Scientific Research Publishing

Journal of Minerals and Materials Characterization and Engineering, 2024, 12, 79-90
https://www.scirp.org/journal/jmmce
ISSN Online: 2327-4085
ISSN Print: 2327-4077

Manufacture of Industrial Refractory Crucibles Based on Clays from Burkina Faso

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How to cite this paper: Barry, K., Sawadogo, Y., Sawadogo, M., Sorgho, B., Seynou, M., Guel, B., Blanchart, P. and Zerbo, L. (2024) Manufacture of Industrial Refractory Crucibles Based on Clays from Burkina Faso. *Journal of Minerals and Materials Characterization and Engineering*, **12**, 79-90.
https://doi.org/10.4236/jmmce.2024.122006

Received: November 29, 2023
Accepted: March 1, 2024
Published: March 4, 2024

Abstract

In Burkina Faso, one of the three largest gold producers in West Africa, foundry activity is often paralyzed when basic equipment such as crucibles and cups are not available or should be imported. However, previous studies have shown the availability of kaolinite-rich clay resources that could be used for the local manufacture of silico-aluminous ceramic crucibles. This work allowed to manufacture industrial ceramic crucibles with local clays and then they were tested in foundry industry. The materials were manufactured from three (03) raw materials including two clays (SAB and ROU) and sand. The chemical and mineral analysis has shown that the raw materials are suitable for the formulation of refractory materials. The results of characterization of the materials formulated showed that the properties of use are appreciable. The porosity of the materials is relatively low (23 - 28 vol%) with a diametral compressive strength between 0.61 and 1.34 MPa. Crucibles sintered at 1250˚C with a stay of 2 hours have a mechanical strength capable of supporting the weight of the ores contained. These crucibles have a refractoriness under load ($T_{0.5}$) above 1141˚C and resist chemical attacks. Tests were carried out in the industry at 1100˚C, and the results were satisfactory.

Keywords

Refractory, Foundry Activity, Compressive Strength, Chemical Resistance

1. Introduction

Refractory materials are widely used worldwide in various industrial fields due to their specific properties such as resistance to thermal shocks and aggressive media, mechanical resistance, low thermal expansion, etc. [1] [2] [3] [4]. They are used as crucibles and cups in the mining company, in the furnace coatings, filters, etc. [1] [4] [5] [6] [7] [8].

In recent years, several studies have been carried out on silico-aluminous refractory materials and their applications because the economic and industrial capacity of a country is closely linked to its metallurgical processing capacity. Foundry activity is virtually paralyzed when basic equipment such as crucibles are not available, should be imported [5]. However, the abundance of natural mineral resources such as refractory clays can favor the local production of these ceramic crucibles [1] [4].

Burkina Faso is one of the three largest gold producers in West Africa. According to the report of the steering committee of the extractive industry from Burkina Faso (ITIE-BF) published on 27 April 2022 [9], annual gold production was estimated at 62.746 tons in 2020 et 66.858 tons in 2021. However, despite the abundance and accessibility of clay resources, mining industries go on importing refractory crucibles for their foundry because no local manufacturing unit is located there. Silico-aluminous refractory crucibles are mainly obtained from clays rich in silica and alumina and sand (quartz) [1] [4]. They are obtained at high temperatures, above 1200˚C. These materials should be thermally resistant to cope with thermal shocks during the application in foundry industry. The objective of this work is to manufacture refractory crucibles for industrial application from local clays and sand. According to previous works, the mass composition of silica and alumina in the starting powder should be in the ranges of 60 wt% - 65 wt% and 30 wt% - 34 wt%, respectively [4] [10]. Some studies, on the other hand, indicate a much wider range of 20 wt% - 45 wt% for alumina and the Fe_2O_3 content should be less than 2.5wt% [1] [11].

2. Materials and Methods

In this study, two clay raw materials (SAB and ROU) rich in silica and alumina and a raw material rich in quartz (Sand) were used for the manufacture of refractory crucibles. All of these raw materials are from Burkina Faso and have been characterized through previous works [12] [13]. SAB was sampled originate from Sabcé located at about 95 km from Ouagadougou. ROU was sampled originate from Roumba located approximately 130 km from Ouagadougou. The sand was collected at Bobo at coordinates 11˚10 North Latitude, 4˚17 West Longitude. After sampling, clay samples were dried in the sun for two days to remove residual moisture and then crushed to a desired particle size. The used quarry sand containing less impurity was directly crushed to the desired particle sizes. Three different formulations were prepared with variable contents of ROU and sand (**Table 1**) in SAB. Thus, the different formulations are obtained by dry mixing using a jar blender for 15 minutes. The mixtures are then moistened with 25wt% of water depending on contents of ROU and sand.

