Effect of metakaolin as partially cement replacement on the compressive strength of standard mortars

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Abstract

This study presents the effect of incorporating synthesized metakaolin on the compressive strength of standard mortars for a constant water/binder ratio of 0.5. Synthesized metakaolin mixtures with cement replacement of 5, 10, 15 and 20% were tested.
From the results, it was observed that 15 % replacement level was the optimum level in terms of compressive strength. Beyond 15 % replacement levels, the strength was decreased but remained higher than the control mixture. Compressive strength of 52 MPa was achieved at 15 % replacement. This investigation has shown that it is possible to produce high strength mortars using local kaolin.

Key-words: metakaolin, mortar

1. Introduction

Metakaolin is a highly reactive pozzolan obtained by calcination of kaolin at high temperature (700-1200°C). It enhances the strength, durability, and workability of Portland cement concrete and other cement-based products.

The use of metakaolin as partial substitute of cement in concrete/mortar pastes has been widely investigated in the previous years. However, the researchers have not been able to reach agreement on a unique conclusion regarding the optimum metakaolin replacement percentage for obtaining maximum strengths of concretes and mortars. Different replacement levels have been reported in the literature:

Rabehi et al. [1] partially replaced cement in mortars with calcined kaolin at levels of 0%, 5%, 10%, 15%, and 20%, by weight. Fixed w/b ratio of 0.5 was used. The results indicated that 5% metakaolin exhibited the highest compressive strength.

Said-Mansour et al. [2] partially replaced cement in mortars with metakaolin at levels of 0%, 10%, 20%, and 30%, by weight. Fixed w/b ratio of 0.5 was used. 0.9% of SP was used for mixture containing 30% metakaloin. The results indicated that 10% metakaolin exhibited the highest 28 days compressive strength while the inclusion of 30% metakaolin reduced it by 5.42%.

Goel [3] partially replaced cement in mortars with metakaolin at levels of 0%, 5%, and 10%, by weight. W/b ratios of 0.46 and 0.5 were used. The results indicated that 5% metakaolin exhibited the highest compressive strength at w/b ratio of 0.46, whilst the inclusion of 10% metakaolin exhibited the highest compressive strength at w/b ratio of 0.5.

Potgieter, Vermaak and Potgieter [4] investigated the optimum compressive strength, at age of 28 days, of mortars containing 0%, 10%, 20%, and 30% South African metakaolin as partially cement replacement. They used fixed w/b ratio of 0.375. The results showed that 10% metakaolin exhibited the highest 28 days compressive strength.

Courard et al. [5] partially replaced cement in mortars with metakaolin at levels of 0%, 5%, 10%, 15%, and 20%, by weight. Fixed w/b ratio of 0.5 was used. The results indicated that 15% Metakaolin exhibited the highest 28 days compressive strength.

Khaleel and Abdul Razak [6] partially replaced cement in mortars with metakaolin at levels of 0%, 5%, 10%, and 15%, by weight. Fixed w/b ratio of 0.32 and various dosages of Superplasticizer were employed. The inclusion of 10% metakaolin exhibited the highest 28 days compressive strength.

Vu et al. [7] partially replaced cement in mortars with metakaolin at levels of 0%, 5%, 10%, 15%, 20%, 25%, and 30%, by weight. W/b ratios ranging from 0.4 to 0.53 were used. 1.3% and 0.5% of SP were employed for mixtures containing w/b ratios of 0.4 and 0.44, respectively. The results indicated that 15% metakaolin exhibited the highest 28 days compressive strength at w/b ratios of 0.44 and 0.47, whilst the inclusion of 20% metakaolin exhibited the highest 28 days compressive strength at w/b ratios of 0.4, 0.5, and 0.53. The inclusion of 30% metakaolin reduced it by 1.38%.

Parande et al. [8] partially replaced cement in mortars with metakaolin at levels of 0%, 5%, 10%, 15%, and 20%, by weight. Fixed w/b ratio of 0.4 was used. The results indicated that 15% metakaolin exhibited the highest compressive strength. The enhancement in the 28 days compressive strength was 50%, and 45% with the inclusion of 15%, and 20% metakaolin, respectively.

Khatib et al. [9] partially replaced cement in mortars with Metakaolin at levels of 10% 20%, 30%, 40% and 50%. Fixed w/b ratio of 0.5 was used. Compared to the control, the results showed an increase in the compressive strength with the inclusion of 10%, 20%, and 30% metakaolin, whilst the inclusion of 40% metakaolin showed slightly lower compressive strength. The inclusion of 50% metakaolin showed approximately 20% reduction in the compressive strength. The inclusion of 20% metakaolin exhibited the highest 28 days compressive strength.

