

Evaluation of Pozzolanic Activity of Illitic and Lateritic Raw Materials from Burkina Faso

ABSTRACT

Aims: Evaluate the potentially use as pozzolan in cement replacement of activated Lateritic and Illitic raw materials from Burkina Faso.

Place and duration of study: Laboratory of Molecular and Materials Chemistry-University of Ouagadougou, National Laboratory of Building and Publics Works-Ouagadougou, Burkina Faso. Between March 2015 and June 2016.

Methodology: The initial two raw materials were characterized to perform its chemical, mineralogical, geotechnical and microstructure properties. The two samples were activated at 680°C during 2 or 5hours to produce amorphous reactive materials. The activated samples were characterized using infrared spectroscopy and the selective dissolution by HF acid. Pozzolanic activity was assessed using saturated lime test, Frattini test, mineralogical analysis and compressive strength test. Mineralogical analyses were carried out with X-ray diffraction and DTA/TG.

Results: The characterization of initial raw materials shown that lateritic material contain kaolinite (71%.wt) and Goethite (18%.wt) as the mains phases and the illitic material consisting by kaolinite (44%.wt) and illite (40%.wt). The activation of samples at 680°C, given amorphous phase (metakaolin ite and silica) which amount around 74%.wt and 61%.wt respectively for lateric and illitic sample. The activated samples fixed at 7days more than 80%.wt of lime. The DTA/TG test shown that at 8days, lateric sample fixed between 33-37% and the illitic sample around 23% of portlandite produced by ement hydratation. The strength activity index, 91% and 97% respectively for lateritic and illitic samples exceeds require minimal value (75%) for pozzolan materials. Lateritic sample with finer particle and high amount of metakaolinite was presents a better pozzolan activity. However it strength is lower than those of Illitic sample. This inversion of previsible strength is due to the high contain of iron oxide in lateritic sample.

Conclusion: The two samples, after heat treatment at 680°C du ring 2hours, can be used as pozzolan for cement partial replacement in the constructions.

Keywords: Illite, Laterite, Pozzolan, Low carbon cement

1. INTRODUCTION

High numbers of scientific works are focused on the search for environmentally low carbon cement which could replace the Portland cement [1].The current Portland cement present some defaults related to **its** environment and durability sustainability. The cement production generates a great quantity of carbon dioxide. The production of one (01) kg of cement, produce on average the same quantity of carbon dioxide caused by the calcite decarbonisation (0.53kg), the burning process (0.33kg) and the electrical requirement

(0.12kg) [2-4]. During the portland cement hydration, portlandite Ca(OH)_2 is produced in great quantity and around 25% remain on the concretes or mortar [4]. This portlandite is responsible for the low resistance of concrete against aggressive environment like acid and the sulfuric solutions. The portlandite can also react with the dioxide carbon to produce calcite in the concrete which caused the expansion and the possible erosion site.

The new long sought cement must then permit not only to enhance environmental sustainability but also to ensure the durability of structures. In this intention, many materials have been proposed to replace partially or totally the Portland cement. The most used and study are the pozzolan. The pozzolan are natural or artificial materials which can react with the portlandite and enhance the mechanical and durability properties of concretes [5,6]. The portlandite is reduced by the pozzolan reaction which increases the resistance of concrete against aggressive environment and increase the mechanical strength by increasing the amount of gel CSH [7,8]. Fly ash, metakaolin, silica fume, zeolite are the main pozzolan materials commonly used in the cement substitution. However these pozzolan are not directly available. They are mostly obtained by thermal, chemical or physical treatment of raw materials. It is the case of the metakaolin, very reactive, but obtained by heat treatment between 500 - 800 °C of kaolinite rich materials [9]. Many results are available on the production and use conditions of metakaolin as pozzolan [10,11], but its availability is not always the same case in many countries. We much work then to evaluate the potential use as pozzolan of other material such as lateritic and illitic materials. A few results are available on pozzolanic activity of lateritic and illitic materials [12, 13]. The results on the use of iron-rich materials as pozzolan are often conflicting. Some conclusive results are obtained on the laterite pozzolanic activity and others **show** that beyond a certain percentage of iron, the mechanical strength **has** greatly reduced. The same discussion has been done on the use as pozzolan of illitic sample activated or not.

The aim of this work is to evaluate the potential use of lateritic and illitic raw materials as pozzolan. The first part of the paper is devoted to geotechnical, chemical and mineralogical characterization of the raw materials. The second part related the activation of the different raw materials. The last part is focused on the evaluation of the pozzolanic activity of the activated materials.

2. MATERIAL AND METHODS

2.1. Materials

Raw clay materials deposits in Burkina Faso (Laye and Rouko) were evaluated in this work to produce a reactive pozzolan. The location of the two sites on the map of Burkina Faso was given by the Fig. 1. The corresponding geographic coordinates are 12°31' North and 1°47' West for Laye and 13°19' North and 1°62' West for Rouko. Laye is located at 35km of Ouagadougou and Rouko at 25km of Kongoussi.

The lime used for the pozzolan activity test was an analytical grade calcium hydroxide (95% minimum purity).

The used cement for the pozzolanic activity and to make the pastes is a CEM I (CPA 45) from Diamond Cement society in Burkina Faso.

