



Effect of metakaolin dispersion on the fresh and hardened state properties of concrete

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ABSTRACT

The use of pozzolanic materials such as metakaolin in mortars and concretes is growing. Their use is usually related to the promotion of hydraulic binder reactions or to the mitigation of expansive reactions that can occur in concrete. Introduction of fine particles such as metakaolins, can have a strong effect on fresh and hardened state properties. This paper aims to study the effect of metakaolin in concrete formulations with a preset workability and to assess the system rheology but also its hardened state properties such as mechanical strength. The effect that the dispersion of metakaolin particles induces on concrete microstructure, particularly in porosity, is discussed. Formulations were prepared with several metakaolin amounts and workability was controlled either with water or a high range water reducer admixture (HRWRA). The use of HRWRA can cause deflocculation of metakaolin particles, allowing workability control in concrete and leading to better efficiency and improved performance.

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1. Introduction

The mortar and concrete development, with special admixtures or with additions such as pozzolanic materials, allows the improvement of the product quality towards specific requirements of modern construction. In this context, further research is needed for mortar and concrete development or optimization.

Nowadays, the use of pozzolanic materials is widely accepted as partial replacement of Portland cement in mortar and concrete production. Amorphous silica is the main component in pozzolanic materials. This amorphous component, in the presence of water, reacts with calcium hydroxide ($\text{Ca}(\text{OH})_2$) to form compounds with cementitious properties. The effectiveness of a pozzolanic material depends on its reactivity, which can depend on two factors, namely, the maximum amount of calcium hydroxide with which the pozzolan can react and, the speed at which the pozzolanic reaction occurs, which is directly related with the material fineness [1]. Because these materials are very thin, they may also present a filler effect, promoting a decrease in the system's total porosity due to the filling of capillary pores and of the interface transition zone (ITZ) between the aggregate and the cement matrix, where the porosity is higher.

Apart from microsilica and fly ashes, metakaolin (MK) is one of the pozzolanic materials that have been most studied in recent times. MK is an artificial pozzolan obtained from the calcination of kaolinitic clays at temperatures around 700–850 °C. Due to its high pozzolanic

activity, the inclusion of MK improves the mechanical properties and durability of concrete [2]. MK presents, as its main component, amorphous silica but also amorphous alumina that in presence of water, react with calcium hydroxide (CH), mainly producing calcium aluminate hydrates and aluminiumsilicate hydrates (CAH and CASH, respectively). A research about the influence of MK on the mortar and concrete properties shows several advantages, specifically an increase of mechanical strength and durability, but also a decrease of shrinkage due to the increase of material density and a better particle packing [3].

Wild and Khatib [4] consider the MK as a different material compared with other pozzolans, not only because of its high pozzolanic reactivity, but also due to its capacity to accelerate the cement hydration reaction. Other authors [5,6] agree that MK seems to have a catalytic effect on the cement hydration, accelerating this reaction.

Most of the work that has been done on the replacement of cement by MK shows that the use of this pozzolanic material leads to improvements in the behaviour of mortar and concrete. The calcium silicate hydrates (CSH) are formed as a gel that penetrates pores, promoting porosity refinement due to the decrease in average pore size. This effect is also observed in the interfacial transition zone (ITZ) between the binder and aggregate, resulting in densification. The refinement of pores and densification of ITZ can justify improvements in the mechanical strength and reduction of capillary water absorption, improved chemical resistance and increased durability [3,6–9].

All these authors agree that the use of MK also promotes a decrease in workability. This effect requires an increase in the amount of mixing water or the use of high range water reducing admixture (HRWRA). The water reducing admixtures improve the workability

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Table 1
Aggregate particle size distribution.

Sieve size (mm)	Natural siliceous sand	Crushed aggregate	Crushed aggregate
31,5	100.0	100.0	100.0
16	100.0	100.0	24.5
8	100.0	100.0	0.6
4	100.0	59.9	0.5
2	99.8	12.2	0.5
1	98.8	4.9	0.5
0.500	82.9	3.8	0.5
0.250	19.4	3.3	0.5
0.125	1.3	0.9	0.4
0.063	0.5	0.3	0.0

Table 2
Metakaolin chemical analysis.

