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Refractory bricks based on Tikaré (Burkina Faso) kaolinitic raw clay material

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Abstract: A raw clay material deposit in Burkina Faso has been used for refractory bricks production in the lab. The extruded raw clay material has been fired at 1400°C to produce a chamotte grog that has been crushed into different particle sizes. These have been mixed with 14%.wt of the raw clay material as a binder. The admixture has then been pressed at 120MPa, dried at 105°C and then fired at 1400 or 1500°C to get cubical specimens. The effect of increasing temperature is clearly shown by the significant difference between the mechanical properties of the bricks fired at 1400 and those fired at 1500°C. The bulk density (2.16g/cm^3), the open porosity (11.99%), the compressive strength (99.2MPa) and the refractoriness under load (ISO $T_{0.5}$ = 1419°C) of the brick fired at 1500°C are in the range of many commercial grade refractory bricks.

Keys words: Refractory bricks, refractoriness under load, mullite, cristobalite, vitreous phase

Formulation de briques réfractaires à base de matières premières kaolinitiques de Tikaré (Burkina Faso)

Résumé : Une matière première argileuse du Burkina Faso a été utilisée pour la production de briques réfractaires au laboratoire. Elle a été extrudée et cuite à 1400°C pour produire de la chamotte qui a été par la suite broyée pour donner de particules de granulométrie différente. La chamotte a été mélangée avec 14% de la matière première crue comme liant. Le mélange est pressé avec 120MPa, séché à 105°C et porté à 1400 et 1500°C pour donner de briques cubiques. L'effet de la température de cuisson est clairement montré par une différence significative entre les propriétés mécaniques des briques cuites à 1400 et 1500°C. La densité apparente (2,16g/cm3), la porosité ouverte (11,99%), la résistance mécanique (99,2Mpa) et la réfractarité sous charge (ISO T0,5 = 1419°C) des briques cuites à 1500°C sont comparables à plusieurs briques réfractaires commerciales.

Mots clés: Briques réfractaires, réfractarité sous charge, mullite, cristobalite, phase vitreuse

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1. Introduction

The need for good quality refractory materials for industrial uses is increasing. From metallurgical industry use to the mining industry through domestic use, refractory materials are widely used in the form of bricks, plates, filters or crucibles [1-2]. They are mainly used for kiln linings, the filtration and handling of molten metals (including gold) at high temperatures. The increase of mining exploration in Burkina Faso has consequences on the use and the import of refractories (including crucibles) for gold metallurgy. This situation is prejudiciable to the best exploitation of mineral resources.

This situation could be improved by the local production of refractories. This is why, we have focused our previous work on the elaboration of refractory bricks from a raw clay coming from a deposit in Burkina Faso. From this earlier work refractory sample bricks with a refractoriness of 1580 °C (as measured by the Seger cone test) have been prepared. However these bricks presented a high porosity (38.51%.wt) and a low compressive strength (15MPa). The two latter properties, despite the good refractoriness, are too low for a potential commercial use of this type of bricks.

That is why, the present work, which is the continuation of the work already undertaken, is devoted to the improvement of mechanical and refractory properties of the sample bricks. This work can be divided in 3 parts. The first part consists on the optimization of the chamotte grog by varying its elaboration temperature. The second part is devoted on the optimisation of the bricks composition by adjusting the nature and the content of the binder. The last part is the elaboration and the characterization of the bricks.

