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Characterization of refractory bricks from selected Cameroonian kaolins

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Abstract

Three clay materials (codded MY3, KG and KK) from western Cameroon were selected and investigated for the production of refractory bricks. Samples MY3 and KG are kaolinite-rich materials, having clay mineral contents of 88% and 72%, respectively, whereas KK is a sand-rich material with a total clay content of 44%. Chamottes were prepared using each clay, and the ground chamottes were later used in the refractory formulation, with each raw clay used as a binding phase. After firing, the X-ray diffraction analyses of all of the refractory bricks indicated the same mineral assemblage made of quartz, cristobalite and mullite. The cristobalite is due to high-temperature conversion of quartz, whereas the mullite is due to clay mineral conversion. The evaluation of the linear shrinkage, physical properties (including bulk density and open porosity) and mechanical testing through refractoriness under load and compressive strength indicates that all of these clays could be used as raw materials for standard clay refractory materials. These results stand as a proof for the potential for these locally available materials to be used as raw materials for refractories that could be locally produced to reduce the cost of access to refractory raw materials faced by industries located in Cameroon.

Refractory materials are high-temperature building materials that are necessary for lining furnaces and high-temperature reactors (Jourdain, 1966; Lapoujade & Le Mat, 1986; Boch, 2001a; Kolli *et al.*, 2007; Poirier, 2011; Minh *et al.*, 2018; Lopez-Perales *et al.*, 2021; Jan *et al.*, 2022). They are strategic materials because they are essential for the production of steel, glass, cement and non-ferrous metals, which are foundations of our civilization (Jourdain, 1966; Aliprandi, 1979; Lapoujade & Le Mat, 1986; Boch, 2001b; Routschka, 2004; Kolli *et al.*, 2007; Amrane *et al.*, 2011; Poirier, 2011; Muhammed *et al.*, 2019; Hossain & Roy, 2021; Lopez-Perales *et al.*, 2021). Historically, clay refractory materials have been obtained cheaply from local resources (Routschka, 2004; Subhojeet *et al.*, 2013; Jan *et al.*, 2022; Vasić *et al.*, 2023). Today, even though they are not considered 'high-tech' materials, they remain useful in many applications requiring high temperatures, such as pottery, earthenware, tableware, sanitary ceramics and brick and tile production applications (Jouenne, 1984; Maitiet *et al.*, 2004; Poirier, 2011; Chaouki, 2014; Bomeni *et al.*, 2018; David *et al.*, 2021). They are also utilized as general-purpose materials, including as linings in the production of steel and cement, amongst other applications.

The policy of sustainable development in Cameroon is mainly orientated towards supporting the value of locally available materials. Many studies have been carried out to identify the potential of local clays for various applications (Nkoumbou *et al.*, 2009; Tassongwa *et al.*, 2014; Nkalih *et al.*, 2015, 2018; Njindam *et al.*, 2018; Pountouenchi *et al.*, 2018; Mouafon *et al.*, 2020). Several exploitable clay deposits were recently found in Foumban located ∼350 km from Yaounde in western Cameroon (Tassongwa *et al.*, 2014). These raw materials are currently used for making typical raw or fired bricks for housing. However, due to their mineralogical composition, some of these clays might have applications in other fields, including ceramics, paper coatings, adsorbents in environmental remediation and refractory bricks.

In the context of improving industrial development, the necessity of achieving minimum loss of energy is challenging. To this end, optimal energy use requires the utilization of high-quality refractory materials. In a developing country such as Cameroon, access to refractories is mainly through imports, and this represents an economic limitation given that this option is not easily affordable for most people. Additionally, in the context of sustainability and environmental challenges, the transportation of imported materials is environmentally harmful. Therefore, we need to develop quality refractories with locally available materials.

Table 1. Chemical and mineralogical composition of the selected kaolin clays (Pountouenchi et al., 2018).

	Chemical composition (%)							Mineralogy composition (%)
	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	Total flux	LOI	Sum	
KK	77.3	15.3	0.9	0.1	1.0	5.4	100	Kaolinite (31), illite (13), goethite (4), quartz (52)
MY3	58.5	28,1	0.5	1.3	1.2	10.2	99.8	Kaolinite (80), illite (8), goethite (1), quartz (6), anatase (4)
KG	57.6	26.1	3.1	1.4	1.0	10.7	99.9	Kaolinite (57), montmorillonite (15), goethite (3), quartz (24), anatase (1)

Total flux = sum of MnO, MgO, CaO, Na_2O and K_2O.
LOI = loss on ignition.

In Cameroon, kaolinite deposits are widely available (Ekosse, 2010; Nkalih et al., 2018), and most of them have been the subjects of various studies regarding the production of construction and building materials (Bomeni et al., 2018; Njindam et al., 2018; Nkalih et al., 2018), environmental remediation uses (Mouafon et al., 2020) or the development of alternative cementitious materials (Tiffo et al., 2020; Ahmed et al., 2023). The present study investigates the possibility of using kaolin clays from Cameroon in the production of dense refractory bricks. The reason for such a study is to optimize the economic potential of a locally available resource for the production of added-value materials. To such an end, it is necessary to choose deposits with sufficient reserves located in readily accessible areas. Such a consideration also contributes to reducing greenhouse gas emissions through limitation of the transportation of raw materials and, in case of positive results, reducing greenhouse gas emissions

due to importing materials that can be locally produced. Additionally, the installation costs for a production plan using local materials would also be reduced. For these reasons, in this study three kaolins from the Foumban aera in western Cameroon were chosen. Chamottes were prepared from each sample at two different temperatures, and these were ground and used together with each raw clay as a binding phase in refractory formulations and finally fired at three different temperatures. Some of the technological characteristics of the obtained refractories were evaluated and the obtained parameters are discussed in terms of potential applications of these refractories.

Materials and methods

Raw materials

Clay materials were collected from three deposits located in two localities situated in western Cameroon: Mayouom for sample MY3 and Koutaba for samples KK and KG. The GPS coordinates for the sample collections are 5°35′27″ N and 10°40′18″ E for KK,

Table 2. Compositions of mixtures used for refractory material synthesis.

			Weight (%)		
Composition		Particle size (μm)	q = 0.5	q = 0.4	q = 0.3
Binder clays	KK, MY3 and KG	Ø < 100	14	20	30
Chamottes	KK, MY3 and KG	100 ≤ Ø < 200	6	8	8
		200 ≤ Ø < 500	12	12	12
		500 ≤ Ø < 1000	12	12	11
		1000 ≤ Ø < 2360	24	22	18
		2360 ≤ Ø < 5000	32	26	21

Ø = diameter.

Table 3. Physical properties of the produced chamottes.

