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POZZOLANIC ACTIVITY OF METAKAOLIN UNDER MICROWAVE TREATMENT

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ABSTRACT

Calcium hydroxide produced by cement hydration decreases durability of concrete placed in aggressive solutions and fibre-reinforced cement composites. Pozzolanic properties of metakaolin allow to avoid this drawback.

In the present study the effect of a microwave curing on lime consumption in metakaolin blended cements was investigated. The microwave treatment conditions were assessed for plain portland cement pastes, then the lime consumption for various metakaolin contents was evaluated by Fourier transform infrared spectrometry and differential thermal analysis. The results were compared with those obtained for room temperature curing.

It was found that the pozzolanic activity of metakaolin increased under microwave treatment. Binders containing 15% metakaolin were free of calcium hydroxide, while 30 or 40% metakaolin were needed at room temperature curing.

Introduction

Thermal treatments or chemical accelerators have been used for several years to reduce the curing period of concrete, especially in precast processes to increase productivity and optimize workshop areas.

Schneider and al. (1,2) used microwave heating to accelerate hydration of cementitious materials. Microwave-treated specimens showed homogeneous structure, high strength, and low porosity, compared to identical oven-dried ones. The microwave technique provides a shorter curing period and a better quality of concrete compared with traditional thermal curing. This was confirmed by Xuequan and al. (3,4), and Hutchinson and al.(5).

As heat is generated quickly inside the material, principally from the dielectric losses of water under the electromagnetic field, homogeneous moderate temperatures can be obtained. The power levels needed are quite low, in order to avoid boiling of water.

Microwave curing has been applied successfully to fibre reinforced cement composites (F.R.C.C.) in a previous work (6). Tests were performed on composites containing metakaolin in order to prevent the deposition of portlandite at the glass fibre/ matrix interface. As there was no more lime in composites containing 20 or 30% metakaolin, further research has been done to quantify the pozzolanic activity of metakaolin under microwave treatment. The present study describes the effect of a microwave treatment on calcium hydroxide consumption in cement pastes containing various amounts of metakaolin.

Experimental

Materials:

OPC was mixed with 0 to 30% metakaolin (MK). Table 1 gives their chemical composition. The quantity of water was adjusted in order to obtain the same workability measured by the flow-table test for all mixtures, as illustrated in fig.1. 200g samples were cast in closed plastic boxes to prevent evaporation of water in the microwave oven.

Metakaolin is produced from kaolin by calcination at 800°C. Its ability to react with calcium hydroxide was used by the authors to develop new matrices that allowed their reinforcement by E-glass fibres, usually destroyed in OPC (7), and improve the long-term properties of F.R.C.C..

TABLE 1: Chemical compositions

oxides (% by weight)	OPC	MK
SiO_2	20.0	51.45
Al_2O_3	4.8	44.77
Fe_2O_3	3.2	0.60
CaO	64.6	0.0
MgO	0.9	0.0
K_2O	0.6	0.07
Na_2O	0.2	0.29
SO_3	3.3	0.0
TiO_2	0.3	2.08
MnO	0.1	0.0
Cr_2O_3	0.01	0.02
P_2O_5	0.3	0.02
loss on ignition	1.7	0.7

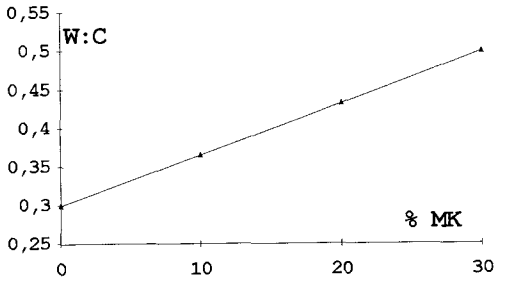

FIG.1
Water:cement ratio in cement pastes.

Heat treatment:

A 2450 MHz microwave oven (model CEM-MDS 81) was used. The maximum microwave output was fixed at 800 W. The fresh cement pastes were treated with different power levels (40-80 W) in order to obtain optimal hydration conditions.

The pozzolanic activity of cement pastes after microwave treatment was compared with a 24 hours curing in a fog room at 20°C. The evolution of the pastes was investigated after 28 days of storage in lime saturated water.

Identification of hydration products:

Samples were ground to be less than 100 µm.

The hydration of cement pastes was investigated by differential thermal analysis. DTA analysis used 600 mg of powder, which was heated at 10°C minute^{-1}.

The lime consumption was studied by Fourier transform infrared spectrometry, using a Perkin Elmer 451 spectrometer. The infrared range 450 to 4,000 cm^{-1} was investigated. The samples for FTIR were prepared as KBr pellets (2 mg of powder in 200 mg potassium bromide solid solution).

