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Rheological and Mechanical Behavior of Mortars with Metakaolin Formulation

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Abstract

This study aims to valorize calcined kaolin powder as an addition to cementitious matrix building materials. The main purpose of this study is to formulate and analyze the performance of metakaolin mortars. The preparation of the metakaolin was carried out by calcining the Kaolin at a temperature of 800 °C for duration of 3 hours. A comparison of the results with a control mortar without addition is established. The observed results showed that the rate of substitution of 10% of cement by metakaolin increases the compressive strength and tensile strength at a young age.

Peer-review under responsibility of the scientific committee of the 3rd World Conference on Technology, Innovation and Entrepreneurship

Keywords: Metakaolin; Mortar; Workability; Setting time; Mechanical strength.

1. Introduction

Thermally activated clays, especially kaolinitic clays, are today revaluated as a source of supplementary cementitious materials to reduce the CO_2 emissions and energy consumption originated in cement production [1].This could be achieved by replacing a part of cement with mineral addition such as fly ash, blast –furnace slag or metakaolin (MK) during concrete mixing. In recent years, the incorporation of MK in building materials is growing [2].

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Arikan et al. [3] state that the main advantage in the use of MK in concrete and cement is its high pozzolanic activity, which is the ability to react with $Ca(OH)_2$ produced during the hydration of Portland cement (PC), forming hydrated calcium silicates and aluminates. Being a very fine material, with 99.9% of particles with size less than 16 µm and an average size of about 3 µm, therefore presenting a high specific surface, metakaolin possesses the ability to accelerate the pozzolanic reaction [4]. Nova John observed that the partial replacement of cement with Metakaolin helps in achieving high strengths in concrete. At 15% replacement of cement with Metakaolin content improves the strength characteristics such as of cube compressive strength, split tensile strength and flexural strength [5].

Kaolin is a naturally occurring clay mineral with kaolinite being the main component having a chemical composition of $Al_2Si_2O_5\,(OH)_4$. It has a layered silicate structure, where oxygen atoms link an octahedral sheet of alumina with a tetrahedral sheet of silica. When kaolinite is calcined at elevated temperatures, dehydroxylation occurs which collapses the crystal structure to produce amorphous metakaolinite ($Al_2\,Si_2O_7$) [6], according the following proposed reaction scheme:

$$Al_2Si_2O_5(OH)_4 \longrightarrow Al_2Si_2O_7 + 2H_2O \tag{1}$$

The objectives of this study were to investigate the substitution of cement by MK up to 10 wt% on the hydration characteristics of MK-blended cement pastes. The physico-chemical properties of cement pastes were determined up to 28 days. The hydration products of some selected samples were investigated by using XRD techniques.

2. Materials and experimental techniques

2.1. Materials Used

The Portland cement type CEM II/A 42.5 from Hammam Dalâa local factory was used in this experimental study. The used cement type has an absolute density, consistency and fineness values of 3.1 g/cm^3, 28 % and 4000 cm^2/g, respectively. The kaolin comes from the wilaya of Mila located in the northeastern region of Algeria. The preparation of metakaolin was carried out by calcining kaolin at a temperature of 850 °C for a period of 3 hours. After calcination, as shown in Fig. 1, the peaks for kaolinite vanished, indicating complete transformation of kaolin to metakaolin. The chemical composition of cement and metakaolin is given in Table 1.