Roy et al. [10] partially replaced cement with metakaolin at levels of 0%, 7.5%, 15%, and 22.5%, by weight. Fixed w/b ratio of 0.36 was used. The results indicated that 7.5% metakaolin exhibited the highest 28 days compressive strength.

Venu Malagavelli, Srinivas Angadi, J S R Prasad and Subodh Joshi [11] partially replaced cement in mortars with metakaolin at levels of 5%, 10%, 15%, 20% weight. They used fixed w/b ratio of 0.3. The results showed that 10 % metakaolin exhibited the highest 7 and 28 days compressive and flexural strengths.

Antoni et al. [12] partially replaced cement in mortars with 30% metakaolin, by weight. Fixed w/b ratio of 0.5 was used. The results showed that the inclusion of metakaolin improved the compressive strength at ages of 7, 28, and 90 days by 7%, 19% and 4%, respectively.

Accordingly, results differ regarding the optimum metakaolin replacement percentage for obtaining maximum strengths of concrete/mortar/pastes.

2. Materials and methods

2.1 Materials used

Materials used in this investigation were:

- Processed kaolin clay, hereafter indicated KT.
- Portland Cement CPJ CEM II/A 42.5 N
- Standardized alluvial sand from El Oued in the southeast of Algeria.
- Alkaline liquid (Activation solution), hereafter indicated SA was prepared in the laboratory.

The processed kaolin KT used for metakaolin synthesis was provided by the SOAKLA Complex of kaolin in El Milia (in eastern Algeria).

The sand used for cement and geopolymer mortar mixtures is normalized siliceous sand conforming to norm ASTM c778-06.

Alkaline activator was prepared in the laboratory by mixing aqueous solutions of sodium hydroxide (NaOH (12M)) and sodium silicate.

2.2 Sample testing

- The chemical compositions of KT kaolin and Cement were determined using a Philips PW 1404 X spectrophotometer.

- X-Ray Diffraction Analysis (XRD) with a Siemens D5000 diffractometer was employed to characterize the processed KT kaolin and the synthesized metakaolin.

-The particle size distribution of the sand was determined by laser granulometry.

- Specific surface areas of cement and MK metakaolin were measured using Blaine testing instrument according to EN 196-6 Standard [13].

- The Fourier Transformed Infra-Red (FTIR) analysis was used to follow the KT kaolin calcination process. It was carried out with a spectrometer equipped with ATR diamond of total attenuated reflectance.

- The compression tests carried out, according to EN 196-3:2005 standard [14] on 40mm x 40mm x40mm cubic specimens was meant to determine the geopolymer optimal formulation and the optimal replacement rate of cement by metakaolin in mortars.

2.3 Thermal treatment of started kaolin clay (KT)

Metakaolin was obtained by calcination of processed kaolin clay in the laboratory. Indeed, the main process important for production high reactivity pozzolana from kaolin clay is calcination.

At this stage of the study, the optimum temperature for heating kaolin in order to obtain Metakaolin with a high pozzolanic index and the heating period are still exactly undetermined. They are still different from one researcher to another which explains the benefits of this study.

The goal of this part of study is to determine optimal calcination parameters for obtaining metakaolin from treated Algerian kaolin clay (KT), for the use as cimentitous material.

In order to obtain optimal calcination parameters, processed kaolin (KT) samples of about 30 g were heat treated in the laboratory furnace at different temperatures (650, 750 and 850 °C) and at different heating times (30, 60, 90, 120 min.).

X-ray diffraction and Fourier transform infrared spectroscopy FT-IR were adopted to investigate the influence of temperature on the metakaolin synthesis process.

After heating, the samples were subjected to room temperature at ambient conditions to avoid crystallization of amorphous metakaolin. The weight of the samples before and after the thermal treatment was measured in order to determine weight loss during calcinations process.

2.5 Effect of synthesized MK-metakaolin as partially cement replacement on the compressive strength of standard mortars

Hereafter we will explore how the synthesized MK-metakaolin affects the compressive strength of standard mortars based on CPJ CEM II/A 42.5 N cement in which MK-metakaolin was used as cement replacement. In addition, the optimum content of MK-metakaolin which exhibited the highest 28 days compressive strength was investigated.

We partially replaced cement in mortar mixtures with MK-metakaolin at levels of 0%, 5%, 10%, 15%, and 20%, by weight. Fixed w/b ratio of 0.5 was used. Beyond 20%, we observed that MK reduced the workability. Formulations studied are listed on table 1.