	CHM KK	CHM KG	CHM MY3
Firing temperature	1550°C	1450°C	1450°C
Bulk density (g cm^{-3})	2.2	2.4	2.3
Open porosity (vol.%)	8.3	10.5	11.9
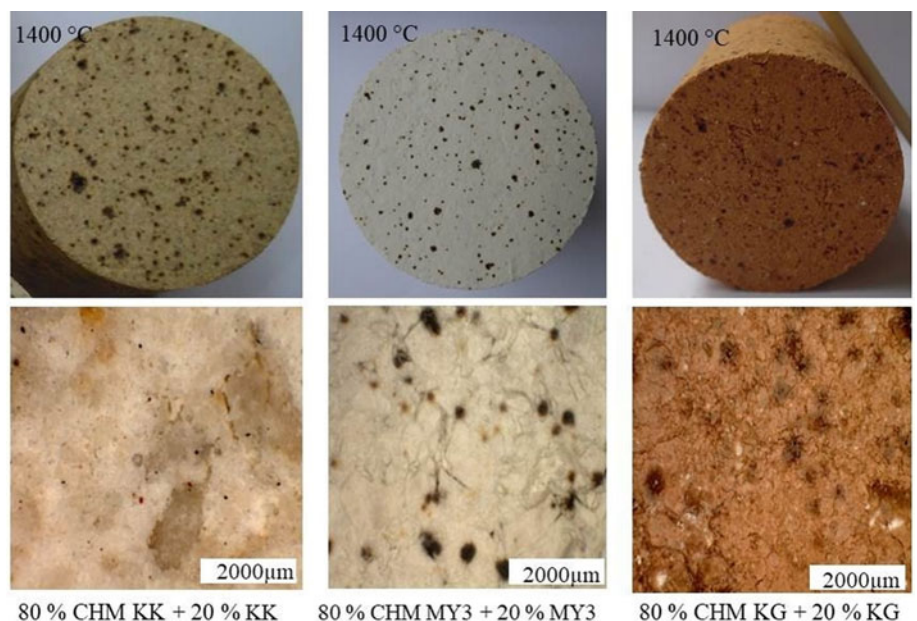
Figure 1. Optical microscopy images of the polished surfaces of some refractories elaborated with the same content of the bonding phase.
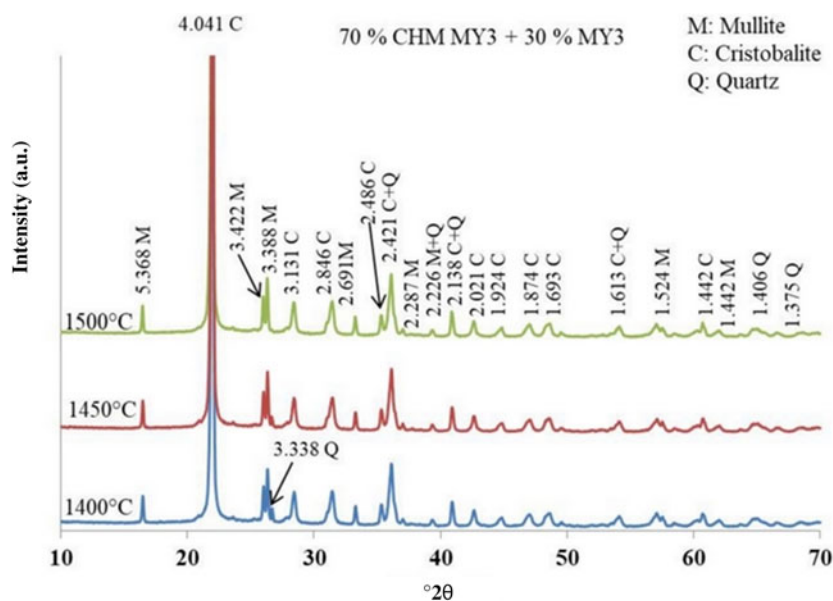
80 % CHM KK + 20 % KK   80 % CHM MY3 + 20 % MY3   80 % CHM KG + 20 % KG

Figure 2. XRD traces of the refractory bricks.

5°35′20″ N and 10°41′26″ E for KG and 5°51′N and 10°59′ E for MY3. The MY3 and KK clays are residuals derived from the alteration of granites and mylonites, which are predominant rocks in these regions, whereas KG is a hydromorphic clay (Tassongwa *et al.*, 2014; Nkalih *et al.*, 2015; Njindam *et al.*, 2018; Pountouenchi *et al.*, 2018). The samples have been described in previous studies (Pountouenchi *et al.*, 2018; Njuhou *et al.*, 2023). A summary of their chemical and mineralogical compositions is reported in Table 1.

Sample KK exhibits a higher SiO_2 content and a lower Al_2O_3 content compared to its counterparts. The KG and MY3 samples have similar SiO_2 and Al_2O_3 contents. The sum of fluxing oxides is low (<1.5% for all of the samples) and is in the following sequence: KK < KG < MY3. These low flux contents indicate that the final materials will probably exhibit high refractoriness and that the fabrication process will require a high firing temperature to obtain materials with good physical and mechanical properties. The high SiO_2 content of sample KK is due to its high sandy content (Table 1). All of the samples exhibit Al_2O_3 contents in the range 10–30% and SiO_2 contents of <85%. Therefore, according to ISO 10081-1, these specimens can be considered as raw materials for manufacturing low-alumina fire clay refractory materials (LF10 group; Norme ISO 10081-1, 2003).

Experimental methods

Clay and chamotte preparation
The raw clays were dried, ground and sieved through a 100 μm sieve. Chamottes were prepared from each clay. They were pressed in $7 \times 7 \times 7$ cm³ steel moulds at 100 MPa with a uniaxial hydraulic press. The KK chamotte was calcinated at 1450°C and 1550°C for 4 h, whereas the MY3 and KG chamottes were fired only at 1450°C for 4 h. The KK chamotte was calcined at two temperatures, because at 1450°C it exhibits a residual open porosity that is too high for its use in the targeted dense materials. After cooling, the chamottes were manually ground in a ceramic mortar and sieved to obtain five different grain-size distributions in the diameter range of 100–5000 μm (Routschka, 2004; Seynou *et al.*, 2013).

For the elaboration of refractory products, a mixture of five particle-size distributions was used (Table 2). The chamottes obtained from the clays KK, KG and MY3 are noted as CHM KK, CHM KG and CHM MY3, respectively.

