Porous refractory ceramics using agrowastes and some kaolinitic clays

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ABSTRACT

In the purpose of making low cost porous refractories bricks, two kaolin named MY3 and KG from the locality of Foumban in Cameroon are used together with a rice husks (Br) and coffee residues (Rc). The formulations made of mixture of chamotte, raw clay as a binder and agricultural wastes in varying proportions, are fired at 1200 °C for 4 h. The addition of chamotte from 10 to 60 % in the studied clays, contributes to a reduction of firing shrinkage. The mineralogy of the products is made of mullite (34–38 %) which is associated to cristobalite (10–19 %), quartz (14–20 %) and amorphous phase (23–39 %). The addition of 10–20 % of agrowastes causes a reduction in the apparent density of 13 % and an increase in the open porosity of 14 %.

1. Introduction

Porous refractories are materials that can be used at very high temperatures, while maintaining their physical forms and chemical identities [1–5]. They are important in industry because they are involved in almost all processes that use heat [1–5]. Our country has several deposits of kaolinitic clay [6–13]. In addition to their ease of extraction, the kaolinitic clays of Mayouom and Koutaba contain relatively high percentage of alumina and a low percentage of impurities [6,7,9]. Although the raw materials are widely available, local needs for porous ceramics are almost entirely covered by imports [6,14,15]. They are obtained by mixing grog, clays as a binding phase and pore-forming agents among which the residues of agro-industrial activity [14–17]. Moreover, the agro-industrial activities are developing considerably and constitute a real source of income for many countries. This development is inevitably followed by a significant increase in discharges, among which some are pollutants. In the development plan implemented by the Cameroonian authorities, the optimal use of natural mineral resources as well as the valorization of agricultural waste plays a significant role in the development of the country [14,15]. Our work is in line with this approach: it aims to optimize the exploitation of clay deposits in the region by defining for each of them their potential for the realization of finished products with high added value. Nowadays, mankind uses many mineral resources for the manufacture of goods. Most natural resources are exhaustible and non-renewable. However, a rare exception is rice and coffee waste. The problem of reuse of these wastes also remains unsolved. The objective of our work is the valorization of rice husks and coffee residues with kaolinitic clay materials of Cameroon for the production of porous ceramic products.

2. Raw materials and methods

2.1. Raw materials

Clay materials used in this study were collected from two deposits located in the Western Cameroon: Mayouom for sample MY3 and Koutaba for samples KG. The GPS coordinates for the samples collection are 5°35′20″ N and 10°41′26″ E for KG and 5°51′N and 10°59′ E for MY3. MY3 clay material is of residual origin and is issued from the alteration of granites and mylonites which are predominant rocks in the region [6,8,9,12] while KG is a hydromorphic clay. All these clay samples were described in previous study [6,10,15] and their chemicals and mineralogical composition are given in Table 1.

Rice husk (Br) and coffee residues (Rc) are organic wastes collected

in Koutaba and Kouoptamo respectively.

2.2. Methods

The raw clay were been finely ground and particle sizes obtained were below 100 μm. Br and Rc were characterized by the X-ray Fluorescence and thermo-gravimetric analysis.

The chamotte was prepared from MY3 clay and is noted CHM. It was pressed in a $7 \times 7 \times 7$ cm^3 steel mold at 100 MPa with a uniaxial hydraulic press and sintered at 1300 °C for 4 h. After cooling, the grog is manually ground in a ceramic mortar and sieved at 100 μm.

Different mixing were realized as follows:

a. Mixture chamotte/clays

The chamotte (CHM) is mixed with each of these clays in the proportions of 10–60 wt %.

b. Mixture chamotte/clays/Br or Rc

The formulations are made by adding 10 to 20 wt % of Br or Rc in the 50 % CHM MY3 + 50 % MY3; 60 % CHM MY3 + 40 % MY3; 50 % CHM MY3 + 50 % KG and 50 % CHM MY3 + 50 % KG mixtures.

To each mixture, 5 % by mass of water was added and the entire mixture was manually mixed. The resulting pastes were pressed at 42 MPa in 3 cm × 3 cm cylindrical mold using a uniaxial hydraulic press. Later on, the products obtained were sintered at 1200 °C during 4 h in an electric furnace with a heating rate of 3 °C/min. After firing, the final products were characterized by evaluating the followings: firing shrinkage (R), bulk density (ρa), open porosity (π), compressive strength (σc), X-ray diffraction and scanning Electron Microscopy.

