

## Characterisation of porcelain compositions using two china clays from Cameroon

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### Abstract

Mayouom and Ntamuka china clays, from Cameroon were used to produce porcelain bodies. Two soft porcelain formulations: PSI (with Mayouom) and PSII (with Ntamuka) and one hard porcelain, PH (with both Mayouom and Ntamuka) were prepared.

The maximum density and flexural resistance for these formulations were obtained at 1200 °C for PSI, 1225 °C for PSII and 1350 °C for PH. Their properties and values were, respectively, density (2.42, 2.58 and 2.59 g/cm<sup>3</sup>), water absorption (0.15, 0.15 and 0.02%), porosity (4.3, 5.3 and 4.2%) and flexural strength (148, 148 and 160 MPa). In addition to varying amounts of liquid phase, the soft porcelain formulations contained mullite and quartz crystals while the hard porcelains contained quartz and more intense mullite peaks. At 1250 °C PSI and PSII presented a self-glazing phenomenon which gave significant brightness and high aesthetic quality. PH with lower alkali (especially Na<sup>+</sup>) did not self-glaze even at 1400 °C. Considering the soft porcelain formulations, the higher amounts of TiO<sub>2</sub> (0.83%) and Fe<sub>2</sub>O<sub>3</sub> (0.31%) in PSI (with Mayoum clay) resulted in a higher sintering effect at lower temperatures, giving lower water absorption and higher resistance. Both Mayouom and Ntamuka china clays were found to be suitable raw materials for the production of porcelain stoneware tiles. Ntamuka could also be used for the manufacture of optimal quality whitewares.

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**Keywords:** Soft–hard porcelain; China clay; Characterisation; Cameroon; Self-glazing; Mullite

### 1. Introduction

Traditional ceramic formulations consist of at least three components that play the three fundamental roles for optimum processing, and hence performance of the final products, kaolin or kaolinitic clay for plasticity, feldspar for fluxing and silica as filler for the structure. Formulations of triaxial porcelain (SiO<sub>2</sub>–Al<sub>2</sub>O<sub>3</sub>–KNaO) usually involve 25 wt.% of plastic component, 25 wt.% silica and 50 wt.% feldspar (generally sodium feldspar) for soft porcelain and 50 wt.% of clay, 25 wt.% silica and 25 wt.% feldspar (generally potassium feldspar) for hard porcelain [1–5]. After suitable production processing, these formulations should lead to high strength performing porcelains with high frost and chemical resistances and low

porosity. The typical microstructure of porcelain ceramics consist of a glassy matrix that contains mullite and dispersed and/or partially undissolved quartz particles.

Commercial feldspar and silica for porcelain contain a maximum of 0.3 wt.% iron oxide as the major impurity. Mullite (3Al<sub>2</sub>O<sub>3</sub>·2SiO<sub>2</sub>) is considered the principal crystalline phase in porcelains and responsible for their microstructure and mechanical properties. The formation of mullite depends mainly on the type and proportion of kaolin used. Pure and well-crystallized kaolinite gives good results of mullitisation at above 1000 °C, while disordered kaolinite forms mullite at higher temperatures, above 1200 °C [6,7]. There is competition between the formation of mullite and crystallisation of the amorphous silica present in the matrix during firing. Moreover, the proportion and nature of the quartz used in the composition of porcelain batch influences the proportion of un-reacted residual quartz during firing. Quartz possesses a higher coefficient of thermal expansion as compared to that of the

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surrounding glassy phase, hence giving rise to thermal stresses which remarkably affect the strength of porcelain [3,8].

Different authors have demonstrated that mullitisation can be increased by catalytic ions such as  $\text{Fe}^{3+}$  and  $\text{Ti}^{4+}$  [9,10]. These metallic ions help in mullite formation by replacing the  $\text{Al}^{3+}$  ions in the glass structure during firing. The presence of small amounts of  $\text{Fe}^{3+}$  and  $\text{Ti}^{4+}$  in kaolin modifies the chemical composition of the ceramic bodies and therefore the sintering behaviour which in the case of porcelain is characterised by mullitisation. The chemical composition of kaolin also affects the rheological properties of the ceramic paste which play an important role in slip casting, the most frequent technique used for the manufacture of whiteware porcelain articles [11].