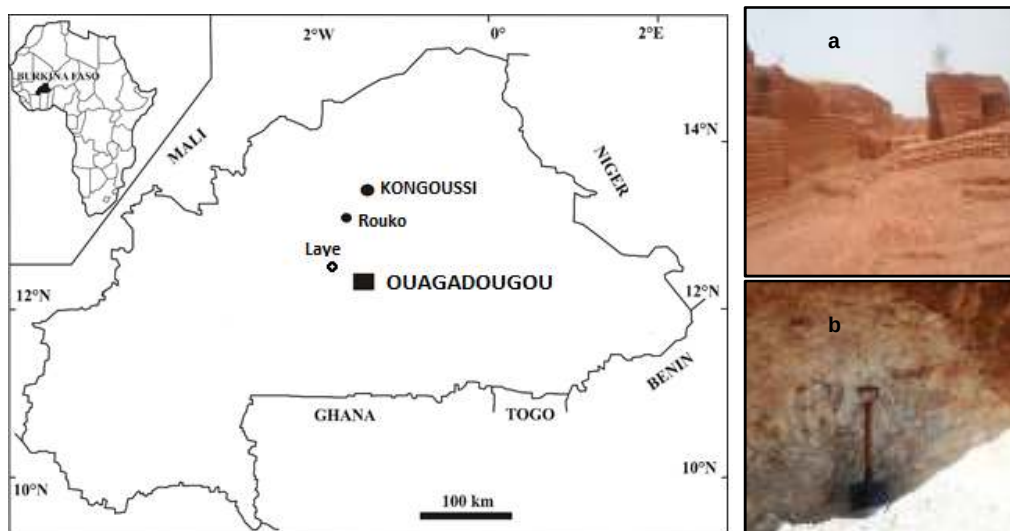


Fig. 1: Location of the sites on the map of Burkina Faso and Images photos: a- Laye, b-Rouko

2.2. Methods

2.2.1. Starting materials characterization

The particle size was determined using a Malvern Mastersizer 2000 based on the measurement technique of laser diffraction. The fineness parameters were performed using the surface specific BET method. The density was determined by hydrostatic weighing of a powder sample in a non-reactive liquid (acetone). The blue methylene value (V_{BS}) was determined on the total sample according to NF EN 933-9 [14]. Atterberg limits (liquid limit, plastic limit and plastic index) have been determined according to NF P 94-051 standard [15].

The image examinations of samples were performed with the Hitachi S2500 scanning electron microscopy.

Mineralogical composition was performed using X-ray diffraction, infrared spectroscopy and thermal analysis. The diffractometer used is a Bruker AXS, operating at 40 kV - 40mA and employing a graphite monochromatic $\text{CuK}\alpha$ radiation. The infrared spectra were recorded in the range of 400 - 4000 cm^{-1} using a Perkin Elmer FT-IR Spectrometer. The thermal analysis (DTA/TG) was carried out using SETARAM instrument operating at 10 $^{\circ}\text{C}/\text{min}$ from ambient to 1200 $^{\circ}\text{C}$. Calcined alumina was taken as a reference.

The chemical composition of the raw clay materials was assessed by Inductively Coupled Plasma - Atomic Emission Spectrometry (ICP-AES).

The crystalline mineral phases percentages contained in the sample were determined according to Yvon's et al. methodology [16] and according to the results of the elementary chemical analysis and those of the mineralogical analysis. The method of calculation uses the ideal compositions of the mineral kaolinite ($2\text{SiO}_2 \cdot \text{Al}_2\text{O}_3 \cdot 2\text{H}_2\text{O}$), illite ($\text{Al}_2\text{O}_3 \cdot \text{SiO}_2 \cdot \text{K}_2\text{O} \cdot \text{H}_2\text{O}$) quartz (SiO_2) and goethite ($\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$).

2.2.2. Activation process of the raw material

The samples were activated by thermal treatment. According to DTA curves, the temperature 680 $^{\circ}\text{C}$ was found to be sufficient for kaolinite - metakaolin conversion in the two samples. To ensure that all the thermal changes were made, we used 5hours as bearing. However, for energy consumption and environmental issues, 2hours as bearing has also been explored. In fact, it is the two bearing which are greatly used in raw clays thermal activation. All the activated materials will be called metakaolin in the rest of this work: ML for

104 Laye (ML1 for 2hours stage, ML2 for 5hours stage) and MR for Rouko (MR1 for 2hours
105 stage, MR2 for 5hours stage).

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107 **2.2.3. Metakaolins characterization**

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109 The density of metakaolins was determined by hydrostatic weighing of a powder sample in a
110 non-reactive liquid.

111 Fourier Transformed Infra Red (FTIR) analysis was performed for detecting the functional
112 groups and characterizing covalent bonding in the metakaolins.

113 The quantification of the amorphous phase was performed by selective dissolution of
114 aluminosilicate glass in dilute hydrofluoric acid (1%) [17]. This method is based on the
115 principle that the dissolution rate of amorphous aluminosilicate compounds is faster than that
116 of crystallized phases. The tests were performed for 40 min with 200 ml of 1% HF (with 3
117 drops of HNO_3) per 1 g of the crushed metakaolin.

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119 **2.2.4. Pozzolan reactivity of metakaolin**

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121 Pozzolan reactivity indicates how quickly after mixing the pozzolan material reacts with
122 calcium hydroxide and the rate at which the reaction takes place. The pozzolan reactivity is
123 complex and its study requires the association of many methods. This reactivity was then
124 assessed using chemical, mineralogical and mechanical activities tests.

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126 **2.2.4.1. Chemical activity**

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128 Saturated lime test and Frattini test are the two chemical methods which were used for
129 chemical activity evaluation.

130 Saturated lime test is a simpler approach [18], in which a fixed quantity of $\text{Ca}(\text{OH})_2$ is
131 available in solution. Samples were prepared with 1 g of metakaolin added to a plastic bottle
132 containing 75 mL of saturated lime solution. The lime solution was prepared by dissolving 2
133 g of hydrated lime in 1L of distilled water. The bottles were then sealed and placed in an
134 oven at 40 °C for 1, 3, 7 and 28 days. Samples were vacuum filtered through a pore size
135 filter paper and allowed to cool to ambient temperature in sealed Buchner funnels. The
136 filtrate was analysed for $[\text{OH}^-]$ by titration against dilute HCl with methyl orange indicator and
137 for $[\text{Ca}^{2+}]$ by pH adjustment to 12.5, followed by titration with 0.03 mol.L⁻¹ EDTA solution
138 using Patton and Reeders indicator. As the quantity of Ca^{2+} ions is accurately known at the
139 beginning of the test and because Ca^{2+} ions only interact with the test material or water, the
140 quantity of lime fixed by the test materials can be quantified. Results are reported as % total
141 CaO fixed per gram of test pozzolan.