The chemical analyses of major elements were carried out by X-ray Fluorescence Spectrometry with an ARL PERFORM-X 4200 XRF spectrometer with a standard error < 1% on samples sintered at 1050 °C.

Thermo-Gravimetric Analysis (TGA) were carried out with a SETARAM Cahn apparatus, operating under the following conditions: heating rate of 5 °C/min, in an air atmosphere from ambient temperature to 1000 °C. The sample weight used was 1 mg.

Firing shrinkage was assessed by comparing the height of the samples before and after thermal treatment.

Bulk density and open porosity are determined using Micromeritic's AccuPyc II 1340 pycnometer. The gas pycnometer is used to measure the solid density using the pressure difference between an empty sample cell and the cell including sample. The increase in pressure is directly related to the volume of gas displaced. To obtain the true density, Helium is used as the analysis gas to fill all open voids. Using solid displacement technique, the structural volume (Vs) was measured. In this technique, a cylinder containing treated glass microsphere that behave as fluid was used. The solid is place in the cylinder and a piston under a given force compact the medium. The distance travelled by the cylinder in presence and in the absence of the solid are used to access the solid volume (Ve) knowing the cylinder volume. The solid volume (Ve) and the structural volume (Vs) are used to compute the connected porosity and density:

- Open porosity: $\pi = \frac{V_e - V_s}{V_e} \times 100$.

- Bulk density: $\rho = \frac{m}{V_e}$, where m is the weight of the tested sample and ρ = the true density from helium displacement using gas pycnometry.

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 parallelepipedic samples (114 × 114 × 64 mm) according to EN 993 standard [18].

The X-ray pattern were recorded using a Philips PW 1729 diffractometer operating at 40 kV- 40 mA and using a monochromatic CuKα radiation. The X-ray patterns were treated with *CRYSTAL* software for mineral identification and semi-quantitative analysis through the Reference Intensity Ratio (RIR) method. The following formula is used by CRYSTAL software to determine the percentage Xp of each phase [19, 20]:

$$X_p = \frac{I_{phase} \ x \ RIR_{phase}}{\sum\limits_{i=1}^{n} I_i \ x \ RIR_i} \ x \ 100$$

With I_{phase} = Diffraction intensity of used reflection from database patterns; RIR_{phase} is the RIR value of the phase; n is the total number of phase; I_i and RIR_i are respectively the diffraction intensity and RIR value of phase i.

The Amorphous phase was quantified by adding a known amount of internal amorphous standard to the sample, then the quantity is deduce from sum of Xp = 1.

Scanning Electron Microscopy (SEM) was used to observe their morphology and size; the observations were carried out on an ESEM Philips XL-30 device at the University of Liege. The images were obtained by a secondary electron detector after metallization with gold powder by plasma spraying for an accelerating voltage scan.

It is important to mention that a ceramic product is qualified as thermal insulating according to the ISO 2245 standard [21] if it has the following characteristics: firing shrinkage < 2 %; 0.4 < bulk density <1.6 g/cm^3 and open porosity >45 % by volume.

3. Results and discussion

3.1. Characterization of raw materials

3.1.1. Chemical analysis

The chemical compositions of the rice husk and coffee residue are listed in Table 2.

The results show that the mineral phase of rice husk consists of silica (29.6 %), while coffee residues contain 4.3 % potassium oxide as main mineral residue. The high content of silica (29.6 %) in rice husk is in line with the work of other researchers who found that, rice husk ash is exclusively composed of silica [22,23]. This silica could play the role of the inert grains, i.e. decrease the shaping water, and reduce the drying shrinkage through the facilitation of excess fluid evacuation out of the ceramic body during drying [24,25]. On the other hand, the K_2O content of about 4.3 % in coffee residues may induced vitrification during firing Which may lead to increase densification and at the same time reduce porosity through pore enclosure arising from the liquid phase that interconnect the grain [24,26]. The loss on ignition is high for both material: 68.6 % for Br and 90.6 % for Rc, this high loss are of interest for

Table 1
Chemical and mineralogical composition of the clays [6].

