. The composition of the different mixtures is summarized in Table 1. The reason for the large variation in W/C ratios is explained in Section 3.1.

Mortar formulations were cast in prismatic moulds with dimensions of 40×40×160 mm to perform mechanical properties and cylindrical moulds with diameter of 41 mm and height of 50 mm to carry out the fire tests. Specimens were left in their moulds for 24 h in a curing chamber at a constant temperature of 20 °C and a relative humidity of 95% and the unmolded mortars were further allowed to cure in the same conditions up to 28 days.

Table 1
Studied mortar compositions.

Mortar	(C/A)	Aggregates (%)		(W/C)
		LG-MH	LG-MC	
100LG-MH	1:4	100	0	2.61
75LG-MH	1:4	75	25	2.03
50LG-MH	1:4	50	50	1.77
25LG-MH	1:4	25	75	1.44
0LG-MH	1:4	0	100	1.27

2.3.2. Testing procedures

Density and porosity were determined according to the Spanish standard UNE 83-312-90 based on the Archimedes Principle. Thermal conductivity and thermal diffusivity at room temperature were determined by a transient line-source technique with a Quickline™-30 instrument. Flexural strength and compressive strength tests were performed in a mechanical testing machine MUTC-200 from Incotecnic at a loading rate of $5\ kg\ s^{-1}$ [16]. High-temperature behaviour was evaluated in a furnace designed according to the standard ISO 1182: Non-Combustibility Test. The cylindrical specimens were introduced in the vertical tube of the furnace. During the preparation of each cylindrical specimen, a type K thermocouple was located in its central point. In addition, the system is provided with two more type K thermocouples to measure the furnace temperature and the temperature at the specimen surface.

The samples were tested under two heating programs:

- Slow program: furnace temperature is controlled to increase at constant rate, from room temperature to 800 °C in 2 h, and then maintained at 800 °C for 1 h. Four specimens of each formulation were tested.
- Fast program: furnace temperature is already at 800 °C when the specimen is introduced, and remains at this temperature for 1 h. Four specimens of each formulation were tested. This program is more similar to a case of real fire. In fact, the standard fire curve ISO 834 reaches 800 °C in only 20 min.

3. Results and discussion

3.1. Magnesium by-products characterization

The chemical composition obtained from XRF is given in Table 2 for each determined element as the most stable corresponding oxide. As expected magnesium is the main element in both by-products. The amount of calcium and silicon is also significant. The diffraction patterns of Fig. 1 show that the main compounds constituting LG-MH are brucite ($Mg(OH)_2$), magnesite ($MgCO_3$) and dolomite ($CaMg(CO_3)_2$), although

Table 2
Average chemical composition of magnesium by-products.

Oxides	LG-MH (%)	LG-MC (%)
MgO	49.57	33.51
CaO	7.98	14.36
SiO_2	3.24	4.55
Fe_2O_3	2.13	2.17
Al_2O_3	0.41	0.23
MnO	0.25	0.09
P_2O_5	0.08	0.05
K_2O	0.08	0.03
TiO_2	0.02	0.02
Loss of ignition (LOI)	34.31	45.79

LOI: loss of ignition at 1000 °C.

