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Effect of Metakaolin as Partial Substitution to Portland Cement on the Mechanical and Durability Properties of High Performance Concrete

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Abstract In Burkina Faso, concrete remains the most used building material. Although several studies have been carried out for development of special concretes that have high performances, all methods for the formulation of concretes do not ensure the appropriate performances needed in buildings. This study is part of a project attempting to develop building materials that satisfy sustainable development in the construction area. It investigates the feasibility of using local materials such as metakaolin in partial substitution of Portland cement for formulation of High Performances Concretes (HPC). The physico-mechanical and durability properties were tested on fresh and hardened HPC incorporating 0-30 % metakaolin with respect to the mass of Portland cement. The results showed that High Performances Concretes containing metakaolin present higher mechanical performance than the reference concrete containing only Portland cement. The compressive and tensile strength reached 88 MPa and 7 MPa at the age of 28 days, respectively. It was also revealed that the incorporation of metakaolin contribute to the reduction of water absorption and porosity, and improvement of the resistance to the acid attacks.

Keywords: *high performance concrete, metakaolin, durability, mechanical performance*

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1. Introduction

Civil construction projects in Africa are becoming more important for developing infrastructures that would sustain the growth of the continent. However, these projects are highly constrained mainly due to the nature of the environment and available building materials, on the one hand, and complexity of the works to be carried out, on the other hand. In order to account for these constraints, it is necessary to use materials offering high mechanical performance and appropriate durability with regard to the aggressive environment.

In general, the most widely used building material in Africa and globally remains concrete. Unfortunately, the raw materials used for its production has strong negative environmental and economic impacts [1,2,3]. Alternative solutions must be found in order to reduce greenhouse gas emissions, using local resources which are more accessible and less polluting, in formulation of concretes in general and particularly high performance concretes for the construction of large superstructures [4].

High performance concretes are characterized by a compressive strength at the age of 28 days (fc28) between 50 and 100 MPa. They have better mechanical and durability characteristics compared to common concretes (fc28 less than 50 MPa). Their formulation requires the use of superplasticizer in order to obtain a good rheology while maintaining a water/binder ratio lower than 0.4 [5]. Most of HPC contain supplementary materials such as silica fume, rice husk ash and metakaolin, which contribute to improving their mechanical strength and durability [6,7,8]. All these properties allow achieving stronger civil engineering structures and reduction of their overall cost. Structural lightening which correlates with the current architectural requirements and long-term maintenance benefits clearly make high performance concrete one of the building materials of the 21st century.

This study contributes to the use of metakaolin produced locally in Burkina Faso as supplementary material to the formulation of HPC. It specifically evaluates the mechanical performances and durability of HPC through a series of laboratory tests on fresh and hardened concrete.

2. Materials and Experimental Methods

2.1. Materials

2.1.1. Cement

The cement used in this study is a CEM II A/L 42.5 produced in Burkina Faso by CIMBURKINA. Table 1 shows its physical properties. The results of analysis showed that this cement contains between 80 and 94 % of clinker and 6 to 20 % of limestone [9].

Table 1. Physical properties of the cement

Properties	Value
Specific weight(t/m^3)	3.22
Apparent weight (t/m^3)	1.03
Blaine specific area (m^2/g)	1.265

2.1.2. Metakaolin

The metakaolin (MK) used in this study resulted from calcination of kaolin from the locality of Saaba-Burkina Faso (12°22′46″N; 1°24′38″W and 317 m altitude). Kaolin (K) was ground and sieved to collect the undersize on 125 microns before calcination at 700 °C for 3 hours at a heating rate of 10 °C/min [10]. The obtained metakaolin has an apparent density of 0.59 g/cm^3 and a specific density of 2.63 g/cm^3, as determined according to the NF E N 1097-7 French standard [11,12]. The specific surface area determined using BET method is 14.8 m^2/g. The chemical composition of MK shows the predominance of silica (57.85 %) and alumina (38.30%), with a molar ratio SiO_2/Al_2O_3 of 2.57 (Table 2).

