The Use of Local Metakaolin in the Formulation of Self-Compacting Concrete, Physico-Mechanical Characterization

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Abstract

Today, self-compacting concrete (SCC) is a material used in Civil Engineering, especially for construction. In order to find new economic solutions in addition to technical solutions, research on economic SCC has been conducted in recent years. However, the very large amount of mineral addition in SCC formulation suggests that the use of natural, local and economic mineral additions seems to be one of the possible solutions for the future of SCC. Thus, work has shown that it is possible to formulate self-compacting concrete containing different percentages of local metakaolin -Bechar region - (5%, 10%, 15%, 20%, 30%) for an economic and ecological purpose with quantities of cement well determined. This work shows that the substitution rates of 05%,10% and 15% of cement with metakaolin increases the compressive and bending strength at young age and in the long term. Durability was also shown at 15% a major increase in the absorption coefficient,and a minor 10% porosity. In addition, non-destructive testing methods were conducted as part of this research to obtain more information on the properties of the self-compacting concrete studied. The results of the tests on the new SCC meet the recommendations of the French Civil Engineering Association (AFGC).

Keywords: self-compacting concrete; local metakaolin; compressive strength; Non-destructive testing; Durability.

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1. Introduction

The environmental impact of cement manufacturing is of increasing concern. An effective way to reduce this impact by using additions from by-products having a pozzolanic character as a partial substitution of cement. for this purpose, important studies have been carried out on the use of various additions for the partial replacement of cement in SCC or in self-placement mortar such as marble powder (Guneyisi *et al.*, 2009; Uysal and Sumer, 2011), limestone powder (Uysa and Yilmaz., 2011), basalt powder (Uysal and Sumer, 2011; Uysal and Yilmaz, 2011), fly ash(Khatib, 2008; Sahmaran *et al.*, 2009) and slag (Boukendakdji *et al.*, 2009; Khatib and Hibbert, 2005).

This strategy will have the potential to reduce costs, save energy and reduce waste volumes. The introduction of metakaolin (the result of kaolin calcination) seems to be becoming an advantage. CO_2 emissions from combustion account for about 8% of global CO_2 emissions (Olivier *et al.*, 2015; Alujas *et al.*, 2015; Andrew, 2018).

Chemical reactions that develop during the manufacture of cement, release carbon dioxide (CO_2) thus contributing to the increase of the greenhouse effect its production requires a high energy consumption and that the production of one tonne of cement re-leases approximately as much carbon dioxide into the atmosphere (Perlot and Rougeau, 2007). While the mineralogical transformations governing kaolin dehydroxylation only generate water vapour. In addition, metakaolin does not compete with cement. It complements and improves it more than it replaces it. The use of MK for concretes has received considerable attention in recent years due to its high pozzolanic reactivity and improved of long-term sustainability (Guneyisi and Mermerdas, 2007). Metakaolin improves the performance of concrete through two actions: the first is physical due to its fineness allowing the filling of pores while the second is chemical due to the pouzzolanic character of the metakaolin (Guneyisi and Mermerdas, 2007). In addition, the incorporation of metakaolin in partial substitution of cement is interesting because it improves several properties (durability, resistance, transfer properties, etc.). Some authors have found that an addition of approximately l5% of Metakaolin confers superior resistance in aggressive environments, such as seawater and acid solutions (Hong-Sam *et al.*, 2007).

2. Materials and methods

2.1 Cement

The cement used is type CEM I 42.5. It is a grey cement resistant to sulphates, result of the grinding of a clinker containing a low level of calcium aluminates with a lower proportion of gypsum than a composite Portland cement. Table 1 shows the chemical composition of cement by XRF. It is a Portland cement type CEM I 42.5 N-SR3. It complies with the national standard NA 442 v 2013 and the European standard EN 197-1 with a C_3A rate < 3%. It has a specific density of 3.01 t/m^3.

Table 1 – Cement chemical composition by XRF.

Component	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	Tio_2	Cl	C_3A	C_4AF
Cement / %	18.77	3.94	6.49	63.02	2.55	2.06	0.02	0.36	0.41	0.027	0.532	19.749

Table 2 - Chemical composition of cement. (Zouini *et al.*, 2023).

Component	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	LOI
Cement / %	20.05	6.40	3.45	61.23	1.2-3	2.06	0.36	0.5-3

Table 3 – Densities of cement from the literature.

Authors	Density reported / (g/cm^3)
Agha et al. 2023 (Agha *et al.*, 2023)	3.05
Rikioui et al. 2021 (Rikioui *et al.*, 2021)	3.14

2.2 Metakaolin

Removal of source rock: Clay mineral kaolinite is formed by the decomposition of feldspar by the action of water and carbon dioxide. Samples of kaolin were taken in the open air.

2.2.1 Preparation of kaolin powder:

- Extraction;
- Grinding with Los Angeles;
- Jaw crusher;
- Sift at 80 µm.