The phenomenon of mullitisation is also influenced by the grade of feldspar which particularly affects the formation of secondary mullite. The quality of feldspar is determined by the amount of orthoclase and albite, respectively in the potassium and sodium feldspar, the most commonly used fluxes in porcelain composition. Nowadays, mixed feldspars are considered suitable fluxing agents for porcelain manufacture since they develop a very viscous liquid phase that embeds the new forming crystals and part of the residual crystals present in the microstructure, enhancing the densification process [12,13].

In this investigation, a chemical approach was applied on soft and hard porcelain compositions with mixed feldspar as fluxing agent to study the behaviour of two china clays from Cameroon. The physico-chemical and mechanical properties of the porcelains obtained were studied. X-ray diffraction (XRD) was used to characterise the mineralogical phases and scanning electron microscopy (SEM) to evaluate the microstructure.

## 2. Materials and experimental procedures

### 2.1. Location of the two china clays

The two china clays are from Mayouom (CH1) and Ntamuka (CH2), both situated on the highlands in the Western region of Cameroon. Mayouom is a village situated at about 80 km from Foumban, the main city of Noun Division (West Province) and Ntamuka is in Bafut 4 km from Bamenda, the main city of Mezam Division (North-West Province).

### 2.2. Raw materials characterization

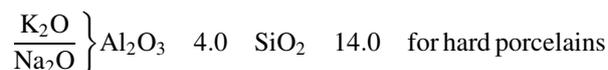
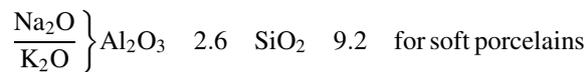
The clays were washed and pulverized into powders ready for batching. Quartz, nepheline and potash feldspar were collected from a ceramic industry in Modena (Italy) and named QUT, NPH and KFD, respectively. These raw materials were procured in ground form with grain size of 100  $\mu\text{m}$  maximum.

Differential thermal analysis (DTA, Netzsch, STA 409) and thermal gravimetric analysis (TGA, Netzsch, STA 409) were carried out using standard procedure with alumina as reference. The chemical composition of the raw materials were performed using inductively coupled plasma-mass spectrometry (ICP-MS) method, those of fired compositions were determined using a software (considering the chemical composition of raw

materials) on the basis of calcined oxides. Mineralogical analyses were carried out with an X-ray powder diffractometer (XRD) (Cu  $\text{K}\alpha$ , Ni-filtered radiation, Phillips Model PW 3710). Particle size distribution was evaluated using Laser granulometer (Masterizer 2004, Malvern Instruments Ltd.).

### 2.3. Porcelain composition: chemical approach

Soft and hard porcelain samples were prepared with the following Seger formulae based on conventional porcelain formulations employing standard raw materials (kaolin, feldspar and quartz) [2]:



The soft porcelain fluxing agent was prepared with a mixture of 12.92% KFD and 86.66% NPH, while that of hard porcelain was 19.28% NPH and 80.71% KFD. As such, the fluxes were of mixed feldspar with the  $\text{K}_2\text{O}/\text{Na}_2\text{O}$  ratio of 0.5 and 4.0, respectively for the soft and hard porcelains under study [12,13]. All compositions were calculated based on the calcined oxides.

### 2.4. Samples preparation

Various batches were dry-milled in a ball mill for 4 h and subsequently wet-milled in a ball mill at 1200 rpm for 40 min with porcelain as grinding media and sodium tripoliphosphate as deflocculant (0.16–0.18% of clay). The suitable water/batch weight ratios were 0.340 for CH1 and 0.304 for CH2. Slurries obtained were of high viscosity and were moulded in open plaster moulds to obtain discs of 40 mm diameter and 5 mm thick for firing and mechanical tests.

The samples were dried at room temperature for 48 h and at 105 °C for 24 h. The samples were fired at different temperatures (1200–1300 °C) and (1300–1400 °C), respectively for soft and hard porcelain compositions in an electric furnace with an average heating rate of 10 °C/min and 60 min of soaking time. The starting temperatures were chosen based on the respective liquidus temperatures of the  $\text{SiO}_2\text{--Al}_2\text{O}_3\text{--Na}_2\text{O}$  and  $\text{SiO}_2\text{--Al}_2\text{O}_3\text{--K}_2\text{O}$  systems.