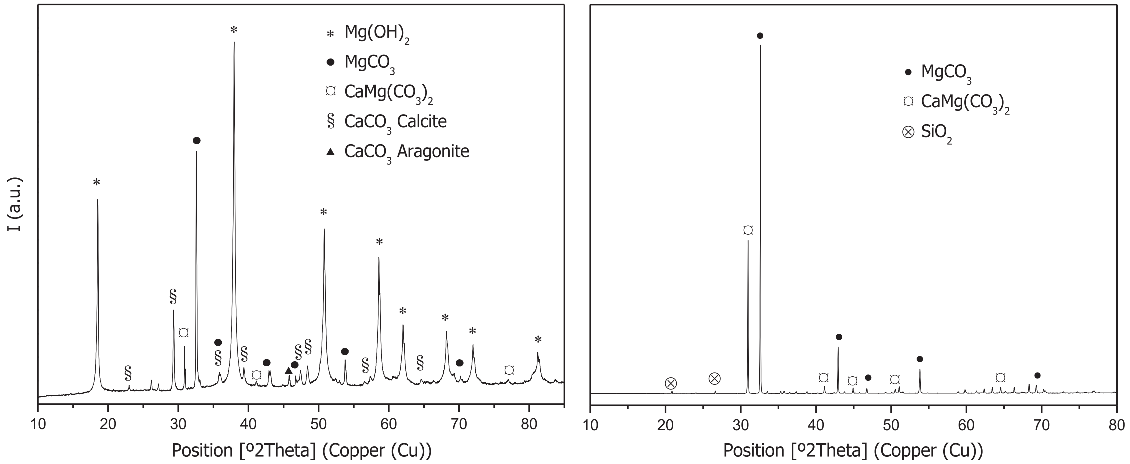

Fig. 1. Diffractograms of the magnesium by-products (LG-MH: left and LG-MC: right).

calcite and aragonite ($CaCO_3$) are also identified, while LG-MC has a composition mainly based on $MgCO_3$ and $CaMg(CO_3)_2$.

The measured density and specific BET surface area were of 2.44 g cm^{-3} and 22.106 m^2 g^{-1} for LG-MH, and 2.96 g cm^{-3} and 1.261 m^2 g^{-1} for LG-MC, respectively. The high specific surface value of LG-MH compared with LG-MC can be explained due to the differences during the production process. As mentioned before, LG-MC corresponds to natural carbonates separated before being introduced in the kiln, showing a low specific surface. However, LG-MH has been collected after suffering a partial calcination in the kiln. This process originates caustic MgO that finally forms $Mg(OH)_2$ with high specific surface. Particle size distributions are presented in Fig. 2. LG-MH shows a wide distribution, reaching the maximum around 100 μm and containing 90% of particles in the range from 5 to 300 μm. The LG-MC distribution is narrower and reaches its maximum at a particle size around 170 μm. Most of the particles, 90%, are in the range from 70 to 350 μm. In both cases, only a small fraction of particles (lower than 5%) is greater than 400 μm. These differences in the size distributions can be visualized in the ESEM images of the two magnesium by-product aggregates presented in Fig. 3. It is worth mentioning that the larger LG-MH particles (Fig. 3-left) could be formed by the agglomeration of the smaller ones. This may be a consequence of the high-temperature process to obtain this by-product. Moreover, a small fraction of submicron particles is observed in both by-products, but is more noticeable in the case of LG-MH. This explains why, as already noted in Section 2.3.1, mortars formulated with a high percentage of LG-MH needed more water than mortars formulated with a high LG-MC percentage. The smaller average particle size in the LG-MH aggregate (Fig. 2) and its high specific surface are responsible of this fact.

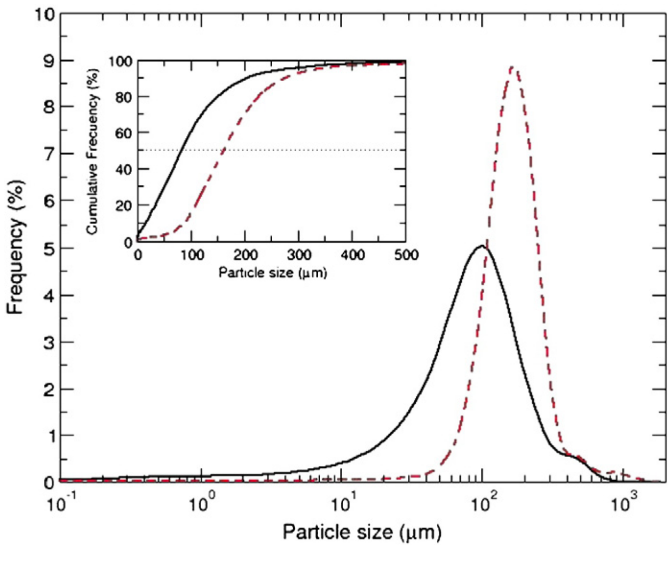

Fig. 2. Particle size distribution of magnesium by-products: LG-MH (black solid line) and LG-MC (red dashed line). Inset shows the cumulative frequency.