Hydration of cement pastes

The optimal microwave curing conditions were obtained from the investigation of different times of treatment for a chosen power level. The weight loss of cement pastes was measured, and then the hardened cement paste was pulverized (100 µm sieve) in order to examine the formation of hydrates by means of infrared spectrometry and differential thermal analysis (DTA).

When the power level reached 10% of the maximal microwave output, an excessive water evaporation from the cement pastes was observed, leading to an incomplete hydration of OPC, while 5% of the maximal microwave output allowed the formation of hydration products (fig.2), with a moderate weight loss.

The optimal time of treatment was determined as follows: it was that leading to the maximal production of calcium hydroxide (fig.3) measured by DTA (endothermal peak area at 540°C).

Moreover, the maximum infrared absorbtion band of the OH^- ions relative to calcium hydroxide at 3640 cm^{-1} was obtained just after microwave heating for about 3 hours, and did not increase after 28 days curing in lime saturated water. These microwave treatment conditions led to quite complete hydration of OPC, and a moderate weight loss of 2.2%.

Plain OPC pastes cured 24 hours in the fog room at 20°C led to lower hydration level. An increase of the lime peak area after 28 days in lime saturated water was observed.

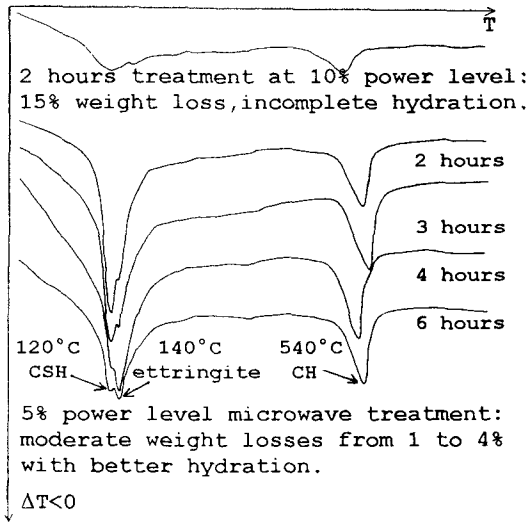

FIG.2: DTA curves of plain OPC pastes.

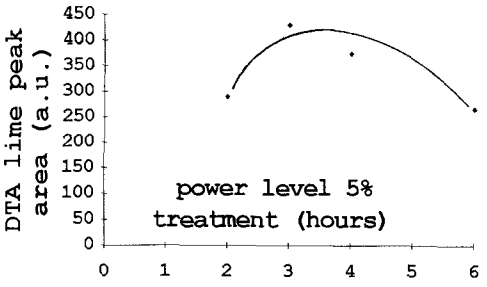

FIG.3: Lime formation versus treatment time.

Pozzolanic activity of metakaolin

Cement pastes including 0 - 5 - 10 - 15 - 20 - 25 and 30% MK were treated 3 hours at power level 5%. A complete lime consumption was observed in the blend containing 15% MK: the DTA $Ca(OH)_2$ decomposition peak completly disappeared (fig.4 and 5).

Cement pastes including 0 - 10 - 20 - 30 and 40% MK were cured for 24 hours at 20°C and 100% RH. Complete hydration lime consumption was obtained for 40% MK (fig.6), and 30% MK after 28 days in lime saturated water (fig.7), where the infrared absorbance of the OH^- ions relevant to calcium hydroxide completly disappeared, showing that the pozzolanic reaction of metakaolin was very slow at room temperature, as shown in previous work.

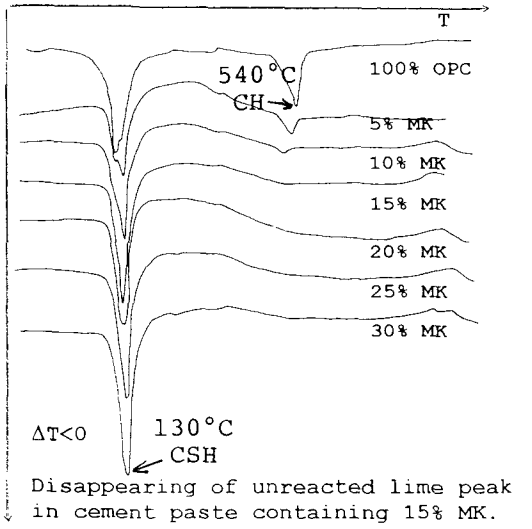

FIG.4: DTA curves of cement pastes.

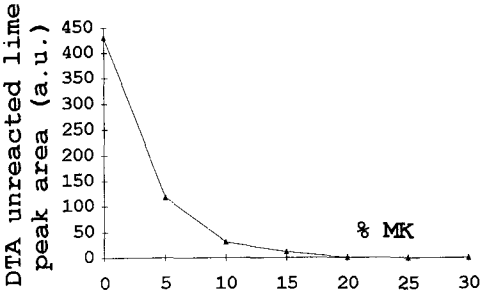

FIG.5: Lime consumption versus MK content.