2. Raw materials and experimental techniques

2.1. Raw material

The clay used as raw material is a clay from Burkina Faso. It has been characterized in our previous work but we recall here some general guidelines. The chemical composition (**Table I**) indicates that the clay is mainly composed of silica (62.42%) and alumina (24.60%) with a small amount of iron oxide (1.73%) and potassium oxide (1.50%). According to ***Sayel M. Fayyed et al*** [3]**,** the usual refractory clays have an Al_2O_3 content between 25 to 45%, TiO_2 between 1 to 4%, Fe_2O_3 not exceeding 2.5% and $Na_2O + K_2O + CaO + MgO$ should not exceed 6%. The chemical composition of our sample indicates that the Tikaré raw clay is chemically suitable for elaboration of refractory bricks. The identified mineral phases are kaolinite (50%), quartz (33%) and illite (13%). The geotechnical properties indicate that the clay used is of rather low plasticity. The cumulative particle size curve (**Figure 1**) indicates that 10.8% of fractions are finer than 2µm.

Table I : Chemical, mineralogical and geotechnical parameters of Tikaré Raw clay material

Chemical composition (%)		**Mineralogical composition (%)**	
SiO_2	62.42	Kaolinite : $Al_2Si_2O_5(OH)_4$	50
Al_2O_3	24.60	Quartz : SiO_2	33
K_2O	1.50	Illite : $KAl_3Si_3O_{10}(OH)_2$	13
CaO	0.08	**Total**	96
MgO	0.21	**Geotechnical properties**	
Fe_2O_3	1.73	Liquid limit, W_L (%)	27.37
Cr_2O_3	0.04	Plasticity limit, W_P (%)	23.05
TiO_2	1.09	Plasticity index, I_P (%)	4.32
ZrO_2	0.09	Methylene blue value (g/100g)	3.86
P_2O_5	0.03	d_{10} (µm)	2
Loss on ignition	8.21	d_{50} (µm)	8
Total	100.00	d_{90} (µm)	40

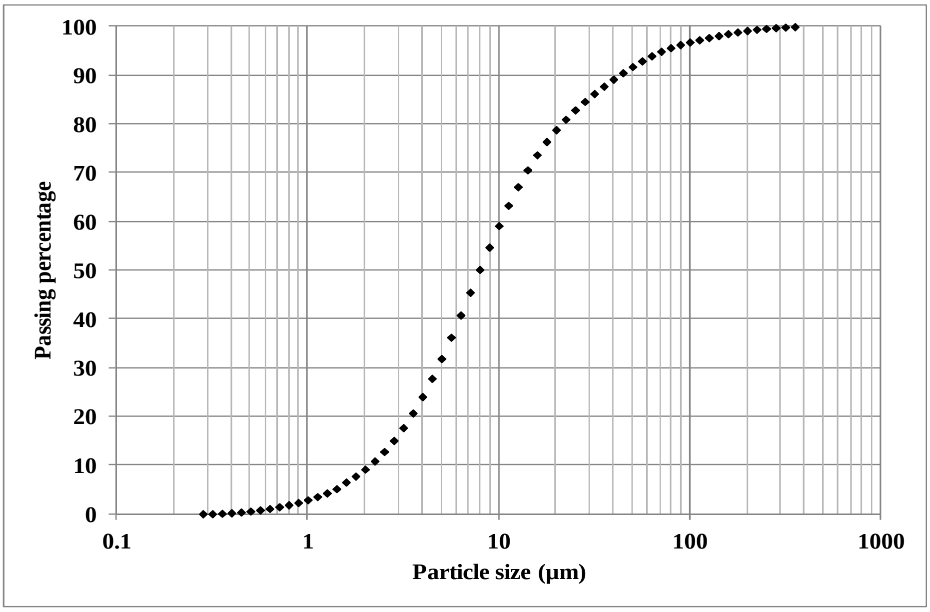

Figure 1: Particle size distribution of the raw clay material

2.2. Experimental techniques

2.2.1. Chamotte grog preparation

The raw clay has been finely ground into particle sizes finer than 100µm. The resulting powder has been moistened at 33%.wt and mixed for 30 minutes in a Winkworth mixer. The resulting paste has been extruded and cut in the form of cylinders.. After drying at 110 °C for 24 hours (**Figure 2**), these pellets have been fired at different temperatures : 1200, 1300 and 1400 °C for 2 hours using a Nabertherm furnace with 2 °C/min as the heating rate. The different obtained chamotte grogs have been tested for density and open porosity. The results, reported in **Table II,** indicate that the sintering temperatures 1200 and 1300 °C are too low to produce dense chamotte grogs. The temperature 1400 °C gives the denser and less porous chamotte grog. This last temperature was then used to prepare larger quantities of the chamotte grog needed for the elaboration of the sample refractory brick.