Refractory material synthesis
Dense refractory material compositions were obtained by mixing ground chamottes (aggregates) with a fine-particle raw clay (bonding phase or matrix). The composition and the particle-size distribution were based on the Andreasen Law (Equation 1):

$$P(\%) = 100(d/D)^q \qquad (1)$$

where P is the fraction (%) of particles smaller than d, q is the exponential factor, d is the smallest particle size and D is the

**Table 4.** Semi-quantitative analysis of the refractory bricks at 1350–1500°C.

Formulation	Temperature (°C)	Amorphous matter (%)	Cristobalite (%)	Mullite (%)	Quartz (%)
70% CHM KK + 30% KK	1400	23	56	20	1
	1450	26	54	19	1
	1500	28	53	19	–
70% CHM KK + 30% MY3	1400	27	46	23	4
	1450	31	45	22	2
	1500	30	46	22	2
70% CHM KK + 30% KG	1400	28	49	22	1
	1450	29	48	22	–
	1500	31	46	22	1
70% CHM MY3 + 30% MY3	1350	30	17	41	12
	1400	34	16	40	10
	1450	39	12	40	9
70% CHM MY3 + 30% KG	1350	29	22	39	10
	1400	32	21	39	8
	1450	34	20	38	8
70% CHM KG + 30% KG	1350	31	32	34	4
	1400	26	33	37	4
	1450	31	35	32	2
70% CHM KG + 30% MY3	1350	27	30	36	7
	1400	25	31	38	6
	1450	25	32	38	5
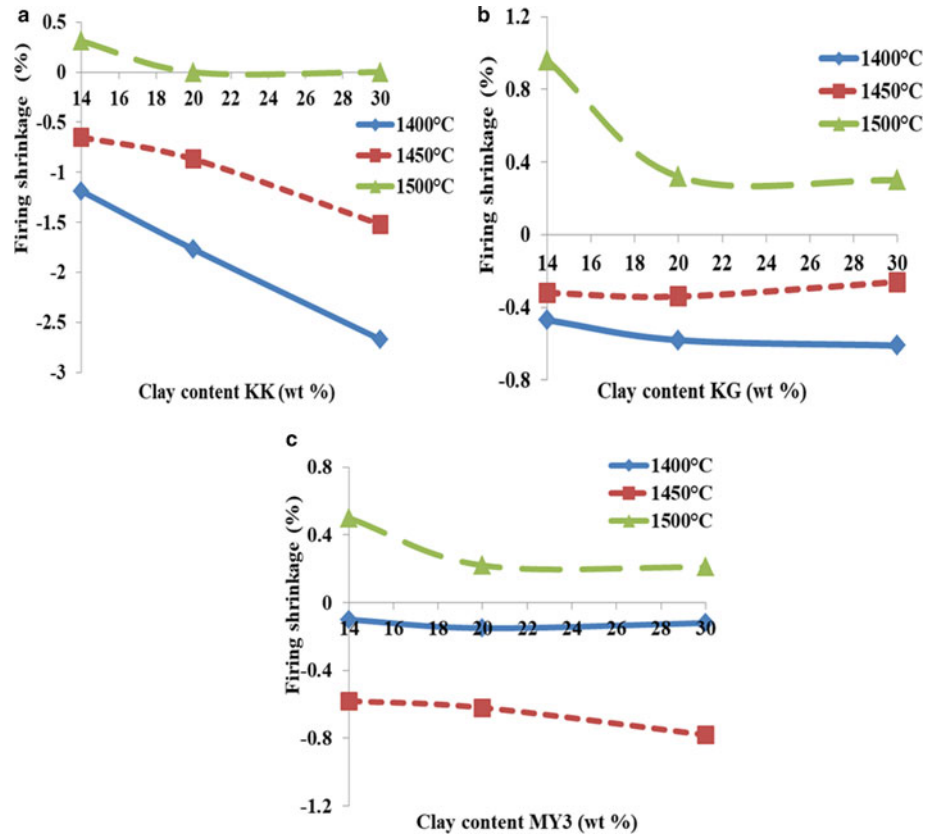
Figure 3. Firing shrinkage of materials containing chamotte KK obtained after firing at 1400°C, 1450°C and 1500°C *vs* (a) KK , (b) KG and (c) MY3 clay content.
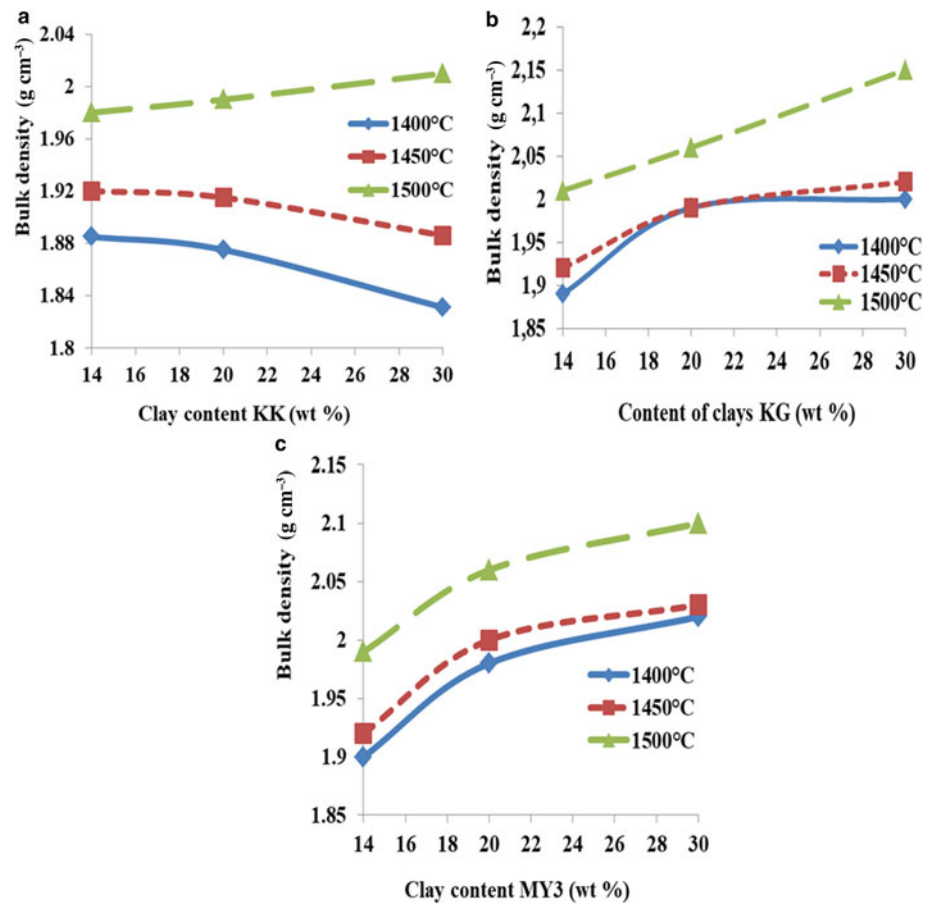
Figure 4. Bulk density of materials containing chamotte KK obtained after firing at 1400°C, 1450°C and 1500°C *vs* (a) KK, (b) KG and (c) MY3 clay content.
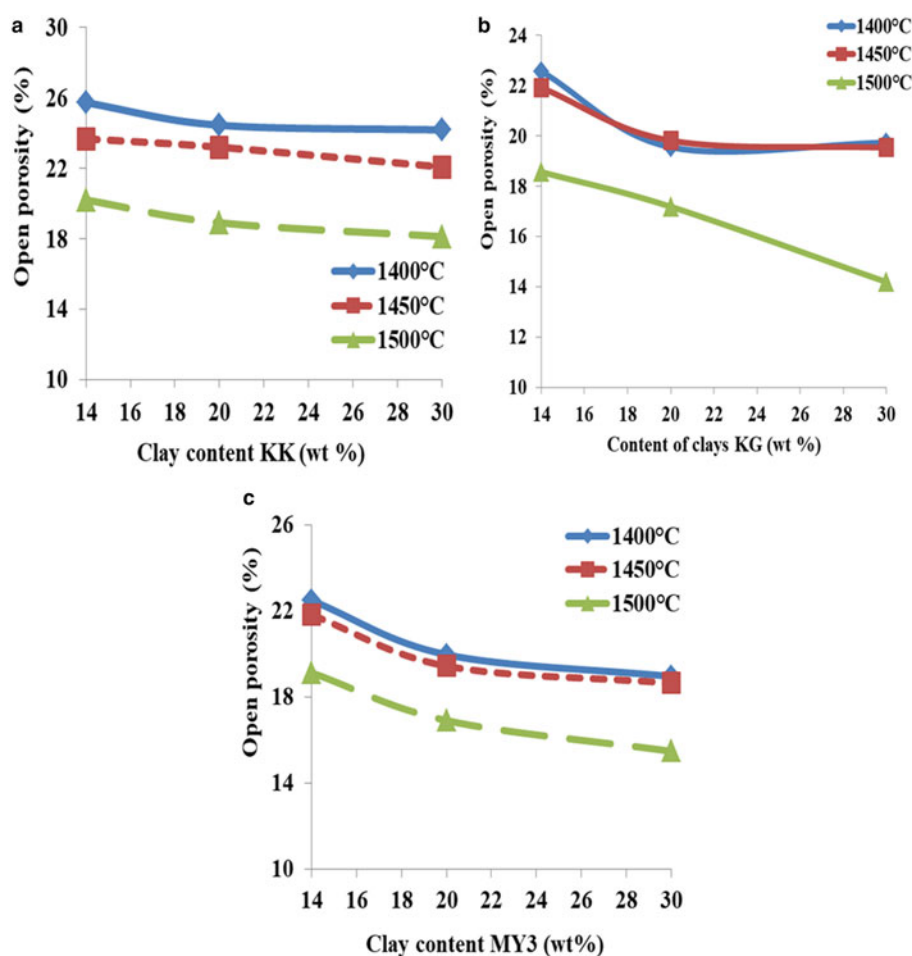
Figure 5. Open porosity of materials containing chamotte KK obtained after firing at 1400°C, 1450°C and 1500°C *vs* (a) KK, (b) KG and (c) MY3 clay content.

