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Study on the addition effect of metakaolin and mechanically activated kaolin on cement strength and microstructure under different curing conditions

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HIGHLIGHTS

- Strengths of MK and AK mortars ordinary cured and autoclaved are compared.
- The hydration products and microstructure of MK and AK pastes are determined.
- Ordinary cured mortar with 20% of MK achieved highest strength.
- 10% AK substitution in both curing conditions led to satisfactory strengths.

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ABSTRACT

The effects of thermally (MK) or mechanically activated kaolin (AK) on the compressive strength of mortars and microstructure of pastes were investigated. Mortar mixtures, in which 10%, 20% and 30% of ordinary Portland cement (OPC) was replaced by either MK or AK, were prepared (w/b of 0.5) and ordinary (age 2, 28 or 90 days) and autoclave cured. Hydration products were determined by X-ray diffraction (XRD) and differential thermal analysis/thermal gravimetry (DTA/TG) analysis, while microstructure was investigated by mercury intrusion porosimetry (MIP).

MK substitution increases the compressive strength of ordinary-cured mortars, as a result of the higher content of reactive silica that caused more pronounced pozzolanic reaction, as well as by effective refinement of their pore structure. Positive effects on the compressive strength could be achieved up to 30% substitution of OPC by MK. The OPC substitution by AK resulted in lower strengths of ordinary-cured mortars, compared to both MK mortars and reference. Higher specific surface area and finer particles of AK were insufficient to compensate, through filler effect, lower pozzolanic reaction and additional negative influence of the kaolinite presence. The highest compressive strength was obtained for mortar with 10% of AK (relative strength of 94%).

In comparison to the reference, autoclaved MK and AK mortars, exhibited lower compressive strength, as a consequence of increasing the hydrogarnet formation, instead of tobermorite. The highest strength was achieved for mortar with 10% of AK.

1. Introduction

Supplementary cementitious materials (SCMs) are becoming an essential component of modern cement and contemporary concrete [1]. The commonly used SCMs are fly ash (FA), ground granulated blast-furnace slag (GGBS), silica fume (SF), rice husk ash (RHA) and metakaolin (MK) [1]. The availability of industrial by-products, such as silica fume and blast-furnace slag, is locally imbalanced and the supplies are quite limited, compared to the worldwide production of cement [2,3]. Today, alternative sources of SCMs, such as calcined clays, are of interest, because of their widespread availability and low prices, as well as some new compounds currently under investigation [4].

MK and calcined clays have been extensively studied since the mid-1990s [5–7]. Research has been generally focused on the optimization of the thermal activation process [8–12] and improvement of the cement-based materials by using MK/calcined clays

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[13–16]. Commonly applied production of MK includes three-stage method that comprises a selection/grinding process, then thermal activation of the raw kaolin for several hours in a rotary kiln, followed by grinding of the calcined material.

The dehydroxylation and structural disorder of kaolinite, obtained by thermal activation, could also be achieved by mechanical activation in grinding equipment, such as vibratory, oscillating and planetary mills [17,18]. Although mechanical activation of kaolin has a lot of advantages, such as simplicity of the process and ecological benefits, the large-scale application of this process for kaolin activation has not been reported so far. The technical, environmental and economical benefits of MK and calcined clay use are well documented in scientific literature [5,19], but only a few data are available for mechanically activated kaolin [20,21]. Pozzolanic activity and differences between thermally and mechanically activated kaolin were the subject of considerable research in recent years [22–25]. However, the data about use of mechanically activated kaolin in cement-based materials, are rather scarce [23].

Nowadays, MK has found utilization in variety types of concrete ordinary cured, such as high-performance concrete, high-strength, lightweight concrete, glass fibre reinforced concrete [1]. A clear conclusion regarding the optimum MK percentage for obtaining maximum strength of concrete/mortar is still not available. After a comprehensive literature review, Rashad [7] concluded that the highest 28 days compressive strength of mortars could be attained with 20% MK (w/b of 0.5–0.53), followed by 15% and 10% respectively.

Additionally, recent studies [26,27] have demonstrated that MK could be successfully applied in the precast industry and that under steam-cured conditions it is possible to substitute up to 25% of cement with MK and still achieve positive effects on the strength. In the literature, there are no available data relative to use both MK and mechanically activated kaolin as SCMs under autoclave curing, which are also used in precast industry. Reports on the microstructure of autoclaved MK pastes are few and scattered [28–30], while data of microstructure of paste with AK is lacking.