2.2.2. Formulation of refractory bricks

The formulation of refractory bricks samples has been done in two steps. The first one is the optimization of chamotte grog particle size distribution and of the firing temperature which allows the improvement of brick properties. The second part is the elaboration of the refractory brick itself.

For chamotte grog composition optimization, the grog has been crushed into different size fractions as shown in **Table III**. With these different size fractions, four compositions of bricks (M1, M2, M3 and M4) were prepared by varying the content of the fine fraction. This fine fraction is composed of fireclay and Tikaré raw clay. 0.4%.wt of organic binder LCA LITHOPIX was added to each mixture that had a water content of 12.5%.wt. The mixtures were kept in hermetic bags for 24 hours. Cylinder specimens (diameter = 30mm) have been shaped by uniaxial compaction (30g of moistened mix per specimen) with a pressure of 85MPa using a GRASEBY SPECAC press. After drying at 100 °C for 24 hours, the specimens have been fired at 1200, 1300 and 1400 °C for 2 hours with 2 °C/min as heating rate.

The final refractory bricks (cubic specimens $7x7x7cm^3$) have been shaped by uniaxial pressing of the optimized composition with a 120MPa pressure using a Max Voggenreiter hydraulic press. The cubic specimens have been fired at 1400 and 1500 °C (6 hours soaking time) after a 2 °C/min heating rate ramp.

Table II : Chamotte grog physico-chemical properties versus the elaboration temperature

Temperature	Open porosity (%)	Bulk density (g/cm^3)	Density (g/cm^3) Helium pycnometer	Calculated
1400°C	23.92	1.87	2.6	2.45
1300°C	30.70	1.77	2.67	2.56
1200°C	41.71	1.55	2.73	2.65

Table III: Composition of the different mixtures used in the chamotte grog optimization

Composition	Particle size	M1	M2	M3	M4
		Composition (%.wt)			
Tikaré raw clay	ϕ< 50µm	2,8	5,6	8,4	14
Chamotte grog	ϕ<106µm	11,2	8,4	5,6	0
	106 < ϕ ≤ 200	6	6	6	6
	200 < ϕ ≤ 500	12	12	12	12
	500 < ϕ ≤ 1000	12	12	12	12
	1000 < ϕ ≤ 2500	26	26	26	26
	2500 < ϕ ≤ 5000	30	30	30	30

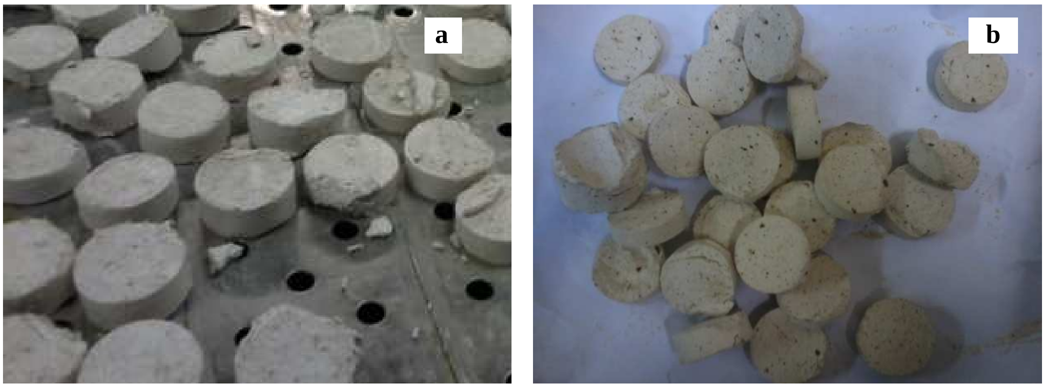

Figure 2: Extruded material: a- dried; b- sintered at 1400°C

2.2.3. Mechanical characterization of the brick

The different fired specimens have been tested for shrinkage (S), water absorption (WA), open porosity (OP), bulk density (BD) and compressive strength (Rc) according to the ISO 10545-3 standard.

