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MINERALOGICAL CHARACTERIZATION OF ETHNOGRAPHIC CERAMICS FROM BANKIM (ADAMAWA, CAMEROON/ WEST CENTRAL AFRICA)

CARACTERIZAÇÃO MINERALÓGICA DA CERÂMICA ETNOGRÁFICA DE BANKIM (ADAMAWA, CAMARÕES/ÁFRICA CENTRAL OCIDENTAL)

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ABSTRACT

The study investigates mineralogical characteristics and thermal threshold at successive steps of Tikar pottery production chaine-operatoire from Bankim (Adamawa, Cameroon). It is a pilot study relying on X-ray diffraction (XRD), thermal analysis (DSC/TG) and Fourier Transform Infrared Spectroscopy (FTIR). The ceramics were collected in the drying and firing stages of the production chain. The work aimed to estimate the drying and firing temperature of the studied pottery sherds. Due to the mineralogical change occurring during the drying or pre-firing and firing stages, the correlation between the three analytical methods suggested that the pre-firing temperature were below 450°C whereas the firing temperature was between 650-800°C in a simple bonfire.

KEYWORDS: Bankim-Ethnographic Ceramics-Mineralogy-Pre firing-Firing Temperatures

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RESUMO

Este estudo investiga as características mineralógicas e o limiar térmico em etapas sucessivas da cadeia de produção de cerâmica Tikar de Bankim (Adamawa, Camarões). É um estudo piloto baseado em difração de raios X (DRX), análise térmica (DSC/TG) e Espectroscopia de Infravermelho de Transformação Fourier (FTIR). As cerâmicas foram coletadas nas etapas de secagem e queima da cadeia produtiva. O trabalho teve como objetivo estimar a temperatura de secagem e queima dos cacos de cerâmica estudados. Devido à mudança mineralógica ocorrida durante as etapas de secagem ou pré-queima e queima, a correlação entre os três métodos analíticos sugerem que a temperatura da pré-queima estava abaixo de 450°C enquanto a temperatura de queima estava entre 650-800°C em uma simples fogueira.

PALAVRAS-CHAVE: Bankim-Cerâmica Etnográfica-Mineralogia-Temperaturas-Pré-queima-Queima

INTRODUCTION

In the process of pottery manufacture, firing is one of the most important stages which leads to the transformation of clay material into ceramic material. During firing some minerals resist whereas others collapse depending on the temperature, mineralogical characterization is one of the mostly used approach to estimate firing temperature of archaeological and ethnographic pottery. The researches carried out on the ethnographic pottery from Bankim have been mostly focused on the knowledge about variety of ceramic traditions, the stylistic-morphological approach and the relation between potters through marriage which has an influence on the variety of pottery techniques in this region.

However, a physico-chemical approach on the pottery tradition from Bankim has not yet been carried out to give information about the clay composition, firing parameters and other aspects. In order to complete the knowledge about the technology of pottery tradition from Bankim, the present work is a pilot study based on the determination of pre-firing and firing temperatures of the traditional ceramics from Bankim by using analytical methods such as X-ray diffraction (XRD), thermal analyses (DSC-TG) and Fourier transform infrared spectroscopy (FT-IR). These methods are widely used for the mineralogical characterization of ancient and modern ceramics in order to give information related to some technological aspects such as their origin/composition or firing temperature (MERCADER et al., 2000: 172; EMAMI et al., 2021: 2). For this reason, X-ray diffraction technique is quite widespread in ceramic studies and has been applied systematically from the 1990s, both in the mineralogical analysis of archaeological ceramics and clays (SANTACREU, 2014: 19). The FT-IR spectroscopy is a common and well established tool for the identifications of the ceramic body, it also reveals the type of clay and temperature of firing and firing conditions (RAVISANKAR et al., 2011: 373). As minerals go through phase transitions at various temperature, the combination of TG and DTA/DSC allows a clear representation of various phases, thus providing an image of the entire firing processs (ARTIOLI, 2010: 55).

LOCALIZATION OF BANKIN AND CERAMIC PRODUCTION BACKGROUND

Bankim is a locality situated in Adamawa division of Cameroon; it belongs to the Tikar area, with the coordinates 6°05 N -11°30 E **(Figure 1)**. Tikar area is a transition region between forest and savannah, thus this region is close to the centre of Cameroon. Eleven ethnic groups are found in Tikar area: the Bamileke, Bamoum, Mambila, Kwanja, Vuté, Wawa, Nizaa, Yamba, Tep, Gbaya and Tikar. Their languages are divided in three linguistic groups: Ubangian for Gbaya and Tikar people, Bantoid languages (Bamileke, Bamoum, Yamba) and Mambiloid languages (Mambila, Kwanja, Vuté, Wawa, Nizaa). Ceramic production is an activity practised by all the groups from this area but the choice of the Tikar one is due to the

fact that this group is the most dominant in the area.