Oxide	Percentage	Oxide	Percentage
SiO ₂	55%	TiO ₂	1.5%
Al ₂ O ₃	40%	CaO + MgO	0.3%
K ₂ O + Na ₂ O	0.8%	Loss on ignition	1%
Fe ₂ O ₃	1.4%		

at a given amount of mixing water or lead to the same workability with a great reduction in water content. The increase in the system fluidity due to the addition of a high range water reducing admixture (HRWRA) is a consequence of its adsorption on the particle surface, which deflocculate, releasing water to lubricate the system and facilitate the air expulsion retained inside the particles agglomerates [1,10,11].

The multiple effects of this addition and admixtures clearly justify further studies. Hence, this work aims to analyse the effects of MK, as a pozzolanic material, on the rheological behaviour and in the hardened state properties of concrete. The joint effect of a high range water reducer admixture (HRWRA) is also discussed.

2. Experimental

2.1. Materials

In this study, the formulation for the control concrete mixture was determined using the Faury method. The control concrete mixture (B) constitution involves Portland cement (CEM type I 42.5R) as a binder, a siliceous natural sand and two types of crushed limestone as aggregate, whose particle size distributions are presented in Table 1.

MK was used as a partial cement substitute in contents of 10, 20 and 30 wt.%. This material is a dehydroxylated aluminium silicate, with a general formula of Al₂O₃·2SiO₂. It is an amorphous non-crystallized material, constituted of lamellar particles. This MK presents a pozzolanic index (measured by the Chapelle test) of 1100 mg Ca(OH)₂/g of MK

and a specific surface area (BET) of 17 m²/g. Table 2 presents the MK chemical analysis.

The determination of the particle size distribution of MK was made using Coulter LS300 equipment [12]. The material is placed in an aqueous solution and is dispersed, using a deflocculating agent, in order to separate and determine the real particle size distribution. Particle size distribution of MK was also measured without deflocculant.

Table 3 shows the different compositions studied. To ensure an approximately constant workability, all the compositions were prepared to achieve a slump (Abrams cone method according to NP EN 12350–2) of 9 ± 1 cm (height reduction). The water reducer admixture (HRWRA) is based in polycarboxylic acid, with a density between 0.67 and 1.1 and solid contents between 28.5 and 31.5%.

The concrete samples in this study were referenced as **B_xMK_yW_zWR**, where B means basic concrete mixture, x is the replacement mass percentage of cement by MK; y is the water/binder (w/b) ratio, where the binder is the total of cement and MK content, and z is the amount of HRWRA as mass% of total solids.

The samples dimensions for mechanical tests were 100 × 100 × 100 mm and they were cured in a chamber at 20 °C and 95% of relative humidity (RH).

2.2. Characterization tests

For characterization, samples were prepared with a mixing procedure performed in a mixing machine suitable for the production of small amounts of concrete (35 kg of solids). This equipment is a pan mixer with a capacity of 50 l which has vertical shaft that attain a speed of 55 rpm. The concrete samples mixing procedure involved the following steps: (i) placement of the required water volume, including the admixture (HRWRA) in the mixing recipient (ii) addition of the binder (cement plus MK) in the water (iii) 2 min mixing at a constant speed (iv) addition of the aggregates (sand and two limestones), with the mixer always working, for a total time of 5 min.

The rheological characterization was made immediately after the concrete mixing process on a compact rheometer (Fig. 1) proper for measuring fresh concretes (Schleibinger BT2 rheometer [13]). The container of the rheometer has a diameter of 30 cm and a depth of 10 cm, allowing a concrete sample volume of 20 l. In the container centre there is a support where the rheometer measuring head is placed for rotation. This measuring head has two 90 mm long pins, located at different distances of the head centre (75 and 175 mm), allowing measurements at different angular velocities. This concrete rheometer allows determination of the material relative yield stress and viscosity from momentum and angular velocity measurements [13,14].