The shrinkage has been determined by means of the equation (1), with Ls and Lc respectively the height of the green and fired specimens.

$$S(\%) = \frac{L_S - L_C}{L_S} \times 100 \quad (1)$$

The open porosity OP is the relation between the volume of open pores to the exterior volume of the specimen. Its value is determined with the equation (2).

$$OP(\%.vol) = \frac{P_3 - P_2}{P_3 - P_4} \times 100 \,. \quad (2)$$

The bulk density BD (g/cm^3) is determined using the following equation (3):

$$BD(g/cm^3) = \frac{P_2}{P_3 - P_4} \,. \quad (3)$$

The water absorption is the relation of the mass of water absorbed to the mass of the fired specimen. It is calculated with the following equation (4) :

$$WA(\%) = \frac{P_3 - P_2}{P_2} \times 100 \qquad (4)$$

In the last three different equations, P_2, P_3 and P_4 are respectively the mass of dry fired specimens, of water satured ones and of the hydrostatic mass of immersed fired specimens.

The mechanical properties of fired specimens have been performed by uniaxial diametric compression until failure of specimens using a TONI Technik apparatus. The value of the compression strength Rc (MPa) is deduced from the relationship (5), with F: the maximum load sustained by the specimen and S the value of the section of the specimen.

$$R_C(MPa) = \frac{F}{S} \qquad (5)$$

Refractoriness of bricks is determined by the method of refractoriness under load according to standard ISO 1893: 2007.

2.2.4. Mineralogical characterization

Mineralogical composition of brick sintered at 1400 and 1500 ° C was estimated by XRD using a Philips PW 1729 diffractometer operating at 40 kV - 40mA and employing a graphite monochromatic CuKα radiation. The diffractograms were treated with CRYSTAL software.

3. Results and discussion

3.1. Optimization of grog composition

The different properties of fired specimens of the four mixtures (M1, M2, M3 and M4) are represented by **Figures 3, 4** and **5**.
Whatever the mixture, the bulk density increases (**Figure 3**) with sintering temperature and increases from the mixture M1 to M4, which corresponds to the increase of the raw clay content in the mixture. In other words, the higher is the clay content in the fine fraction of the mix, the higher is the bulk density.

The open porosity (**Figure 4**) decreases while increasing the sintering temperature and with the increase of the amount of raw clay in the mixture. These results are agreement with those of the bulk density.

During the sintering process, there is a formation of a liquid phase at the grains boundaries. The amount of this liquid phase is depending on the increase of sintering temperature. It is partly resulting from the transformation of plastic minerals such as kaolinite. With the increase of the raw clay content, the amount of binder is increased and therefore the liquid phase becomes more important. This phase is to a large extent responsible for the increase of the density and of the mechanical strength of the sintered products.