### 2.5. Characterisation of the samples

To characterise the fired specimens, water absorption, was evaluated by 2 h immersion in boiling water and a further soaking for 24 h in ambient temperature. True density ( $D_T$ ) was determined by helium picnometer and apparent density ( $D_A$ ) by Archimedes method. The porosity ( $P\%$ ) was then calculated using Eq. (1):

$$P(\%) = \left( \frac{D_T - D_A}{D_T} \right) \times 100 \quad (1)$$

Table 1  
Mineralogy and particles size distribution of raw materials

Raw materials	Major phase	Minor phases	Particles >100 $\mu\text{m}$	Particles <40 $\mu\text{m}$	Particles <10 $\mu\text{m}$
CH1	Kaolinite	Illite, quartz, Ilmenite	0	84.97	56.29
CH2	Kaolinite	Quartz	0	92.08	65.52
QUT	Quartz	–			
NPH	Nepheline syenite	–			
KFD	Orthoclase	Quartz			

The microstructure of the fired specimens was studied by SEM (PHILIPS XL40) on freshly fracture polished and gold-coated sections, and backscattered electron images were collected. Energy dispersion X-ray fluorescence spectroscopy, EDS, (X\_EDS INCA, Oxford Inst.) was carried out to identify crystalline phases and their distribution.

XRD analyses were carried out to determine the mineralogical compositions.

Ten discs per composition were used for flexural tests as described in the ASTM standard F394-78 [14–16]. The thickness and the diameter of the specimens were determined using an electronic micrometer. The discs were centred and supported on three steel spheres (2.67 mm diameter) positioned at 120° apart on a circle (9.5 mm diameter), loaded in a material testing machine (MTS, type 810, USA) with a crosshead speed of 3 mm/min.

### 3. Results and discussion

#### 3.1. Raw materials and porcelain compositions

The main phases identified from XRD patterns and particle size distribution of the raw materials are reported in Table 1 and the chemical analyses in Table 2. DTA and TGA of the two china clays are plotted in Fig. 1. The DTA curves show endothermic peaks at 578 and 574 °C and exothermic peaks at 992 and 988 °C for CH1 and CH2, respectively. CH2 presents another exothermic peak at 1280 °C not visible for CH1, probably due to the interaction between kaolinite and others minerals such as illite and ilmenite. The peak at about 1200 °C attributed to a second generation mullite, is usually observed in kaolinite in addition to those at about 573 and 980 °C [4]. This confirms that kaolinite is the major mineral in both china clays. The low amount of alkalis ( $\text{K}_2\text{O} + \text{Na}_2\text{O}$ ), 0.92% in CH1 and 0.1% in CH2, indicates the high degree of kaolinitization in these

Table 2  
Chemical analysis of raw materials

Constituents (wt.%)	CH1	CH2	QUT	NPH	KFD
$\text{SiO}_2$	44.35	61.84	99.06	56.94	68.18
$\text{Al}_2\text{O}_3$	33.87	24.88	0.42	26.32	19.43
$\text{Fe}_2\text{O}_3$	1.67	0.69	0.03	0.11	0.06
CaO	0.06	0.00	0.03	0.42	0.12
MgO	0.19	0.08	0.02	0.05	0.04
$\text{Na}_2\text{O}$	0.10	0.04	0.31	11.37	0.80
$\text{K}_2\text{O}$	0.82	0.06	0.08	4.19	11.13
$\text{TiO}_2$	4.40	0.58	0.02	0.00	0.03
L.O.I	13.90	11.80	0.13	0.59	0.22

two deposits, since kaolin is formed from the alteration of feldspathic rocks [17].

The chemical analyses of the porcelains obtained is presented in Table 3. PSI and PSII are soft porcelains in which CH1 and CH2 are the respective plastic materials employed. PH is a hard porcelain with CH1 and CH2 in equal proportions as plastic agent.