A characteristic Bingham fluid presents a relation of torque (T) with rotation speed (N) such as $T = g + hN$, where g (N.mm) and h (N.mm.min) are coefficients related to yield stress and plastic viscosity, respectively [15–18]. Together with the rheometer tests, concrete samples workability was also assessed by the slump measured by the Abrams cone method.

Table 3
Different studied concrete formulations.

Composition (% mass)	Cement (%)	MK (%)	Sand (%)	Limestone A (%)	Limestone B (%)	Water (w/b)	HRWRA (% total solids)
B_0.6W	16.90	0.00	20.40	43.10	19.60	0.60	0.00
B_10MK_0.65W	15.21	1.69	20.40	43.10	19.60	0.65	0.00
B_10MK_0.6W_0.08WR	15.21	1.69	20.40	43.10	19.60	0.60	0.08
B_10MK_0.6W_0.1WR	15.21	1.69	20.40	43.10	19.60	0.60	0.10
B_20MK_0.7W	13.52	3.38	20.40	43.10	19.60	0.70	0.00
B_20MK_0.6W_0.15WR	13.52	3.38	20.40	43.10	19.60	0.60	0.15
B_30MK_0.75W	11.83	5.07	20.40	43.10	19.60	0.75	0.00
B_30MK_0.6W_0.2WR	11.83	5.07	20.40	43.10	19.60	0.60	0.20

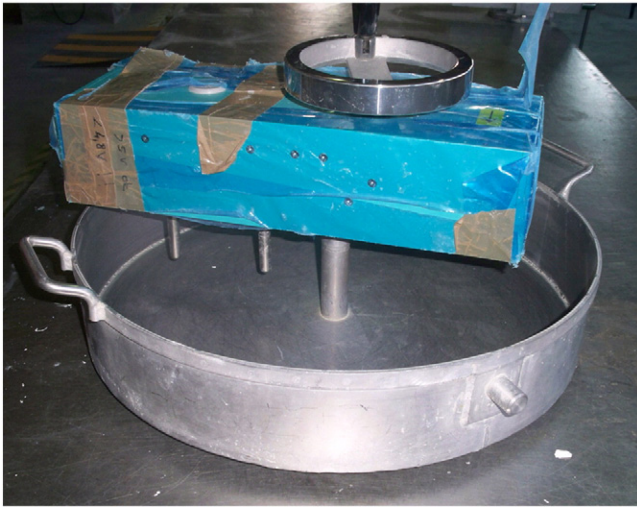


Fig. 1. Concrete rheometer (Schleibinger BT2).

The evaluation of hardened state properties involved compressive mechanical strength test according to NP EN 206-1, using a testing machine (FORM+TEST type Beta 2 3000D) on three samples (100×100×100 mm) of each composition. Porosity measurements were also carried out on hardened samples by mercury intrusion porosimetry (MIP), which allows assessing not only the total porosity but also the pore size distribution. Samples for porosimetry analysis presented a mass between 1.5 and 3 g. Their preparation involved the separation of the mortar part from the rest of the concrete (coarse aggregate). Only the mortar part was analyzed.

Complementary, thermal analysis (differential and thermogravimetric analysis) was also used to calculate the consumption ratio of $\text{Ca}(\text{OH})_2$ in the concrete due to the MK introduction. Thermal analysis samples had a mass of 3 mg. Small portions of various locations were removed from the large concrete sample, disaggregated together and passed through a 63 μm sieve. Thus, the aggregate is removed and only the cementitious matrix was analyzed.

3. Results and discussion

Several aspects must be considered in order to achieve this work objective, the study of the effect of MK on the fresh and hardened state properties of concrete. One of them is the ability to disperse the MK particles throughout the concrete matrix in order to obtain the best results. As a matter of fact, pozzolanic fine particle systems like MK tend to agglomerate and this may lead to changes in the fresh and hardened state properties of mortars and concretes. Fig. 2 shows the MK particle size distribution with and without the use of a dispersing agent.