The manufacture of crucibles was carried out by uniaxial pressing of the moist mixture using a mechanical press in a metal mold; a very smooth inner wall allowing a controlled force to be applied. After shaping, the green crucibles obtained are dried on air in laboratory at 25˚C for 72 hours, followed by a drying in steamer at 80˚C for 12 hours and then at 105˚C for 24 hours. After drying, the green crucibles were sintered at 1250˚C with a stage time of 2 hours or 3 hours and a cooling rate of 5˚C·mn^{-1}.

The references of the final crucibles obtained are reported in **Table 1**. The green and sintered crucibles are represented in **Figure 1**.

The raw materials were mainly characterized by chemical analysis carried out by ICP and mineralogical analysis by XRD like indicate in **Table 2** and **Figure 2**. This made it possible to see the conformity of raw materials for the refractory crucibles manufacture.

The two samples (SAB and ROU) contained kaolinite, illite and quartz as major phases. In addition to these phases, ROU contains a significant quantity of bentonite, which is favorable to the densification of the material during sintering.

The manufactured materials were characterized and tested in foundry industry for their application. The characterization parameters are mainly bulk density, porosity, water absorption, mechanical strength, mineralogy (XRD) and chemical attack tests.

Table 1. Different formulations and references of final crucibles.

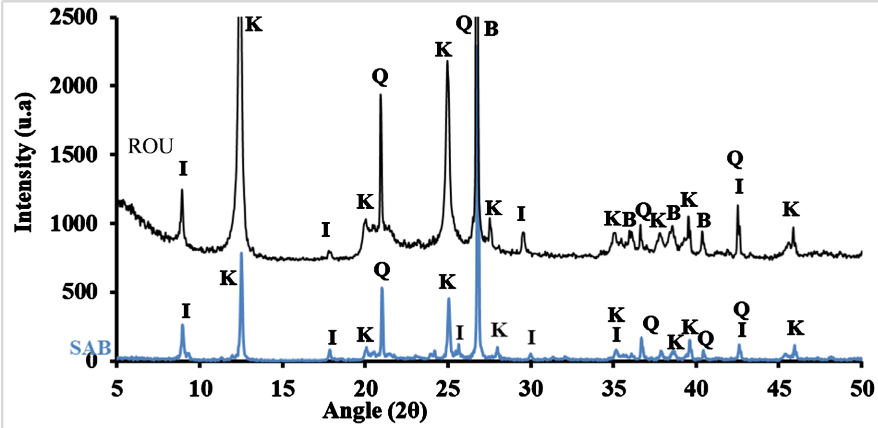
SAB (wt%)	ROU (wt%)	Sand (wt%)	Crucibles reference
85	5	10	A
80	10	10	B
75	10	15	C

Figure 1. Pictures (a) green crucibles and (b) sintered crucibles.

Table 2. Oxides chemical composition of clay raw materials SAB, ROU and sand [12] [13].

Oxides (%)	SiO_2	Al_2O_3	CaO	K_2O	MgO	Na_2O	TiO_2	Fe_2O_3	LOI*
SAB	69.09	20.55	-	1.45	0.12	0.49	0.95	0.85	6.43
ROU	56.51	23.24	1.46	1.77	0.54	0.10	0.66	4.11	9.71
Sand	86.67	9.82	0.13	0.07	0.05	0.16	0.3	0.1	2.70

LOI*: Loss of ignition.



K = kaolinite ; I = illite ; Q = quartz ; B = bentonite

Figure 2. XRD patterns of raw materials [12] [13].

The nature of the crystalline phases within the raw clay materials and the sintered materials was examined by powder X-ray diffraction (XRD) experiments using a D8 Advance DaVinci diffractometer, operating in Bragg–Brentano geometry with CuKα1 radiation (λ = 0.1540598 nm). The operating voltage and current were maintained at 40 kV and 40 mA, respectively. The determination of the peaks was carried out by the DIFFRAC.SUITE EVA software equipped with the ICDD reference databases (PDF2, PDF4 plus), PDF4 AXIOM and COD.