Table.1: The chemical, physical properties of cement and Kaolins

<table>
<tr><th></th><th colspan="2">Cement (%)</th><th colspan="2">Kaolins (%)</th></tr>
<tr><td>SiO_2</td><td colspan="2">20.7</td><td colspan="2">48</td></tr>
<tr><td>Al_2O_3</td><td colspan="2">04.75</td><td colspan="2">36.5</td></tr>
<tr><td>Fe_2O_3</td><td colspan="2">03.75</td><td colspan="2">1.01</td></tr>
<tr><td>CaO</td><td colspan="2">62.92</td><td colspan="2">0.07</td></tr>
<tr><td>MgO</td><td colspan="2">01.90</td><td colspan="2">0.30</td></tr>
<tr><td>SO_3</td><td colspan="2">01.98</td><td colspan="2">-</td></tr>
<tr><td>Cl</td><td colspan="2"></td><td colspan="2">-</td></tr>
<tr><td>K_2O</td><td colspan="2">-</td><td colspan="2">2</td></tr>
<tr><td>Na_2O</td><td colspan="2">-</td><td colspan="2">0.10</td></tr>
<tr><td>TiO_2</td><td colspan="2">-</td><td colspan="2">0.05</td></tr>
<tr><td>L.0.I</td><td colspan="2">-</td><td colspan="2">12</td></tr>
<tr><td colspan="5">Mineralogical composition of cement</td></tr>
<tr><td rowspan="2">Phase content (%)</td><td>C_3S</td><td>C_2S</td><td>C_3A</td><td>C_4AF</td></tr>
<tr><td>60</td><td>25.55</td><td>2</td><td>12.45</td></tr>
<tr><td colspan="5">Particle size analysis of Kaolins (%)</td></tr>
<tr><td></td><td>Min</td><td>Value</td><td colspan="2">Max</td></tr>
<tr><td>> 53um</td><td>-</td><td>-</td><td colspan="2">0.25</td></tr>
<tr><td>> 8um</td><td>14</td><td>19</td><td colspan="2">24</td></tr>
<tr><td>< 2um</td><td>34</td><td>39</td><td colspan="2">44</td></tr>
</table>

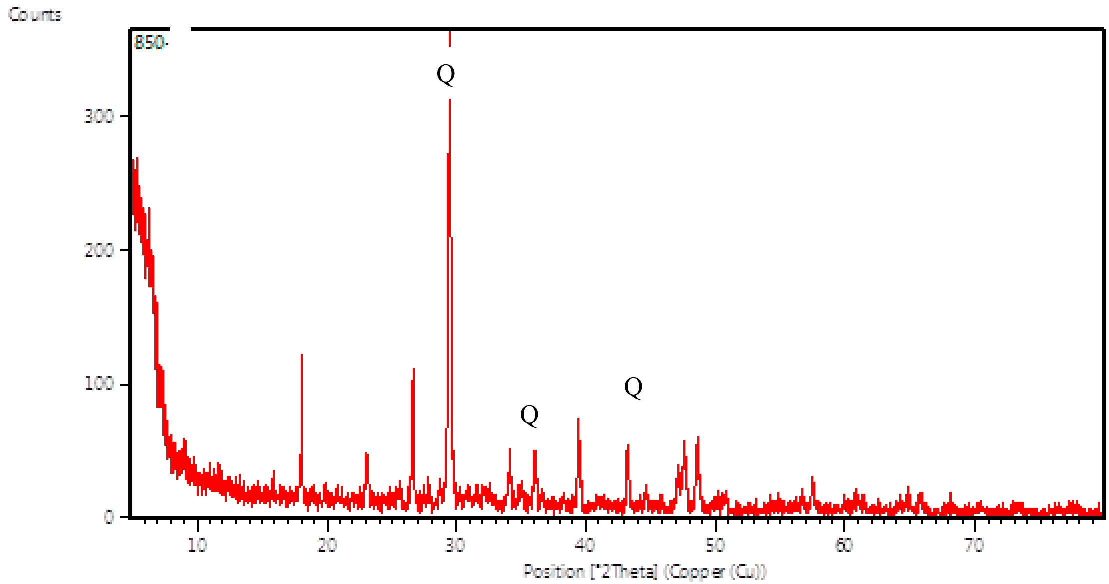

Fig 1: XRD pattern of metakaolin

The sand used in the mortar is dune sand with water absorption of 1.82% and a specific gravity of 2560 kg/m^3, respectively.

2.2. Mortar sample preparation

Mortar samples were constructed by replacement of 10% cement with activated materials by weight. The mortars were designed using sand to cementitious material ratio (S/C) of 3.0 and W/C of 0.5. The mortar was mixed mechanically, cast in moulds and then compacted with the aid of a vibrating table. After casting, the specimens were covered with ahessian for 24 hours under laboratory conditions before demoulding. Prior to testing, all the specimens were cured in lime saturated water for 28 days. The final compositions of mortars with addition, after optimization is reported on table 2.