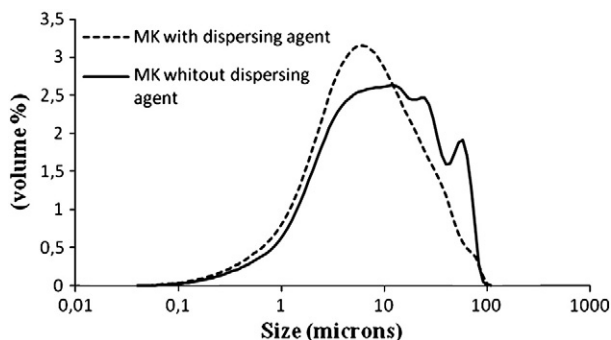


Fig. 2. Metakaolin particle size distribution with and without a dispersing agent in water.

Table 4
Rheological parameters (h, g) and slump for concrete formulations.

Compositions	Slump (cm)	h (N.mm.min)	g (N.mm)
B_0.6W	8.5	4689	702
B_10MK_0.65W	8.0	4704	533
B_10MK_0.6W_0.08WR	8.0	5643	761
B_10MK_0.6W_0.1WR	10.0	4549	527
B_20MK_0.7W	9.5	4873	427
B_20MK_0.6W_0.15WR	8.0	4648	836
B_30MK_0.75W	9.0	3878	479
B_30MK_0.6W_0.2WR	8.5	5001	500

When no dispersing agent is used the particle size distribution presents a deviation towards larger sizes due to the presence of agglomerates. A variation towards smaller sizes is observed on particle size distribution due to the agglomerates dispersion by a deflocculating agent. This observation can present implications on the fresh and hardened state behaviour when cement is replaced by MK in the concrete formulations.

Table 4 shows the results on workability (slump) and rheological parameters (g and h) evaluated for the different studied concrete formulations.

In these compositions, where the MK content was varied, the water content (W) and the admixture content (HRWRA) were used to keep the workability at a constant slump of 9 ± 1 cm. One can observe that when MK content is increased by replacing the cement, the water or the HRWRA content must be increased in order to keep workability constant. When increasing HRWRA content, one can also reduce the water content simultaneously, since these admixtures are water reducing admixtures.

It is possible to observe that the rheological parameters are sensitive to changes in the slump (Fig. 3). The g and h values behave in an opposite way regarding the slump value. An increase in viscosity (h) or yield stress (g) reflects as a decrease of slump, since the material became more rigid or less fluid.

Regarding the use of HRWRA, their main action is to reduce yield stress allowing a reduction of mixing water content but maintaining the workability [19–23]. However, the cement chemical composition,

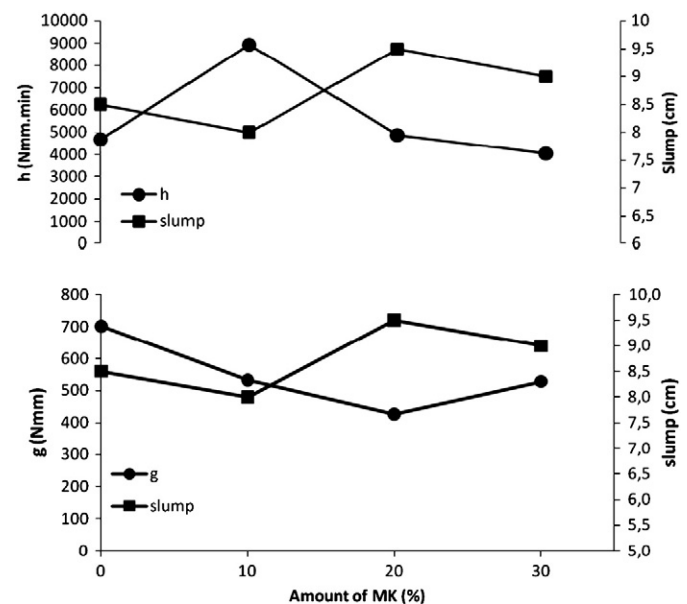


Fig. 3. Relation between slump and rheological parameters for compositions with different MK contents. Workability was kept at 9 ± 1 cm of slump by the water content (w/b ratio of 0.6, 0.65, 0.7 and 0.75 respectively for 0, 10, 20 and 30% MK concretes).