Water absorption was determined by the boiling water impregnation method [2]. It determines the water content filling the water-accessible pores of sintered materials. It was calculated from the relation (1). The bulk density (d) was assessed by the hydrostatic weighing method based on Archimedes' thrust [3] [14]. It was assessed from the relation (2). Open porosity was assessed from the results of bulk density and water absorption [14]. It was calculated from the relation (3).

$$\text{Water absorption}(\%) = \frac{m_2 - m_1}{m_1} \times 100 \tag{1}$$

$$\text{Bulk density}(\%) = \frac{m_1}{m_2 - m_3} \tag{2}$$

$$\text{Open porosity}(\%) = \text{Water absorption} \times \text{Bulk density} = \frac{m_2 - m_1}{m_2 - m_3} \times 100 \tag{3}$$

In these equations, m_1, m_2 and m_3 are respectively the mass of fired specimens, satured mass in water of sintered specimens and hydrostatic mass of sintered specimens.

Diametral compression tests were carried out with an "LLOYD Easy Test EZ 20" universal mechanical testing device, which was configured in compression and controlled by the NEXYGEN PLUS 3.0 software. The speed of elongation was set at 1 mm/min.

$$\sigma_R = \frac{2F}{\pi Dh} = 0.636\frac{F}{Dh} \tag{4}$$

where F, D and h are respectively the load to rupture, the specimen diameter and the specimen thickness.

The acid and basic attacks were carried out by total immersion of the specimens in solutions of hydrochloric acid (HCl, 20 vol%) and sodium hydroxide (NaOH, 30 mg/L) in a hermetically sealed container [1] [15] [16]. After each 7 days of immersion, the specimens are removed and dried at 60˚C in an oven then weighed to evaluate mass variations. The operation is repeated until the 21st day.

The refractoriness was evaluated through the sag under load in compression of the blocks made from the raw materials used to make the crucibles. The test was carried out by measuring the hot resistance under load (RUL). ISO 1893:2007 specifies a method for determining the deformation of dense, insulating shaped refractory products which are subjected to a constant load under conditions of gradual rise in temperature (determination of sag under load), by a differential method [17]. A uniaxial hot-pressing process was used with a constant load of 0.2 MPa and a heating ramp of 5˚C/min. The uniaxial strain was recorded with temperature up to 1500˚C.

3. Results

Figure 3 shows the evolution of water absorption, density (**Figure 3(a)**) and porosity (**Figure 3(b)**) of the crucibles depending on the sand and ROU content and also the sintering stage time. The obtained values of water absorption vary between 16.62% and 19.05% while the bulk density is between 1.82 and 2.12 (g·cm^{-3}). Porosity varies in the same direction as water absorption and decreases when the sintering dwelling time of sintering increases. It is between 23.38% and 27.93% for the dwelling time of 2 hours (P2) against 23% and 25.1% for that of 3 hours (P3).