142 Frattini test procedure is specified by the standard EN 196-5 [19]. Twenty grams of test
143 samples were prepared consisting of 80% CEM-I and 20% of the test pozzolan and mixed
144 with 100 ml of distilled water. After preparation, samples were left for 8 days in a sealed
145 plastic bottle in an oven at 40 °C. After 8 days, samples were filtered and titrated for $[\text{OH}^-]$
146 and $[\text{Ca}^{2+}]$ using the same procedure as in the saturated lime test.

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148 **2.2.4.2. Mineralogical reactivity**

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150 The filtrate sample obtained during Frattini test was finely grounded and tested for
151 mineralogical exploration. The powders were then subjected to X-ray diffraction and
152 DTA/TG, and the pozzolan reactivity was defined as the portlandite consumption. X-ray
153 diffraction and DTA permit to follow the formation or disappearing of the phases. The
154 consumed portlandite by metakaolins quantity was evaluated with TG analyses.

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156 **2.2.4.3. Mechanical activity index**

The pozzolanic activity of the metakaolins was also measured by mechanical test (pozzolanic activity index). Mortars (4x4x16cm³) were prepared according to the French standard NF EN 196-1 [20]. The different mortars were composed of three parts in mass of sand, one part of cement and a half part of water. The cement replacement by metakaolin was expressed as the fraction of cement mass in the control mix. Replacement rate was 25wt. %. The mortars bars were removed from their moulds 24 hours after casting. The mortars were cured at 20°C in water. The compressive strength was tested for three mortars for different rate replacement at 28days. The mechanical activity index is determined as the ratio between the compressive strength of blend and compressive strength of reference.

3. RESULTS AND DISCUSSION

3.1. Starting raw materials

The chemical composition of the raw materials is shown by the Table 1. The main oxides present on the raw materials are SiO₂ and Al₂O₃. Laye and Rouko samples contain respectively particular amount of Fe₂O₃ and K₂O. The sum of reactive oxides SiO₂ + Al₂O₃ + Fe₂O₃ exceed the minimum 50% requirement for pozzolan materials according to the standard ASTM C-618[21]. However the Fe₂O₃ amount in Laye exceeds the maximum 3% value as indicated by some authors.

The X-ray diffraction patterns of raw materials (Fig. 2) showed the presence of kaolinite (Al₂Si₂O₅(OH)₄), and illite ((K,H₃O)Al₂Si₃AlO₁₀(OH)₂) as clay phases in Rouko and only kaolinite in Laye. At these clay phases are added non – clay such as quartz (SiO₂), and exclusively goethite (FeO(OH)) in Laye sample.

The FTIR analysis presented by the Fig. 3 corroborated the X-ray diffraction results. The characteristic bands of kaolinite are shown by the bands ν_{OH^-} (at 3695, 3668, 3652, 3620cm⁻¹), ν_{Al-OH} (at 938, 913 cm⁻¹) and $\nu_{Si-O-Al}$ (at 537 cm⁻¹). Illite is shown by the bands ν_{OH^-} (at 3620 cm⁻¹), $\nu_{Si-O-Si}$ (at 1031cm⁻¹) and ν_{Al-OH} (at 834, 664, 644 cm⁻¹) [22]. The quartz characteristic bands are $\nu_{Si-O-Si}$ (at 1164, 797, 778 cm⁻¹) [23].

Differential thermal analysis (Fig. 4-a) shows principally two similar peaks for the two samples. The first around 400-600°C correspond to the decomposition of kaolinite into metakaolinite, and the second between 900 and 1050 °C is attributable to the formation of Al-spinel or mullite phase. Illite phase in Rouko is identified by the shoulder between 680 and 880 °C. Goethite in Laye is shown by the endothermic peak between 200 and 400°C. Thermogravimetric analysis (Fig. 4-b) shows clearly the illite and goethite transformation respectively in Rouko and Laye.

The semi-quantitative composition of the raw materials is shown by the Table 2. Laye consist of Kaolinite (71wt.%), goethite (18wt.%) and quartz (8wt.%). Its composition is very interesting for pozzolan use. The activation of kaolinite will give a metakaolinite which presents good pozzolanic activity. The quartz also improves the cement hydration and participates then on the pozzolan reaction [24]. The goethite can also improve the pozzolan reactivity as show by some authors but at relatively feeble quantity. In our case, the goethite percentage is very high. Should be a handicap for the use of Laye as pozzolan? Concerning Rouko, it contain approximately the same amount of kaolinite (44.wt. %) and illite (40.wt.%). Illite present a lower pozzolan reactivity than kaolinite and its activation temperature is higher than those of kaolinite. Rouko is then particular by its composition.