Figure 1 - Device for the preparation of metakaolin.

2.2.2 Kaolin heat treatment:

Metakaolins are obtained by calcination of kaolinic clay (hydrated alumina silicate) at temperatures between 600 and 900°C. The calcination temperature depends on the degree of purity in kaolin (otherwise known as kaolinite). Calcination causes deshydroxylation and destruction of the initial crystalline structure of kaolinite (calcined kaolinite). The heat treatment time used is five hours. (Badogiannis and Tsivilis, 2009).

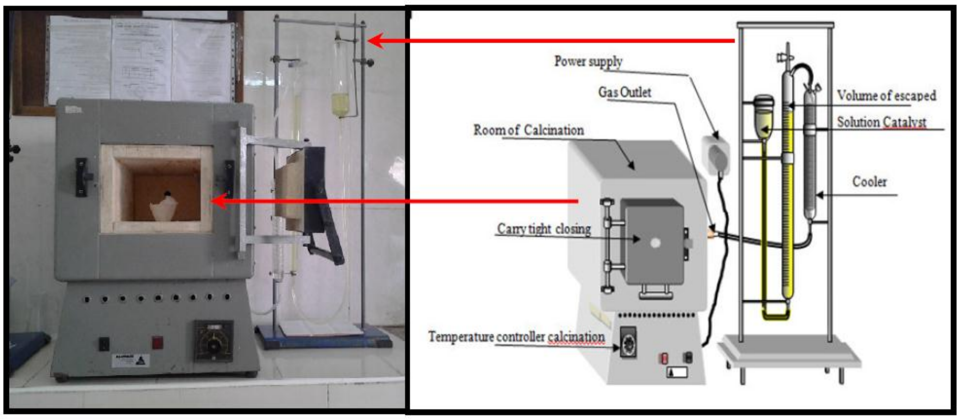

Figure 2 - Gas measuring device (muffle furnace – Calcimeter).

$$Al_2O_3.2SiO_2.2H_2O \longrightarrow Al_2O_3.2SiO_2.2\ (1\text{-}\alpha)\ H_2O + 2\ \alpha\ H_20 \quad (1)$$

450°C to 800°C

Kaolinite ⟶ Metakaolin

α is the dehydroxylation coefficient.

- It can be obtained by calcination of Kaolin (primary) or Kaolinic clays (secondary Kaolins);
- It is an artificial pozzolane with a particular reactivity to lime.

$$Kaolin \longrightarrow Métakaolin + 2\ H_2O\ gaz \quad (2)$$

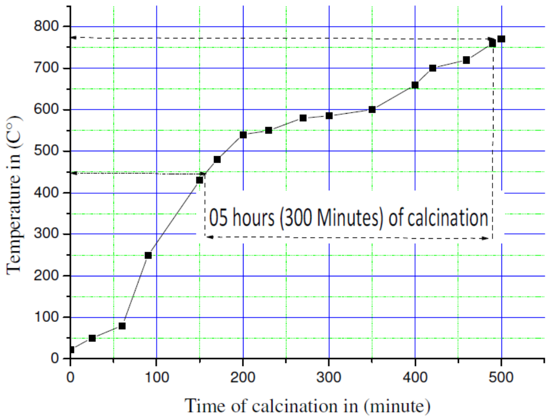

Figure 3 - Cycle thermal processing of the Tabelbala kaolin.

2.2.3 Mass loss during incineration:

Table 4 – Loss of mass of different samples.

crude kaolin /gr	Kaolin after calcination / gr	quantity of water / gr	quantity of water / %
435	396.9	38.1	08.76
408.9	370	38.9	09.51
430	395.5	34.5	08.02
452.2	411.7	40.5	08.96
513.5	467.9	45.6	08.88
1030.7	900.2	130.5	08.73
400.1	365	35.1	08.77

According to the table above, the mass loss varies between 8 and 10% regardless of the mass of the sample.

2.2.4 XRF Chemical Analysis:

Table 5 – Chemical composition of kaolin and MK by XRF.

component	Kaolin / %	MK / %
SiO_2	46.84	50.36
Al_2O_3	28.09	29.83
Fe_2O_3	04.95	05.53
Cao	01.70	01.61
MgO	00.27	00.29
SO_3	00.50	00.41
K_2O	01.32	01.54
Na_2O	00.12	00.13
Cl	00.161	0.004

2.2.5 Density:

The mean values of metakaolin densities reported in the literature are in the order of 2.5 kg/dm^3 (Table 6). These values may fluctuate depending on the mineralogical composition of the parent rock but also depending on the method of manufacture of the metakaolins. Indeed, thermal treatments cause a profound change in the mineralogical composition and morphology of particles. According to the work of Measson. (1981) (Measson, 1981) the maximum temperature of the heat treatment of metakaolins is also a factor influencing the density.