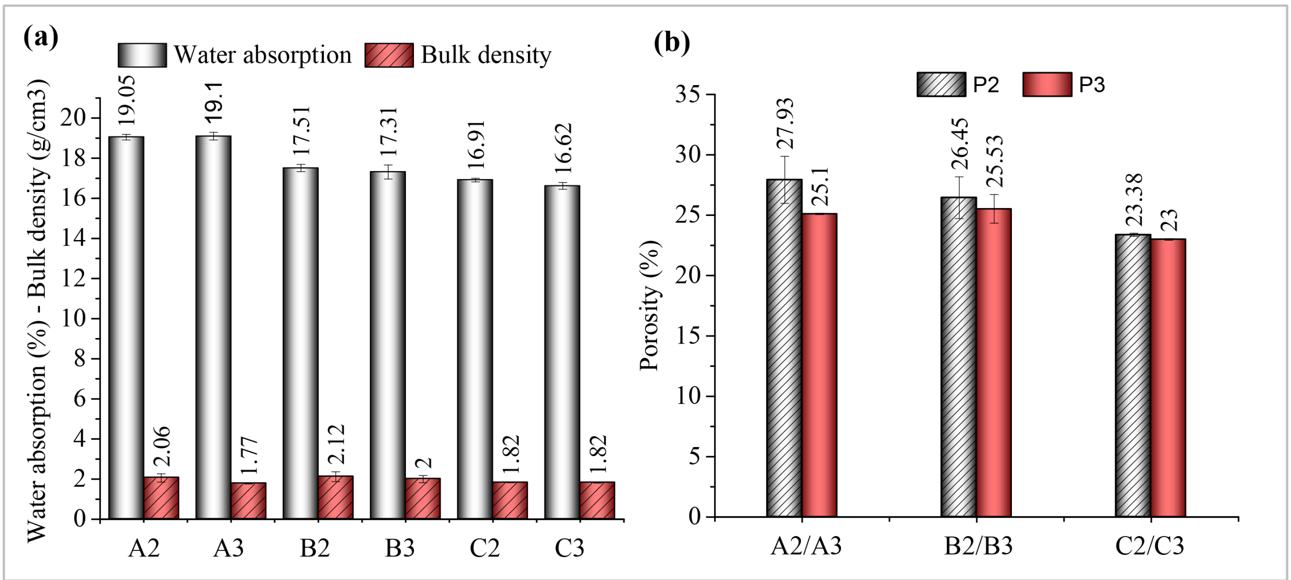


Figure 3. Evolution of (a) water absorption, bulk density and (b) crucible porosity as a function of formulations and dwelling time of sintering.

The strength of the sintered materials was assessed by diametral compression called Brazilian test. The results obtained (**Figure 4**) show an increase in the fracture stress with the increase of the dwelling time of sintering. The compressive strength varies between 0.61 and 1.10 MPa for 3 hours of stage (P3) and between 0.73 and 1.34 MPa for 2 hours of stage (P2). It is also found that the diametral compressive strength increases with the content of ROU and sand.

The test with basic (NaOH, 30 mg·mL^{-1}) and acidic (HCl, 20 vol%) solutions was carried out for 21 days. We see that there is no mass loss on all samples. The elaborate crucibles therefore resist chemical attacks.

The refractoriness under load (RUL) with temperature is plotted in **Figure 5** for the different materials. It is the most used technique to characterize the maximum working temperature under compressive stress [18].

A calibration of creep values ($\Delta L/Lo_{cor}$) was obtained using dense alumina as a reference sample. The expansion of the curve around 500˚C is due to the transformation of quartz *α* and β which is accompanied by an increase in volume, therefore an increase in the height of the material.

Reference temperature corresponding to fixed values of relative deformations (0.5, 1, 2, 5 length%) are obtained in each corrected curve, and they are reported in **Table 3**. These reference temperatures are very useful for the comparison of the behavior with temperature of different refractories under load (RUL).

The results of the mineralogical analysis of the crucibles are shown in **Figure 6**. These results show that all of the materials sintered at 1250˚C, whatever the stage time of sintering (2 or 3 hours), are composed of the same formed mineral phases. We can note mainly mullite and cristobalite. There is also a presence of residual quartz after sintering of materials which could be beneficial for mechanical strength of crucibles.

4. Discussion

The alumina content in clay raw materials SAB and ROU is in the range of 20%

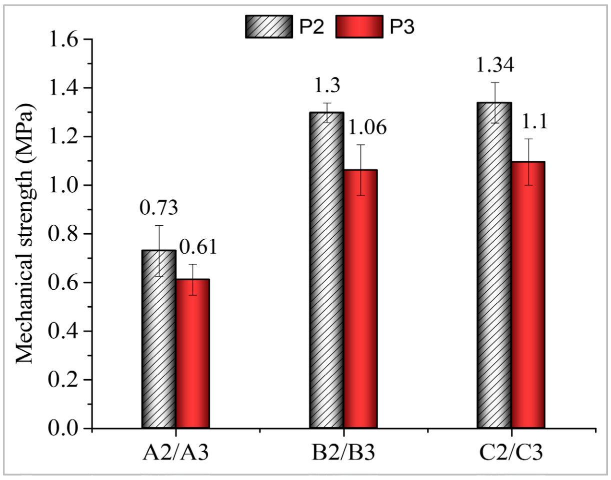


Figure 4. Evolution of diametral compressive strength values of crucibles according to stage time and sand content.