The thermogravimetric curves of LG-MH and LG-MC as well as their corresponding derivatives are shown in Fig. 4. In the case of LG-MH, the TG curve shows a weight loss of 35%. In addition to the loss due to water evaporation, three other decreases in weight were observed, which can be attributed to the decompositions of $Mg(OH)_2$, $MgCO_3$ and $CaMg(CO_3)_2$, respectively.

$$Mg(OH)_{2(s)} \xrightarrow{T=320\text{–}420\,^{\circ}C} MgO_{(s)} + H_2O_{(g)} \tag{1}$$

$$MgCO_{3(s)} \xrightarrow{T=500\text{–}600\,^{\circ}C} MgO_{(s)} + CO_{2(g)} \tag{2}$$

$$CaMg(CO_3)_{2(s)} \xrightarrow{T=670\text{–}780\,^{\circ}C} MgO_{(s)} + CaO_{(s)} + 2CO_{2(g)}. \tag{3}$$

In the case of LG-MC, two significant weight loss are observed, which can be attributed to the decompositions of $MgCO_3$ and $CaMg(CO_3)_2$.

The percentages of each weight loss are shown in Table 3. Relating these results with the XRF chemical analysis of the by-products it can be concluded that in both cases the magnesium and calcium come from the hydroxides and carbonates identified in the TGA. The results of differential scanning calorimetry (DSC), shown in Fig. 5, also corroborate these endothermic decompositions. A mixture of both by-products (50% LG-MH and 50% LG-MC) is included in the same figure. It is necessary to point out that, in addition to the endothermic decompositions, an exothermic peak appears in the DSC of LG-MH. This exothermic peak is reported by Formosa et al. in Ref. [17] and it is due to the presence of tars providing from the calcinations of the petcoke used as fuel in the calcination process of LG-MH. This exothermic peak seems to be counteracted as the percentage of LG-MC in the aggregate increases. This fact can be observed in Fig. 5 for the DSC of the mixture 50LG-MH (50% LG-MH and 50% LG-MC) which the exothermic peak has been completely counteracted by the endothermic decomposition of $MgCO_3$ contained in LG-MC.