Table 6 – Densities of metakaolins from the literature.

Authors	Density reported / (kg/dm^3)
Gruber *et Al.* (2001) (Gruber *et al.*, 2001)	2.50
Courard *et Al.* (2003) (Courard *et al.*, 2003)	2.54

Particle size analysis (NF P 94-057):

Figure 4 - kaolin on the left and metakaolin on the right.

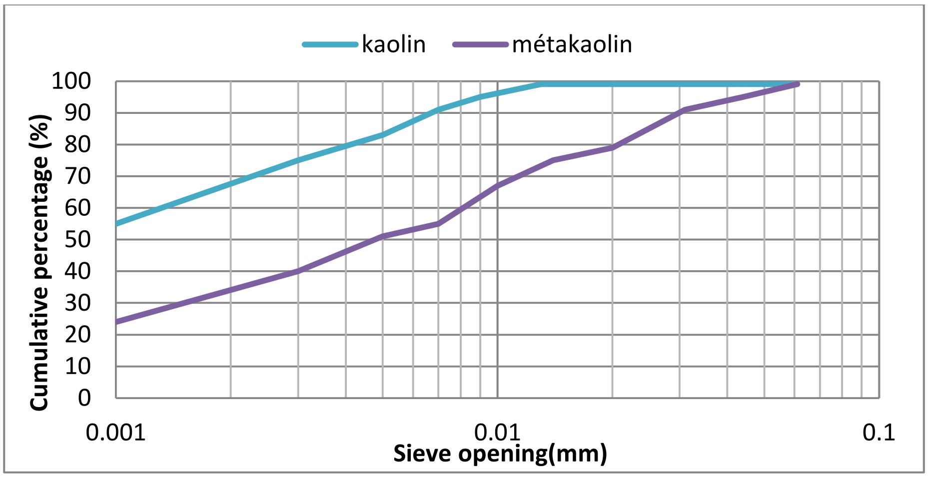

Figure 5 - Particle size analysis of kaolin and metakaolin by sideementometry.

2.3 Aggregates

Aggregates (sand and gravel) are the skeleton of concrete. They are defined as a set of mineral grains between 0 and 125 mm in size and must be chemically inert to cement, water and air. The nature of the rocks constituting the deposits is responsible for the intrinsic properties (density, resistance, porosity, reactivity, etc.) of the aggregates. (Baron and Ollivier, 1996)

The aggregates used in this study are crushed aggregates from a quarry at Hassi el 20 in Bechar. They are of three granular classes: Sand 0/3, Gravel 3/8 and Gravel 8/15.

Table 7 – Physical and mechanical characteristics of aggregates.

	Sand 0/3	Gravel 3/8	Gravel 8/15
Bulk density / (g/cm^3) **[NF P18-555]**	1.782	1.380	1.580
True density / (g/cm^3) **[NF P18-555]**	2.633	2.660	2.659
Equivalent sandy / (%) **[NF P 18-598]**	84.80	/	/
Fineness modulus [NFP 18-540]	02.61	/	/

2.3.1 Particle size analysis:

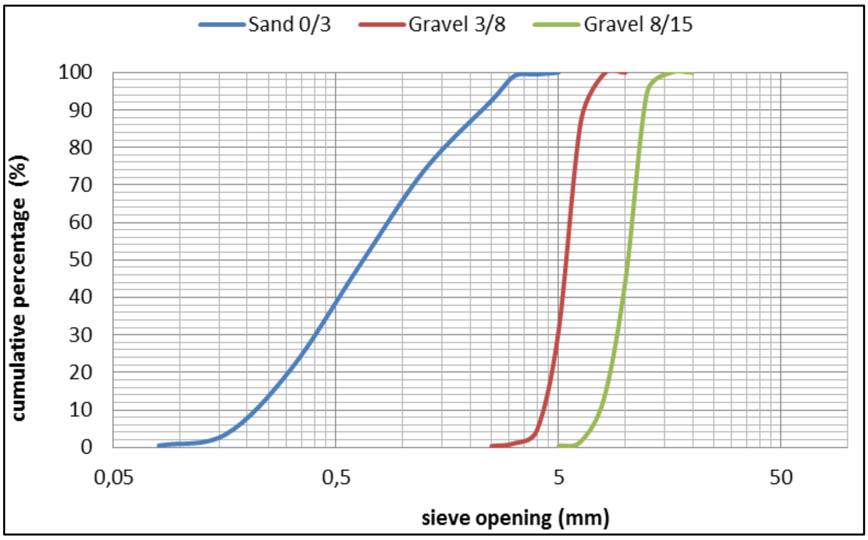

Figure 6 - Particle size distribution of aggregates.

2.4 Mixing water

The water used in this study is spring water.