The evolution of the compressive strength (**Figure 5**) confirms the bulk density and open porosity. With the increase of sintering temperature and of the binder amount (Tikaré raw clay) the

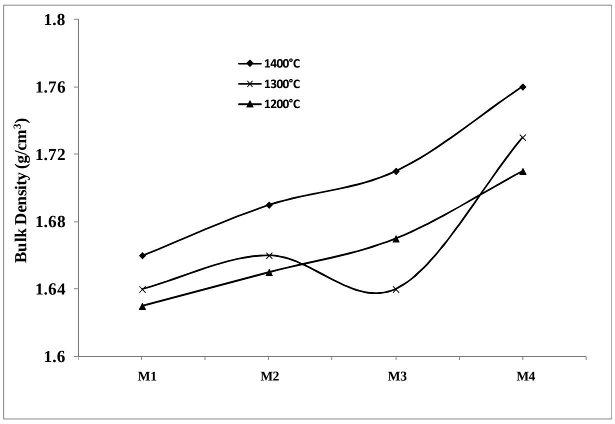

Figure 3 : Evolution of bulk density during the sintering of the different mixtures

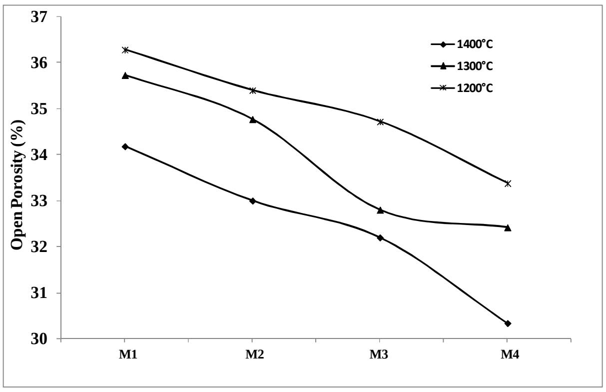

Figure 4 : Evolution of open porosity during the sintering of the different mixtures

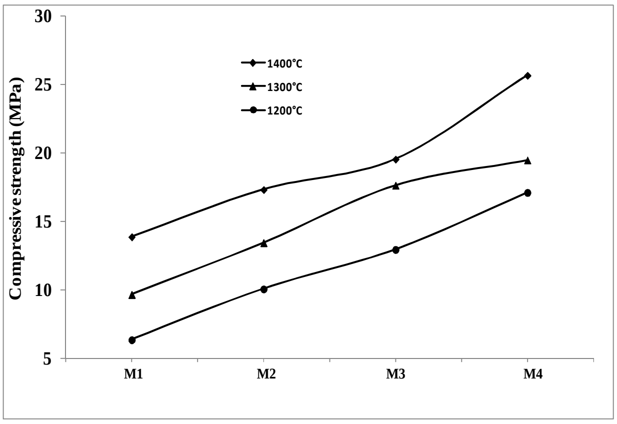

Figure 5 : Evolution of compressive strength during the sintering of the different mixtures

liquid phase becomes more and more important and reduces the porosity of the sintered material. This reduction gives then a consolidated material with better mechanical properties.

The mixture M4 corresponding to 14%.wt of Tikaré raw clay as finer component and sintered at 1400°C, presents the best mechanical properties (Bulk density : 1.76g/cm^3 ; Open porosity : 30.34% ; Compressive strength : 25.64MPa). This mixture has been used for making the final refractory bricks.

3.2. Properties of refractory bricks

The different results of the characterization tests of refractory brick samples (**Figure 6**) are shown in **Table IV**.

Figure 6 : Image of fired bricks : A- 1400 and B- 1500°C

Table IV : Some properties of refractory bricks

Temperature (°C)	1400	1500
Bulk density (g/cm^3)	1.77	2.16
Open porosity (%)	29.94	11.99
Apparent density (g/cm^3)	2.52	2.45
Compressive strength (MPa)	17.49	99.2
Firing shrinkage (lin.%)	1	5
Ultrasonic velocity (m/s)	2474	4200
$T_{0.5}$	1373	1419
T_1	1416	1470
T_2	1441	1486

We note a significant difference between the bricks fired at1400 and those fired at 1500 °C. At 1400°C and with 120MPa as pressure of brick elaboration, the difference between the last specimens sintered at 1400°C with 85MPa as pressure is very limited.