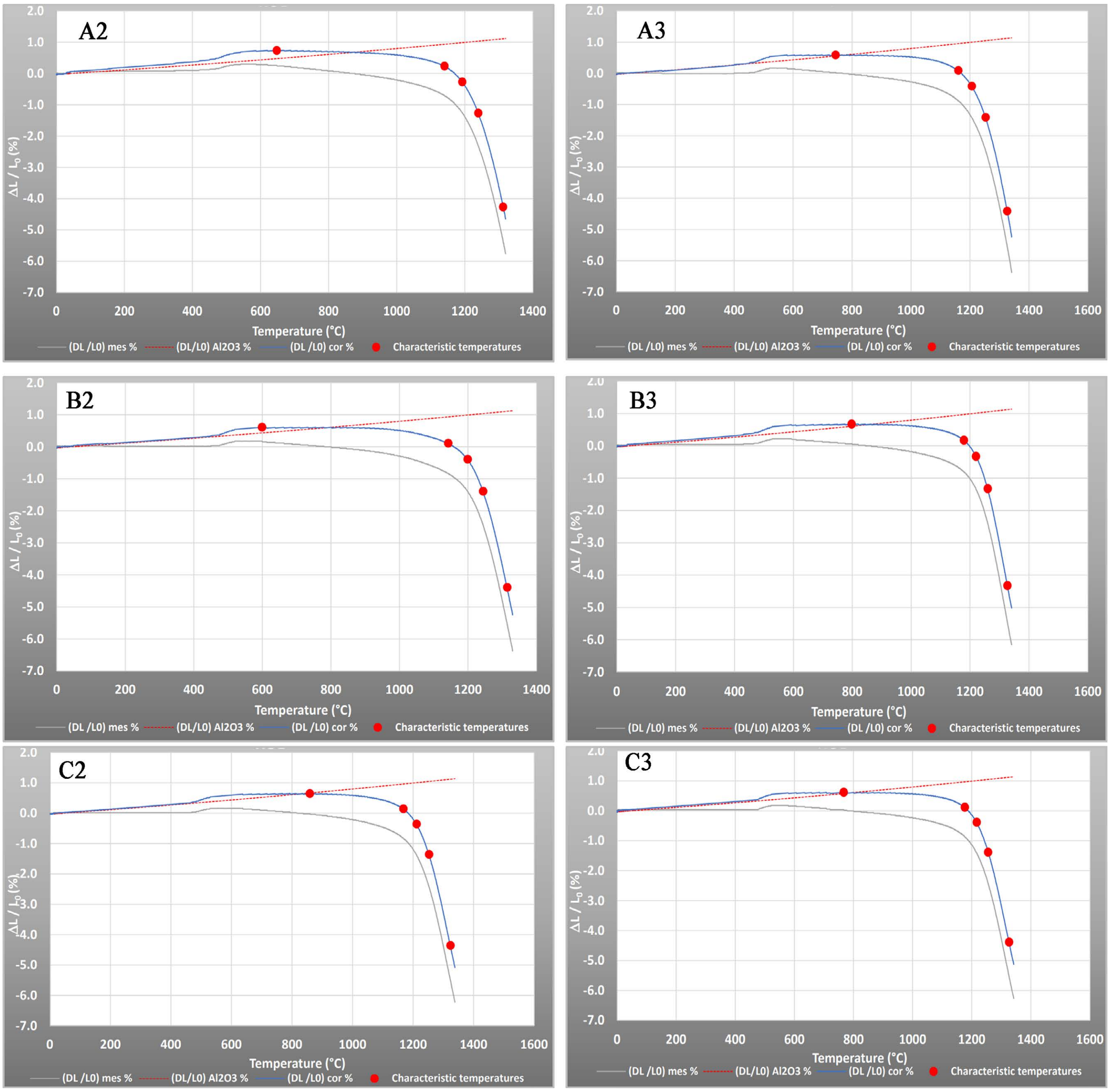


Figure 5. Experimental and corrected refractoriness under load curves.

Table 3. RUL Characteristic temperatures of sintered specimens.