	SiO$_2$	Chemical composition (%)						Mineralogy composition (%)
		Al$_2$O$_3$	Fe$_2$O$_3$	TiO$_2$	Total Flux	LOI	sum	
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	10.7	99.9	Kaolinite (57), montmorillonite (15), goethite (3), quartz (24), anatase (1)

Note: Total Flux: sum of MnO, MgO, CaO, Na$_2$O and K$_2$O.

Table 2

Chemical composition (wt. %) of the rice husk and coffee residue.

Oxydes majeurs	SiO$_2$	Al$_2$O$_3$	Fe$_2$O$_3$	MgO	CaO	K$_2$O	TiO$_2$	SO$_3$	P$_2$O$_5$	LOI	Total
Br	29.6	0.1	0.1	0.2	0.2	0.7	0.8	< Id	0.4	68.6	100.5
Rc	0.5	0.3	0.1	0.2	0.5	4.3	0.02	0.2	0.4	90.6	97.2

LOI: Loss on Ignition.

the use of this waste as porogenic agents.

3.1.2. Thermogravimetric (TG) analysis of rice husk and coffee residues

Fig. 1 shows the thermogravimetric curves and their derivative DTG for the rice husk and the coffee residues used.

The TGA curves show a large mass loss domain from about 100 °C up to 600 °C (an even beyond for Rc). In this mass loss domain, at least two slopes are easily observable. The TGA curves derivation (DTG) is used to find mean temperature in each domain. These curves allow to observed a first mean at around 110 °C, a second at around 250 °C and a third one around 450 °C. The first at 110 °C corresponds to the dehydration of hydroxyl groups in the organic constituents [13] with a loss of mass of 5.9 % for Br and 5.6 % for Rc. The second endothermic peak at 260 °C is associated to the thermal decomposition of non-crystalline region of hemicellulose and cellulose as a major organic component in the rice husk and coffee residues [27]. The third temperature (around 450 °C) is due to the ultimate decomposition of cellulose residue due to crystalline region of this material associated to lignin decomposition. Several researchers [28,29] reported that hemicellulose decomposes mainly at 150–350 °C which is the least stable component of rice husk and cellulose decomposes between 275 and 350 °C. The reported decomposition temperature here (250 °C–450 °C) is in line with a composition mainly made of cellulose and hemicellulose in both Br and Rc. The third mean decomposition temperature given that lignin is thermally more stable and under goes gradual decomposition between 370 and 600 °C [30]. The mass percent losses associated are significant 57.7 % for Br and 84.4 % for Rc. These mass losses are coherent with the loss on ignition from chemical analysis (Table 2).

3.2. Product characteristics

3.2.1. Physical characterization of products from clay/mixture: choice of the optimal clay/chamotte mixture to be used for porous refractory making

Fig. 2 shows the evolution of firing shrinkage, bulk density and open porosity as a function of the amount of chamotte added, for a firing at 1200 °C.

From this result, it is found that the firing shrinkage and bulk density decrease (Fig. 2a b) with the amount of chamotte while the open porosity increases (Fig. 2c). At 0 % chamotte, the densification of KG clay is higher than that of MY3. From a previous study [6] it is reported

that MY3 and KG exhibiting similar Al$_2$O$_3$ and SiO$_2$ contents, hence the densification difference may be associated to the difference in iron and titanium oxides content. This content is greater for KG (4.5 % as compare to MY3 1.8 %) and probably contribute to densification through the promotion of vitrification which promotes increase density and porosity lowering. The finer particle size for KG (65 % of < 2 μm) will also contribute to improve grain contact and hence the densification as compare to MY3 where the < 2 μm fraction is just 23 %. Regarding the Shrinkage, the fact that KG shrinkage is higher than that of MY3 is due to the difference in vitreous phase formation in both materials which is associated to the difference in fluxing oxide content. However, it is noted that this gap decreases with the addition of chamotte which act as skeleton controller as it will not undergo sintering modification during the firing of the refractory body. The densification of these materials results from the inert grains action of the chamotte, but also from the effect of the crystallization of the mullite and the transformation of quartz into cristobalite [31–33]. At 60 % chamotte, the physical properties of the two clays are similar. This behavior can be explained by the stability of the chamotte, at the considered firing temperature, whose only action is that of inert grains [1,2,31–41]. With regard to these behaviors, the mixtures of (60 % CHM + 40 % clay binders) were retained for the continuation of the study.