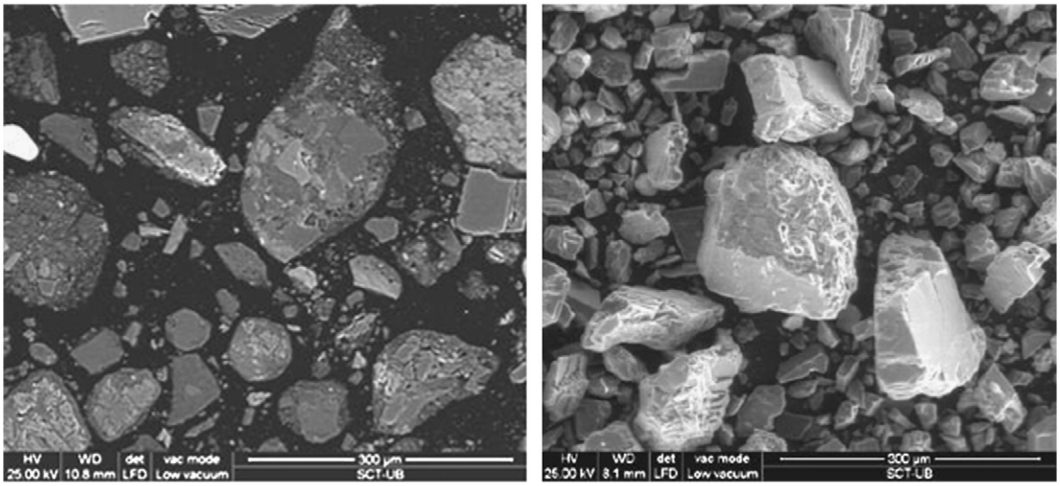

Fig. 3. ESEM images of the magnesium by-product aggregates: LG-MH (left) and LG-MC (right).

3.2. Mortars: physical and mechanical properties

Mortar samples were prepared as stated in Section 2.3.1, according to the formulations referred to in Table 1. As can be observed in the ESEM micrograph showed in Fig. 6, the fracture surface of the mortar 50LG-MH presents a homogeneous distribution of aggregate particles which surface seems to have a good cohesion with the Portland cement matrix. Some physical properties: density, porosity, thermal conductivity and thermal diffusivity were determined at room temperature (see Table 4). It can be observed that an increment in the percentage of LG-MH leads to a higher porosity and, consequently, to lower values of density, conductivity and diffusivity. This fact can be explained by the high amount of water needed in the preparation of the mortars with high percentage of LG-MH. However, it has to be taken into account that the density range determined in the specimens formulated with magnesium by-products is slightly high to be used as wall coating mortars.

Compressive Strength (CS) and Flexural Strength (FS) of mortars are illustrated in Fig. 7. It is observed that CS and the FS decrease with increasing LG-MH content, as was expected because of their higher porosity. In order to obtain the same consistency and workability in all mortars, the W/C ratio was significantly higher for mortars with LG-MH aggregate due to its elevated specific surface. The great amount of water needed to obtain the desirable consistency is not used to form the hydrated phases of Portland cement and remains adsorbed into the particle pores. During the curing process, a fraction of this water is desorbed and removed by capillarity towards external surface particles, decreasing the cohesion with the binder matrix, increasing the porosity of mortars and, as a consequence, decreasing the mechanical properties. As can be observed the W/C ratio and the porosity decreases as the percentage of LG-MC aggregate increases obtaining CS values around 8–9 MPa for the mortar 0LG-MH (see Fig. 7) which are in accordance to the standard UNE-EN 998-1:2003.

3.3. High-temperature behaviour

Cylindrical specimens of the different mortar formulations were tested in the non-combustibility furnace. Figs. 8 and 9 show the temperatures, measured at the central point of each specimen, corresponding to the slow program (Fig. 8) and the fast program (Fig. 9). In the profile of the curves shown in this type of graph, each endothermic decomposition identified in the TGA/DSC analysis (see Figs. 4 and 5) is seen as a flat region or *plateau*. During the endothermic decompositions mortars take heat from the environment keeping the temperature constant into the specimens forming a *plateau*. The first *plateau* occurs at around 100 °C and corresponds to water evaporation. This is longer for specimens with a higher percentage of LG-MH due to the higher W/C ratio. At about 400 °C, there is a second *plateau* associated with the decomposition of the $Mg(OH)_2$ contained in the LG-MH aggregates. Thus, the length of this plateau increases as the LG-MH contents increase. $MgCO_3$ is present in both by-products, although with a higher percentage in the case of LG-MC. Therefore, one *plateau* at about 600 °C was expected for all the formulations, being longer for higher LG-MC contents. However, due to the exothermic reaction previously observed in the DSC analysis of LG-MH (Fig. 5), this *plateau* disappears at high percentages of LG-MH. The exothermic effect even produces a significant increase in temperature in the 100LG-MH sample, and a smaller effect in the 75LG-MH samples. However specimens 50LG-MH and below do not exhibit any indication of