#### 3.2. Vitrification behaviour

The maximum density for the soft porcelains were 2.42 g/cm<sup>3</sup> at 1200 °C and 2.58 g/cm<sup>3</sup> at 1225 °C, respectively for PSI and PSII (Fig. 2). At these temperatures both porcelains had the same water absorption value of 0.15% (Fig. 3) but different porosities 4.23% for PSI and 5.30% PSII (Fig. 4). Both water absorption and porosity reduce with increasing firing tempera-

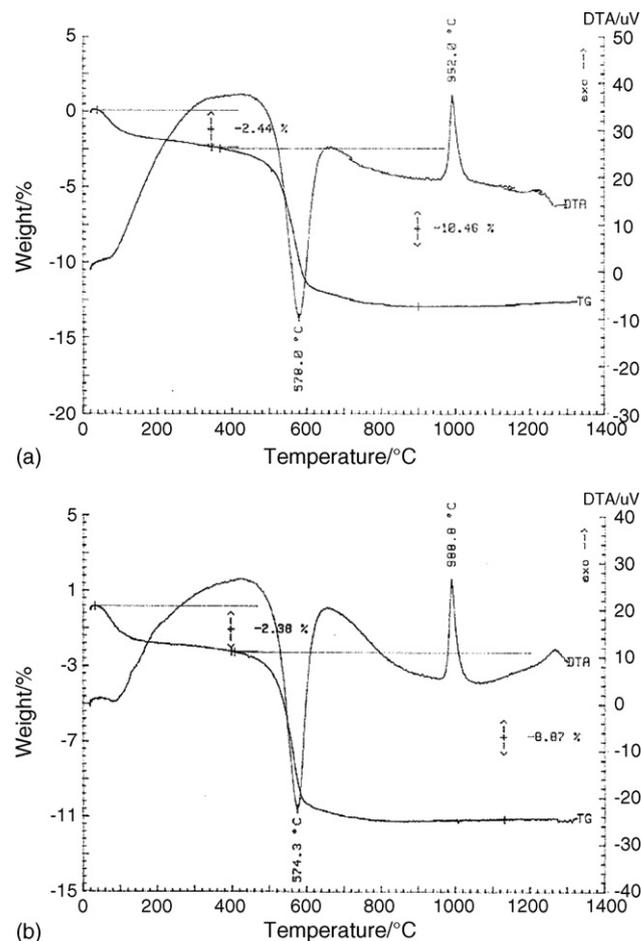


Fig. 1. DTA and TG curves of the two china clays: (a) CH1 and (b) CH2.

Table 3  
Chemical analysis of the fired products

wt.%	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	ZrO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	MnO	MgO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>
PSI	71.66	19.12	0.83	0.00	0.37	0.01	0.06	0.21	5.16	2.58	0.01
PSII	72.37	19.29	0.15	0.00	0.22	0.01	0.05	0.20	5.16	2.56	0.01
PH	69.50	24.05	0.82	0.00	0.34	0.01	0.04	0.23	1.17	4.37	0.01

ture. This is due to the increasing formation of glass phase to the detriment of the crystalline ones. The liquid phase aids in sintering, there by increasing consolidation at higher temperatures. At 1250 °C and above, the soft porcelains developed significant liquid phase that presented a phenomenon of self glazing, exhibiting good brightness and translucency. At almost all temperatures, PSI showed lower water absorption and porosity than PSII, probably due to the presence of high impurities in the Mayouom clay employed. From the chemical compositions the sum of Fe<sub>2</sub>O<sub>3</sub> + TiO<sub>2</sub> is 1.19% for PSI (Mayouom) and 0.38% for PSII (Ntamuka). Many studies have described the influence of these mineralisers in enhancing the process of sintering of ceramic matrix and the formation of

mullite [9,10]. Ti<sup>4+</sup> and Fe<sup>3+</sup> play important role by either substituting Al<sup>3+</sup> or by their integration into the structural interstices of the matrix.

It is well known that the amount of quartz used in the composition of porcelain directly influences the quantity of amorphous phase after firing [6,9]. However, the higher degree of sintering (lower water absorption) in PSI can be attributed to larger amount of quartz (added) and impurity content (Fe<sub>2</sub>O<sub>3</sub> + TiO<sub>2</sub>) compared to PSII.