largest particle size. Theoretically, a q value of 0.5 leads to the greatest compactness; however, values ranging between 0.3 and 0.5 are commonly used (Routschka, 2004; Seynou *et al.*, 2013). The investigated compositions are listed in Table 2. For each formulation, the amount of raw clay used as a binder is expressed in the percentage mass of the total mass of the mixture.

The chamotte CHM KK was mixed with 14 wt.% ($q = 0.5$), 20 wt.% ($q = 0.4$) and 30 wt.% ($q = 0.3$) KK clay. Subsequently, the KK binder phase was substituted with the same proportions of MY3 and KG since KK is richer in SiO_2 and lower in impurities, shows a low level of densification up to 1500°C and does not constitute a good binding phase for refractory brick production (Pountouenchi *et al.*, 2018; Pountouenchi, 2020).

The chamottes CHM MY3 and CHM KG were mixed with the same proportions of MY3 and KG clays, respectively. The KG and MY3 binding phases were not substituted by other clays because they both have similar thermal behaviours and KK exhibits too high refractoriness (Pountouenchi *et al.*, 2018; Pountouenchi, 2020).

Some 4 wt.% water was added to the mixtures followed by manual mixing. Samples were pressed in a steel mould using a uniaxial hydraulic press. For the sintering investigation, cylindrical samples (30×30 mm) pressed at 100 MPa were used, whereas for mechanical properties 50×50 mm cylindrical samples were obtained by pressing at 120 MPa. The refractories produced from chamotte KK were fired from 1400°C to 1500°C, whereas those produced using chamottes MY3 and KG were fired from 1350°C to 1450°C for 4 h.

Characterization of the fired refractory materials

After firing, the materials were characterized for their microstructure, mineralogical composition, shrinkage, bulk density, water absorption, open porosity, compressive strength, refractoriness under load and thermal shock resistance. The microstructure was observed with a Leica DM750 optical microscope using polished surfaces to improve microstructure observation. The mineralogical composition of the fired products was determined using X-ray diffraction (XRD) with a Philips PW 1729 diffractometer using Cu-$K\alpha$ radiation (1.548 Å) at 40 kV and 40 mA. The XRD traces were treated with the *CRYSTAL* software for mineral identification. Firing shrinkage was evaluated as the difference between sample thicknesses before and after thermal treatment. Bulk density and open porosity were determined using the water displacement method according to EN 993-1 standard (Norme ENV 993-11, 1997).