Table1

Various Mix proportion of MK-Metakaolin Blend mortar

Material	**Mix**				
	MK0	**MK5**	**MK10**	**MK15**	**MK20**
CPJ CEM II/A 42.5 N cement (g)	450	425	400	375	350
MK (g)	0	25	50	75	100
Water (g)	225	225	225	225	225

The mortars were mixed in a 5L mixer according to EN 196-1 and ISO 679:2009 standards with the mixing water. Standard sand (EN 196-1 and ISO 679:2009 standards) of a particle size between 0 and 2 mm was used. The mortar specimens were cast in 4x4x4 cm cubic metal moulds. All specimens were cured at 20°C and 95% R.H. for 24 hours and then demoulded and stored under the same conditions until testing.

The compression tests were performed on cubic specimens of dimensions 4x4x4 cm (fig.1) according to EN 196-1 standard.

Fig.1 MK-metakaolin/Portland cement mortar specimens

3. Results and discussion

3.1 Raw materials characterization

3.1.1 Sand sieve analysis

Figure 2 plots the particle size distribution of the sand. As it can be seen, sand is mainly constituted of fine aggregates.

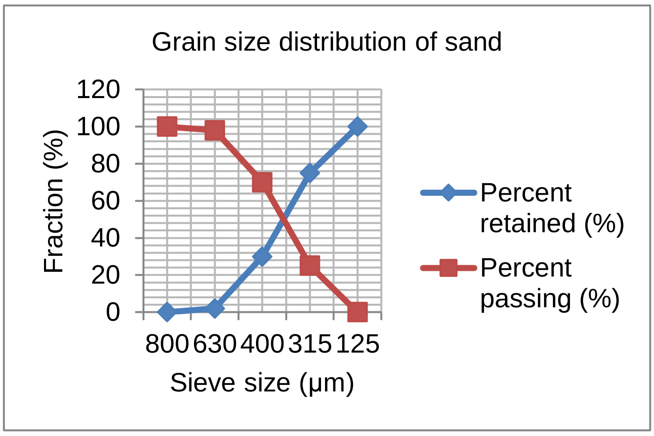

Fig.2. Grain size distribution of sand

3.1.2 Chemical analysis of raw materials

The chemical analysis of KT kaolin, Portland cement was determined by X-Ray Fluorescence (XRF). Results are given in Table 2.

Table 2

Chemical composition of raw materials

Chemicals	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	LOI[(1)]	I.R[(2)]	TiO_2
Cement	20.34	5.37	3.00	61.69	1.80	2.20	0.76	0.14	0.97	1.12	-
Kaolin (KT)	49.30	33.00	2.38	0.08	0.40	-	2.93	0.09	-	-	0.24

(1) LOI: Loss in ignition; (2) I.R: Insoluble Residue;

3.1.3 FTIR characterization

FTIR technique was used to show the functional groups contained in Kaolin and Metakaolin. In order to confirm the characteristic bands of kaolinite in raw sample and the absence of these bands in thermally treated samples, a FTIR spectrophotometer Nicolet 6700 Thermo Scientific was used. IR spectra obtained for KT Kaolin starting clay and thermally treated samples are presented in Figures 3a, 3b, 3c and 3d. The results of IR spectroscopy of KT starting clay (Figure 3a) show the characteristic bands of kaolinite [15]: OH− at 3700, 3600 cm^{-1}; Al–OH at 913 cm^{-1}; Si–O at 1032, 1008, 469 cm^{-1} and Si–O–Al^{VI} at 538 cm^{-1}.