Hard porcelain (PH) maintained a high density of 2.59 g/cm<sup>3</sup> between 1325 and 1350 °C. Its porosity and water absorption decreased with increasing temperature giving about 4 and 0.05%, respectively at 1350 °C, in conformity with hard porcelain standards [2]. The water absorption becomes zero at 1375 °C and above, with a density of 2.36 g/cm<sup>3</sup> (at 1375 °C), within acceptable limits for hard porcelains. The translucency was the best at the highest temperatures but the samples did not self glaze even at 1400 °C. This could be attributed partly to the lower alkali content 5.54% (Na<sub>2</sub>O + K<sub>2</sub>O) as compared to about 7.76% for soft porcelains and partly to the relatively higher alumina content in the hard porcelains that increases viscosity and refractoriness. With small ionic radii, alkali ions (Na<sup>+</sup> and K<sup>+</sup>) can easily diffuse to the surface at the high temperatures used, especially when forming is by slip casting.

Regarding the fluxes used, the soft porcelains contained more Na<sub>2</sub>O from nepheline syenite than K<sub>2</sub>O from the orthoclase in their compositions, and vice versa for the hard porcelains. The respective ratios of K<sub>2</sub>O/Na<sub>2</sub>O was 0.5 for soft porcelains and 4 for hard porcelain. Nepheline syenite is a stronger fluxing agent than feldspars, due to its low-SiO<sub>2</sub>

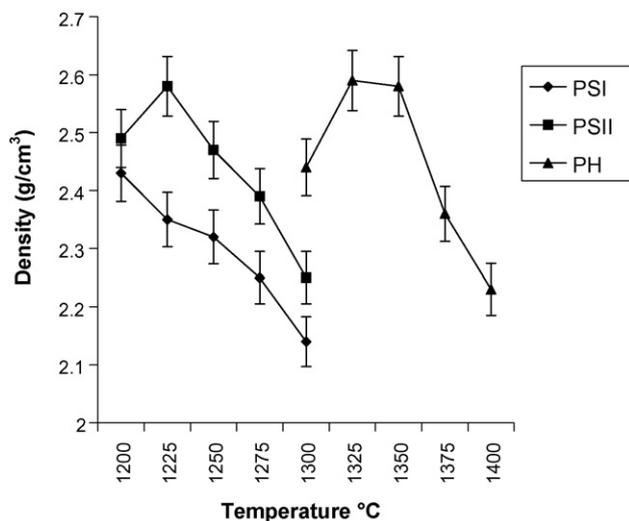


Fig. 2. Bulk density of porcelain compositions (g/cm<sup>3</sup>).

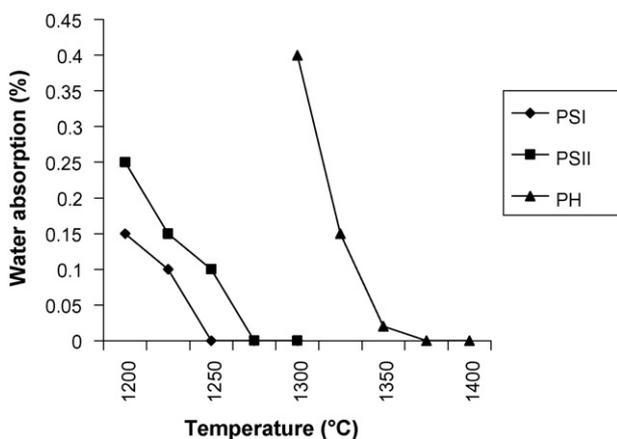


Fig. 3. Water absorption of porcelain bodies (%).

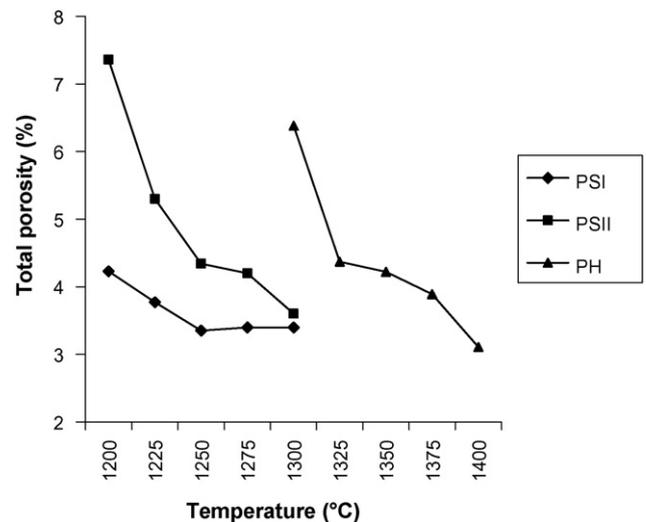


Fig. 4. Apparent porosity of porcelain compositions (%).