For the mechanical, refractoriness under load and thermal shock tests, 50×50 mm cylinders (with an uncertainty of ±0.5 mm) were used. The compressive strength was measured using a Toniversal 645–656 brand hydraulic testing machine (crushing speed of 0.005 N/mm^2.s) from the maximum load sustained by cylindrical samples according to the EN 993 standard (Norme NBN EN 993-5, 1999). The refractoriness under load was evaluated using the differential method with increasing temperature according to the EN-ISO 1893 standard (Norme NBN EN 993-8, 1997). A uniaxial hot-pressing process was used with a constant load of 0.2 MPa and a heating rate of 5°C min^{-1}. The uniaxial strain was recorded at 15 s
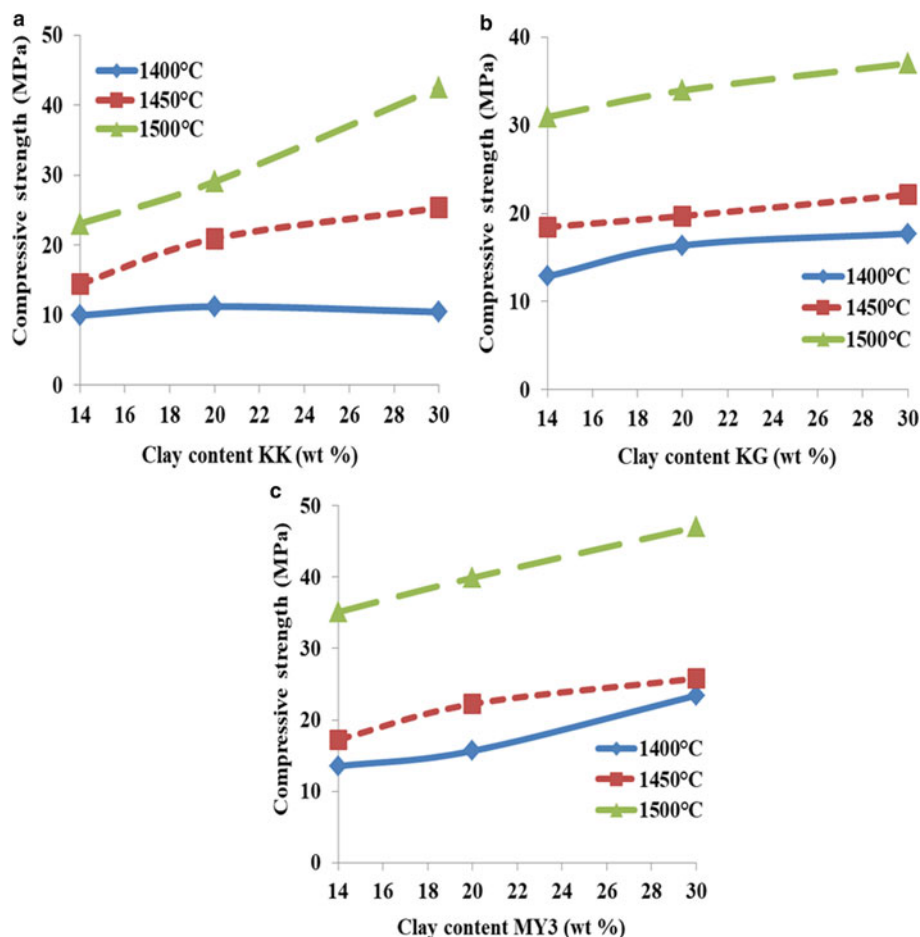
Figure 6. Compressive strength of materials containing chamotte KK obtained after firing at 1400°C, 1450°C and 1500 °C *vs* (a) KK, (b) KG and (c) MY3 clay content.

intervals up to 1600°C. The temperatures ($T_{0.5}$, T_1, T_2 and T_5) corresponding to sagging percentages of 0.5%, 1.0%, 2.0% and 5.0% of the maximum height of the specimen were noted. Thermal shock was used to evaluate the potential use of a refractory as an internal wall material for continuous-use furnaces and was determined according to the EN 993-8 standard (Norme NBN EN 993-1, 1995). The samples were heated at 950°C for 15 min in a Nabertherm electric fournace and immediately quenched in a flowing water bath at 25°C. The test was repeated up to sample decohesion. A maximum of 30 cycles were applied.

Results and discussion

Chamotte characterization

Table 3 lists the physical properties of the fired clay samples.

At 1450°C, the chamotte KK exhibits a high residual open porosity (41.8%), which renders the obtained fired product unsuitable for use in the fabrication of the targeted dense materials. A higher firing treatment at 1550°C led to a low porosity (8.3 vol.%) consistent with its use for the targeted dense materials. At 1450°C, chamottes MY3 (10.5%) and KG (11.9%) exhibit low open porosity (10–12%) and high density (2.3–3.5 g cm^{-3}), which are consistent with the targeted application. The fact that KK does not develop a chamotte of interest for dense products at 1450°C might be due to the difference in flux contents (Table 1). The higher

flux contents in KG and MY3 probably favour an increase in density and decrease in porosity associated with vitreous-phase formation, in which capillary diffusion induces pore enclosure and favours densification at lower temperatures than in KK. It is suggested that the addition of fluxes to starting materials for chamottes might reduce the firing temperature and hence reduce energy use during their production.

Characterization of fired products

Optical microscopy

Figure 1 shows optical microscopy images of the polished surfaces of some refractories. The refractory products obtained from chamottes (KK and MY3) and from KK and MY3 clays as bonding-phase sinters are white with tiny dark red spots (Fig. 1) due to the low colourant oxide contents (Fe_2O_3 and TiO_2) in the raw materials (Table 1). By contrast, those with KG clay as the bonding-phase sinter were coloured due to the abundance of Fe_2O_3 and TiO_2 in this sample (Table 1), leading to the formation of coloured solid solutions after firing.

Mineralogical analysis of the refractory products

The composition of refractory products reflects the composition and transformation of both the aggregates and the bonding phase. The aggregates (chamotte) were pre-fired and their mineralogical composition was transformed before firing. Therefore, they are more stable (in terms of composition and volume), but because their stability has not been specifically tested before,
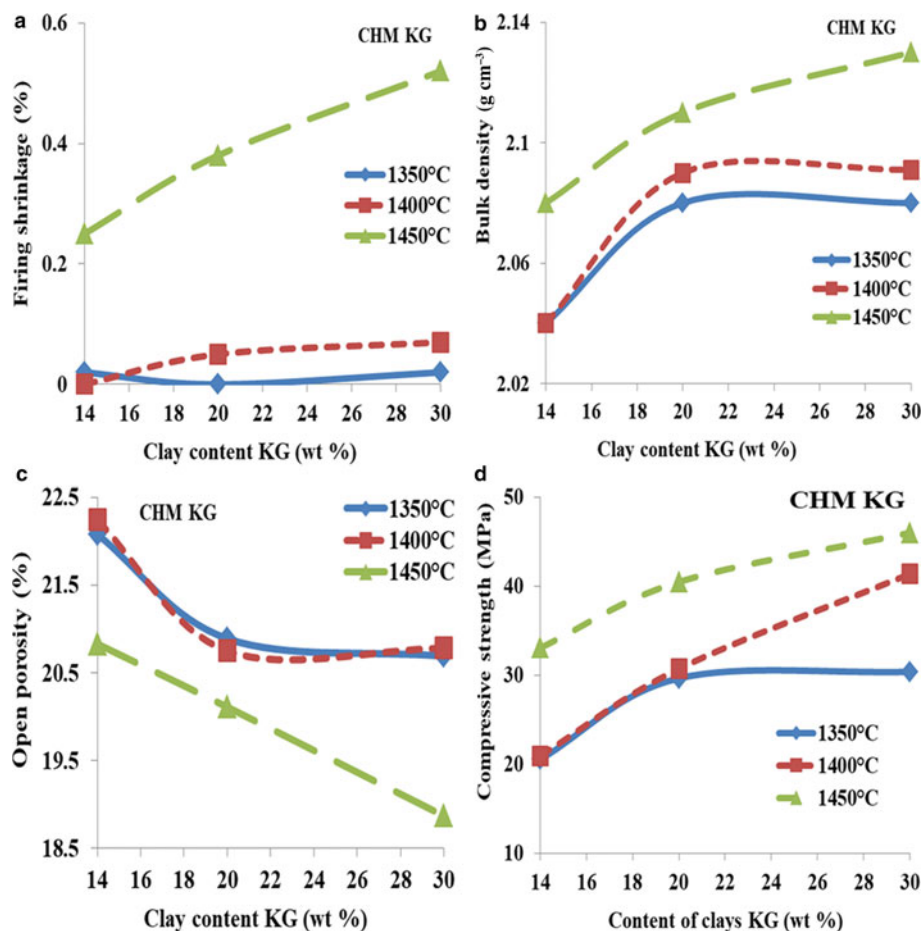
Figure 7. (a) Firing shrinkage, (b) bulk density, (c) open porosity and (d) compressive strength of materials containing chamotte KG obtained after firing at 1350°C, 1400°C and 1450°C *vs* KG clay content.