Valentine Roux and Etienne Zangato (2013) conducted a project called ANR DIFFCERAM in the Tikar area with the aim to establish the first report concerning ceramic production tradition in relation with the different ethnic groups found in this area. Therefore, ethnographic research has been conducted in this region, the samples used in this study resulted from the research conducted by Valentine Roux and Etienne Zangato. During their research, inquiry was focused on the observations of chaine operatoire (extraction of raw material, preparation and modelling techniques), historical account of different traditions in relation with the transmission processus and the technical terms used by each social groups. From these works is clearly seen that potters from Tikar area belong to three principal ethnic groups: Mambila, Yamba and Tikar.

These groups produced pots which have the same morphology and function as well as the same way of treating the clay and firing, but they differ in the modelling techniques. Four different ceramic traditions have been found in this region according to the modelling techniques. For each tradition and following the experimentation work in this region, the collection of the clay, the preparation of the paste and the treatment of the raw material are identical: the clay material is extracted in the humid state and conserved in the pits of covered basins, the preparation of the paste is done by pounding in a mortar and adding water and mixing. At the same moment, purification of the paste is also done by removing the coarse elements. The mounting is generally done by hand or moulding using stick of palm tree. Once the vessel is mounted, the decoration is done by using a vegetal cordelette. Then the vessel is dried in the sun during 20 to 30 min. After the drying stage, some potters practice the pre -firing for 15 min. Afterwards, the vessels are placed on the fire and covered with herbs and plants: a typical simple bonfire.

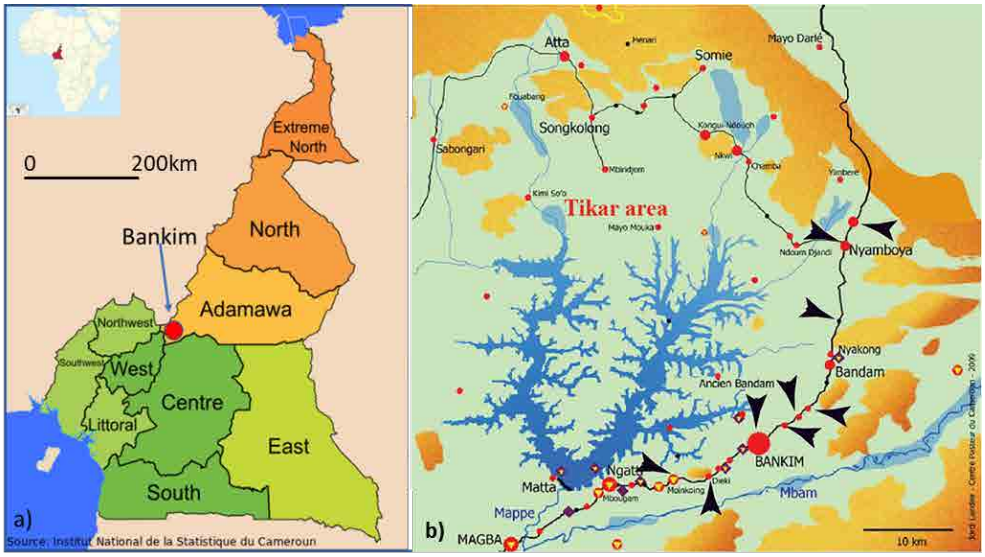

Figure 1. Localisation of Bankim a) Bankim in the Cameroonian map b) Bankim is an excavated site from the Tikar area (Valentine Roux and Zangato, 2013).

MATERIALS

As mentioned above, the samples used in this study resulted from the research conducted by Valentine Roux and Etienne Zangato (2013). For this study, as a pilot study, five pottery sherds from the vessels collected in Bankim during drying, pre-firing and firing stages of the production chain were analysed. These samples are named PS, PP1, PP2, PC1 and PC2 **(Figure 2 and Figure 3)**. The sherd PS comes from the drying stage, the sherds PP1 and PP2 from the pre-firing stage whereas the sherds PC1 and PC2 from the firing stage. They present different colours, thickness and ornaments (pointing, tracing and impressions). Table 1 below shows their characteristics.