Temperature \ Specimen	A2	A3	B2	B3	C2	C3
T_0 (˚C)	647	744	599	798	859	767
$T_{0.5}$ (˚C)	1141	1161	1142	1179	1168	1177
T_1 (˚C)	1193	1206	1199	1220	1212	1217
T_2 (˚C)	1240	1254	1244	1260	1253	1256
T_5 (˚C)	1313	1327	1315	1327	1324	1326

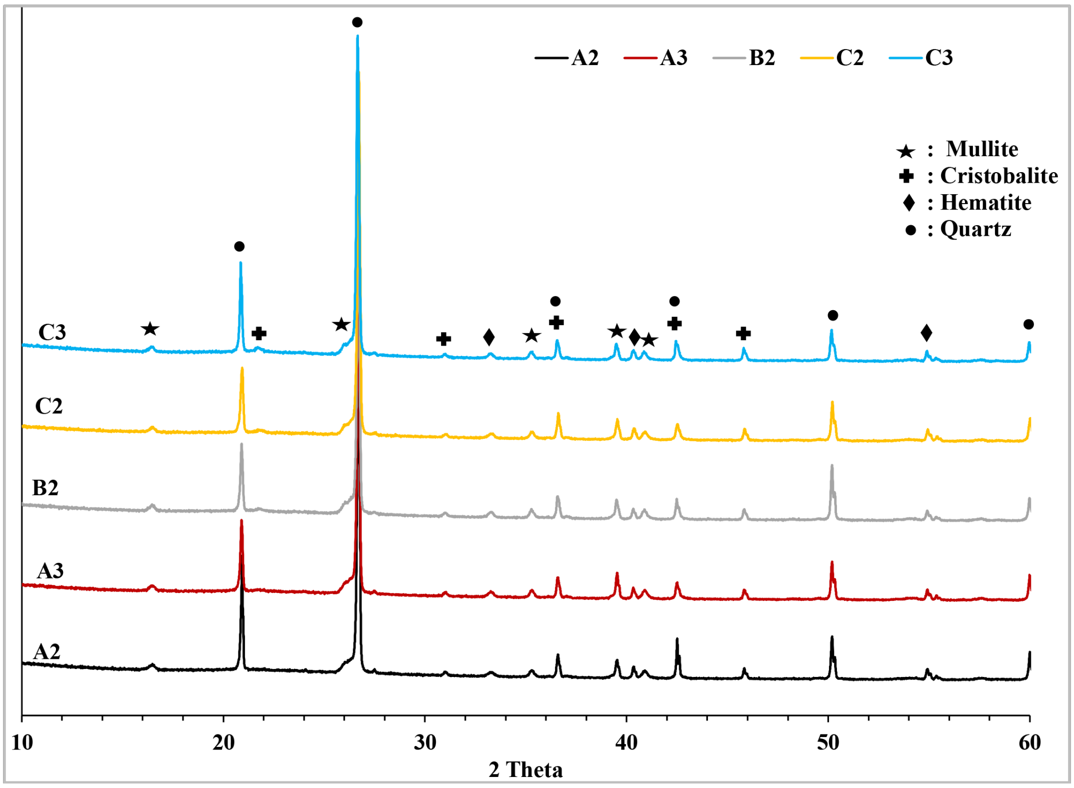


Figure 6. XRD patterns of crucibles.

to 45% indicating that these raw materials are suitable for the manufacture of refractory materials [1] [11]. Firing these refractory materials at low temperatures justifies the use of ROU which provided little melting oxide and bentonite necessary for the moderation of the sintering temperature with regard to the high cost of energy in Burkina Faso. Bentonite is a very specific clay sought in ceramics for its very high plasticity which is linked to the shape and size of its particles. The addition of sand to the mixture improves the mechanical strength of the green materials (crucibles) needed to withstand the handling during the demolding and drying process.

The decrease in porosity in the sintered materials, can be explained by the densification of the material matrix during sintering (**Figure 3(b)**). Indeed, the increase in the content of ROU (melting oxides and mineral) and the stage time favors the viscous flux and the movement of particles [7] [19]. C2 and C3 have lower porosity than those of materials B2 and B3 due to viscous flux from the free silica of the added sand.

Unlike porosity, the diametral compressive strength decreases when the stage time is increased (**Figure 4**). This would mean, in addition to the mullite formed, the residual quartz contained in the sintered materials contributes to the improvement of mechanical strength. The increase in the stage time causes the melting of the residual quartz progressively without causing the germination of mullite crystals which can improve mechanical strength [20] [21] [22]. The evolution of the mullite crystals depends essentially on the sintering temperature and the amount of melting oxides [20] [21] [23], in agreement with the results presented in **Figure 6**. The addition of ROU leads to a supply of melting oxides that can cause the germination of mullite crystals, and thus the increase in mechanical strength [7] [23].