their possible transformation during their second firing cannot be excluded. However, the thermal behaviour during firing of the products is probably due to the bonding phase (unfired clay; Djangang et al., 2008).

Figure 2 presents representative XRD traces of materials fired at various temperatures. Semi-quantitative analysis of the refractory bricks is given in Table 4. All materials contain mullite, cristobalite, abundant amorphous matter and sometimes some residual quartz. The amorphous phase is typically present in refractory products after firing, which is in accordance with previous work (Aydın, 2004; Martín-Marquez et al., 2009; Minh et al., 2018; David et al., 2021). This is due to melting at high temperatures of Al-silicates and fluxing agents, which promotes the formation of a vitreous phase during cooling. Furthermore, metakaolin decomposes to form γ-alumina and amorphous SiO_2 according to the following equation: $Al_2O_3.2SiO_2 \rightarrow Al_2O_3 + 2SiO_2$. Part of this amorphous SiO_2 remains in the amorphous phase at the end of the firing process when the cooling of the kiln is controlled and set at a sufficiently rapid rate (5°C min^{-1}). Cristobalite forms mainly from the quartz transformation associated with high-volume expansion. Mullite crystallization is due to the reaction between Al_2O_3 and SiO_2 according to the following equation: $3Al_2O_3 + 2SiO_2 \rightarrow 3Al_2O_3.2SiO_2$ (Kumar et al., 1999; Boch, 2001a; Chen et al., 2004; Kornmann, 2005; Djangang et al., 2007; Sahnoune et al., 2008).

Mullite forms mainly due to clay mineral transformation. For the same reason, a large fraction of the amorphous phase also forms due to clay transformation at the beginning of the reaction.

The MY3 samples exhibit a higher content of fluxing agents. This explains the higher level of densification of the MY3-based products (Kumar et al., 1999; Kornmann, 2005; Parvesh et al., 2007; Pountouenchi et al., 2018; Muhammed et al., 2019; Pountouenchi, 2020; David et al., 2021; Njuhou et al., 2023). MY3 products also have the highest mullite content due to the higher Al_2O_3 content in the raw clay material. The apparent low cristobalite content for MY3-based products is due to the abundant mullite formed by reaction between Al_2O_3 and SiO_2. The high cristobalite content of KK materials can be explained by the high quartz content and the low alumina content of the clay raw material. This results in the limited formation of mullite, which allows for the excess amorphous SiO_2 to transform into cristobalite, with the remaining being amorphous.

Physical and mechanical properties of the fired products

Figures 3–8 present the physical and mechanical properties of the final refractory bricks *vs* firing temperature. The negative shrinkage (expansion) of the materials (Fig. 3) results from three mechanisms: expansion due to crystallization of cristobalite and mullite; densification due to appearance of a molten phase; and flow into the pores (liquid sintering). Expansion begins at lower temperatures and liquid sintering occurs at higher temperature (Routschka, 2004; Njindam et al., 2018).

With respect to clay addition, the shrinkage decreases or remains stable depending on the temperature. There is also a balance between liquid sintering and crystallization (Nkalih et al., 2018; Pountouenchi et al., 2018), which explains the increase in
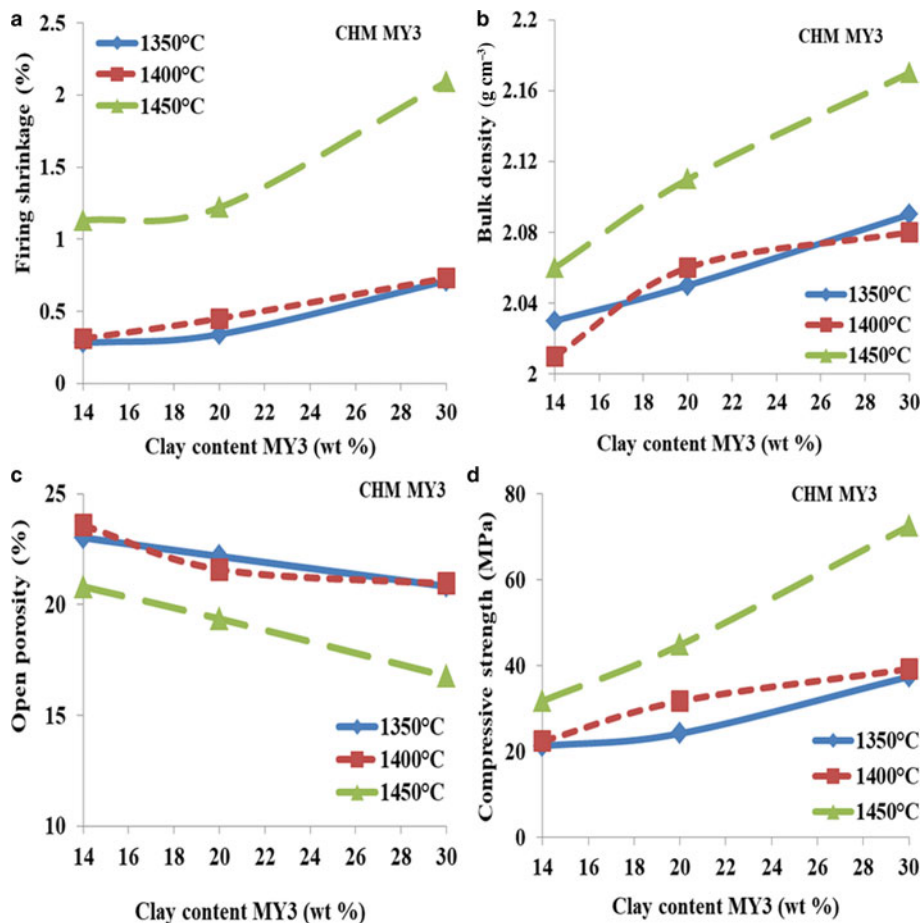
Figure 8. (a) Firing shrinkage, (b) bulk density, (c) open porosity and (d) compressive strength of materials containing chamotte MY3 obtained after firing at 1350°C, 1400°C and 1450°C *vs* MY3 clay content.

shrinkage with temperature. Higher temperatures promote an increase in the liquid-phase content. Greater shrinkage is observed in the KG clay for the binder phase (Fig. 3b) and less shrinkage is observed in the KK clay (Fig. 3a).