Figure 2. Drying and firing stages of the vessels a) drying stage in the sun; b) pre-firing stage; c) beginning of firing; d) end of firing/ bonfires (photo Ossima, 2016).

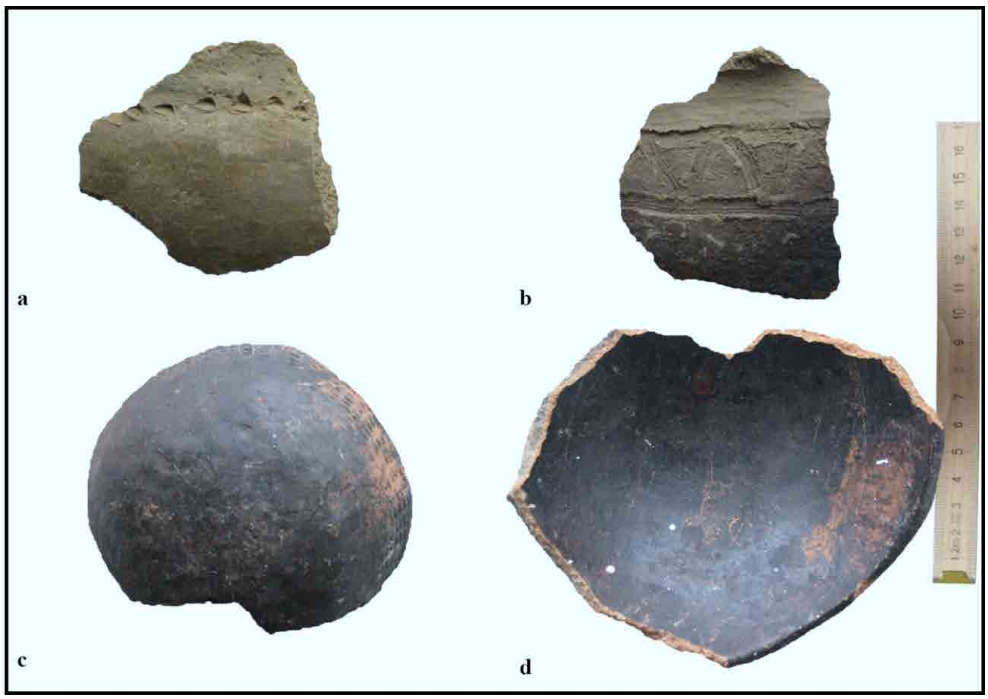

Figure 3. Representative samples: a) dried; b) prefired; c) and d) fired samples (photo Epossi, 2021).

Table 1. Characteristics of studied samples.

				Surface colours			Firing Atmosphere
samples	**stage**	**thickness (cm)**	**ornament**	**external**	**internal**	**fracture**	
PS	drying	1.5-2.3	pointing	Yellowish	Yellowish	Yellowish	oxidising
PP1	pre-firing	1.5-1.8	tracing	Light grey	Light grey	Greyish	oxidising
PP2	pre-firing	1.3-1.5	tracing	Light grey with some dark areas	Light grey	Greyish	oxidisnig
PC1	firing	0.8-1.0	Impression--maize corn	black	black	Red-black-red	Reducing and then oxidising
PC2	firing	0.7-0.9	Impression--roulette	black	black	Red	oxydising

METHODS

X-ray diffraction method is based on identifying minerals by their crystalline structure, X-ray diffraction analysis is a method of identifying minerals by their crystalline structure through the Bragg equation: $n\lambda = 2d \sin\Theta$ (Artioli 2010: 50–51). When Xrays are aimed onto a specimen, the atomic planes (d) of its constituent minerals diffract (reflect) the waves along certain directions or angles (Θ) that are picked up by a detector. A series of intensities or peaks of the rays at these angles, known as a diffraction pattern, is created**.** The peaks, plus the wavelength (λ) of the rays, allow the mineral to be identified by its characteristic lattice spacings (d) (Rice 2015: 484)**.** The analyses can be qualitative, semi quantitative or quantitative. The X-ray diffraction (XRD) patterns were obtained with a Bruker D8-Advance Eco 1Kw diffractometer (Copper Kα radiance, λ=1.5418 Å, V=40 KV, I=25 mA) with Lynx-eyed X energy dispersive detector. The analyses were carried out on the bulk material (non-oriented powder with grounded particles <50 µm). The XRD patterns were recorded over the 2-70° (2θ) angular range for the bulk material. The step sizes considered for this analysis was 0.02° (2θ), whereas the time per step chosen was 48 seconds. The identification of mineral phases was carried out using Eva software.