The results of chemical attacks show a change in mass which would be due to the formation of metal precipitates of chloride or hydroxide on the surface of the materials following the phenomena of metal pickling and corrosion [1]. This indicates there is a slight surface deterioration of the materials during their immersion whatever the used chemical solution. In general, the materials would be more sensitive in basic media according to the used concentrations, with regard to mass variations.

The creep strain with temperature under a constant load is quantified with the values of fixed temperatures reported in **Table 3**. Particularly, the temperature $T_{0.5}$ is the temperature corresponding to the onset of softening and at T_2, there is a noticeable strain. For all materials, $T_{0.5}$ is above 1100˚C, the melting temperature of ore in mining companies. $T_{0.5}$ is therefore considered a reference temperature [24]. The refractoriness of materials depends on the chemical composition of the samples [25]. However, the alumina content is just only one of the refractoriness key factors since it decreases strongly with the content of some elements as Na_2O, K_2O, MgO, TiO_2, Fe_2O_3. But the presence of these fluxes (in moderate quantities) allows the densification of the material at relatively low temperatures. It also depends on the sintering temperature and the sintering stage as shown in **Figure 5** and **Table 3**. In general, $T_{0.5}$ increases with the sintering stage and decreases with the porosity value.

Table 4 shows some characteristics of an elaborate crucible made from SAB (85 wt%) + Sand (25 w%), without addition of ROU, sintered at 1250˚C for 2 hours. By comparing these values with those in **Figure 3(b)** and **Table 3**, we see that the addition of ROU reduces porosity but also refractoriness under load. This is due to its content of fluxing oxides and bentonite.

Figure 7 shows the images of the crucibles after tests carried out in industry at 1100˚C and the results are satisfactory. It can be seen that there is no deformation or stripping of the internal surface in contact with the ore. This confirms that the crucibles have good thermal resistance and are also resist to chemical attack.

5. Conclusion

The objective of this work was to manufacture refractory crucibles for industrial applications. The results of chemical analysis (oxides composition) have shown that the used raw materials are suitable for the formulation of refractory crucibles.

Table 4. Some characteristics of a crucible without ROU.

Porosity	Density	Diametral compressive	RUL $T_{0.5}$
29.12 vol%	2.05 g/cm^3	1.32 MPa	1173.6˚C

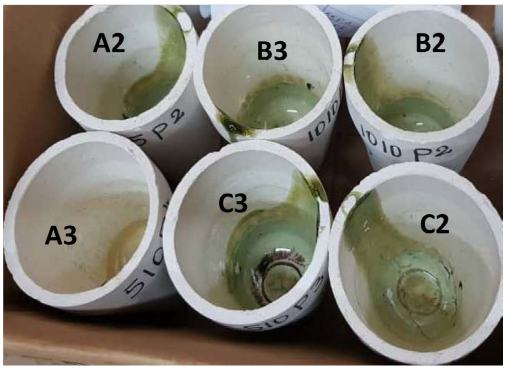


Figure 7. Images of the crucibles after tests at 1100˚C.

Porosity of crucibles obtained decreases when the sintering dwelling time is increased. However, this increase does not seem to be mechanically profitable because the diametral compressive resistance decreases. Whatever the sintering dwelling time and the composition of the mixtures, all of the manufactured materials are essentially composed of mullitic phase and have good resistance in aggressive media. The results show that from a mechanical point of view, the materials sintered at 1250˚C with 2 hours of soaking are more resistant. For all materials, $T_{0.5}$ is above 1100˚C, the melting temperature of ore in mining companies. To confirm this, these crucibles have been successfully tested in gold foundry industry. The compilation of these results, considering the cost of energy in Burkina Faso and tests carried out in industry during this study showed that crucibles B2 could be manufactured locally with the used clays to supply foundry industries. In our future study we plan to raise the sintering temperature in order to improve the mechanical properties.

Acknowledgements

This work benefited on equipment acquired under the project "FRITTE" project of the National Fund for Research and Innovation for the Development (FONRID) of Burkina Faso. The authors express many thanks for financial supports that this work has received during some analyzes carried out outside the Joseph KI-ZERBO University.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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