Table 2. Chemical Composition of Metakaolin [10]

Oxide [%]	SiO_2	Al_2O_3	CaO	Fe_2O_3	K_2O	Na_2O	MgO	MnO_2	TiO_2	P_2O_5	LOI
MK	57.85	38.30	0.05	2.30	0.23	0.11	0.09	0.01	0.09	0.02	1.01

2.1.3. Aggregates

The local sand was used for this study. It was sieved on 5 mm and then washed to eliminate the fine particles such as clay particles. The granulometric analysis performed according to the NF P 94-056 French Standard shows the grain size between 0.1-5 mm (Figure 1).

Table 3. Physical properties of Aggregates

Properties	Value	
	Sand	Gravel
Specific weight (t/m^3)	2.72	2.69
Apparent weight (t/m^3)	1.68	1.47
Fineness modulus (%)	2.10	-
Coefficient of Hazen (Cu)	3.20	-
Coefficient of curvature (Cc)	0.80	-

The used gravel is a granitic rock extracted from a local quarry. It has granular size in range of 5/10 mm after sieving and washing to eliminate smaller particles (Figure 1). The physical properties of the aggregates are summarized in Table 3.

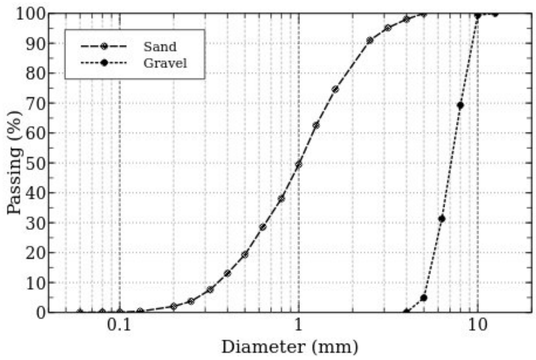

Figure 1. Particle size distribution of aggregates

2.1.4. Water and Superplasticizer

The mixing water was tap water provided by the national office for water and sanitation (ONEA)in Burkina Faso. The superplasticizer known by its commercial name of viscoconcrete was used in this study

2.2. Mix Design and Sample Preparation

The method proposed by Feret was used for the mix design (Equation 1). The goal is to produce concrete which has a compressive strength of 85 MPa at 28 days of curing and slump of class S4 on the Abrahams' cone.

$$f_{c28} = G \times \sigma_{l28} \times \left(\frac{V_C}{V_C + V_v + V_e} \right)^2 \quad (1)$$

Where G is the granular coefficient, σ_{l28} the true strength of the cement, V_c the volume of cement, in the present study V_c correspond to the volume of the binder (cement + metakaolin), V_e the volume of water and V_v the volume of occluded air.

The binder consists of cement and metakaolin at different substitution ratios (Table 4). The aggregates consist of a binary mixture of granular classes 5/10 for granitic gravel and 0/5 for sand. The mass ratio of water/binder was kept constant at 0.225 for all formulations. The superplasticizer was adjusted to maintain a constant slump class (S4) on the Abram's cone, between 16 et 21 cm, in accordance to the standard NF EN 206-1 [13]. Thus, six (6) concrete formulations were proposed by partially substituting the cement with 5-30 % metakaolin, plus a reference formulation with 100 % Portland cement (Table 4). Cylindrical specimens having diameter x height of 10×20 cm were made and cured in water saturated with lime for 1, 7 and 28 days. After curing, the specimens were tested according to the test campaign described below.