Differential thermal Analysis (DTA) or DSC (differential scanning calorimetry) is coupled with Thermogravimetry analysis (TG). Differential thermal analysis is based on the measurement of changes in the temperature of clays or ceramics heated experimentally to a temperature up to 1000°C. When a clay/ceramic is heated, it undergoes a series of changes in which heat is given off (exothermic reactions) or absorbed (endothermic reactions). Thermogravimetry is based on the weight lost during the heating of the sample.

Thermal analyses DSC/TG were carried out using 20 to 22mg of material/powdered samples in the Physico-chemistry Material Research Laboratory at the University of Yaounde 1, Cameroon. The samples were placed in Al_2O_3 crucibles and the measurements were done with Al_2O_3 as standard in ambient air from 24 to 1000°C with a heating rate of 10°C/min. The LINSEIS SPAPT 1000 apparatus was used.

The FT-IR spectroscopy is a vibrational method which provides some information of the crystalline and the molecular networks of the samples. It is advisable since it allows the identification of the crystalline or amorphous phases existing in the samples (Santacreu 2015: 41). The Infrared spectrum of a sample is collected by passing a beam of infrared light through the sample described by Fabbri (FABBRI, 2012: 438) as follow: "The examination of the transmitted light reveals how much energy was absorbed by the sample at each wavelength. This can be performed by means of a monochromatic beam, which changes in wavelength over time. In such a way, a transmittance or absorbance spectrum can be produced, showing at which IR wavelengths the sample absorbs. Analysis of these characteristics of absorption reveals details about the molecular structure of samples." Moreover, FT-IR spectroscopy can also be applied for the study of organic materials on the contrary of X-ray Diffraction which can be only used in the study of crystalline materials. The analyses of the studied samples were performed with a Nicolet iS5 FTIR Spectrometer using a ATR mode on a Germanium crystal in the wavelength regions 4000-500 cm^{-1} and 500-400 cm^{-1}.

RESULTS AND DISCUSSIONS
X-RAY DIFFRACTION XRD

The mineralogical composition of the five samples shows quartz and feldspar (microcline and / or albite) in all the samples. Kaolinite is present in the samples PS, PP1, PP2, and absent in the samples PC1 and PC2. Table 2 gives the summary of the qualitative mineralogical composition. X-ray diffractograms of the samples PS, PP1, PP2, PC1 and PC2 are presented in **Figure 4**).

Table 2. Qualitative mineralogical composition of the samples (X: means Present / Abs: Absent).

Minerals	**Samples**				
	PS	**PP1**	**PP2**	**PC1**	**PC2**
Quartz	X	X	X	X	X
Feldspar	X	X	X	X	X
Kaolinite	X	X	X	Abs	Abs