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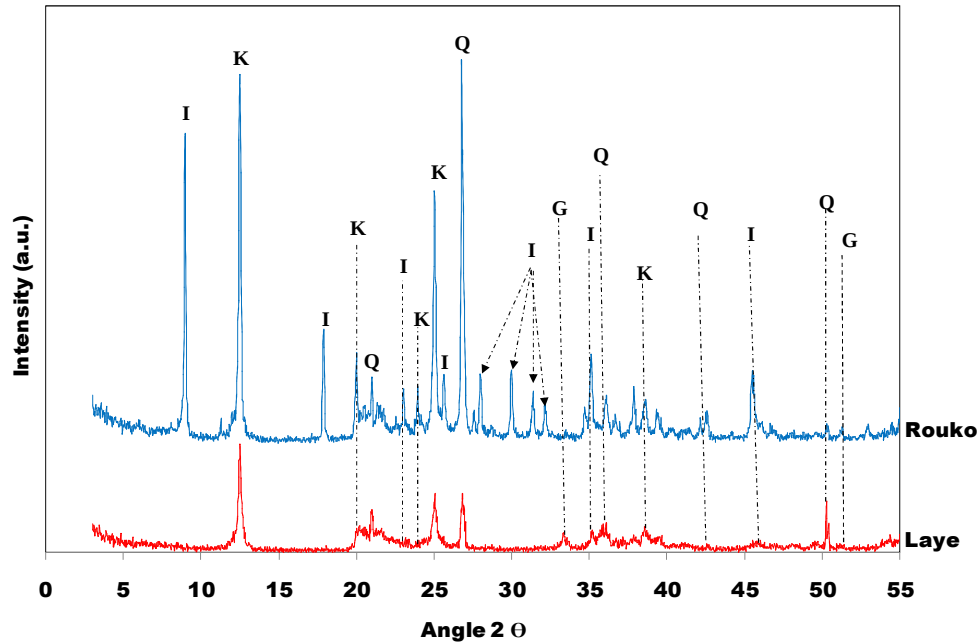
Table 1: Chemical composition (% by mass) of raw materials

Oxides(%)	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MnO	MgO	CaO	Na ₂ O	K ₂ O	TiO ₂	P ₂ O ₅	LOI	Total
Rouko	50.00	32.47	1.61	0.01	0.80	0.07	0.13	4.62	1.37	< D.L.	8.70	99.78
Laye	41.50	28.61	16.32	0.01	0.08	< L.D.	< D.L.	0.10	1.10	0.05	12.14	99.91

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D.L. : Detection Limit ; LOI : Loss On Ignition



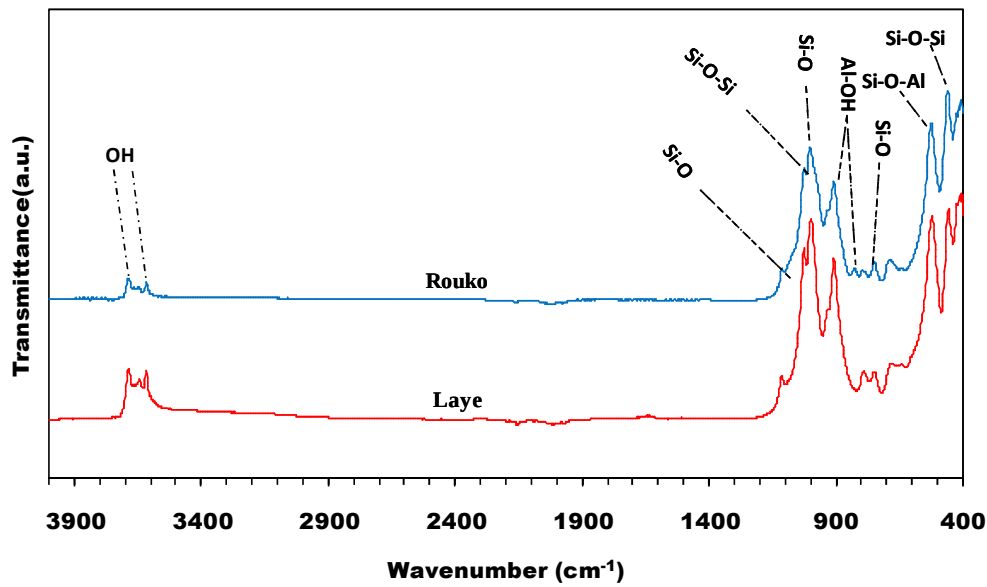
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Fig. 2: X-ray diffraction of raw materials. K= kaolinite ; I= illite ; G= goethite et Q= quartz



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Fig. 3: Infrared spectra of raw materials

Table 2 presents the geotechnical results of the two samples. According to Casagrande [25], the Atterberg limits show that the samples are plastic clay materials. This result is corroborated by the methylene blue values. The particle size curves are presented by the Fig. 5. Laye particle size is very varied with finer particles than Rouko which seems to be composed by one granular family.

The BET specific surface value of Laye ($29\text{m}^2/\text{g}$) is conforming to the other value and show that the principle phase in Laye is the kaolinite. However, this surface for Rouko is low and did not correspond to the mineralogical composition.

The SEM images are given by Fig. 6. The image shows platelets in great quantity on the SEM image of Rouko. These platelets are characteristic forms of kaolinite and agreed with mineralogical results. Laye SEM image shows an agglomeration of particles with small size characteristic of clay particle. Laye SEM image is not very clear due to the presence of iron oxides in important quantity.

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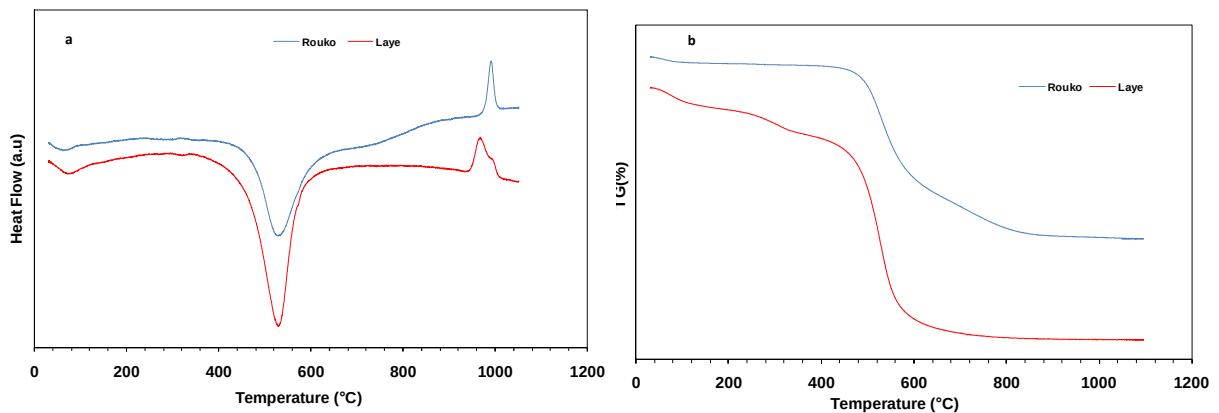