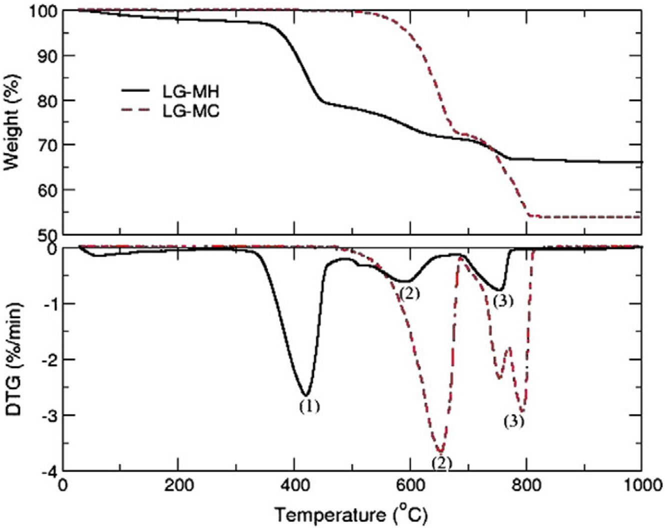

Fig. 4. TG results and their derivatives for LG-MH (black solid line) and LG-MC (red dashed).

Table 3
Thermogravimetric analysis (TGA). Loss of weight associated to the different compounds.

Compound	LG-MH (%)	LG-MC (%)
H_2O	3	0
$Mg(OH)_2$	18	0
$MgCO_3$	7	28
$CaMg(CO_3)_2$	6	18
Total	34	46

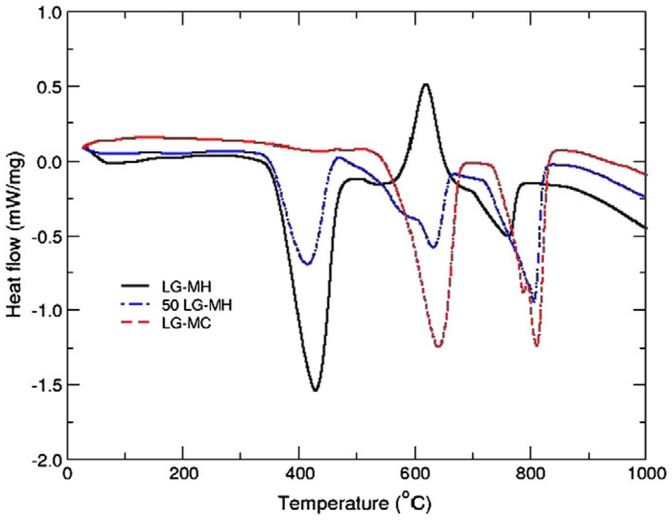

Fig. 5. DSC for LG-MH (black solid line), LG-MC (red dashed line) and a mixture of LG-MH and LG-MC (blue dot-dashed line).

the exothermic reaction. Above 800 °C a similar behaviour is expected attributable to $CaMg(CO_3)_2$ decomposition.

Both Figs. 8 and 9, indicate that mortars with high LG-MH content behave better at lower temperatures, and mortars with high LG-MC content do so at higher temperatures. In order to quantify this fact, the time required for each formulation to reach a fixed temperature has been determined. Results for the fast program are shown in Fig. 10.

It can be observed that, up to 600 °C, the curves grow as the LG-MH percentage increases, which means a better behaviour as a fire-protecting mortar. Above 600 °C, the decreasing curve indicates a better behaviour for lower LG-MH percentages. Thus, considering the whole temperature range, formulations 50LG-MH and 75LG-MH would be the most suitable for fire-protecting applications. Furthermore, previous studies comparing mortars formulated with these magnesium by-products as aggregates with that formulated with vermiculite as aggregate, demonstrated that the presence of the by-products led to greater delay times [18].