3.2.2. Characterization of porous products from clay/chamotte/organic waste mixtures

3.2.2.1. Physical characterization. They are obtained by substituting part of the 60 % CHM +40 % (KK, MY3) mixture by the Br and Rc samples in the proportions 10; 15 and 20 %. Table 3 shows the physical and mechanical characteristics of the products obtained after firing at 1200 °C.

The results show that firing shrinkage and open porosity increase with the added amount of Br or Rc, while apparent density and compressive strength decrease. Firing shrinkage is slightly more pronounced when MY3 clay is substituted by KG and Br by Rc. This is due to the fact that KG clay promote a better densification of the ceramic body than MY3 clay due to its finer particle size, which was previously highlighted in our work [6,15]. Indeed, firing shrinkage and densification are largely controlled by the most active fine particles and thus the total clay content [6], but also by "impurities" such as iron and titanium
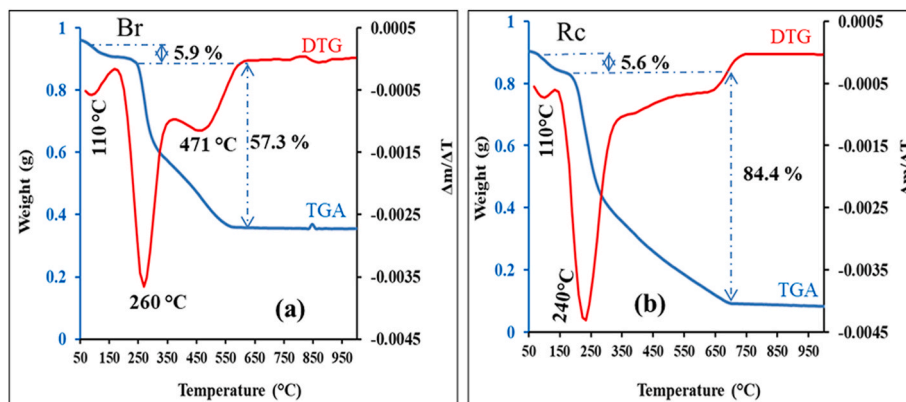

Fig. 1. Thermogravimetric analysis curves (ATG/DTG) of the agrowastes used: (a) rice husk (b) the coffee residues.
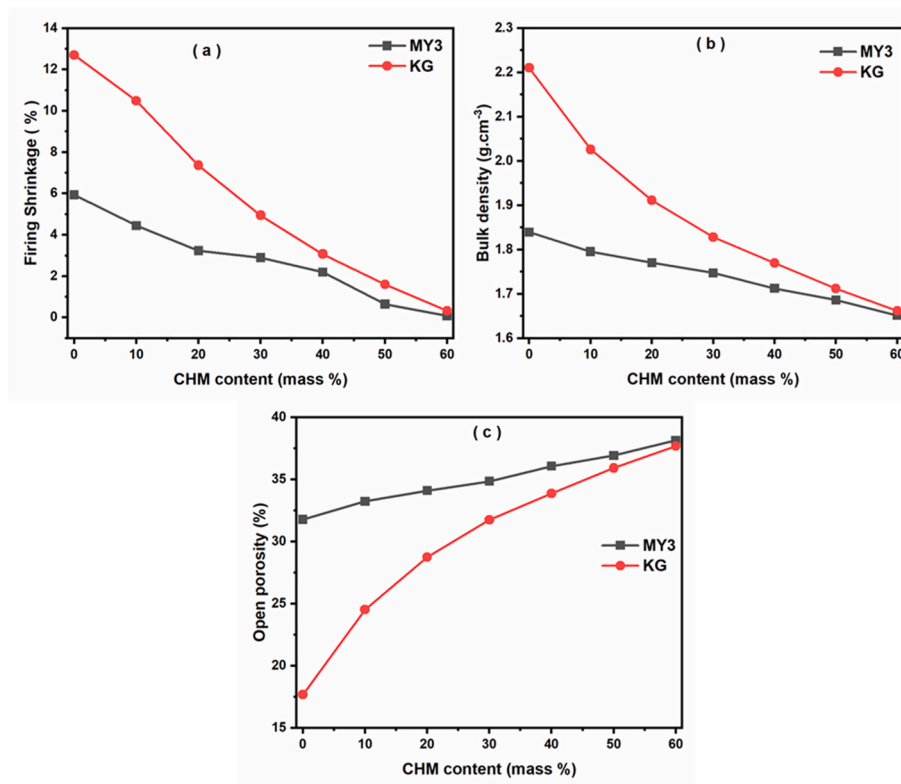
Fig. 2. Evolution of firing shrinkage (a), bulk density (b) and open porosity (c) as a function as a function of the amount of grog added.