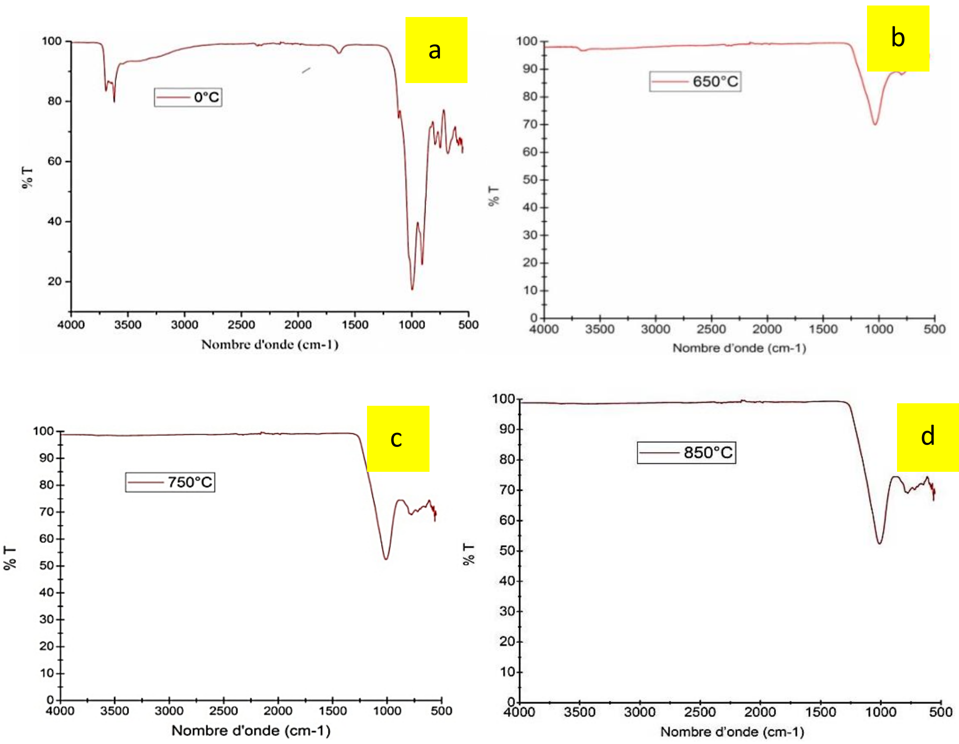

Fig. 3. IR spectra of starting kaolin (a) and calcined Kaolin for 90 min at: 650°C (b), 750°C **(c)** and 850°C (d)

Absence of the detectable Al–O–H bands at 3700 and 3620 cm^{-1}, due to KT deshydroxylation is evident from figure 3c and figure 3d. Absence of the band at 539 and 913 cm^{-1} and the appearance of a new band at 800 cm^{-1} can be related to the change from octahedral coordination of Al^{3+} in kaolinite to tetrahedral coordination in metakaolinite. The bands at 1100 and 1200 cm^{-1} are assigned to amorphous SiO_2.

After KT calcination, absence of bands at 1113, 1024, 998 and 908 cm^{-1} and apparition of the band about 1044 cm^{-1} on the MK spectral, confirm the process of Kaolinite-Metakaolinite conversion, as well as the presence of amorphous silica in metakaolin. Absence of the band at 908 cm-1 after kaolinite amorphisation at 750 °C and the appearance of the band at 546 cm^{-1} (Al - O, Al^{IV}) can be related to the change from octahedral coordination of Al^{3+} in kaolinite to tetrahedral coordination in metakaolinite. [16]

3.2.1 XRD Analysis

In order to confirm disappearance of kaolinite peaks, after thermal treatment, the XRD patterns of starting and calcined kaolin were compared (Figures 4a and 4b). It is evident that the major mineral constituents of the starting clay are kaolinite, quartz, Muscovite and albite minerals (Fig. 4a). After calcination at 750 °C, Kaolin was transformed into an amorphous phase, which is Metakaolin (2θ/20–35), in addition to the formation of Anorthite after its reaction with CaO decomposed from Calcite, and other minerals like Quartz and Leucite resulting from the transformation of Muscovite (Fig. 4b).

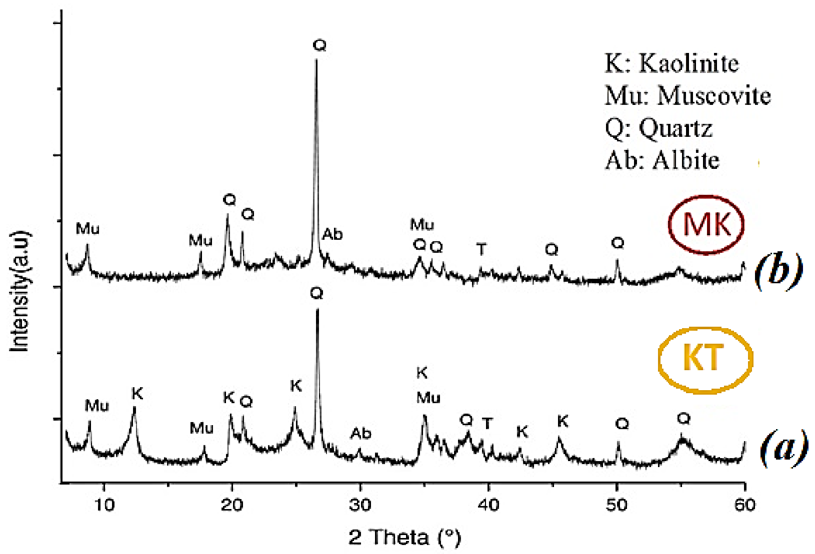

Fig. 4 XRD patterns of KT starting kaolin clay (a) and thermally treated KT kaolin at 750°C for 90 minutes (b)