In KK-bonded materials, the low shrinkage (high expansion) is due to the higher quartz content (Table 1) and thus the higher cristobalite (Table 4) content after transformation associated with

expansion. This also explains the large volume increase at lower temperatures when the clay quantity increases (Fig. 3a). The KG- and MY3-bonded materials show greater shrinkage (Fig. 3b,c) due to their lower quartz contents (Table 1) and larger amounts of the molten phase at high temperatures (Pountouenchi *et al.*, 2018).

A direct correlation exists between open porosity and densification (shrinkage). Open porosity decreases with densification
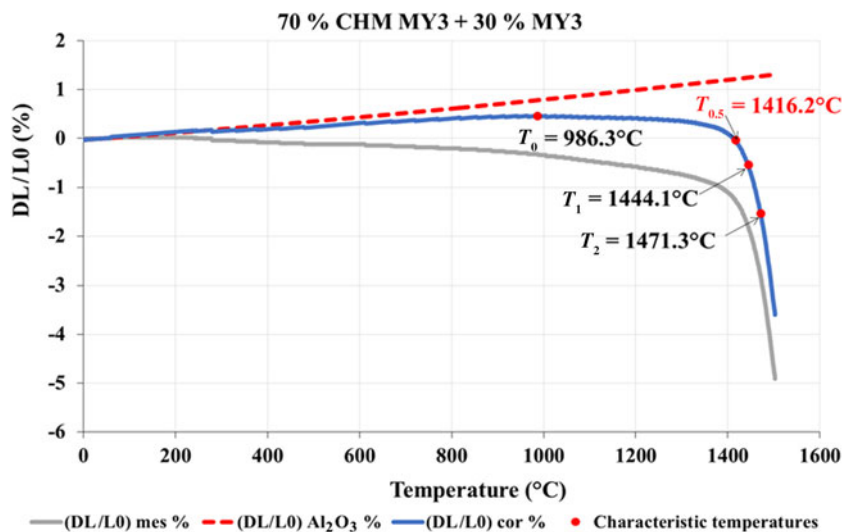
Figure 9. Example of a refractoriness under load curve for MY3. DL/L0 = the rate of material expansion/shrinkage; DL/L0 cor % = the corrected rate of material expansion/shrinkage; DL/L0 mes % = the measured rate of material expansion/shrinkage.

Table 5. Refractoriness under load of some refractories produced with certain quantities of the binder phase.

	T_0	$T_{0.5}$	T_1	T_2	T_5
			Refractoriness under load		
Firing temperature (°C)			1450		
70% CHM KK + 30% KK	909	1333	1398	1450	1494
70% CHM KK + 30% MY3	875	1335	1411	1452	–
70% CHM KK + 30% KG	929	1390	1452	1467	–
Firing temperature (°C)			1400		
70% CHM MY3 + 30% MY3	986	1416	1444	1471	–
70% CHM MY3 + 30% KG	973	1395	1428	1455	–
70% CHM KG + 30% KG	1125	1350	1393	1434	–
70% CHM KG + 30% MY3	1107	1360	1407	1444	–

Table 6. Physical properties of refractory products before and after the thermal shock tests (30 cycles).

Refractory product	70% CHM MY3 + 30% MY3	70% CHM MY3 + 30% KG
Bulk density (g cm^{-3})		
Before the thermal shock	2.2	2.1
After the thermal shock	2.1	2.1
Open porosity (%)		
Before the thermal shock	16.8	18.2
After the thermal shock	18.6	20.8
Compressive strength (MPa)		
Before the thermal shock	72.5	39.2
After the thermal shock	54.4	27.1

(Figs 5, 7c & 8c). Conversely, density increases with decreasing porosity (Figs 4, 7b & 8b). Density values result from open porosity and the phase composition.

The compressive strength evolution at different temperatures *vs* composition (Figs 6, 7d & 8d) indicates that these values are inversely proportional to open porosity (Figs 5, 7c & 8c). Materials with low open porosity exhibit more efficient bonding and a smaller quantity of defects (pores), which explains their greater mechanical properties. A comparison between the three chamottes shows that chamotte KK undergoes less shrinkage, indicating its evolution during the second firing cycle and cristobalite crystallization with volume expansion.