Fig. 4: Thermograms of the samples: a- DTA; b-TGA

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Table 2: Mineralogical composition and geotechnical parameters of raw materials

	Laye	Rouko
Mineralogy ,wt. %		
Kaolinite	71	44
Illite	1	40
Quartz	8	12
Goethite	18	2
Total	98	98
Geotechnical parameters		
Density (g/cm^3)	2.71	2.72
BET surface area (m^2/g)	29	6.74
Liquid limit $W_L(\%)$	43	41
Plastic limit $W_P(\%)$	21	20
Plasticity index $I_P(\%)$	22	21
Methylene blue value V_{BS}	1.33	1.5

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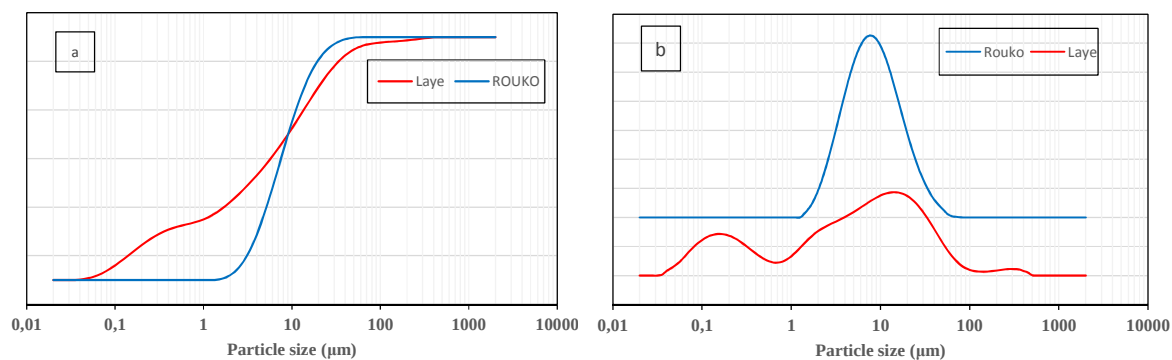


Fig. 5: Particle size distribution of the samples: a- Cumulative; b- Volume

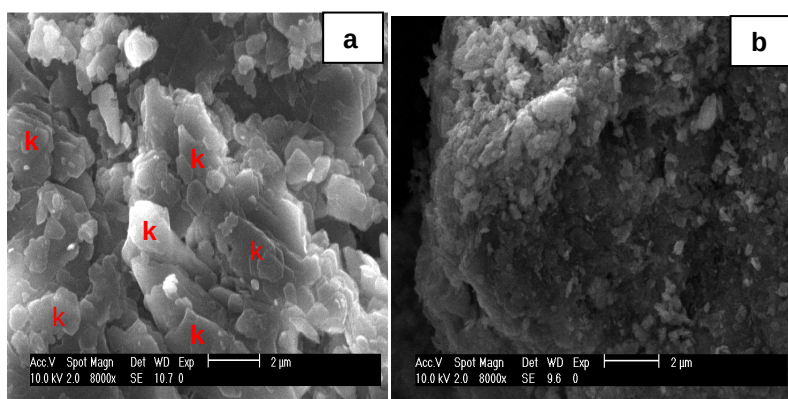


Fig. 6: SEM micrographs of clays: a- Rouko, b- Laye
K : kaolinite

3.2. Metakaolin characterization

The dehydroxylation of kaolinite into metakaolinite is confirmed by the infrared spectra (Fig. 7) by the loss of the bands of kaolinite ν_{OH} at $3620 - 3700\text{cm}^{-1}$. The kaolinite transformation into metakaolinite is corroborated by the disappearance of the kaolinite doublet ν_{Al-OH} at $913 - 938\text{cm}^{-1}$. The large band around 1070cm^{-1} is assigned to amorphous silica in the metakaolins.

For all the sample the amorphous quantity (Table 3) is high and predict a very reactive materials. ML are more amorphousness than MR.

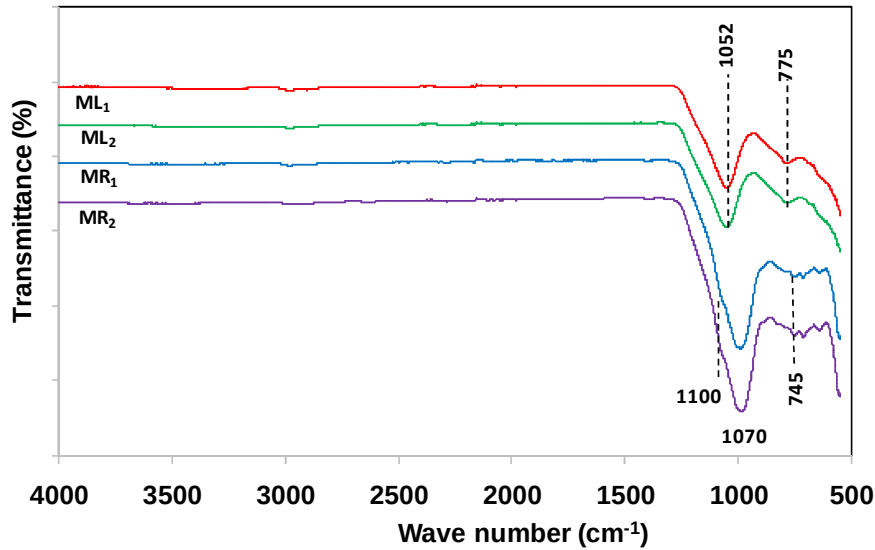


Fig. 7: Infrared spectra of metakaolins

Table 3: Physical characteristics of the metakaolins

Parameters	ML1	MR1	ML2	MR2
Density (g/cm ³)	2.73	2.54	2.51	2.35
% Amorphous	73	63.5	76	60