Table 4. Mass proportions of different constituents in 1 m^3 of concrete

Components		Mix designs						
		PC	05%MK	10%MK	15%MK	20%MK	25%MK	30%MK
Gravel (kg)		834	834	834	834	834	834	834
Sand (kg)		788	788	788	788	788	788	788
Binder	Cement (kg)	679	645	611	577	543	509	475
	Metakaolin (kg)	0	34	68	102	136	170	204
Water (kg)		153	153	153	153	153	153	153
Superplasticizer (mass % of binder)		3.5	3.5	3.5	3.5	3.5	3.5	3.5

2.3. Methods

2.3.1. Testing the Compressive Strength

The compressive test intended to determine the compressive strength of the concretes. The specimens were tested after 1, 7 and 28 days of curing by applying an increasing load until the occurrence of the fracture. The compressive strength (Rc in MPa) was calculated as the ratio between the fracture load (P in N) and cross section area (S in mm²) of the specimen (Equation 2). The fracture load (P) is the maximum load recorded during the test, which was carried out in accordance with French standard NF EN 12390-3 [14].

$$R_c = \frac{P}{S}. \tag{2}$$

2.3.2. Testing the Tensile Strength

This test aims to determine the indirect tensile strength of the specimens. The test was carried out by applying to the test-tube a compression force along two opposite generatrices, according to standard NF EN 12390-6 [15]. This compression force induces tensile stresses in the plane passing through these two generatrices. If h is the height (mm) of the specimen, d its diameter (mm) and P the applied load (N), the splitting failure stress (MPa), R_t, is determined with the formula in Equation 3:

$$R_t = 0.637\frac{P}{d \times h}. \tag{3}$$

2.3.3. Pundit Test: Dynamic Modulus of Elasticity

The pundit test is a non-destructive test aiming at evaluating the quality of the concrete. The principle of the test consists on measuring the speed of sound within the concrete using an ultrasonic speed tester (Figure 2). The dynamic modulus of elasticity (E_d in MPa) of the concrete is evaluated by Equation 4 which relates it to the density of the concrete (ρb in kg/m^3), the Poisson's ratio of the concrete (ν) taken equal 0.15 [16] and longitudinal velocity of the sound (V in km/s).

Figure 2. Pundit test setup

$$E_d = V^2 \rho_b \frac{(1+\nu)(1-2\nu)}{1-\nu}. \tag{4}$$

2.3.4. Water Accessible Porosity

The water accessible porosity measures the percentage of voids inside the mass of the specimen which are connected to the surface. The test was performed according to standard NF EN 18-459 [17] by saturating the specimens with water at 20°C. The test principle is consisted of determining the mass of specimen in dry state and in saturated state by hydrostatic weighing. The calculation of the water accessible porosity expressed as a volumetric percentage is given in Equation 5:

$$P_{water} = \frac{M_{air} - M_{dry}}{M_{air} - M_{water}} \times 100. \tag{5}$$

With P_{water}: the water accessible porosity in %, M_{air}: mass of the saturated specimen weighed in air in kg, M_{dry}: the mass of the specimen dried at 105 ± 5 ℃ up to constant mass in kg and M_{water}: mass of the saturated specimen weighed while submerged in water in kg.

2.3.5. Capillary Absorption

The capillary absorption measures the ratio of absorption of water by capillary suction of the unsaturated concrete samples, brought into contact with water without hydraulic pressure. In this study, the test is conducted according to the AFPC-AFREM procedure [18]. The test is performed on truncated cylindrical specimens with a height between 60 and 100 mm and a diameter of 100 mm. At the end of the test, the absorption coefficient is determined by Equation 6.

$$C_a = \frac{M_X - M_0}{A} \tag{6}$$

With C_a: coefficient of capillary absorption in kg/m^2, M_x: mass of the specimen at a given time in kg, M_0: initial mass of the specimen in kg and A: surface of the specimen in contact with the water in m^2.

2.3.6. Acid Attack

The acid attack test consisted of measuring the mass loss of the concrete specimens immersed in a nitric acid solution (HNO_3) concentrated at 5 %. HNO_3 was chosen because it is one of the acids mainly found in the analysis of acid rain [19,20]. After curing, the specimens were immersed in solution of nitric acid. For each specimen, the loss of mass is measured after 1, 7, 14, 21 and 28 days of immersion in the acidic solution.