This work is a part of an extensive research program regarding utilization of thermally and mechanically activated kaolin in cement-based materials. This research included application of Serbian kaolin for the production of MK [31], optimization of the conversion of kaolin to MK [11], investigation of the properties of mortars with added MK [32,33], formation of pozzolanic material by mechanical activation [34,20] and the effects of mechanical and thermal activation on pozzolanic activity of kaolin containing mica [25].

Following our previous research [25], present work investigates the effects of thermally or mechanically activated kaolin on the compressive strength of mortars and microstructure of pastes. Mortars, in which 10%, 20% and 30% of ordinary Portland cement was replaced by either thermally (MK mortars) or mechanically activated kaolin (AK mortars), were prepared. The effects of ordinary curing (age 2, 28 or 90 days) and autoclave curing on the mechanical properties of mortars were compared. It is known that the strength of autoclaved mortar is comparable to the strength of mortar ordinary-cured for 28 days [35]. However, autoclave curing was chosen based on previous results obtained in research with mechanically activated kaolin [36,37]. Pastes were prepared for determination of the hydration products and for measurements of porosity and pore size distribution. The crystalline hydration products were determined using XRD, while DTA/TG analysis revealed the temperature ranges for the decomposition of different phases in the cement paste. The total porosity, as well as the pore size distribution, was determined using MIP. The similarities and differences between the microstructure and compressive strength of MK and AK mortars for different curing conditions were discussed.

2. Materials and methods

2.1. Raw materials

Ordinary Portland cement, CEM I 42.5R was supplied from Lafarge BFC, Beočin Serbia. A summary of its chemical and mineralogical composition and some physical properties is presented in Table 1.

MK and AK were produced from the Serbian kaolin with high content of impurities (~40 wt%) in our laboratory as follows. MK was obtained by thermal activation of kaolin at 700 °C for 60 min, while AK was prepared by mechanical activation for 20 h in a conventional horizontal ball mill. A more detailed description of materials and activation conditions is given previously [25]. Main physical and chemical properties of the MK and AK are presented in Table 2.

CEN Standard sand, complying SRPS EN 196-1:2008, distilled water and superplasticizer Sika ViscoCrete TECHNO 20S were also used.

2.2. Design, mixing, curing and testing

2.2.1. Mortar mixtures

Mortar mixtures, in which 10%, 20% and 30% of cement was replaced by MK or AK, were prepared in addition to a reference mixture (Ref. M) which used only OPC as binder (0% cement replacement). The weight ratio of cementitious material to sand of 1:3 and the water-to-binder ratio of 0.5 were maintained. The mixture composition is presented in Table 3.

Mixing was performed in a bench-mounted mixer in accordance with SRPS EN 196-1:2008. The workability was adjusted (consistency of 164 ± 10 mm) by the addition of appropriate amount of superplasticizer (in dry superplasticizer weight by mass of binder). The MK mixtures consume higher amount of superplasticizer, in order to achieve same workability, probably due to the agglomeration or flocculation of MK particles [38,25].

In MK mixtures, a previously blended mix of cement and MK was added to the water containing superplasticizer, followed by the gradual addition of sand. Same order of operation could not be used with AK mixtures, as they became plastic and lost their workability. Hence, a blended mix of cement and AK was added to the water and continuously mixed with sand, and at the very end, the superplasticizer was added.

The specimens were cast in the moulds (three prisms size 40 × 40 × 160 mm) using a vibration table.

2.2.2. Curing

In order to compare the effects of different curing on the mechanical properties of mortars, ordinary and autoclave curing were applied. In both cases, samples were first stored in the mould in a moist atmosphere for 24 h (temperature of 20.0 ± 1.0 °C and a relative humidity of 95%). Thereafter hardened samples were removed from the moulds and:

- Ordinary cured in water at temperature 20.0 ± 1.0 °C, until the testing ages (2, 28 and 90 days) or
- Autoclave cured at constant temperature and pressure of 216 °C and 2 MPa for 4 h. Then the autoclave heater was turned off and the chamber was allowed to cool naturally.