ML1 :Laye at 680°C for 2hours, ML2 : Laye at 680°C for 5hours;
MR1 :Rouko at 680°C for 2hours, MR2 : Rouko at 680°C for 5hours

3.3. Pozzolan activity of metakaolin

3.3.1. Chemical activity

The saturated lime test results are shown by Fig. 8. The bearing time did not significantly effect the percentage of fixed lime. At the first 24hours, more than 50% of lime are fixed by the two metakaolins (MR and ML). The fixed lime at this age is highest than the lime fixed(~35%) by the commercial metakaolin at the same period. Between 1day and 7days, the pozzolan reactivity increase strongly and more than 80% lime are fixed. After 7days, the pozzolan reactivity is not also faster but, at 28 days more than 90% lime are fixed and is similar to some other metakolin. During all the reaction period, ML fixed lime more than MR. This better behavior of ML is a consequence of it finer particles, it chemical and mineralogical composition. The fineness of particle increases the pozzolan reactivity. For finer particles, the specific surface is high and the surface contact between the reactive compounds increase and give a better reaction. The reactive oxides SiO₂, Al₂O₃ and Fe₂O₃, quantity in Laye (86.43wt. %) is highest than Rouko (84.08wt. %) and can explain one hand the difference on the percentage of fixed lime. ML contains another hand, a highest percentage of amorphous phase which explain it better reactivity than MR.

The saturated lime test is corroborated by the Frattini test (Fig. 9).The entire metakaolins is located below the portlandite solubility curve [26].

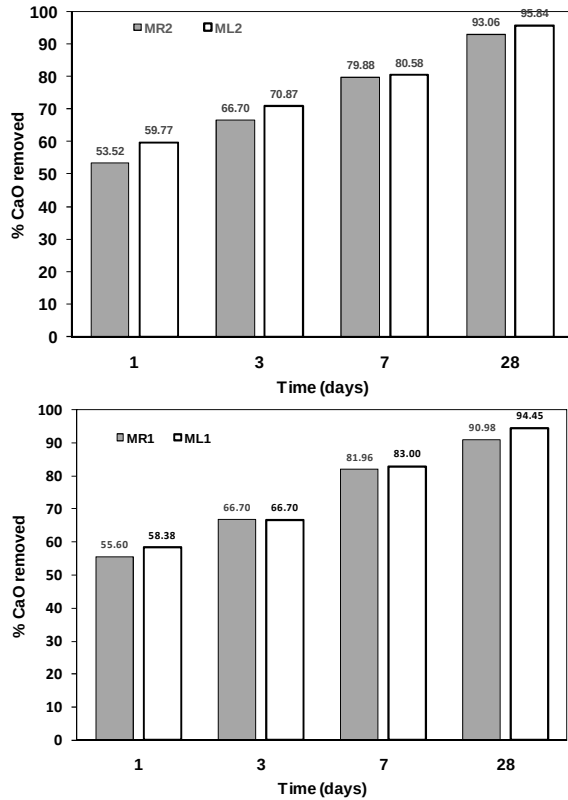


Fig. 8: Saturated test lime results a- for MR₁ and ML₁, b- for MR₂ and ML₂

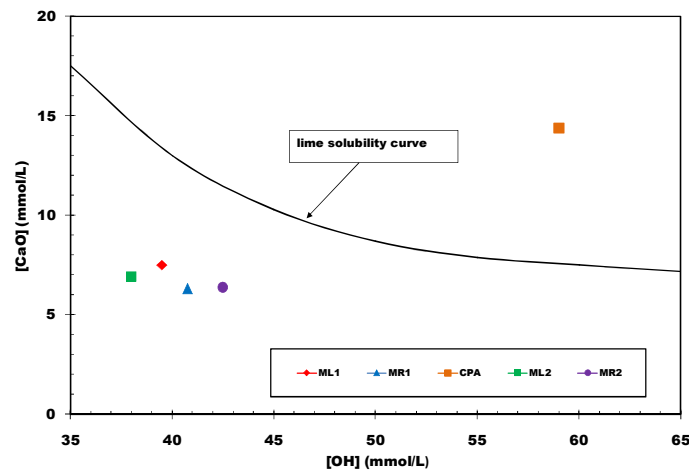
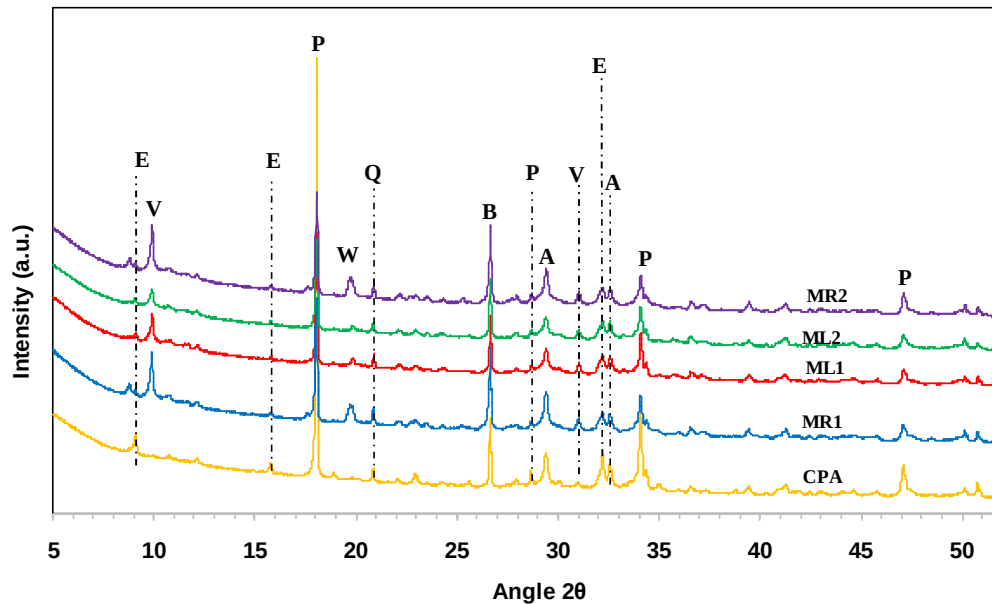