Table 8 – Chemical analysis of the mixing water.

component	symbol	quantity / (mg/l)
Calcium	Ca	55
Magnésium	Mg	17
Potassium	K	0.5
Sodium	Na	≥12
Bicarbonates	Hco_3	210
Sulfates	So_4^{2-}	33
Chlorures	Cl	≥15
Nitrates	No_3^-	4.6
Nitrites	No_2^-	0
Silices	Sio_2	12
Dry residue at 180°c	/	372
PH	PH	7.8

2.5 Superplasticizer EN 934-2

It increases the workability, without reducing the long-term resistance of the mortar or concrete paste. The "water reducer" function is shown by the reduction of the E/C ratio for the same workability. This increases the mechanical performance of the mixture (Maher, 2003). The super plasticizer Sika ViscoCrete-665 used in this study is a versatile super plasti-cizer/high water reducer based on acrylic copolymer.

Table 9 – Sika® ViscoCrete®-665 Data Sheet.

Type	Sika® ViscoCrete®-665
Density	1,085 ± 0,015
PH value	5 ± 1,0
Total Ion Content Chloride	≤ 0,1%
Dosage	0,4 à 2% of the weight of the binder or cement depending on the desired fluidity and performance.
Sodium Oxide Equivalent	≤ 1,0%

3. Concrete formulation

We have already met the necessary conditions to guarantee the autoplacibility while relying on the compositions proposed in the specialized literature or the criteria recom-mended by the French Association of Civil Engineering (AFGC, 2008), it is a question of choosing the proportions of the constituents in 1 m^3 of the concrete, we propose by trial and error a formulation based on the following ranges:

- Ratio (gravel/sand) that must be close to 1;
- Ratio (water/cement) equal to 0.5;
- The cement dosage is rather high (the cement dosage is 520 kg/ m^3);
- The superplasticizer dosage must guarantee the fluidity of the mixture;
- The percentage of metakaolins (0%, 5%, 10%, 15%,20% and 30%) of cement mass;
- 300 l/m^3 ≤ volume of paste ≤ 400 l/m^3.

For this purpose we used standard steel moulds of prismatic shape (4x4x16) cm^3 and cylindrical shape (10x20) cm^2 which were removed 24 hours after casting and kept under water at an ambient temperature of 20°C. For each formulation, three samples were tested.

Table 10 – Composition of SCC with and without metakaolin (kg/ m^3).

Design- -ations	Cement CEM I	Meta- -kaolin	Aggregates			Superpla- -sticizer	Mixing water
			Sand 0/3	Gravel 3/8	Gravel 8/15		

	42,5					Sika® Visco Crete®-665	
SCC 0%	520	0	900	150	580	03.90	260
SCC 05%	494	26	900	150	580	04.66	260
SCC 10%	468	52	900	150	580	06.22	260
SCC 15%	442	78	900	150	580	06.39	260
SCC 20%	416	104	900	150	580	06.92	260
SCC 30%	364	156	900	150	580	08.30	260

4. Experimental procedures

4.1 Tests on fresh concrete

Slump-flow L– box Stability in sieve Density

Figure 7 - Fresh concrete tests as per "AFGC".

4.2 Tests on Hardened Concrete

4.2.1 Mechanical Testing: The bending tensile strength was determined using a 3-point bending machine according to [NF P18-407]. The two parts obtained after the rupture of each test piece will be subjected to the compression test.The standard compression test [NF P 18-406] consists of breaking the sample body between the two plates of a compression press. After this compression test, the split tensile test [NF P18-408] which consists in placing the cylinder (10x20) cm^2 horizontally between the press trays and the load in-creases until the rupture by indirect tensile force, which appears as a split along the vertical diameter of the cylinder.

a)Flexion b) Compression c) Splitting

Figure 8 - Various mechanical tests.

4.2.2 Water Accessible Porosity Test (NF P18-459): The main interest of metakaolins as ultrafine cementitious matrices lies in their ability to increase the compactness of the granular skeleton. Due to their small size, these fine particles can fill the gaps left by the cement grains. This filling capacity is related to the size, shape and degree of agglomeration of metakaolin particles. (Gaboriau *et al.*, 1996). In studying the evolution of the porosity of compacted powders have shown that particle morphology has a direct impact on their ability to fill a given volume.

$$\varepsilon = \frac{M_{air} - M_{dry}}{M_{air} - M_{w}}\ x\ 100\% \qquad (3)$$

M_W: The mass in grams weighed under water;

M_{air}: The mass of the test piece saturated with water;

M_{dry}: The mass in the air of the dry specimen.

4.2.3 Capillary Water Absorption Test (AFPC-AFREM, 1997): The capillary absorption test is performed after 28 days of treatment on test tubes to see the effect of the pouzzolanic reaction which is a slow reaction. The coefficient of absorption of water by capillarity w is calculated from the water absorbed between 1 and 24 hours according to the following relationship:

$$w = \frac{Meau}{S} \qquad (4)$$

W: absorption coefficient in [kg/m^2];

M_{eau}: mass of water absorbed;

S: section in contact with water in m^2.

