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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.

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 ($Ca(OH)_2$) 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_2O_3	40%	$CaO + MgO$	0.3%
$K_2O + Na_2O$	0.8%	Loss on ignition	1%
Fe_2O_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 $Al_2O_3.2SiO_2$. 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 m^2/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	Cement	MK	Sand	Limestone	Limestone	Water	HRWRA
(% mass)	(%)	(%)	(%)	A (%)	B (%)	(w/b)	(% 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