content, absence of quartz and high Na<sub>2</sub>O. At 1250 °C, potassic feldspars have been observed to delay fusibility because of an incongruent fusion giving rise to a new crystalline phase [18,19]. The thin glaze on the self-glazed porcelains could influence mechanical properties [20].

3.3. XRD, microstructure and flexural strength

Mullite, quartz and cristobalite were the only three crystalline phases obtained on the XRD of the porcelain

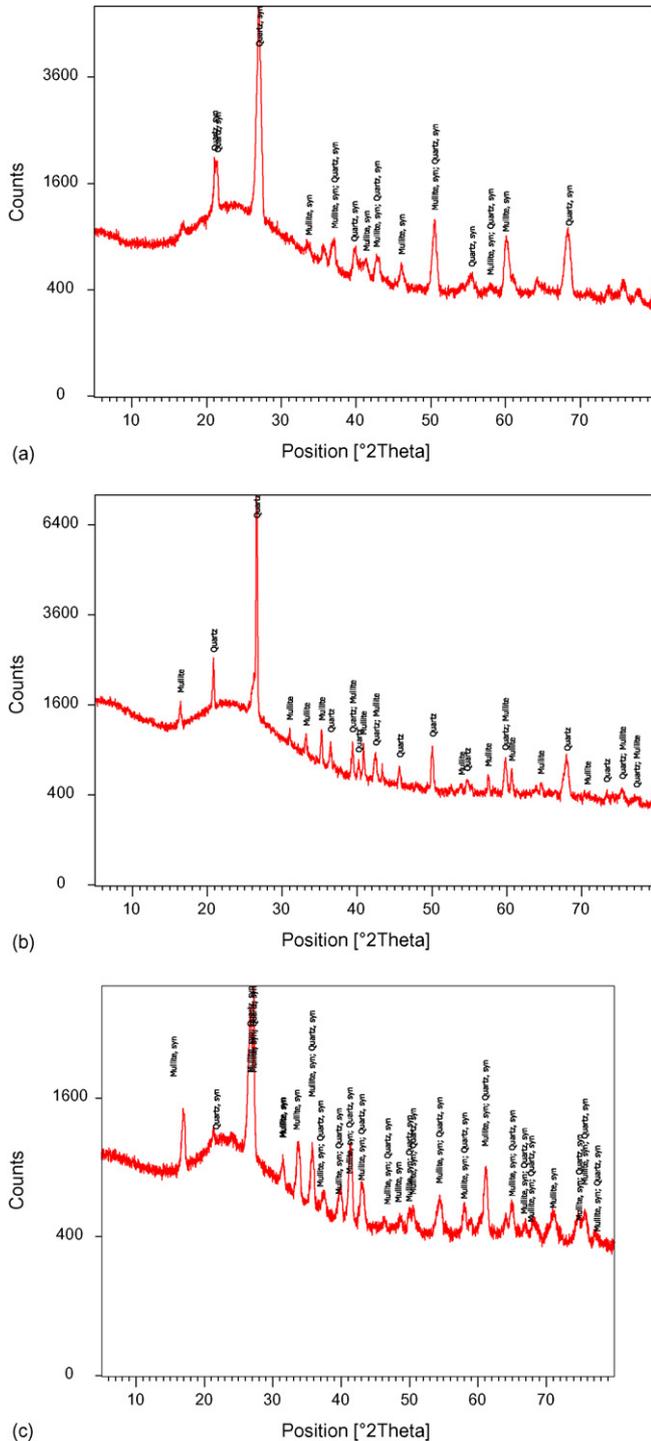


Fig. 5. XRD patterns of the porcelain compositions: (a) PSI, (b) PSII and (c) PH.