Fig. 9: Frattini test results

3.3.2. Mineralogical activity

The x-ray diffraction patterns of the product after Frattini test are shown by the Fig. 10. The portlandite peak intensity decreases greatly on the samples containing the metakaolins. This decrease of portlandite peaks indicates the pozzolan reactivity of metakaolins. New phases

324 characteristics to reaction between metakaolin and portlandite are detected. The main
 325 crystallized are ettringite and calcium aluminate hydrates such as C_4AH_{13} and C_3ASH_6 [27].
 326 The DTA (Fig. 11-a) analysis is used to detect the non crystallized phase which X-ray cannot
 327 shown and the TG analysis (Fig. 11-b) to evaluate the amount of consumed portlandite. DTA
 328 shows around 100°C the peak of dehydration of the gel CSH. Between 400 and 480°C the
 329 peak of portlandite is shown and the pozzolan reactivity of the metakaolin is clearly identified
 330 by the decreasing of this peak for the sample containing metakaolin. ML seems to be more
 331 reactive than MR as shown by the chemical tests. TG curves permitted to evaluate the
 332 percentage of the portlandite consumption (Table 4) at 8days by the metakaolins. ML fixed
 333 more portlandite than MR as shown the previous results. The bearing 5hours did not shown
 334 a significant different with 2hours bearing. For energy issues, the 2hours are then the better
 335 bearing.
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337
 338 **Fig. 10: X-ray diffraction of residues after Frattini test**

339 P : portlandite (CH), Q : Quartz, E: ettringite ($C_6A\bar{S}_3H_{32}$); A :Alite; V: C_4AH_{13} ; W: C_3ASH_6 ; B: Belite
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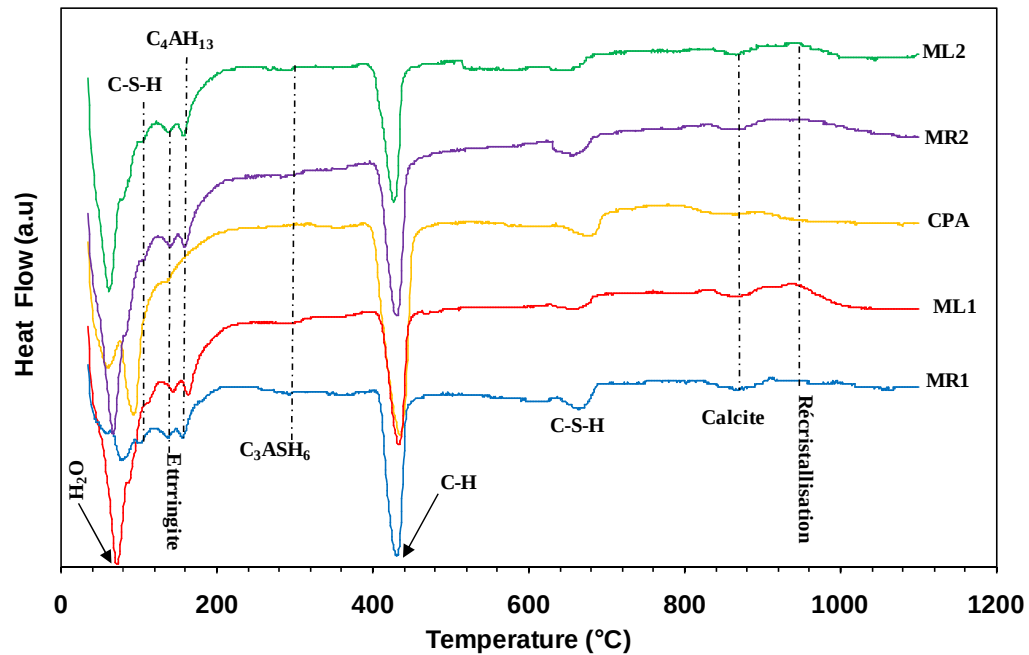