Table 2: The mortar composition

Mix (g)	M0	MK
Cement	450	405
Sand	450	
Metakaolin	-	45
Water/Cement	0.50	

Workability: Slump values of mortar sample have been tested for different sample of concrete mix. It was evaluated by partially replacing metakaolin powder in place of cement in proportions of 0% and 10%. It was evident that the workability of a mortar mix was decreased with increase in the metakaolin powder content. As per NF EN 12350-5 [7] slump test was used to examine workability of the all mixtures.

*Compressive strength :*For the compression strength test, three samples were used for each mixture, and the average strength values of these samples were determined as compressive strengths for the mixtures with a square section of 40 mm × 40 mm and a length of 160 mm in accordance with NF EN 196-1 [8].

Porosity: The protocol of porosity accessible to water conforms to the recommendations of AFREM [9] group. The open porosity allows us to appreciate the evolution of hydration and structuration of hydrated products; this is a key for identification of the most sustainable concrete [9]. The porosity test is carried out on test pieces of dimensions 4× 4×16 cm^3, by applying the following steps:

1) Drying in an oven at 105 °C of the sample for at least 24 hours until obtaining a constant mass. Then they were weighed once dry (*A*);
2) Immersion of the sample in water for 24 hours;
3) Heating to boiling for 5 hours, then weighing the sample in air (weight "*C*");
4) Finally, hydrostatic weighing (*D:* weight of saturated samples subjected to Archi-medes).

The porosity was calculated by the formula: P (%) = [(C-A) /(C-D)].100

3. Results and discussion

3.1. Properties of fresh mortar

3.1.1. Workability

Workability of control mortar mixture and blended mortar mixtures incorporating metakaolin powder with cement and sand are shown in figure 2.The replacement of cement with 10% metakaolin has has no effect on workability. It was observed that slump decreases with the replacement of cement by 10% metakaolin powder. This result explains the fine filler effect of the metakaolin powder and its higher specific area as compared to Portland cement. Deepankar (2019) made similar observations when the supplementary cementitious materials, particularly those with a high surface area like metakaolin and silica fume require relatively higher water content and due to this slump decreases [10].

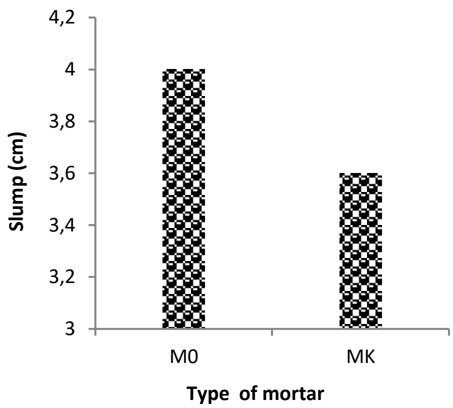

Fig 2**:** Effect of metakaolin powder as cement replacement on workability of mortar

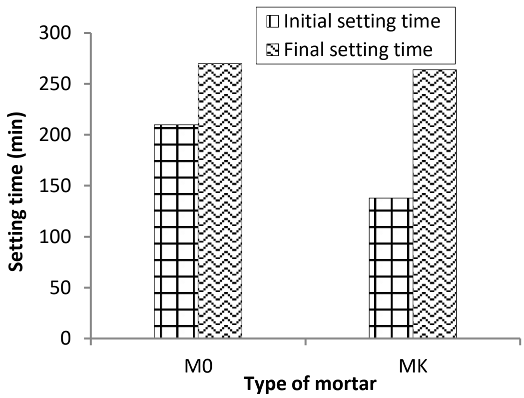

Fig 3: Setting times of binder paste mixture

3.1.2. Setting times

Standard consistency is indicated in Figure 3 for cement pastes with and without metakaolin powder. It decreased slightly with the use of metakaolin powder in place of cement. The decrease in consistency was observed 5% on replacement of cement by 10% with metakaolin powder. The test result shows less water requirement in blended cement paste made with metakaolin powder in comparison to control cement pastes. The cement-sand mixtures prepared by partial substitution of marble powder showed a considerable decrease in water to cement ratio. No detrimental result was apparent due to loss on ignition on water requirement of marble cement paste.

Fig. 3 shows the initial and final setting times of mortar mixtures, it is clear from the figure that initial and final setting time increases with the decrease in metakaolin powder content. The cement paste mixture M0 has higher initial setting times i.e. 210min, as compared to (MK), final setting time for cement paste mixtures M0 and MK was 270 and

264 min, respectively. The hydration process of binder paste mixtures was delayed with the increase in final setting time. This is due to variance in the chemical composition of metakaolin powder and cement. Deepankar (2019) and Abdelli et al. (2017) made concurrent observations [10-11].