Figure 9 - Capillary water absorption test.

4.3 Non-destructive testing

4.3.1 Ultrasonic test NF EN 12504-4:

The non-destructive tests were carried out on_cylindrical test pieces (10x20 cm^2) at the age of 28 days. For each specimen, the ultrasonic test was carried out in two directions by direct transmission so that the direction of measurement of the transit time was perpendicular to the direction of manufacture. The speed of ultrasonic propagation is given by the formula:

$$V = \frac{L}{T} \quad (5)$$

V: ultrasonic speed (m/s); L: Length of test piece (m); T: wave propagation time (s).

4.3.2 Sclerometer test NF EN 12504-2:

The resistance Rc is calculated directly from the mean value of the rebound measurements applied by a Silver Schmidt type ST/PC (N/L) sclerosis meter on the faces of the test piece. The strength of the concrete tested Rc can be related to the sclerometric index as follows Dupain and Saint-Arroman. (2009). (Dupain and Saint-Arroman, 2009)

$$Rc_I = \frac{I2}{32} \quad (6)$$

5. Results and Discussion

5.1 Fresh SCC

Table 11 – Values of the various fresh tests.

Designations	Slump-flow (cm)	L-box (%)	Stability in sieve (%)	Density (kg/m^3)

SCC 0%	74	85	08.35	2320
SCC 05%	72.50	83.70	07.16	2310
SCC 10%	80	87	09.20	2301
SCC 15%	78	85	08.80	2290
SCC 20%	72	81.30	06.16	2285
SCC 30%	77	83	06.95	2255

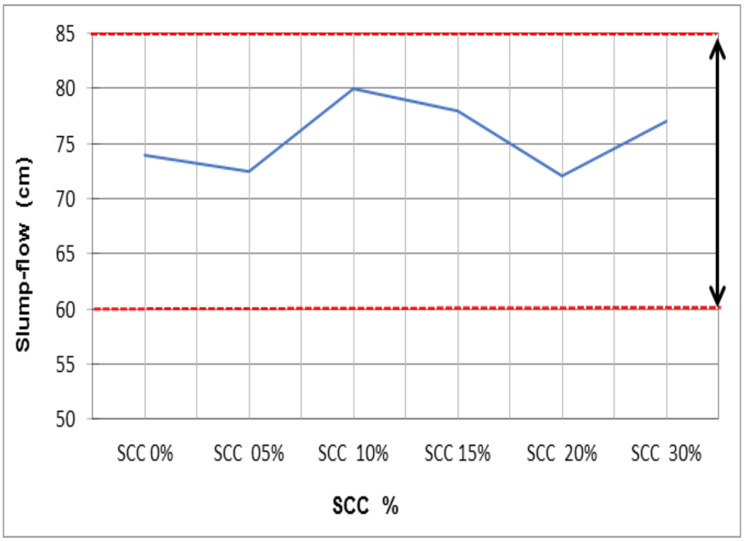

Figure 10 - slump-flow of SCC with and without metakaolin.

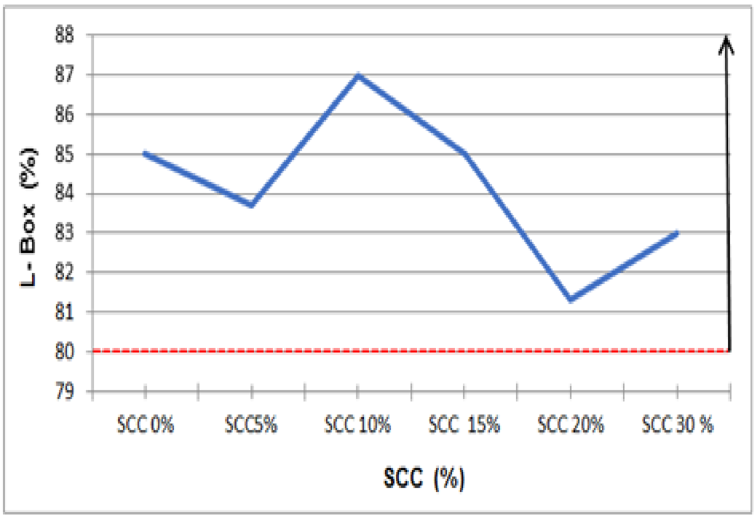

Figure 11 - Effect of MK addition on SCC Dynamic Segregation.