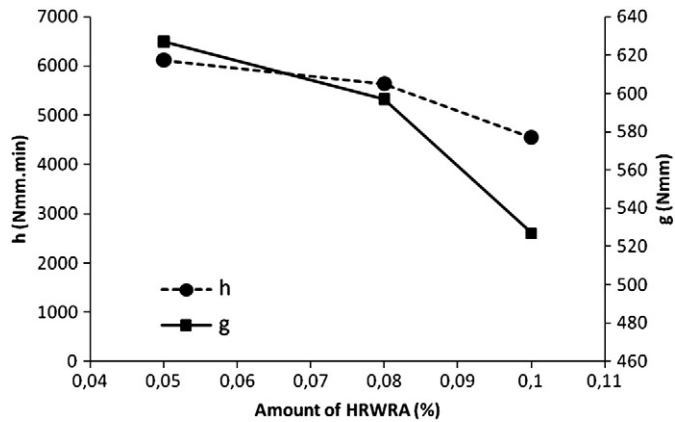


Fig. 4. Effect of HRWRA amount on a composition with a w/b ratio of 0.6 and 10% of cement replacement by MK.

its specific surface area, the presence of other admixtures as well as the hydrate phases, formed during early hydration, affect the behaviour of HRWRA in pastes, mortars and concretes [24]. Some authors [25–27] show that the presence of HRWRA can also decrease plastic viscosity. In what concerns the role of pozzolanic materials on mortars and concrete properties, Galias [28] concludes that, without the use of a HRWRA, those fine particle materials do not contribute to the compaction of the cementitious matrix.

In order to better understand the admixture effect, Fig. 4 shows the effect of HRWRA amount (0.05, 0.08, 0.10% WR) increase in a composition with the same amount of water and the same amount of MK, regardless of the workability. The increase in the system fluidity due to the addition of a water reducing admixture (HRWRA), promotes a decrease on the rheological parameters (h and g), since HRWRA adsorption on the particles surface causes its deflocculation and separation. In this way there is a higher relative amount of water to lubricate the system that becomes more fluid.

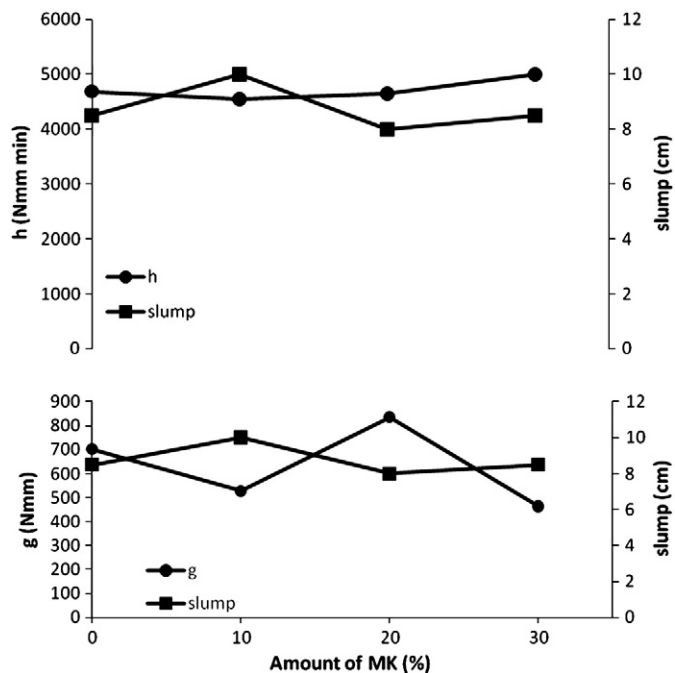


Fig. 5. Relation between slump and rheological parameters for compositions with different MK contents and same w/b ratio (0.6). Workability was kept at 9 ± 1 cm of slump by the HRWRA content (0, 0.10, 0.15 and 0.20% HRWRA respectively for 0, 10, 20 and 30% MK concretes).