3.2. Properties of hardened mortar

3.2.1 Porosity

Figure 4, shows the porosity results of mortar containing metakaolin. It was observed that, the specimens with metakaolin to 10% showed lower porosity values than the control mortar at 28 days.

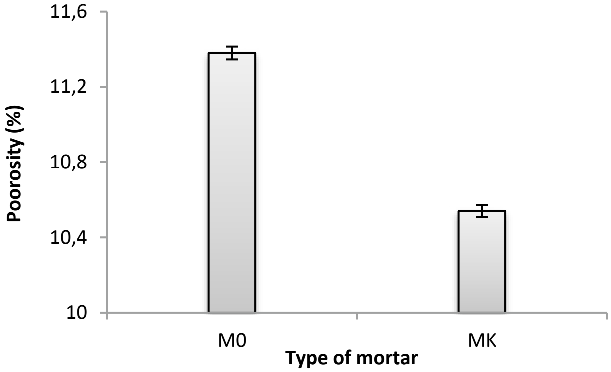

Fig 4: Porosity of mortars used

Generally, the porosity values decreased as the replacement levels increased to 10%. The low porosity values of the blended specimens could be ascribed to pore refinement due to micro-filler effect and pozzolanic reaction. These results may be due to the fact that metakaolin powder have high fineness, very small particle size and also pozzolanic reaction products C-S-H leading to a decrease in solid volume as well as an increase in number of very small pores and thus an increase in porosity. These results agree with what has been reported in the literature by Rashwan *et al* .[12].

3.2.2. Compressive strength

The preliminary compressive strengths of cement mixture with MK treated at 850 °C for 3 h are shown in Fig. 5. As can be seen, the strengths values for the mortar specimens with 10% metakaolin were higher than that of control (0%) at 28 days.

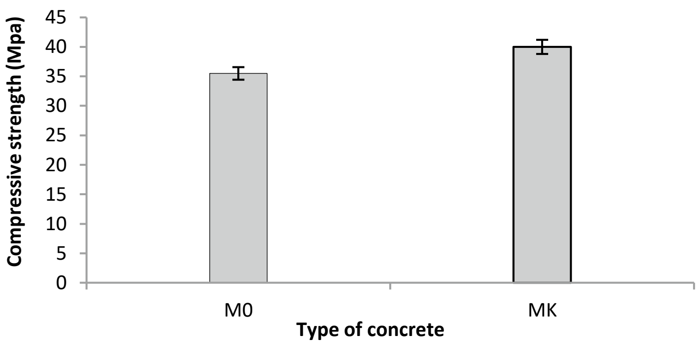

Fig 5: Compressive strength of mortars

According to the research of Mansour et al. (2011) , There are three elementary factors influencing the contribution that MK makes on strength development when it is partially replaced with cement. These are the filler effect, the acceleration of OPC hydration, and the MK pozzolanic reaction. The filler effect, which results in more efficient paste packing, is immediate, the acceleration of OPC hydration has maximum impact within the first 24 h, and the pozzolanic reaction makes the greatest contribution to strength somewhere between 7 and 14 days of age [13]. When mixed with cement, metakaolin had a pozzolanic effect. The pozzolanic reaction occurs in solution (dissolution/precipitation) between the silicate and aluminate generated from metakaolin dissolution, and the calcium hydroxide CH from the cement hydration, producing more CSH. Hence, the pozzolanic reaction generates a denser cement matrix [14-15].

4. Conclusion

Based on the experimental investigations the following conclusions are drawn:

- Algerian MK is an active mineral admixture that could be used in cement concrete products. It has a good effect on the mechanical properties of cement mortars.
- Thermal treatment at 850°C for 3 h is efficient for the conversion of kaolin to metakaolin. At these conditions the produced metakaolin exhibits the highest pozzolanic reactivity;
- Similarly replacing cement with metakaolin by 10% increases the workability ;
- Substitution of cement by MK decreases the initial and final setting times up to 34%
- From the above compressive strength results, it is observed that mortars have achieved an increase in strength for 10% replacement of cement by metakaolin at the age of 28 days when compared to control mortar.

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