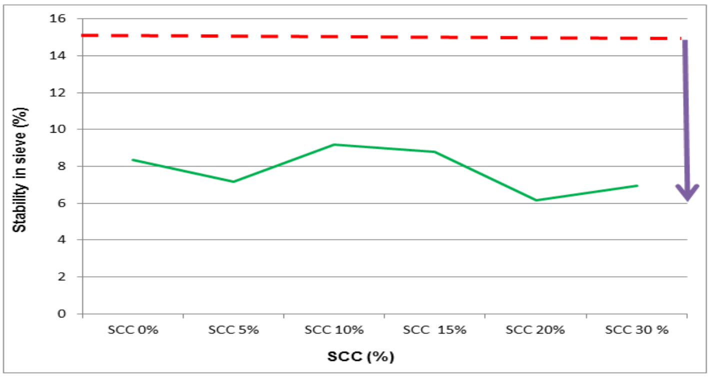

Figure 12 - Stability in sieve.

a) Self-compacting concrete (SCC) can be found to meet the spreading criteria (range 60-85 cm). They are consistent with AFGC. (2008) recommendations.

b) All SCC gives values that fall within the BAP domain (82% ≤ H_2/ H_1≤ 87%), and comply with the AFGC. (2008) recommendations (H_2/H_1) above 80%.

c) The results of the sieve stability tests presented in Table 11 show that the compositions tested have a satisfactory stability of the milk weight (P <15%) so there is no risk of segregation.

Based on the results presented in the figures above, it is observed that all SCC meet the criteria recommended by AFGC. (2008) (AFGC, 2008).

d) On the basis of these experimental results, it is noted that the higher the substitution rate, the lower the density value, regardless of the time frame.

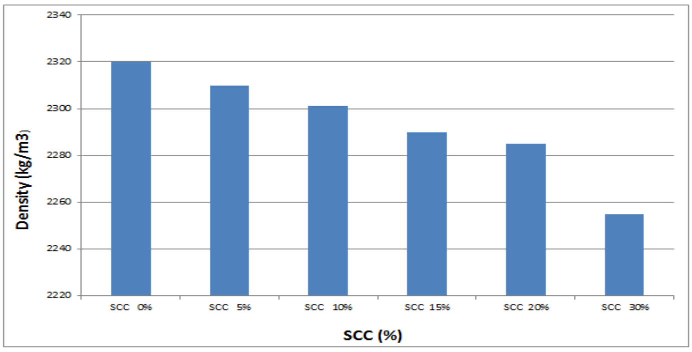

Figure 13 - Density of SCC.

5.2 Hardened BAP

5.2.1 Tensile strength by bending: Figure 14 shows the results of the variation in the tensile strength of concrete obtained by bending 3 points at 7, 28, 90 and 180 days depending on the cement substitution grade by metakaolin. We observe that cement-based concrete with MK has developed higher resistances than cement-based concrete without metakaolin. As may be seen in the previous figure The highest value of tensile strength by long-term bending (180 days) was obtained for concrete incorporating 10% MK with a strength value of 8,07 MPa which represents 12.63% of its resistance to compression. Concrete containing 20% and 30% metakaolin showed almost similar tensile strengths by bending of all the tested concretes. (Kim *et al.*, 2007) found that the bending strength of concrete increases with the increase of the cement substitution content by metakaolin for substitution content between 05%, 10% and 15% MK, This can be explained by the improvement of adhesion between cement paste and aggregates related to the densification of the zone of transitions cement paste and aggregates (Asbridge *et al.*, 1996). Suggested that the transition zone is diminished, as metakaolins improve the adhesion between cement paste and aggregates (Asbridge *et al.*, 1996).

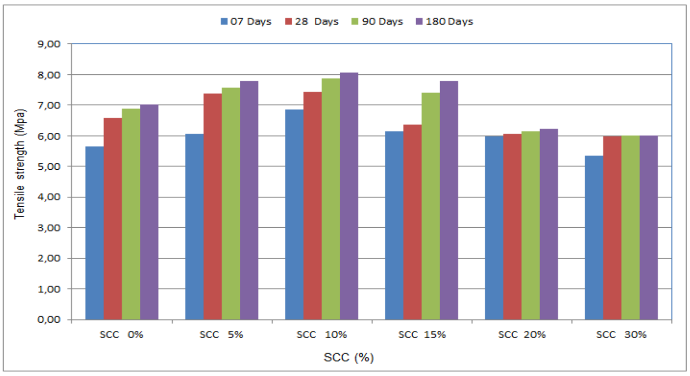

Figure 14 - Effect of MK addition on tensile strength by flexion.

5.2.2 Compressing strength: We can observe a different evolution of resistance over time between concrete in-corporating MK and concrete with portland cement alone. Concrete containing 10% metakaolin, showed a maximum compressive strength of all tested concrete.

The improvement of the compressive strength of the MK-incorporating concrete is related to the three main factors for which the MK contributed improved the hardened concrete strength: the "filling" effect, the effect of cement hydration acceleration and MK pozzolanic reaction with portlandite (Ca(OH)2) (Wild *et al.*, 1996). This is due to the fact that the metakaolin particles fill the voids between the cement grains usually occupied by water, which allows a better filling of the voids by the particles.