Table 3

Characteristics of CHM/clays/(Br and Rc) mixtures at 1200 °C (R: linear shrinkage, ρ: bulk density, π: open porosity and σc: compressive strength).
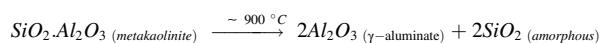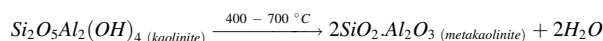
Formulations	R (%)	ρ (g/cm³)	π (%)	σc (MPa)
90 % (60 % CHM + 40 % MY3) + 10 % Br	0.89	1.52	42.80	4.95
85 % (60 % CHM + 40 % MY3) + 15 % Br	1.15	1.39	47.52	2.35
80 % (60 % CHM + 40 % MY3) + 20 % Br	1.61	1.29	50.84	/
90 % (60 % CHM + 40 % KG) + 10 % Br	1.16	1.52	42.68	4.94
85 % (60 % CHM + 40 % KG) + 15 % Br	1.28	1.38	47.59	2.42
80 % (60 % CHM + 40 % KG) + 20 % Br	1.63	1.31	50.08	/
90 % (60 % CHM + 40 % MY3) + 10 % Rc	0.80	1.49	43.73	4.76
85 % (60 % CHM + 40 % MY3) + 15 % Rc	1.01	1.39	47.42	2.81
80 % (60 % CHM + 40 % MY3) + 20 % Rc	1.23	1,29	50.99	/
90 % (60 % CHM + 40 % KG) + 10 % Rc	0.86	1.49	43.34	4.95
85 % (60 % CHM + 40 % KG) + 15 % Rc	1.04	1.39	47.06	2.92
80 % (60 % CHM + 40 % KG) + 20 % Rc	1.51	1.30	50.23	/

oxides which are in higher amounts in KG compared to MY3 and may promote the appearance of the vitrified phase [2,6,15,41]. It's important to mention that the melting temperature and the amount of liquid phase can be linked to the nature and quantity of impurities which induce a decrease in the melting temperature and a large amount of liquid phase. The addition of 10–20 % rice husk in various compositions causes a decrease in apparent density of the order of 13 % and an increase in open porosity by volume of 14 %. At 15 % Br or Rc, firing shrinkage is less than 2 %, apparent density is between 1.3 and 1.5 g/cm³, open porosity in volume is greater than 45 % and compressive strength is between 2 and 5 MPa. These characteristics are similar to those of insulating refractory products made with low alumina content according to ISO

standard 2245, 1984. If the 20 % rice husk compositions have better insulating characteristics than the 15 % ones, they nevertheless exhibit some cracking defects. Thus, to avoid these phenomena, we have considered, for subsequent analyses, the compositions with 15 % rice husk as the optimal formulation.

3.2.2.2. Mineralogical analysis using powder X-ray diffraction. The XRD patterns of the processed products are given in Fig. 3 and their semi-quantitative analysis is presented in Table 4.