bodies fired at 1250 °C for PSI and PSII and 1350 °C for PH (Fig. 5). The quartz content (main phase) was more abundant in the soft porcelains than in the hard porcelain in which cristobalite was observed at temperature above 1350 °C (here not reported). It is reported that amorphous silica obtained during transformation of metakaolin to mullite crystallises at about 1350 °C to give cristobalite [4]. In addition, in the soft porcelain more quartz was required to adjust their body compositions, 34.70 and 27.32%, respectively for PSI and PSII, while only 11.70% was used for PH. The mullite peak is more intense in the hard porcelain than on the soft ones. It has been observed that there is less mullitisation in silica-rich porcelain bodies and that high alumina (24% in PH and 19% in PSI and PSII) results in more mullite formation [9]. The variation of flexural strength with temperature (Fig. 6) for all three porcelains follows the same allure with that of their densities (Fig. 2).

The highest flexural strength (that corresponds to the highest densities) were 148 MPa for PSI (1200 °C) and PSII (1225 °C) and 167 MPa for PH at 1350 °C. The decreasing densification at higher firing temperatures could explain the flexural strength decrease in PSI and PSII. In PH, the flexural strengths increase up to 1350 °C before dropping. The increase in flexural strength with temperature development up to 1250 °C for soft and 1350 °C for hard should be the result of increasing in amount of mullite along with the liquid phase, responsible for the densification processes and quartz dissolution. The increased amount of liquid phase at higher temperature certainly affects negatively the mechanical strength. Generally in ceramic compositions containing clays and feldspars, increased temperatures result in higher mullite formation, thereby improving mechanical properties. However, it has been observed that, at even higher temperatures, mullite crystals become coarse, consequently decreasing strength [21–23]. The mullitisation reactions in porcelain-sintering processes include transformation of metakaolinite to mullite that generally produces amorphous silica. This silica is present in the glassy matrix and can react with dispersed alumina ions (Al<sup>3+</sup>) to form secondary mullite or recrystallise to SiO<sub>2</sub>. This competition can result in microstructures that are not always favourable for mechanical body strength. The microstructure of the three

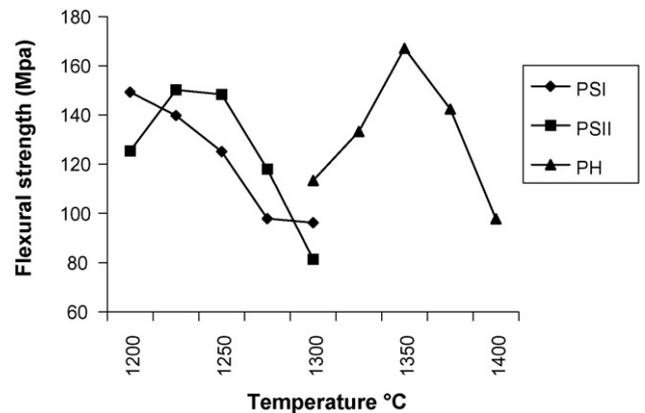


Fig. 6. Flexural strength of porcelain compositions.