3. Results and Discussion

3.1. Characteristics of the Fresh Concrete

The apparent density and slump were measured on various formulations of fresh concretes. The results of the tests are given in Table 5.

Table 5. Properties of the fresh concrete

Compositions	Apparent density (kg/m^3)	Slump (cm)
PC	2457.27	19.1
05% MK	2452.04	20.9
10% MK	2441.40	21.0
15% MK	2444.88	20.7
20% MK	2421.32	21.0
25% MK	2416.68	20.2
30% MK	2409.20	20.4

We notice a decrease in the apparent density as the substitution ratio increases. This is related to the specific density of cement which is higher than that of metakaolin. The values of the slump of the concrete vary a little and remain in agreement with the hypotheses of the formulation. The formulated concretes have a slump between 19 and 21 cm.

3.2. Mechanical Properties

3.2.1. Compressive Strength

Figure 3 shows the evolution of the average values of the compressive strength (tested on three specimens) for all formulations at 1, 7 and 28 days of curing.

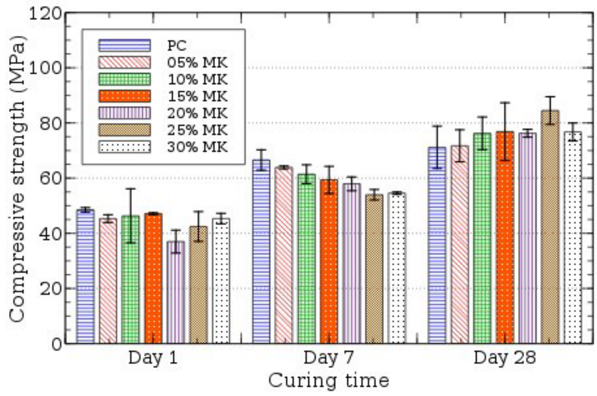

Figure 3. Evolution of the compressive strength

It can be noticed that after 1 day of curing, all formulations have almost the same compressive strength. After 7 days, there is a slight decrease in the compressive strength with the increase of the substitution ratio of the cement by the metakaolin. However, the trend reverses after 28 days. At this age, the results show that the values of the compressive strength increase with the substitution ratios (10 %, 15 %, 20 %, 25 % and 30 %). The incorporation of metakaolin improves the compressive strength of concrete at 28 days of curing. This is explained by the pozzolanic nature of metakaolin, whereby metakaolin reacts with the portlandite formed from the hydration of cement to form secondary cementitious products [21]. Wild et al. [22] reported that the incorporation of metakaolin enhances the adhesion between the paste and the aggregates resulting in increasing the mechanical strength of the concrete. The composition with the most significant increase of the compressive strength at 28 days is the one with a substitution ratio of 25%. For the concrete containing 30 % of metakaolin, the compressive strength tends to decrease but remains higher than that of the reference concrete. The same trend has been observed on mortars in the literature [11]. This decrease can be explained either:

-By the compactness of the granular skeleton which decreases for a substitution ratios higher than 25%, whereby the metakaolin might be heterogeneously dispersed and tend to agglomerate.

-By the excess amount of metakaolin compared to that of available portlandite. The additional amount of metakaolin would not lead to the formation of cementitious phases contributing to the mechanical strength of the material.

The evolution of the compressive strength over time for the reference concrete (without metakaolin) is controlled by the speed of hydration of the cement. For concretes containing metakaolin, this evolution depends on the rate of hydration of the cement, which is responsible for the formation of portlandite and kinetic of the pozzolanic reactions of metakaolin and portlandite. Indeed at 1 day of curing, the compressive strength reached the maximum value for the reference concrete which is related to the early hydration effect. At 28 days, the strength of the reference concrete is lower than that of the concrete with 25 % metakaolin. Thus 25 % can be considered the optimum substitution ratio to reach the maximum compressive strength in the present study unlike Poon et al. [23] and John [24] who find an optimum ratio of 10 %. These results suggest that the compressive strength at early age (1 day curing) is related to cement hydration. The effect of metakaolin becomes obvious on increasing the compressive strength between 7 and 28 days of curing. This corresponds to a strong development of pozzolanic reaction which contributes to further densification of the cementitious matrix and thus to the increase of the compressive strength.