Table 5
Mechanical compression strength at 7 and 28 days.

Compositions	Rc (7 days) (MPa)	Rc (28 days) (MPa)
B_0.6W	32.8 ± 0.5	38.1 ± 0.9
B_10MK_0.65W	32.3 ± 0.6	37.0 ± 0.6
B_10MK_0.6W_0.08WR	34.6 ± 0.2	39.8 ± 0.4
B_10MK_0.6W_0.1WR	32.9 ± 0.3	42.3 ± 0.7
B_20MK_0.7W	26.3 ± 0.6	34.3 ± 0.7
B_20MK_0.6W_0.15WR	37.5 ± 0.5	44.6 ± 0.3
B_30MK_0.75W	20.2 ± 0.7	30.3 ± 0.8
B_30MK_0.6W_0.2WR	36.5 ± 0.5	45.1 ± 0.3

Observing in Fig. 5 the effect of MK content (0 to 30%MK) on compositions with the same workability and mixing water (0.6w/b), it is possible, with the adequate HRWRA amount (from 0.08 to 0.2%) to control the rheological parameters and slump in order for them to be fairly constant. Indeed, Fig. 5 shows that it was possible to keep workability (slump values of 9 ± 1 cm) as well as the h parameter at a range from 4500 to 5000 N.mm.min and the g parameter between 500 and 850 N.mm.

Regarding the effect of MK on the hardened state properties of concrete, Table 5 shows the results of the mechanical compression test performed on the several concrete formulations.

As described above, the MK particles are very small, with high surface energy, and so they tend to agglomerate. The agglomerates are a random group of particles, with low compacity and retained air inside. Due to this, the agglomerates act as large particles with lower reactivity. Thus, in these conditions, the decrease of the compressive strength with the increase on the MK content is easy to understand due to the presence of these agglomerates, when no dispersing admixture (HRWRA) is used in the formulations (Fig. 6), and also because the increase in water content is needed to compensate the MK increase.

Fig. 7 shows the same results but with concrete formulation where HRWRA was added to control workability and, as a simultaneous result, to disperse the MK fine particles. In this case, the values of mechanical strength become higher than those of the compositions without HRWRA, proving the importance of the MK particles' dispersion.

First, it must be noticed that, when MK content was increased, higher amounts of HRWRA were used to maintain workability and ensure the needed dispersion of MK particles. Second, from Fig. 7, it is also possible to conclude that mechanical strength increases by the introduction of MK, although cement is being strongly replaced (up to 30 wt.%). This is due to the high pozzolanic activity of this MK that contributes to keep the mechanical strength of these concretes.

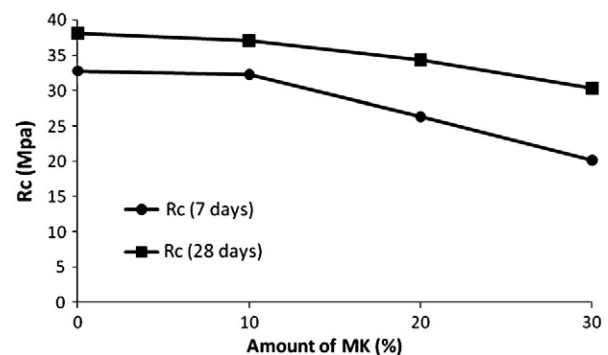


Fig. 6. Compressive strength values (7 and 28 days) for compositions with different amounts of MK. The workability was kept constant by changing water content.