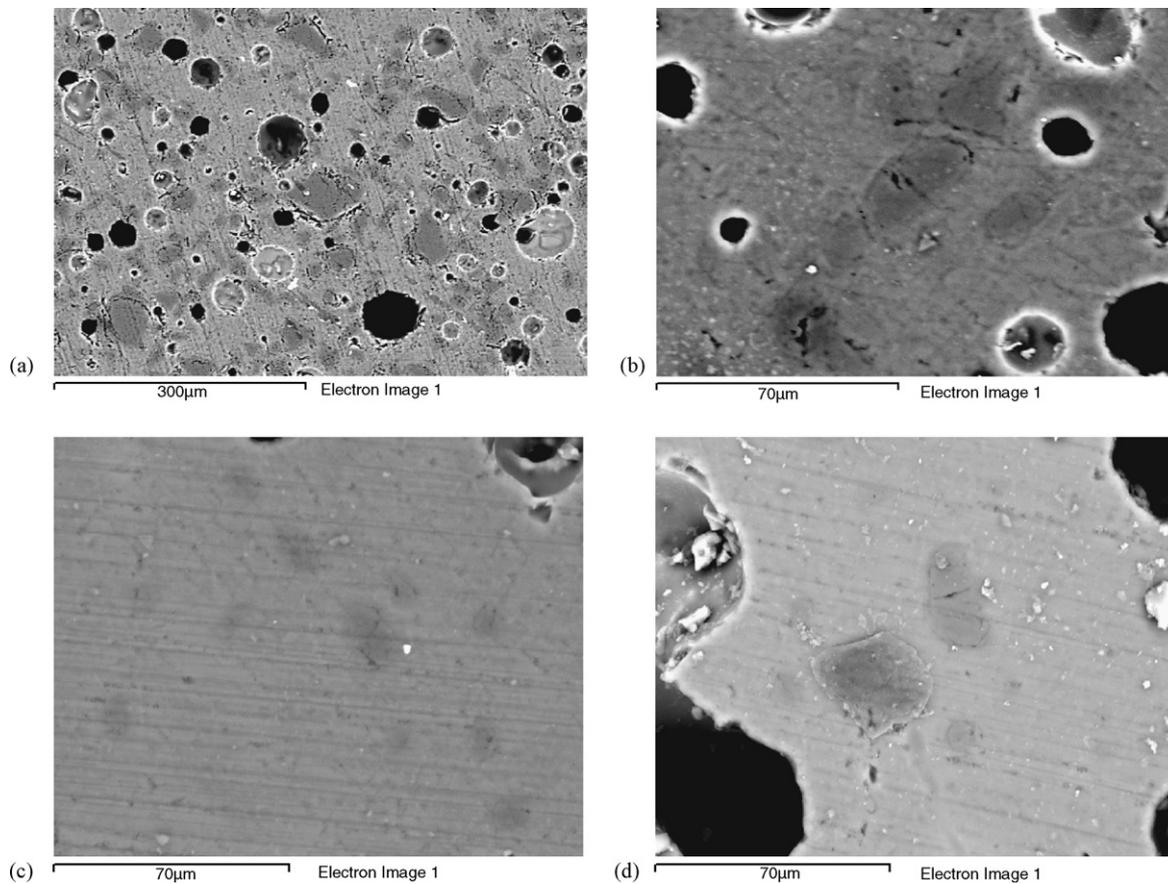


Fig. 7. Microstructure of porcelain bodies (BEI images): (a) PSI at 1275 °C; (b) PSII at 1275 °C and (c) PH at 1350 °C and (d) PH at 1400 °C.

porcelain bodies as observed on SEM are shown in Fig. 7. The microstructures exhibit homogeneous matrix with relatively low porosity. Quartz grains are seen to be dispersed in the soft porcelains. The matrix has a relatively high Si/Al ratio compared to that of PH, revealed by EDS spectra analyses. From the micrographs, it is observed that the undissolved quartz grains possess cracks around them, generating some stress that results in lower mechanical strength.

In the soft porcelain, residual quartz amount was observed to decrease with higher temperatures, due to its dissolution. In the hard porcelain, less quartz was observed, due to the initial lower amount of quartz used and the conversion of part of the SiO<sub>2</sub> in cristobalite.

#### 4. Conclusions

Soft and hard porcelain with excellent technical characteristics were produced with two different china clays from Cameroon. From the results obtained in this work, it can be concluded that the two china clays from Cameroon are suitable as clay for porcelain bodies. The properties of the final products show that:

- Soft porcelain with low clay content and higher proportion of fluxing agent can be produced in the range of temperature of 1200–1225 °C with average density of 2.4 g/cm<sup>3</sup>, water absorption less than 0.1% and flexural strength of 149 MPa.

- Hard porcelain bodies with higher clay content and relatively low proportion of fluxing agent can be produced in the range of temperature of 1325–1350 °C and flexural strength of 167 MPa
- Therefore the use of China clays with TiO<sub>2</sub> and Fe<sub>2</sub>O<sub>3</sub> content permits a decrease of 25 °C in firing temperature. This should reduce production costs which makes its utilisation very attractive, especially for tiles where the white colour is not required.

For porcelain white ware the china clay from NTAMUKA should be better indicated due to its white colour after firing in addition to its silica content that is more suitable for low temperature. The phenomenon of self-glazing can be exploited to produce good aesthetic products with soft porcelain compositions when there is not strong requirement for mechanical properties.

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