The results show that the processed products consist mainly of mullite, quartz and cristobalite as newly formed mineral phases and an amorphous phase. Mullite is formed as a result of reactions decomposition of phyllosilicates (mainly kaolinite) during sintering. The equations below illustrate the formation steps of these minerals [6,33–35].

$$Si_2O_5Al_2(OH)_4 \text{ }_{(kaolinite)} \xrightarrow{400-700 \text{ }°C} 2SiO_2.Al_2O_3 \text{ }_{(metakaolinite)} + 2H_2O$$

$$SiO_2.Al_2O_3 \text{ }_{(metakaolinite)} \xrightarrow{\sim 900 \text{ }°C} 2Al_2O_3 \text{ }_{(\gamma-aluminate)} + 2SiO_2 \text{ }_{(amorphous)}$$

$$3Al_2O_3 \text{ }_{(\gamma-aluminate)} + 4SiO_2 \text{ }_{(amorphous)} \xrightarrow{\sim 1000 \text{ }°C} Si_2Al_6O_{13} \text{ }_{(mullite)} + 2SiO_2 \text{ }_{(amorphous)}$$

$$Si_2Al_6O_{13} \text{ }_{(mullite)} + 4SiO_2 \text{ }_{(amorphous)} \xrightarrow{\sim 1150 \text{ }°C} Si_2Al_6O_{13} \text{ }_{(mullite)} + 4SiO_2 \text{ }_{(cristobalite)}$$

Cristobalite result from high temperature conversion of silica from the raw material as well as silica from kaolinite conversion into mullite.

However, the intense mullite diffraction peaks observed on the X-ray diffractograms of the fired products is justified by phyllosilicate contents of the raw clays which are >70 % for both KG and MY3. The semi-quantitative analysis of the products mineral composition confirmed this predominance with mullite content of 34–38 %. It is observed that the mullite development is lower in bricks using Rc as compared to Br of
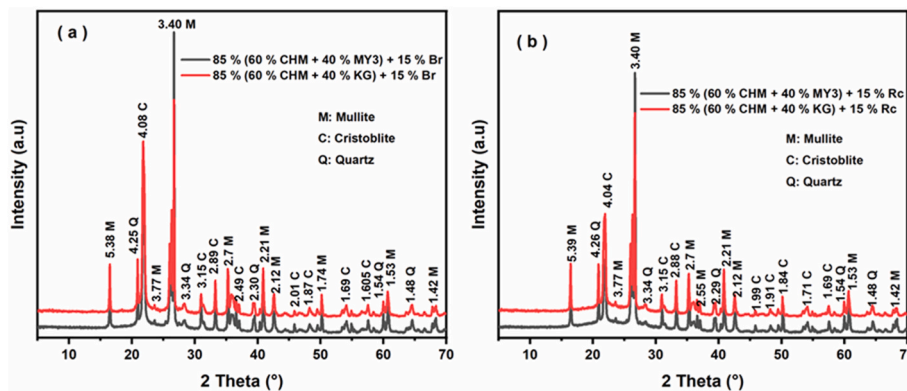
Fig. 3. X-ray patterns of the optimal compositions of the elaborated insulators: (a) using rice husk (b) using coffee residues.

Table 4
Semi-quantitative analysis of the processed products (by wt. %).

Cristalline phases	Amorphous phases	Mullite	Cristobalite	Quartz
85 % (60 % CHM + 40 % MY3) + 15 % Br	23	38	19	20
85 % (60 % CHM + 40 % KG) + 15 % Br	24	38	22	16
85 % (60 % CHM + 40 % MY3) + 15 % Rc	39	33	10	18
85 % (60 % CHM + 40 % KG) + 15 % Rc	39	34	13	14

about 3 %. The same observation is made for cristoballite and amorphous phase for respective difference of 4 % and 3 %. These difference can be attributed the difference in the amount of vitreous phase formation due to difference in flux content arriving from the mixture with agrowaste. In such a consideration, the difference in K_2O between Br and Rc (Table 2) can reasonably account for this although one may considered the mixing inhomogeneity. The formation of more vitreous phase when Rc is used may embedded the crystalline mullite, quartz or cristoballite leading to their diffraction peaks lowering.