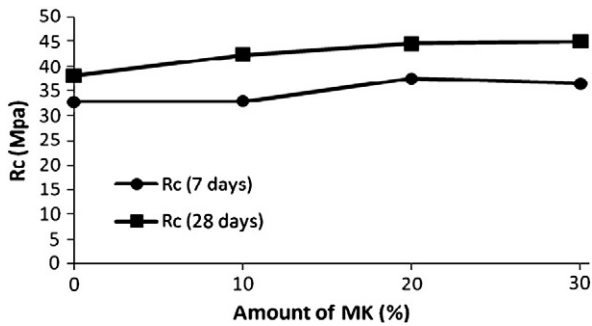


Fig. 7. Compressive strength values (7 and 28 days) for compositions with different amounts of MK but the same water content. The workability was kept constant by changing HRWRA content.

The variation on mechanical strength observed with the increase on MK content on concrete samples is also thought to be related to porosity variation in these samples' microstructure. Hence, porosimetry measurements were performed to assess this relationship. Table 6 shows porosity and mean pore size for concrete samples prepared in this work with 7 and 28 days of curing.

It is possible to observe that, in the concrete samples without HRWRA, there is an increase in total porosity with the increase in MK and water contents, which are responsible for the observed variation of mechanical strength reported in Fig. 6. Furthermore, it is possible to confirm that the presence of HRWRA in the formulation acts as a dispersing agent for MK particles leading to lower values of porosity in the MK containing concretes. However, these HRWRA-containing compositions still present higher porosity values than the control concrete mixture (B_0.6W sample). This effect may be explained by the fact that the HRWRA content (set to get desired slump workability) is not enough to separate all particles and some MK is still agglomerated, with retained air inside responsible for the higher porosity. Nevertheless, even in such conditions, the partial cement replacement by MK in HRWRA-containing formulations is quite viable, even presenting some mechanical strength increase with the MK content.

This role, played by MK content, is governed by its pozzolanic activity. To follow this, complementary thermal analysis was performed and it was possible to observe that a strong consumption of $\text{Ca}(\text{OH})_2$ exists in the MK containing concrete. The pozzolanic reaction of the calcium hydroxide with an aluminium silicate such as MK generates compounds of hydraulic nature that contribute to the mechanical strength, preventing its decrease even with such a reduction in cement content.

Fig. 8 shows an example of thermal gravimetric and differential thermal analysis of concrete samples where, besides other reactions, the presence and amount of $\text{Ca}(\text{OH})_2$ can be followed by its decomposition peak around 450–500 °C. Table 7 presents the calculated amounts of $\text{Ca}(\text{OH})_2$ from thermal analysis for a series of concrete

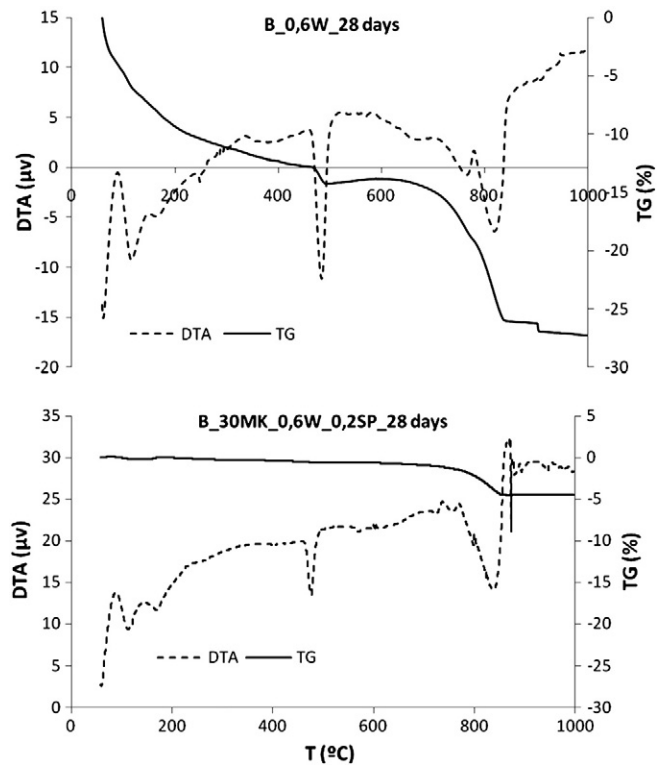


Fig. 8. Differential and thermal gravimetric analysis on concrete samples containing 0 and 30 wt.% MK at 28 days.