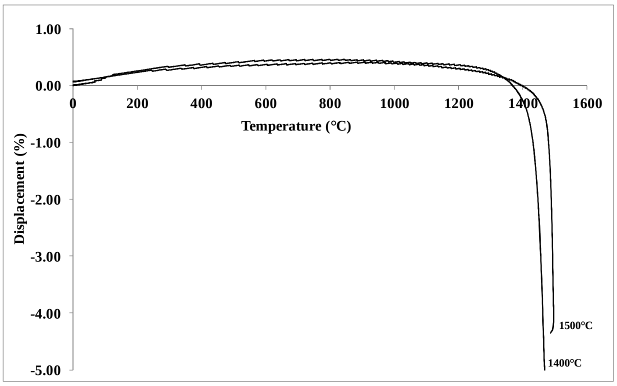

Figure 7: Refractoriness under load curves of fired bricks

This result shows that the pressure didn't improved significantly the mechanical properties. However, the difference is very high between the brick sintered at 1400°C and those sintered at 1500°C. The effect of increasing of temperature is very beneficial for the mechanical properties. The properties of brick obtained at 1500°C with a bulk density (2.16g/cm^3), open porosity (11.99%) and compressive strength (99.2MPa) are comparable to those commercial refractory bricks as indicated in ***Otto Feuerfest booklet*** [4].

The curves of refractoriness under load are given in the **Figure 7**. From these curves, the calculated temperatures ($t_{0.5}$, t_1 and t_2), are summarized in **Table IV**. The temperature $t_{0.5}$ is the temperature corresponding to the start of brick softening. This temperature for all the bricks is not less than 1300°C as recommended by a previous work [3]. However, the effect of sintering temperature is remarkable. The ISO T0.5 is 1373°C for brick fired at 1400°C and becomes 1419°C for the brick fired at 1500°C. This T0.5 temperature depends on the mechanical properties of the fired brick, and in particular on the porosity. When the porosity increases, the temperature T0.5 decreases.

3.3. Mineralogical composition

Figure 8 presents the X-ray diffraction of fired bricks at 1400 and 1500°C. The mineralogical composition of the fired bricks is summarized in **Table V**. The two fired bricks are composed essentially of vitreous amorphous phase, cristobalite, mullite and quartz. With the increase of firing temperature, the vitreous phase quantity increases, at the contrary of cristobalite and quartz. The higher firing temperature enables the transformation of a bigger part of cristobalite and of almost all the quartz into a vitreous amorphous phase. The thermo-mechanical behavior of refractory materials is mainly governed by the