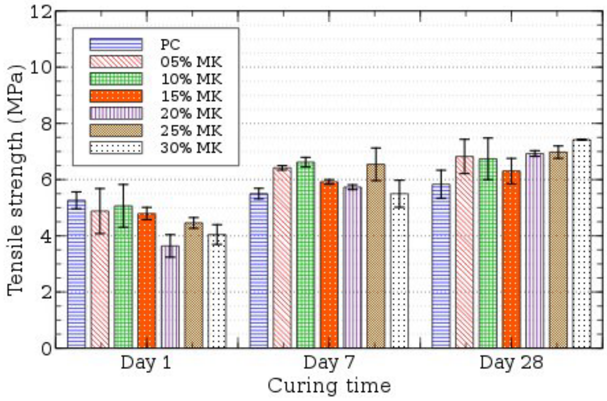

Figure 4. Indirect tensile strength

3.2.2. Indirect Tensile Strength

The indirect tensile strength presented in Figure 4 behaves similar to compressive strength (Figure 3). Indeed it is shown that the samples incorporating metakaolin have a lower tensile strength than the reference concrete at 1 day of curing; but they offer better strength at 7 and 28 days of curing. The highest tensile strength at 28 of days curing is obtained for a substitution ratio of 30 %. Few studies report indirect tensile strength on concretes incorporating metakaolin. Nevertheless, the works of Rougeau and Borys [25] as well as that of Kulkarni and Vipat [26] showed similar results.

3.2.3. Dynamic Modulus of Elasticity

Figure 5 shows the evolution of the dynamic modulus with respect to metakaolin substitution ratio. The results indicate a decrease in the dynamic modulus on all concretes containing metakaolin compared to the reference concrete. Since the dynamic modulus is a function of the density, a correlation between these two parameters is observed. The evolution of the dynamic modules for the different formulations has the same trend as the corresponding ultrasonic speeds, as previously reported by Khatib and Clay; De Larrard and Helson et al. [22,27,28,29].

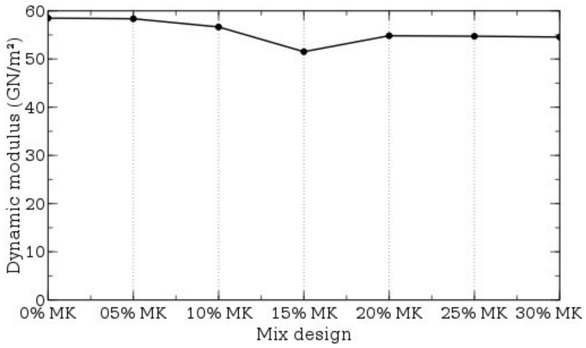

Figure 5. Dynamic modulus

3.3. Durability

3.3.1. Water Accessible Porosity

Figure 6 shows the evolution of the water accessible porosity with the substitution ratio. The results indicate that incorporation of metakaolin tends to decrease the water accessible porosity in concretes. The optimal substitution ratio was found to be 30 %. This is explained by the densification of the cementitious matrix due to the supplementary products from the pozzolanic reactions. The formation of supplementary hydrates reduces the water absorption capacity of the concrete. These results agree with those of Ramezanianpour et al. [30] and Colak [31].

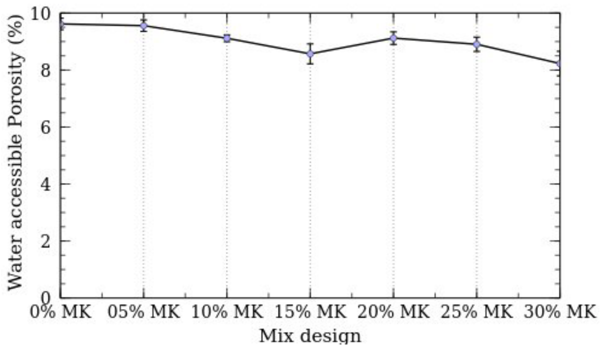

Figure 6. Water accessible porosity

3.3.2. Capillary Absorption

Figure 7a shows the evolution over time of the coefficient of capillary absorption for the different substitution ratio. These results show that this coefficient decreases with the increase of the metakaolin substitution ratio. The water capillary absorption is the highest for the reference concrete and decreases with increasing ratio of metakaolin reaching the minimum at 30 % substitution ratio.