After determining optimal calcination parameters, a sufficient quantity of the KT was calcined in a 3m^3 gas furnace. The calcined kaolin is then ground into fine powder in a ball mill and then sieved through a sieve with apertures of 80 µm. Figure 5 shows the synthesized MK-metakaolin powder.

Fig.5. Crushed MK-metakaolin

The main physical and chemical characteristics of the synthesized MK metakaolin were investigated.

3.2 MK metakaolin characterization

3.2.1 Main physical properties: Absolute density and Specific surface area

The main physical properties values of Portland cement and metakaolin are reported on table 3. It should be noted in particular that the MK metakaolin specific surface value is over 15 times higher than Portland cement. By contrast, the absolute dry density of cement exceeds that of MK metakaolin.

Table 3

Main physical characteristics of Portland cement and the synthetized Metakaolin

Property	MK Métakaolin	CPA-CEM II/A 42.5N cement
absolute dry density (g/cm3)	2.70	3.10
specific surface area (m^2/g)	15.00	0.34

3.2.1 XRF analysis

The chemical composition results of metakaolin MK determined by X-ray fluorescence (XRF) is listed in Table 4.

Table 4

Chemical composition of Synthetized Metakaolin

Type	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	TiO_2
Percent by mass	55.08	36.87	2.66	0.09	0.45	3.27	0.10	0.27

3.3 Effect of MK metakaolin as partially cement replacement on the compressive strength of standard mortars

Effect of MK-metakaolin as partially cement replacement on the compressive strength of standard mortars was investigated. Formulations studied are listed on table 6 above.

Figure 6 shows evolution of the compressive strength of mortar at 7 and 28 days for different formulations, as a function of the metakaolin replacement levels.

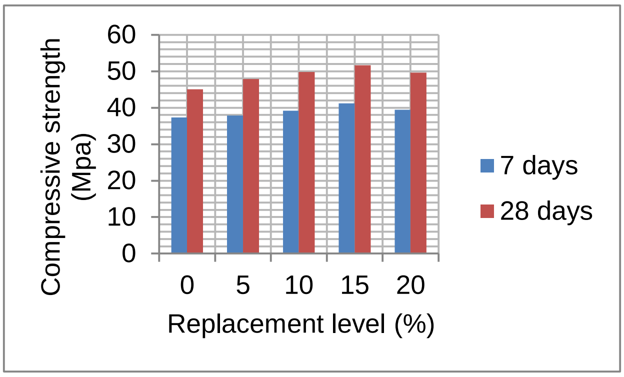

Fig.6. Effect of metakaolin (MK) on the compressive strength of mortars

The results indicated that 15% Metakaolin exhibited the highest 28 days as well as the 7 days compressive strength followed by 10% metakaolin, 20 % metakaolin and 5% metakaolin respectively.

Realistically, the optimum percentage of metakaolin that exhibited the maximum compressive strength is dependent on the cement type and content, type and dosage of admixtures, and also on the age of testing. In addition, some authors believed that the w/b ratio is the main factor that governs the optimum content of metakaolin [7] while others believed that the calcination temperature and its period is the main factor that governs the optimum metakaolin content. As well, other authors believed that the chemical composition constitutes the major factor that affecting the optimum metakaolin content.

In general the optimum percentage of metakaolin that exhibited the maximum compressive strength is dependent on the cement type and content, type and dosage of admixtures, and also on the age of testing [12].

Accordingly, results differ regarding the optimum metakaolin replacement percentage for obtaining maximum strengths of concrete/mortar/pastes. However, the vast majority of authors agree that the pozzolanic reaction between metakaolin and Calcium Hydrates (CH) improves the mechanical characteristics (compressive strength) [5, 10, 17] mainly at early ages [18].

4. Conclusion

The conclusions of this study can be summarized as following:

1) Metakaolin, pozzolanic additive, may be obtained by calcination of processed Algerian kaolin clay from El Milia at a temperature of 750 °C with a heating time of 90 min.

2) The optimum replacement level of CEM II/A 42.5 N cement by MK-metakaolin is 15 %, which gave the highest compressive strength in comparison to that of other replacement levels.

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