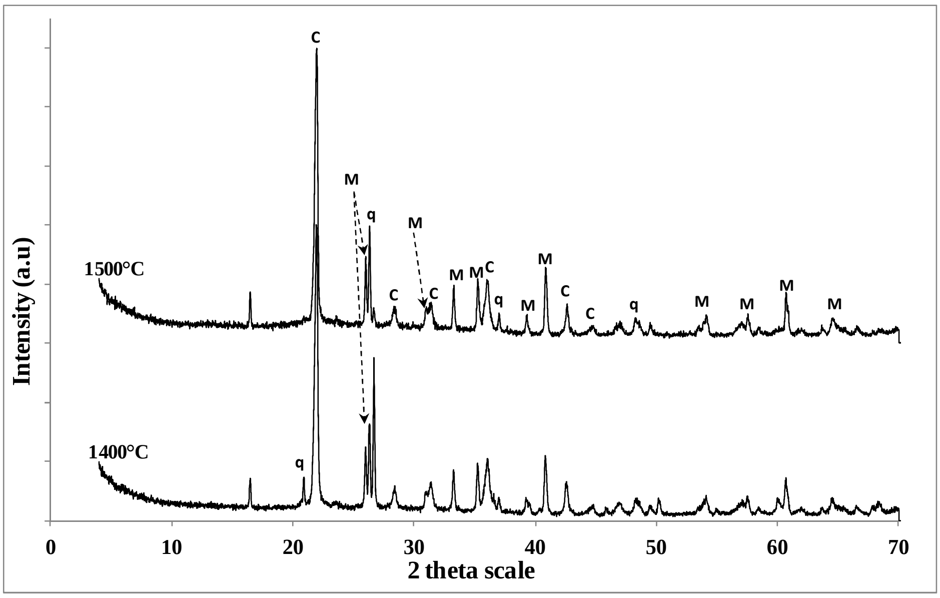

Figure 8 : X-ray diffraction of fired brick : M : mullite ; q : quartz α ; C : cristobalite;

Table V : Mineralogical composition of the refractory bricks

Mineral phases	**Percentage**	
	1400°C	**1500°C**
Glassy amorphous phase	42	51
Cristobalite	30	26
Mullite	22	22
Quartz	7	1
Total	100	100

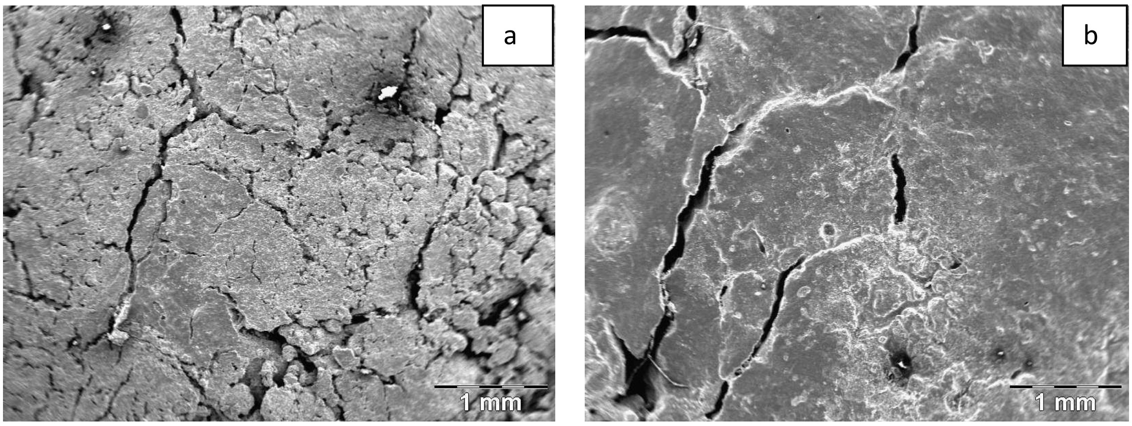

Figure 9 : SEM image of refractory bricks : a- 1400°C, b-1500°C

vitreous phase as show the results of ***M Kolli et al*** **[5]**. The vitreous phase improves the bricks properties firstly by allowing the thermal / residual stresses to accommodate or relax during up quenching or down quenching. Secondly, the vitreous phase can be used to promote crack-tip shielding by bridging cracks under thermal cycling conditions. The mullite quantity didn't change with increasing of temperature. It didn't influence greatly the behavior of refractory brick at high temperature. It plays an important role on the densification of material at temperature around 1100 – 1200°C by reducing the porosity. The firing soaking step during the elaboration of the refractory bricks is very important since it confers to the final product its mineralogical characteristics (chemical reactions between components, grains growth) and physical characteristics (densification, mechanical strength, durability and refractoriness) **[6-8].**

The SEM images (**Figure 9**) confirm the mechanical results. At 1400°C, the refractory bricks are less dense than the bricks at 1500°C. The brick fired at 1400°C is composed by many connected pores contrary to the brick fired at 1500°C.

4. Conclusion

The refractory bricks samples elaborated from Tikaré raw clay material and fired at 1500°C for 6hours present good mechanical and refractoriness properties. The main characteristics values are similar or better to many commercial refractory bricks. This work shows the suitability of the Tikaré raw clay to produce a refractory brick which can be used by the industry.

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