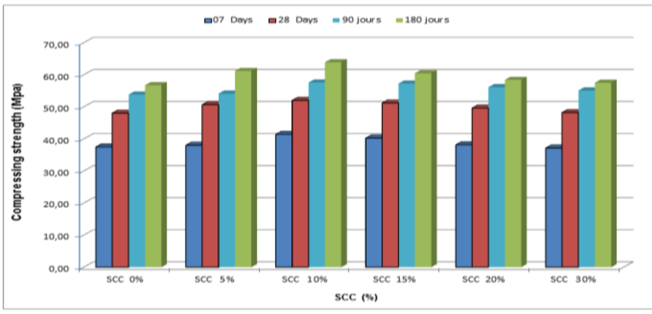

Figure 15 - Effect of MK addition on compressing strength.

5.2.3 Tensile strength by splitting: Figure 16 shows the 28-day split tensile strengths obtained for concrete containing cement alone and with metakaolin. We can observe a different evolution of resistance over time between concrete incorporating MK and concrete with cement alone.

Concrete incorporating 15% MK at 28 days showed the best resistance to tensile by splitting (8.31 MPa) once compared to all other concrete tested, with a gain in strength by filling the control concrete.

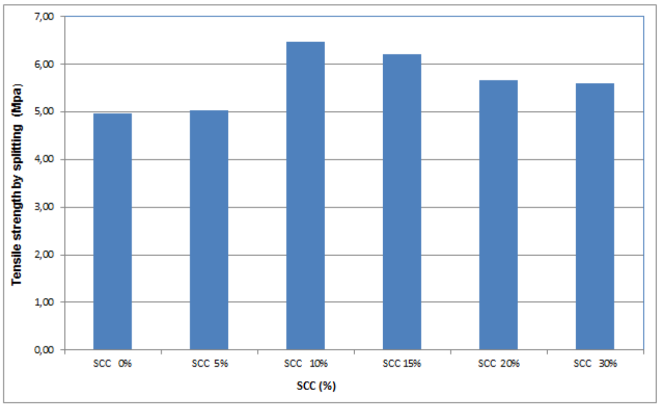

Figure 16 - Mechanical resistance by splitting of the different SCC compositions.

5.3 Physical Test

5.3.1 Water-Accessible Porosity Test: The evaluation of the effect of metakaolin on the porosity accessible to water Figure 16 shows at 28 days cure that the control concrete (SCC 0%) is more porous than the concretes containing metakaolin. Indeed, it has a porosity of about 14.69% against a major value of 14.38% for concrete with 05% metakaolin, and a minor value of 14.12% for concrete of 10%. The decrease in porosity in SCC 05% and 10% containing metakaolin can be explained by the reduction in material permeability, pore size and percentage of $Ca(OH)_2$ (Salhi *et al.*, 2016).

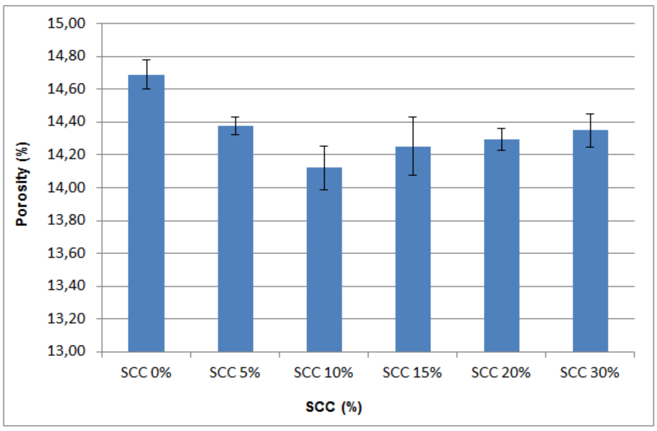

Figure 17- Effect of MK addition on porosity.

5.3.2 Capillary water absorption test: The evaluation of the effect of metakaolin on capillary absorption of concretes after 28 days of treatment is shown in Figure 18. We find that concrete containing 10% metakaolin absorbs more than other concretes with the exception of the control concrete, the absorption is major against a minor absorption of concrete containing 30% metakaolin.

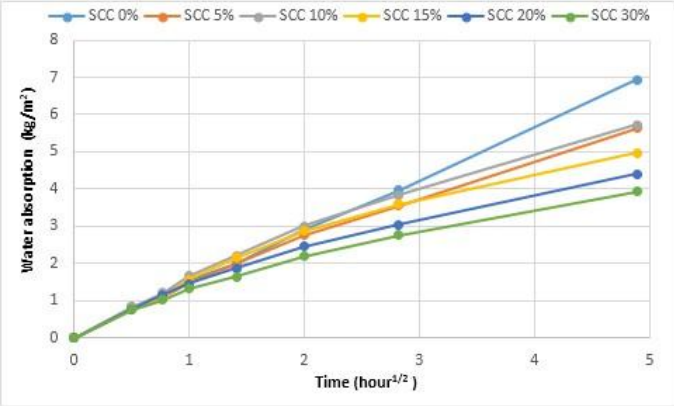

Figure 18- Capillary absorption over time after 28 days of treatment.