Fig.11- a: Differential thermograms (DTA) of residues after Frattini test

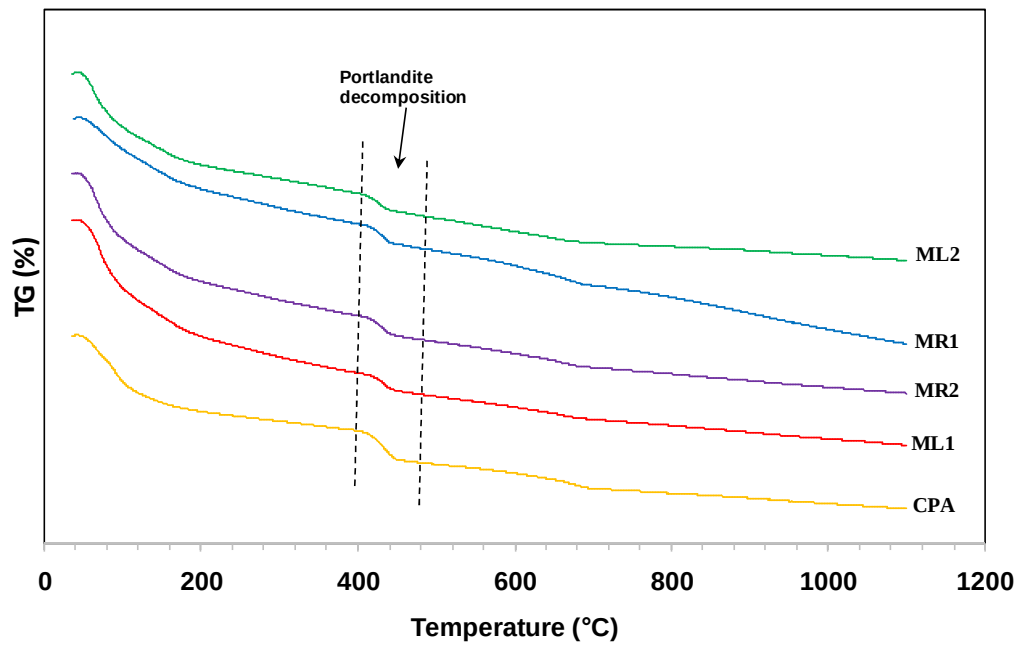


Fig.11-b: Thermograms TGA of residues after Frattini test

Table 4: Percentage of fixed portlandite (CH) by the metakaolins at 8days

Samples	Loss on mass	% of fixed CH
CPA	2.03	-
ML ₁	1.29	36.45
ML ₂	1.34	33.99
MR ₁	1.56	23.15
MR ₂	1.55	23.64

3.3.3. Mechanical activity

Compressive strength values for 28days with 25wt. % replacement of cement are given by Fig. 12. Performance for all the concrete is lower than that of the reference. This decrease can be explained by the significant quartz content of the MK studied. High quartz content can be linked to a relatively small strength activity index. However, the different results are not alarming because the strength is higher than the minimum strength required at 28days. Nevertheless, the mechanical index of concrete containing MR is high than those which containing ML. This result was not predicable according to chemical and mineralogical test. Some hypothesis can be drawn to explain this situation. Firstly we can suppose that, the mechanical strength is a sum of pozzolan reactivity and the filler effect of the additives. This assumption cannot be acceptable because Laye has presented the best pozzolan reactivity and contain the finer particle, then the best filler effect than Rouko.

Secondly, we can think that the strength is strongly depending on the quartz amount and thirdly the iron oxide influence the mechanical strength. According to quartz amount in the two samples, Rouko must have a feeble strength than Laye or is not the case. The third assumption seems to be true. The mechanical behavior is influenced by the quantity of iron oxides. This result is in agreement with previous studies which shown effectively that with iron oxides more than 2.7wt.% the strength decrease. The high iron quantity in Laye reduced it pozzolan and filler effect on the mechanical performance of specimen.

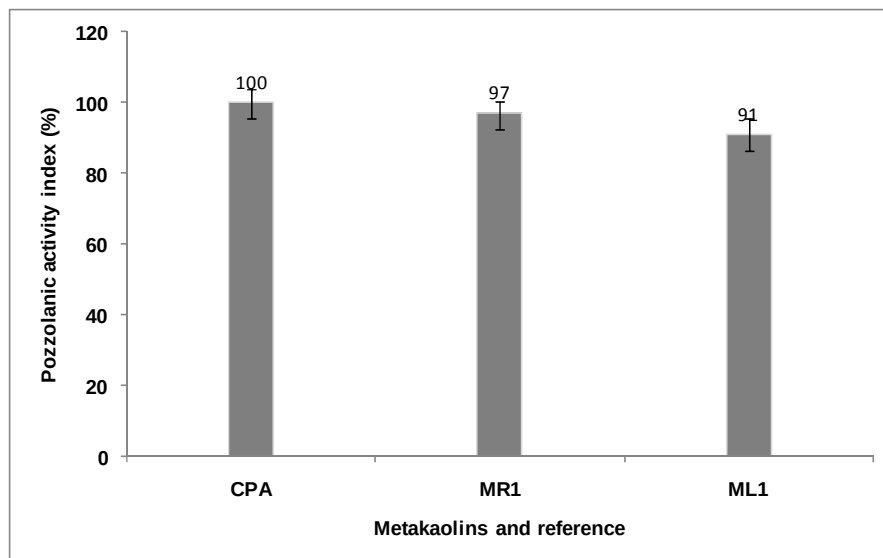


Fig. 12 : Pozzolanic activity index of the metakaolins after 28 days for 25%.wt cement replacement

4. CONCLUSION

The study raw clays materials (Laye and Rouko) are composed by kaolinite, goethite and quartz for Laye and kaolinite, illite and quartz for Rouko. These different mineralogical compositions are suitable for pozzolan uses.

The activated sample at 680°C fixed lime or portlandite with very interesting rate. The sample activated at 680°C during 2hours has shown a similar lime fixation with the sample activated at the same temperature but with 5hours. The bearing period did not affect significantly the materials reactivity with lime or portlandite. ML pozzolanic reactivity has better than MR.

The mechanical behaviour is the sum of pozzolan reactivity and filler effect but is strongly affected by the associates' minerals. Quartz in some percentage decreases the mechanical strength. It is the same case of iron mineral such as goethite which for some percentage decreases also the mechanical strength of the concretes. Nevertheless, the pozzolan index at 28days for the concrete with replacement of 25wt.% by the sample activated at 680 °C during 2hours is conform to requirement value (75% at 28days).

The two samples can be used before activation at 680°C as pozzolan in cement replacement.

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