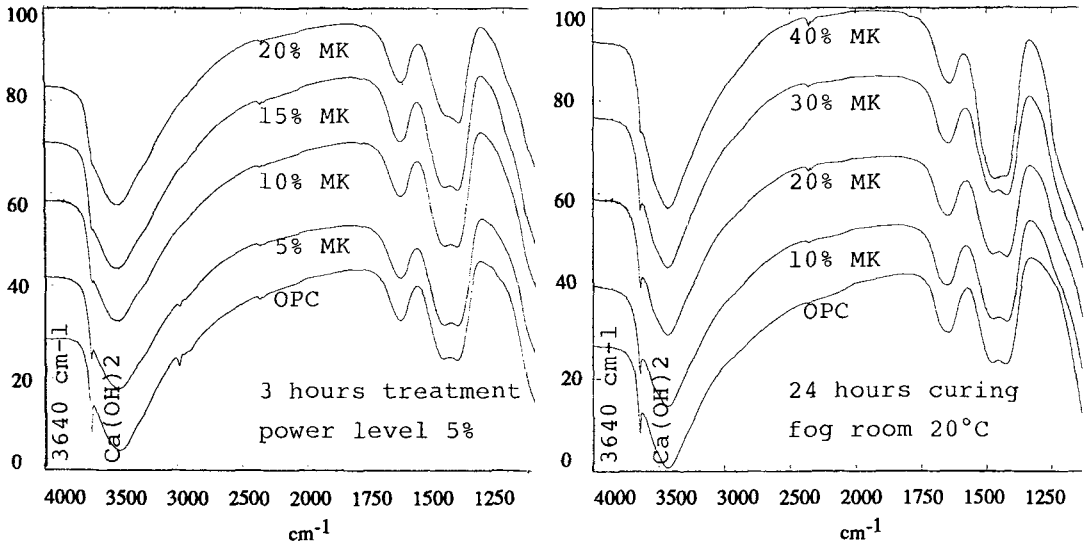

FIG.6: FTIR spectra of cement pastes just after treatment.

Conclusion

Pozzolanic reaction of metakaolin can be consequently promoted by microwave heating. As heat is generated quickly inside the cementitious material, thermal acceleration of the reaction is more efficient, allowing both reduction of the necessary amount of metakaolin (15% instead of 30-40% in normal conditions) and of the water:cement ratio (0.4 instead of 0.5).

The present results are quite interesting for the

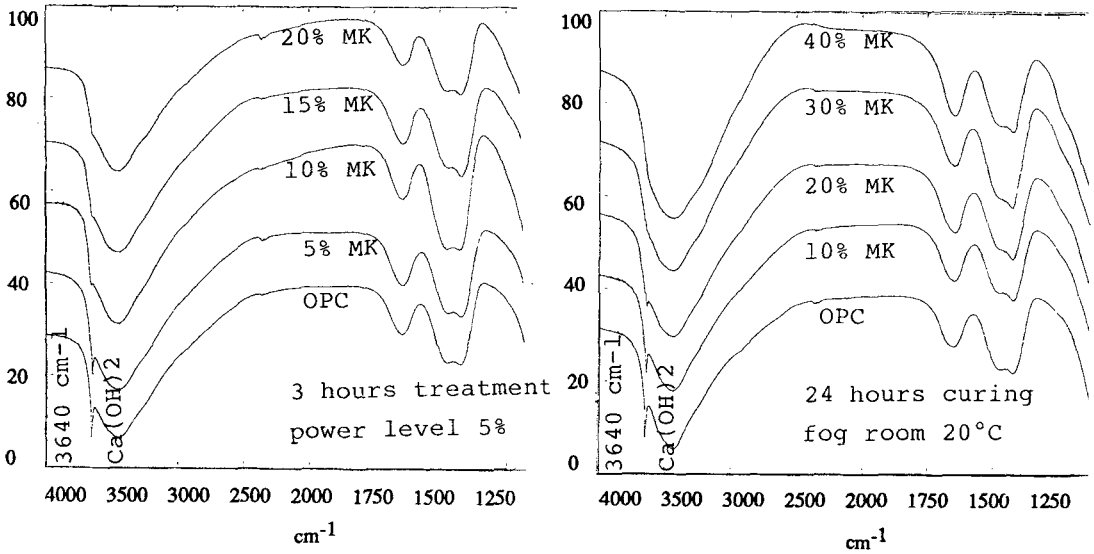

FIG.7: FTIR spectra after 28 days in 20°C lime saturated water.

development of fibre-reinforced cement composites, in which microwave heating allows the reduction of the curing period. Moreover, as the kinetics of pozzolanic reaction is enhanced, further research is engaged to develop new matrices including lower metakaolin contents.

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