3.2.2.3. Scanning electron microscopy of processed products. Fig. 4 shows the morphology of the products elaborated at 1200 °C. The microstructure of the processed products does not show any significant difference for the same amount of binder phase after sintering at 1200 °C for 4 h. However, the existence of mullite crystals with a random
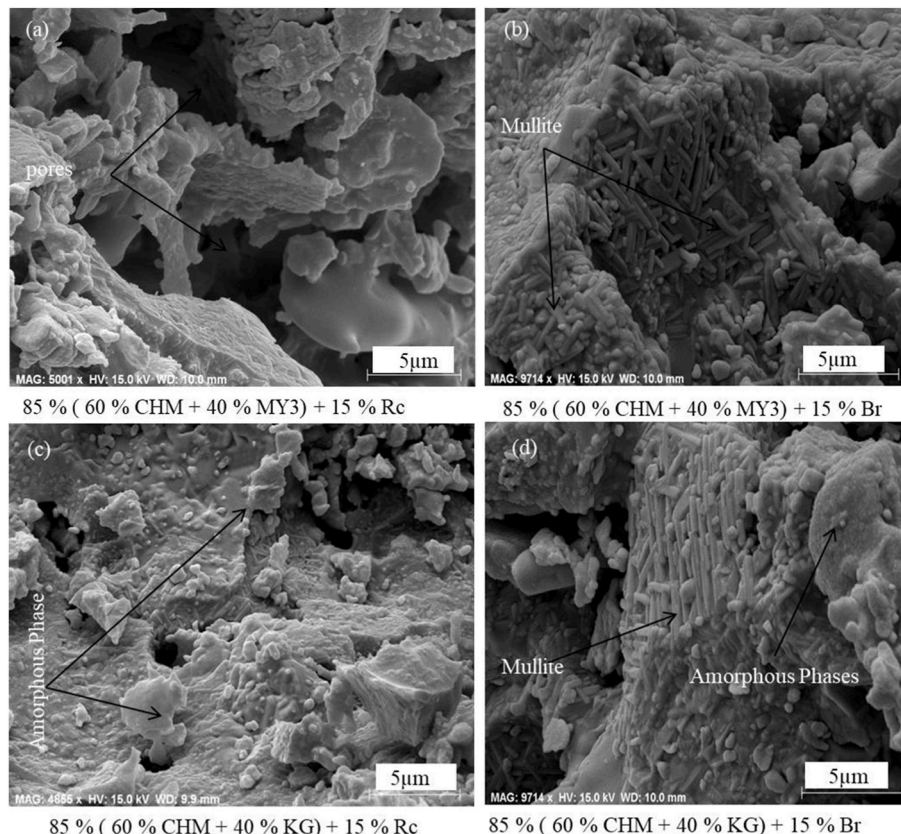

Fig. 4. Scanning electron microscopy of insulating refractory products.

distribution is observed. The structure of the refractory products shows the presence of larger pore sizes. These larger pores are in line with the departure of the organics material upon firing. Also, some void surfaces are observable and may be associated to some grains pull out at the surface during the sample preparation. For all the samples a vitrified structure is observed. This vitrificate surface is due to the high temperature treatment rather than flux content which is low. At high temperature even silica oxide fused and contributes to the vitrification. The rod-shaped mullite particles observed for the products with binder phase KG appear larger than those observed for MY3. This difference is probably associated to the difference in vitrification that enhances the observation of mullite grain. Also, the observed rods like structure are partially due to iron oxide (hematite) which content is greater in KG than in MY3.

4. Conclusion

This work is a contribution to the elaboration of ceramics from Cameroonian kaolinitic clays and agricultural waste. The results obtained show that, the physical and mechanical properties of the elaborated products can be related to the quantity of clays and impurities. The addition of chamotte from 10 to 60 % in KG and MY3 clays leads to a decrease in firing shrinkage and apparent density inducing an increase in open porosity. The mixture of 60 % CHM +40 % clays has shrinkage close to zero and is suitable for the fabrication of the targeted materials. The incorporation of Br and Rc in this mixture indicates, the elaborated products have the optimal composition [85 % (60 % CHM + 40 % (MY3, KG) + 15 % (Br, Rc)] at 1200 °C. The mineralogy by X-ray diffraction of these products indicates a predominance of mullite to which are associated cristobalite, quartz and an amorphous phase. They have the following characteristics: firing shrinkage < 2 %; bulk density 1.4 g/cm^3; open porosity 47 % by volume and compressive strength 2.8 MPa. These characteristics may be comparable to those of shaped insulating refractory products with low alumina content according to ISO 2245. The main applications of these products can be thermal insulation in the manufacture of kilns for production of pottery, clay bricks.

Declaration of competing interest

The authors have declared no conflict of interest.

Acknowledgements

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