Previous studies have shown that cementitious additions such as fly ash (FA), silica fume (SF), metakaolin (MK) can either contribute to reducing the capillary absorption of concrete, (Rackel, 2011) or maintain the porous network at the same level as the cement they substituted (Badreddine, 2004) through the development of the pozzolanic reaction.

5.4 Non-destructive testing

5.4.1 Ultrasonic pulse velocity: The figure below shows the evolution of the propagation speed of the ultrasonic waves of the different self-compacting concretes. It is noted that all concretes have an analog kinetics with regard to the evolution of the ultrasonic speed over time, but with different values, these values essentially depend on the composition of the concrete, the type of adjuvant and its percentage; the ultrasonic speed gives us information on the compactness and homogeneity of the concrete and therefore a good indication on the mechanical behaviour of these concretes.

These results are in coordination with those obtained from the crushing tests (Figure 15); hence the concretes with high speeds have logically high resistances. The best result is obtained with SCC 10%.

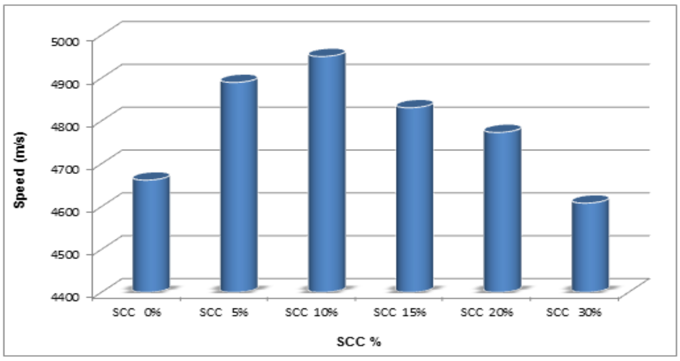

Figure 19- Evolution of the propagation speed of the UPV of the SCC.

5.4.2 Sclerometer Test: In the figure above we notice an increase in the concrete rebound index in the presence of metakaolin up to a rate of 10% MK above which a slight decrease is observed up to the SCC 30%.

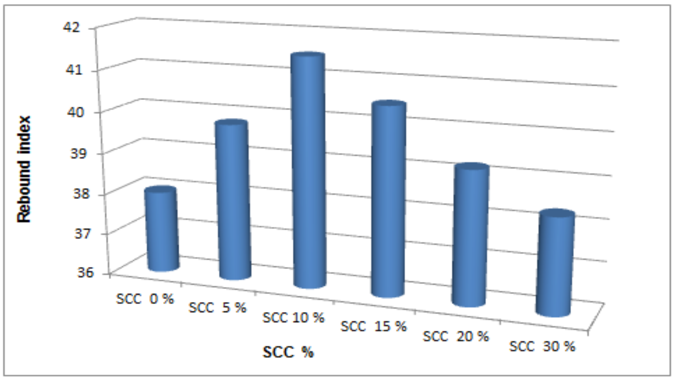

Figure 20- Rebound index according to the different SCC.

6. Conclusion

The exploitation of kaolin and its transformation into metakaolin allow to replace a part of cement in the formulation of concrete and this economically.

In this study the importance of the incorporation of metakaolin in concrete was shown. The concretes suffer a loss of maneuverability following the incorporation of metakaolin hence the need to use a superplasticity. This study also showed that me-takaolin has a much more positive role on concrete. The results obtained in this work lead us to the following conclusions:

- For the transformation of kaolin into metakaolin, a temperature between 600°C and 900°C is necessary. In our study the calcination is 850°C;
- The density of metakaolins depends heavily on their specific surface, particle size and chemical and mineralogical compositions;
- It was found that all the concrete tested met the criteria of self-compacting concrete (slump-flow, L-box and sieve stability);

- The improvement of the strength of the concrete by the metakaolin begins from 7 days of age;
- Concrete incorporating metakaolin is characterized by a porosity, lower than that of concrete based on portland cement because of the densification of the concrete micro-structure by the metakaolin linked to the formation of new hydrates;
- The metakaolin improves the performance of the concrete through two actions: the first is physical due to its fineness allowing the filling of the pores while the second is chemical due to the pouzzolanic character of the metakaolin. Indeed, substitutions of 05, 10, 15, 20 and 30% of the cement with metakaolin increases the resistance to compression and tensile strength;

Finally, it can be concluded that metakaolin is an effective substitute capable of re-ducing the cement content to obtain equivalent concrete, The aim is to reduce the cost of cement and thus to reduce environmental problems in cement production.

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