As some studies showed, the addition of metakaolin can contribute to reducing the capillary absorption of concrete through the development of pozzolanic reactions [32]. The pozzolanic reaction forms new products which make the capillary network more complex, making it difficult for water penetration.

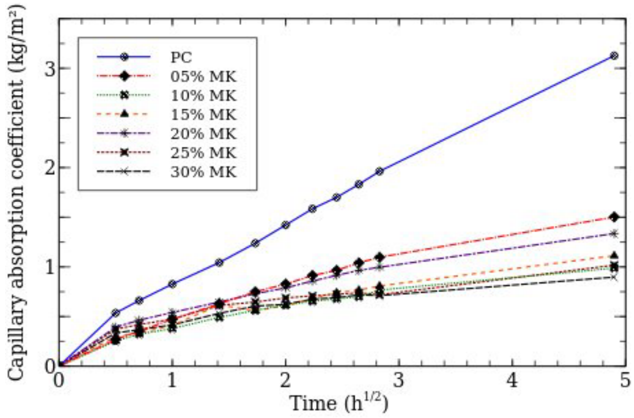

Figure 7a. Evolution of the coefficient of capillary absorption

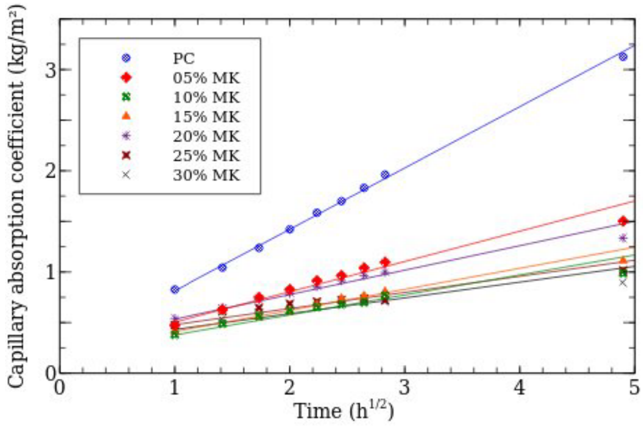

Figure 7b. Evolution of sorptivity

The more the substitution ratio of metakaolin is increased, the more the capillary porosity is affected and the lower is the water absorption coefficient. Khatib and Clay [27] confirmed this observation by comparing the water absorption coefficient with the porosity distributions of concretes.

The sorptivity was determined as the slop of coefficient of capillary absorption between 1 and 24 hours in accordance with Badogiannis et al. [33] (Figure 7b). It is directly related to the absorption rate and qualitatively reflects the pore size in the concrete (Table 6). Having the lowest coefficient of absorption, samples with the highest metakaolin substitution rate (30 % substitution by mass of cement) was found to have the lowest sorptivity and therefore the lowest mean pore radius.

Table 6. Evolution of water capillary absorption and sorptivity

Compositions	Water capillary absorption coefficient at 24 hours Ab_{24h} ($kg.m^{-2}$)	Sorptivity ($kg.m^{-2}.h^{1/2}$)	Linear correlation coefficient (R^2)	*Pore radius (r)
PC	3.127	0.597	0.999	+ + + + + +
05% MK	1.502	0.262	0.968	+ + +
10% MK	0.988	0.151	0.958	+ +
15% MK	1.112	0.171	0.956	+ +
20% MK	1.335	0.204	0.978	+ +
25% MK	1.017	0.125	0.952	+
30% MK	0.897	0.114	0.911	+

*Qualitative estimation of capillary pore radius: ++++++: the largest; +: the smallest.