For the KK chamotte, the porosity of the produced specimens for all binders and for all temperatures varies between 14% and 25%. The compressive strength is lower (10–26 MPa) at 1400°C and 1450°C for all binders and higher (35–50 MPa) at 1500°C. The maximum compressive strength is achieved with the mixture using 30% clay as a binder. At this clay content, the compressive strength varies from 40 to 50 MPa. This is explained by the difference in fine and 'reactive' clay minerals contents, which promotes densification and lower open porosity (Kumar *et al.*, 1999; Maiti *et al.*, 2004, Haddar *et al.*, 2020; Pountouenchi, 2020). The Al$_2$O$_3$ content in the clays, especially in MY3, exhibited a positive correlation with the compressive strength of refractory bricks. This observation is consistent with previous work demonstrating that a higher Al$_2$O$_3$ content promotes the formation of the crystalline mullite phase, contributing to the reinforcement of refractory materials' structure (Kumar *et al.*, 1999; Maiti *et al.*, 2004). However, the flux content in the clays also played a crucial role. Clays MY3 and KG, having higher flux contents, displayed higher densification and lower porosity. This resulted in increased mechanical strength, as the presence of flux promoted melting at lower firing temperatures, facilitating sintering and material consolidation (Njoya *et al.*, 2017; Pountouenchi *et al.*, 2018; Pountouenchi, 2020). The products produced with chamotte KG (CHM KG) and chamotte MY3 (CHM MY3) display better characteristics than the materials made from chamotte KK (CHM KK). For the KG chamotte (Fig. 7), open porosity varied from 18% to 22%, bulk density varied from 2.0 to 2.1 g cm^{-3} and compressive strength varied from 20 to 50 MPa, and for the MY3 chamotte (Fig. 8), open porosity varied from 16% to 23%, bulk density varied from 2.0 to 2.2 g cm^{-3} and compressive strength varied from 20 to 73 MPa amongst the sintered products. These characteristics are similar to those found in the literature and correspond to refractory bricks with low Al$_2$O$_3$ contents (group LF10; 10% < Al$_2$O$_3$ < 30%) according to ISO 10081-1 norm (Norme ISO 10081-1, 2003; Pountouenchi *et al.*, 2018).
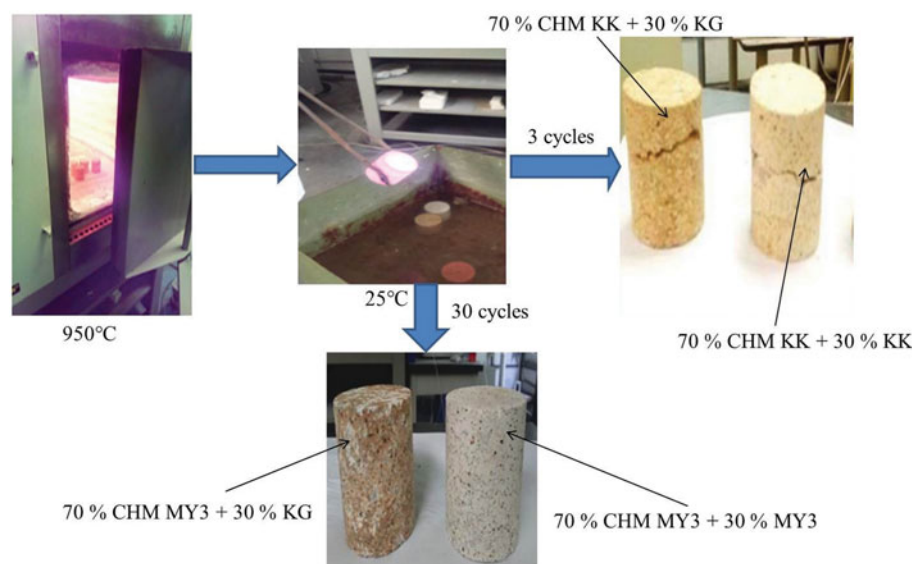
Figure 10. Thermal shock and its effect on the various samples.

Refractoriness under load

Figure 9 presents some examples of the evaluation of refractoriness. The measured temperatures T_0, $T_{0.5}$, T_1, T_2 and T_5, which correspond respectively to 0%, 0.5%, 1%, 2% and 5% levels of collapsing, are listed in Table 5. T_0 is the temperature corresponding to the onset of brick shrinkage and $T_{0.5}$ is a reference temperature generally presented in datasets. All materials have $T_{0.5}$ ranging between 1330°C and 1416°C. The only material with KK bonding (i.e. chamotte KK) exhibits a low $T_{0.5}$ value (1333°C). Materials with MY3 bonding exhibit $T_{0.5}$ values of 1335°C (chamotte KK), 1360°C (chamotte KG) and the highest $T_{0.5}$ value 1416°C for the MY3 chamotte. Materials with KG bonding exhibit $T_{0.5}$ values of 1390°C (chamotte KK) and 1395°C (chamotte MY3), which are close the highest observed $T_{0.5}$ value (i.e. that of the CHM MY3 material). The value for chamotte KG is 1350°C, which is lower than the other values observed.

The results of the refractoriness under load tests demonstrate that MY3 and KG clays have higher values of refractoriness under load temperature ($T_{0.5}$) compared to KK clay. This relationship suggests that clays with a higher $T_{0.5}$ value, such as MY3 and KG (Table 1), possess a superior ability to maintain their structure and mechanical strength under high thermal loading conditions. This conclusion is in accordance with other studies that have shown Al_2O_3 to be a key element in enhancing the thermomechanical resistance of refractory materials (Pountouenchi *et al.*, 2018; Muhammed *et al.*, 2019; Vasić *et al.*, 2023).

In summary, the composition of clays, with variations in Al_2O_3 and flux contents, significantly influenced the refractory properties under load. A higher Al_2O_3 content promotes the formation of reinforcing crystalline phases, whereas the presence of flux contributes to material densification and consolidation. These findings improve our understanding of the underlying mechanisms determining the mechanical performance of refractory bricks and provide a solid foundation for formulating optimized refractory materials (Kumar *et al.*, 1999; Muhammed *et al.*, 2019; Pountouenchi, 2020; Sawadogo *et al.*, 2020; Vasić *et al.*, 2023).

Thermal shock

The thermal shock tests were implemented according to the EN 993-8 standard (water quenching; Norme NBN EN 993-8, 1997) for products containing 30% clay binders (Fig. 10). Thermal fatigue was estimated by measuring density, open porosity and the compressive strength of materials that resisted more than 30 cycles of firing (Table 6).

The refractories produced from chamottes KK and KG do not resist the thermal shock tests, with some damage occurring during the first and second cycles and cracks appearing from the third cycle (Fig. 10). This can be explained by the very high cristobalite content (Table 4) and its α–β transformation associated with 2–3% volume changes (Routschka, 2004; Pountouenchi, 2020). The refractories produced from chamotte MY3 resisted thermal shock because they have the lowest cristobalite content and a high mullite content. The latter samples developed cracks from the 25th cycle. A reduction of 25% in compressive strength is observed for the MY3 bonding phase and a reduction of 31% is observed for the KG bonding phase.

Conclusions

This study assessed kaolins from western Cameroon as raw materials for dense refractory brick-making. The rationale behind the study was to enable local production of refractories to reduce the cost of access to refractories in Cameroon. Three kaolin samples coded MY3, KG and KK from Cameroon were selected based to their reserves and accessibility. Each clay was used for a chamotte and as a binding phase. The characteristics of the obtained products are coherent with the classification of the LF10 group (Norme ISO 10081-1, 2003). It was then concluded that refractory bricks of the LF10 group can be prepared from the three clay samples. The sintering behaviour, final mineralogical composition and mechanical properties were associated with the Al_2O_3, SiO_2 and fluxing agent contents in the starting clay compositions. Compared to the other materials, refractory products using chamotte from MY3 clay exhibit greater resistance to thermal shock. This difference can be explained by its relative low cristobalite content. The flux content influences the firing temperature for chamottes. Hence, to reduce energy consumption in this process, enrichment of flux in the raw clay prior to chamotte-making should be considered.

Some of the main results arising from this work need further study to improve our understanding of refractory-making. The density of the obtained refractories was related to porosity development, whereas deeper analysis of the relation between porosity and product density might be needed regarding the particle-size distribution of the starting sample.

The results of this study are of interest regarding developing a refractory production plan using the tested clays. This will add value to Cameroonian clayey materials and also contribute to reducing the environmental impacts associated with the transportation of imported materials.

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