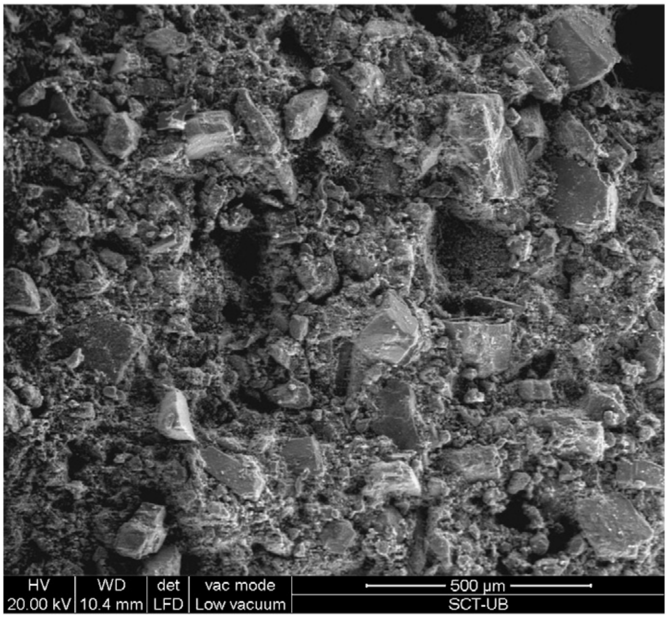

Fig. 6. ESEM micrography from the fracture surface of the 50LG-MH mortar.

Table 4
Mortars formulated with magnesium by-products. Physical properties.

Mortar reference	Density (g/cm^3)	Porosity (%)	λ (W/mK)	α ($\times 10^{-6}$) (m^2/s)
100LG-MH	2.46	56.7	0.24	0.17
75LG-MH	2.48	50.3	0.32	0.22
50LG-MH	2.56	46.3	0.41	0.30
25LG-MH	2.65	41.8	0.65	0.40
0LG-MH	2.74	37.1	1.47	0.89

4. Conclusions

The magnesium by-products used in this work are formed by different hydroxides and carbonates that undergo endothermic decompositions in the range between 300 and 800 °C. One of them, LG-MH, can be considered as a low-grade magnesium hydroxide, while the other, LG-MC, is mainly constituted of magnesium carbonates. These by-products are obtained in different stages of the industrial calcination process of natural magnesite. As a result of all these differences, the specific surface of LG-MH is significantly higher than that of LG-MC. Therefore, mortars formulated with LG-MH require higher W/C ratios, which leads to a decrease of their mechanical properties.

Despite the exothermic peak exhibited by LG-MH around 600 °C, it is possible to formulate fire-protecting mortars combining LG-MH and

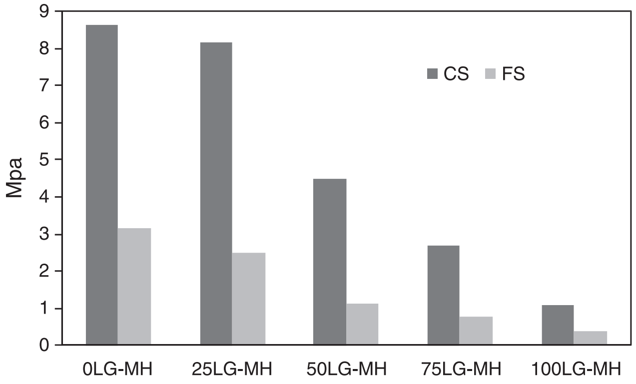

Fig. 7. Mechanical properties of mortar formulations at 28 days. (CS: Compressive Strength; FS: Flexural Strength).

