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

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Authors' contributions

This work was carried out in collaboration between all authors. Author IS designed the study, wrote the protocol, participated in the writing the first draft of the manuscript and provided the literature. Author MS managed the analyses of the study and wrote the first draft of the manuscript. Authors LZ, YM and RO managed the literature searches and managed the interpretation of results. All authors read and approved the final manuscript.

Article Information

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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 5 hours to produce amorphous reactive materials. The activated samples were

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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 (metakaolinite and silica) which amount around 74%.wt and 61%.wt respectively for lateric and illitic sample. The activated samples fixed at 7 days more than 80%.wt of lime. The DTA/TG test shown that at 8 days, 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 during 2 hours, 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 (0.53 kg). the burning process (0.33 kg) and the electrical requirement (0.12 kg) [2-4]. During the portland cement hydratation, portlandite Ca(OH)₂ is produce 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 it disponibility 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. MATERIALS AND METHODS

2.1 Materials

Raw clay materials deposits in Burkina Faso (Lave 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. Lave is located 35 km at of Ouagadougou and Rouko 25 at km 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 made the pastes is a CEM I (CPA 45) from Diamond Cement society in Burkina Faso.

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 – 40 mA and employing a graphite monochromatic CuK α 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°C/min from ambient to 1200°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. [16] methodology 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 $(2SiO_2.Al_2O_3.2H_2O)$, illite $(Al_2O_3.SiO_2.K_2O.H_2O)$ quartz (SiO_2) and goethite $(Fe_2O_3.H_2O)$.



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

2.2.2 Activation process of the raw material

The samples were activated by thermal treatment. According to DTA curves, the temperature 680°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 5 hours as bearing. However. for energy consumption and environmental issues, 2 hours 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 Laye (ML1 for 2 hours stage, ML2 for 5 hours stage) and MR for Rouko (MR1 for 2 hours stage, MR2 for 5 hours stage).

2.2.3 Metakaolins characterization

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

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

The quantification of the amorphous phase was performed by selective dissolution of aluminosilicate glass in dilute hydrofluoric acid (1%) [17]. This method is based on the principle that the dissolution rate of amorphous aluminosilicate compounds is faster than that of crystallized phases. The tests were performed for 40 min with 200 ml of 1% HF (with 3 drops of HNO₃) per 1 g of the crushed metakaolin.

2.2.4 Pozzolanic reactivity of metakaolin

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

2.2.4.1 Chemical activity

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

Saturated lime test is a simpler approach [18], in which a fixed quantity of $Ca(OH)_2$ is available in

solution. Samples were prepared with 1 g of metakaolin added to a plastic bottle containing 75 mL of saturated lime solution. The lime solution was prepared by dissolving 2 g of hydrated lime in 1 L of distilled water. The bottles were then sealed and placed in an oven at 40°C for 1, 3, 7 and 28 days. Samples were vacuum filtered through a pore size filter paper and allowed to cool to ambient temperature in sealed Buchner funnels. The filtrate was analysed for [OH] by titration against dilute HCl with methyl orange indicator and for [Ca²⁺] by pH adjustment to 12.5, followed by titration with 0.03 mol.L⁻¹ EDTA solution using Patton and Reeders indicator. As the quantity of Ca^{2+} ions is accurately known at the beginning of the test and because Ca2+ ions only interact with the test material or water, the quantity of lime fixed by the test materials can be quantified. Results are reported as % total CaO fixed per gram of test pozzolan.

Frattini test procedure is specified by the standard EN 196-5 [19]. Twenty grams of test samples were prepared consisting of 80% CEM-I and 20% of the test pozzolan and mixed with 100 ml of distilled water. After preparation, samples were left for 8 days in a sealed plastic bottle in an oven at 40°C. After 8 days, samples were filtered and titrated for [OH] and [Ca²⁺] using the same procedure as in the saturated lime test.