samples. Wild and Khatib [29] state that the portlandite ($\text{Ca}(\text{OH})_2$) content generated by the hydration of cement pastes and mortars is around 15 to 20% (relative to the mass of cement) but, this value also depends on curing time, water/cement ratio and is highly influenced by the local environment when aggregates are present. These authors refer that the aggregate influence is still not well understood in terms of portlandite production during hydration. Nevertheless, the values on Table 7 for the basic concrete are in the cited range.

From these results, it is possible to observe that $\text{Ca}(\text{OH})_2$ amount falls considerably with the introduction of MK, confirming its high pozzolanic activity even in less favourable conditions where no HRWRA is used and agglomerates are present. An increase in this activity is obtained when HRWRA is used, confirming the importance of breaking the MK particle agglomerates.

4. Conclusions

MK is one of the artificial pozzolanic additives that are currently used in many mortar and concrete formulations. Its role and its effects are highly dependent on its activity but also on formulation features such as workability and also on the way it is mixed in the formulation. In this work, the role of MK was evaluated in terms of the concrete fresh and hardened state properties. Regarding this role, the effects on the fresh state were discussed for a preset range of workability,

Table 6
Porosimetry results (total porosity and mean pore diameter) at 7 and 28 days.

Compositions	Average pore diameter at 7 days (μm)	Average pore diameter at 28 days (μm)	Porosity 7 days (%)	Porosity 28 days (%)
B_0.6W	0.10	0.04	13.65	8.56
B_10MK_0.65W	0.03	0.07	14.47	13.32
B_10MK_0.6W_0.08WR	–	0.05	–	11.01
B_10MK_0.6W_0.1WR	0.04	0.04	12.89	10.02
B_20MK_0.7W	0.03	0.05	17.15	17.64
B_20MK_0.6W_0.15WR	0.04	0.03	13.32	10.08
B_30MK_0.75W	0.04	0.03	16.21	17.22
B_30MK_0.6W_0.2WR	0.04	0.04	13.19	11.93

Table 7
Calculated $\text{Ca}(\text{OH})_2$ amount from thermal analysis.

Formulation	Curing time	$\text{Ca}(\text{OH})_2$ content (% by mass of cement)
B_0.6W	7 days	11.9
B_0.6W	28 days	15.0
B_30MK_0.75W	28 days	5.1
B_30MK_0.6W_0.2WR	7 days	5.5
B_30MK_0.6W_0.2WR	28 days	1.6

as is usually demanded in practical situations. The consequences in the hardened state were also assessed.

HRWRA and water content were used to preset workability (slump) and rheology analysis was performed with a specific concrete rheometer that allows the determination of two rheological parameters (yield stress and viscosity). These parameters are sensitive to slump variations. HRWRAs can cause deflocculation on fine particle systems such as MK and affect the water content available to lubricate the flowing system.

For compositions with no HRWRA, where slump was controlled by increasing water content, there is a decrease of mechanical strength and an increase of porosity due to the presence of agglomerates and water content increase. The fact that no greater decrease on strength was observed was due to the pozzolanic activity of the MK that was high even when agglomerates are present.

When HRWRA is used, dispersion of MK agglomerates is more effective and this reflects on fresh and hardened state properties. In this case, water content was always the same and porosity was lower and mechanical strength increased even with MK replacing the cement up to 30 wt.%. This was due to dispersion of MK and its pozzolanic activity. This was followed by the $\text{Ca}(\text{OH})_2$ content, determined by thermal analysis, that was the lowest when the HRWRA was used in concrete formulations.

This work allowed concluding on the relevance of using HRWRA to control workability in concrete formulations including MK, or other fine particle systems, in order to achieve a good dispersion and a better efficiency.

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