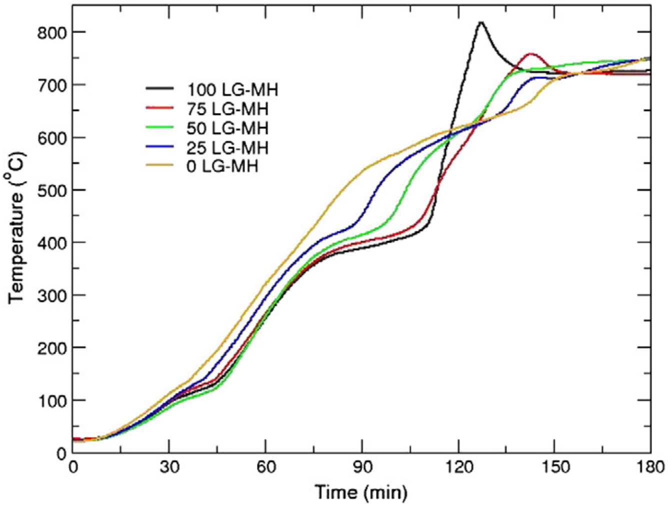

Fig. 8. Temporal recording of the internal temperatures, in samples tested under the slow program in the non-combustibility furnace.

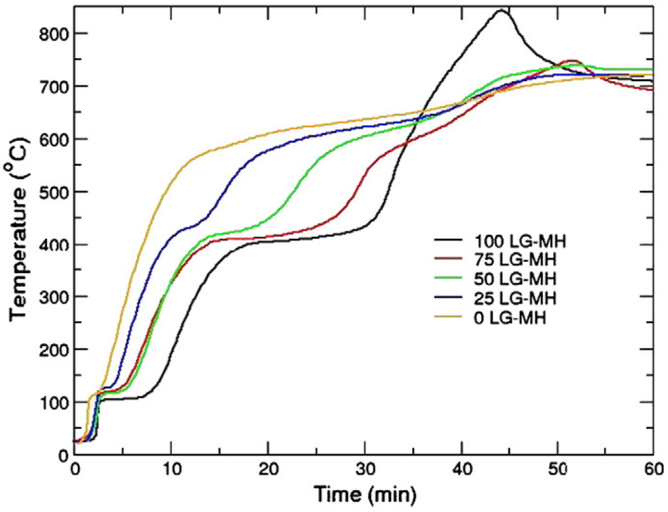

Fig. 9. Internal temperatures, as in Fig. 8, but for fast program.

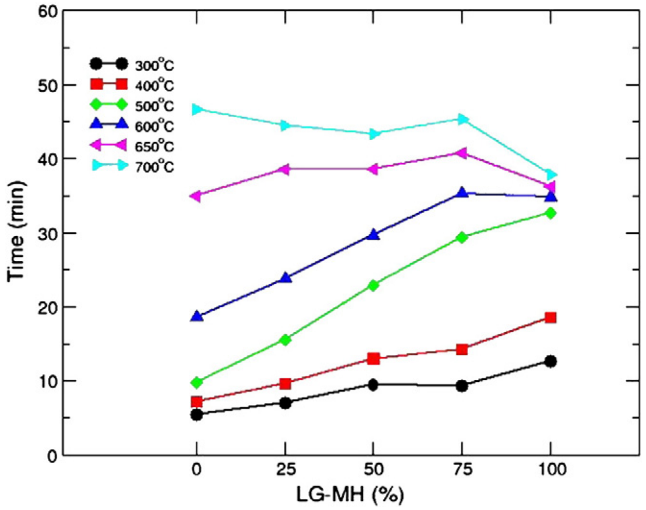

Fig. 10. Time at which a fixed temperature is reached, as LG-MH percentage increases.

LG-MC. Furthermore, mixtures of these aggregates give rise to mortars with a good agreement of mechanical and thermal properties. Mortar 50LG-MH has the optimal formulation considering both mechanical properties and temperature behaviour.

It can be concluded that the magnesium by-products evaluated in this study can be employed as aggregates in fire-protecting mortars. However, better mechanical properties would probably be obtained if only a partial substitution is performed. The combination with light aggregates would help to reduce the mortar density.

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