2.2.4.2 Mineralogical reactivity

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

2.2.4.3 Mechanical activity index

The pozzolanic activity of the metakaolins was also measured by mechanical test (pozzolanic activity index). Mortars ($4 \times 4 \times 16 \text{ cm}^3$) 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 28 days. 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 $(Al_2Si_2O_5(OH)_4)$, and illite $((K,H_3O)Al_2Si_3AIO_{10}(OH)_2)$ as clay phases in Rouko and only kaolinite in Laye. At these clay phases are added non – clay such as quartz (SiO_2) , 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 v_{OH} (at 3695, 3668, 3652, 3620 cm⁻¹), v_{AI-OH} (at 938, 913 cm⁻¹) and $v_{SI-O-AI}$ (at 537 cm⁻¹). Illite is shown by the bands v_{OH} (at 3620 cm⁻¹), $v_{SI-O-SI}$ (at 1031cm⁻¹) and v_{AI-OH} (at 834, 664, 644 cm⁻¹) [22]. The quartz characteristic bands are $v_{SI-O-SI}$ (at 1164, 797, 778 cm⁻¹) [23].

Differential thermal analysis (Fig. 4a) 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 AI-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. 4b) 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 guartz (8wt.%). It 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 hydratation 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 Lave 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 it activation temperature is higher than those of kaolinite. Rouko is then particular by it composition.

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.

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

Oxides(%)	SiO ₂	AI_2O_3	Fe ₂ O ₃	MnO	MgO	CaO	Na₂O	K ₂ O	TiO ₂	P_2O_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	< D.L.	< D.L.	0.10	1.10	0.05	12.14	99.91
D.L.: Detection Limit; LOI: Loss On Ignition												



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



Fig. 3. Infrared spectra of raw materials

Table 2. Mineralogical composition	and geotechnica	I parameters of	f raw materials
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	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 de W _P (%)	21	20
Plasticity index $I_P(\%)$	22	21
Methylene blue value V _{BS}	1.33	1.5



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



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

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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 v_{OH-} at 3620 – 3700 cm⁻¹. The kaolinite transformation into metakaolinite is corroborated by the disappearance of the kaolinite doublet v_{AI-OH} at

 $913 - 938 \text{ cm}^{-1}$. The large band around 1070 cm⁻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 is more amorphousness than MR.



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



Fig. 7. Infrared spectra of metakaolins

Table 3.	Physical	characteristics	of the	metakaolins
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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 2 hours, ML2: Laye at 680°C for 5 hours; MR1: Rouko at 680°C for 2 hours, MR2: Rouko at 680°C for 5 hours

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 24 hours, more than 50% of lime is 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 1 day and 7 days, the pozzolan reactivity increase strongly and more than 80% lime are fixed. After 7 days, 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 and particles, it chemical 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, AI_2O_3 and Fe_2O_3 , 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 characteristics to reaction between metakaolin and portlandite are detected. The main crystallized are ettringite and calcium aluminate hydrates such as C_4AH_{13} and C_3ASH_6 [27].

The DTA (Fig. 11a) analysis is used to detect the non crystallized phase which X-ray cannot shown and the TG analysis (Fig. 11b) to

evaluate the amount of consumed portlandite. DTA shows around 100°C the peak of dehydratation of the gel CSH. Between 400 and 480°C the peak of portlandite is shown and the pozzolan reactivity of the metakaolin is clearly identified by the decreasing of this peak for the sample containing metakaolin. ML seems to be more reactive than MR as shown by the chemical tests. TG curves permitted to evaluate the percentage of the portlandite consumption (Table 4) at 8 days by the metakaolins. ML fixed more portlandite than MR as shown the previous results. The bearing 5 hours did not shown a significant different with 2 hours bearing. For energy issues, the 2 hours are then the better bearing.



Fig. 10. X-ray diffraction of residues after Frattini test

P: Portlandite (CH), Q: Quartz, E: Ettringite (C₆A \bar{S}_3H_{32}); A: Alite; V: C₄AH₁₃; W: C₃ASH₆; B: Belite



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



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

Table 4. Percentage of fixed portlandite (C	H)
by the metakaolins at 8 days	

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 28 days 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 28 days. 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 mechanichal strength is a sum of pozzolan reactivity and the filler effect of the additives. This assumption cannot be acceptable because Lave 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 2 hours has shown a similar lime fixation with the sample activated at the same temperature but with 5 hours. 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 28 days for the concrete with replacement of 25wt.% by the sample activated at 680°C during 2 hours is conform to requirement value (75% at 28 days).

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

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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