This test confirms the results of the water accessible porosity and shows that there is a reaction in the concrete specimens containing metakaolin which improves the porous network thereof. Indeed, according to Cassagnabère et al. [34], the absorption of water is induced by the capillary pores whereas the sorptivity reflects the global evolution of the capillary pore size. Washburn's theory [35] showed a relationship between water accessible porosity, sorptivity, and mean capillary pore radius. From the results in Table 6, it could be concluded that increasing the substitution ratio of cement with metakaolin reduces capillary pore size. This is inherent to the pozzolanic reaction of metakaolin in the presence of portlandite resulting from the hydration of cement.

3.3.3. Acid Attack

Figure 8 shows the loss of mass of the different compositions resulting from the acid attack. The results show that the reference concrete (PC) undergoes a greater loss of mass than that incorporation metakaolin. This loss of the mass decreases with increasing substitution ratio of metakaolin.

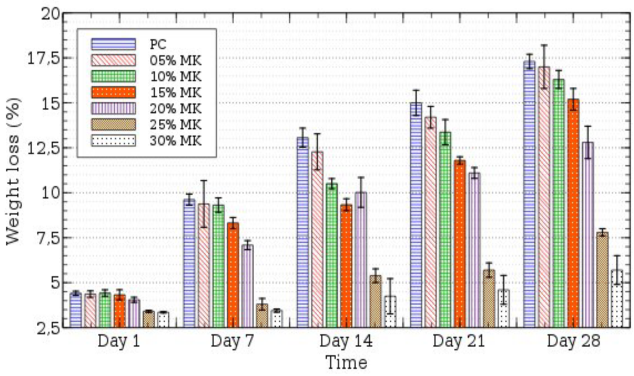

Figure 8. Evolution of the weight loss due to acid attack

The mass loss is more important for the reference concrete and is explained by a decalcification of the cement matrix related to the dissolution of the hydrates in the matrix whose main component is calcium [31]. Different types of hydrates have very different dissolution rate: calcium hydroxide is more soluble than calcium silicates hydrates (C-S-H) and the solubility of C-S-H decreases with decreasing C/S ratio. Concretes made with metakaolin and low water/binder ratio have high compactness and therefore good resistance to acid attack [36,37]. Therefore metakaolin improves the resistance to acid attacks. This is basically achieved by improving the compactness of the cement matrix which limits the penetration of aggressive agents and through the pozzolanic reaction which transforms the calcium hydroxide (most sensitive to dissolution) into C-S-H more stable chemically.

4. Conclusion

The aim of this study was to investigate the feasibility of high performance concretes (HPC) incorporating metakaolin locally sourced from Burkina Faso, i.e. concretes with compressive strength between 50 MPa and 100 MPa at 28 days of curing. The results showed that the concrete incorporating metakaolin achieves very good properties in fresh state such as density and workability exceeding 2400 Kg/m^3 and slump greater than 20 cm. The mechanical properties of hardened concrete have shown that the compressive and indirect tensile strength of concrete containing metakaolin are greater than those of the reference concrete at 28 days of curing. The compressive strength of concrete with 25 % substitution of cement by metakaolin exceeded 80 MPa which is far greater that the minimum value of 50 MPa required for HPC. Nevertheless, at early age between 1 and 7 days of curing, the reference concrete (PC) presents higher strength than the concrete with metakaolin substitutions. The improvement of the compressive strength of concrete containing metakaolin is mainly related to the pozzolanic reaction between portlandite and metakaolin which take place over time. Moreover, different tests showed the improvement of different indicators of the durability when metakaolin was incorporated in the concrete. The incorporation of metakaolin results in decreasing the water accessible porosity, capillary absorption and mass loss due to acid attack. From this study, it can be concluded that concrete incorporating 25 % metakaolin substitution achieves optimal mechanical and durability performances.

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