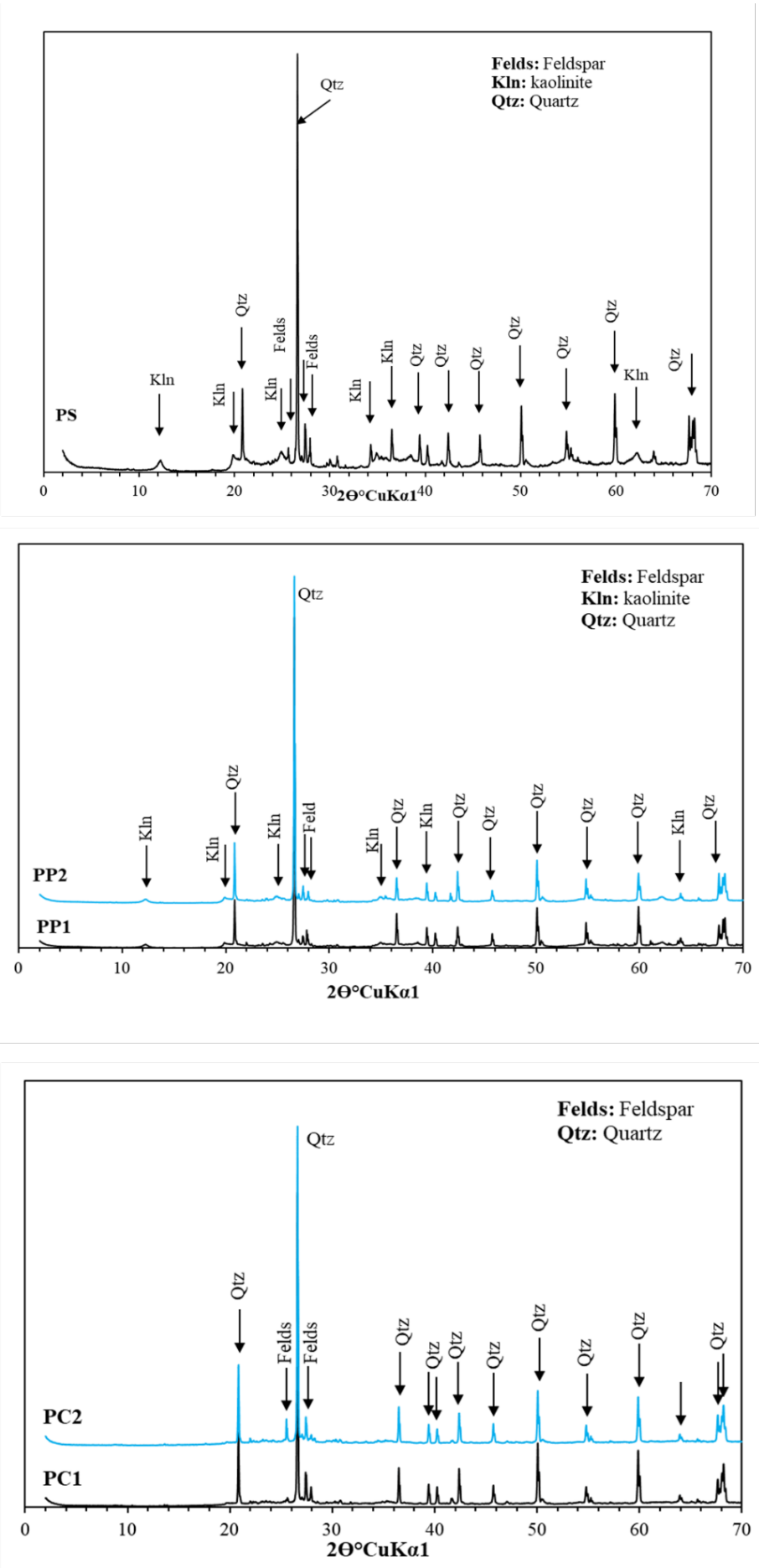

Figure 4. Representative XRD patterns of the samples PS, PP1, PP2, PC1 and PC2.

THERMAL ANALYSES DSC/TG

The thermograms of the five samples show an endothermic peak below 100°C, which corresponds to the interstitial water contained in the samples or dehydration of absorbed water (MEYVEL et al., 2012: 339) **(Figure 5 and Table 3).**

The samples PS, PP1 and PP2 present an important endothermic peak in the range 470°C to 480°C, followed by a great loss of weight between 350-650°C (6.14%, 7.53% and 4.86% respectively for each sample) which indicates the dehydroxylation of clay mineral e.g., kaolinite to metakaolinite. The presence of an exothermic peak around 902°C and 911°C in samples PP1 and PP2 indicates the transformation of metakaolinite to spinel or mullite (MANOHARAN et al., 2012: 416).

The samples PC1 and PC2 show a different thermal behaviour in comparison with the samples PS, PP1 and PP2, since they do not present an endothermic peak around 480°C. It is observed a weak endothermic peak around 571°C which indicates the transformation of quartz alpha to beta quartz. For the sample PC1, the two exothermic peaks at 813°C and 901°C reveal the formation of mullite or spinel. However, the sample PC2 does not show the peak around 813°C. This difference can be explained by a different source of the raw materials used to produced PC1 and PC2. Thermograms of the samples PS, PP1, PP2 and PC1, PC2 are presented in **Figure 5**.

Table 3. Thermal characteristics of the samples (X: means Present/ Abs: Absent).

Thermal Characteristics	**Samples**				
	PS	**PP1**	**PP2**	**PC1**	**PC2**
Endothermic peak <100°C	X	X	X	X	X
Loss of weight < 100°C TG%	-3.84	-3.73	-3.38	Abs	-1.54
Endothermic peak btw 470-480°C	X	X	X	Abs	Abs
Loss of weight between 470-480°C TG%	-6.14	-7.53	-4.86	Abs	Abs
Polymorphism of quartz	Abs	Abs	Abs	X	X
Exothermic peak around 800°C	Abs	Abs	Abs	X	Abs
Exothermic peak around 900°C	Abs	X	X	X	X

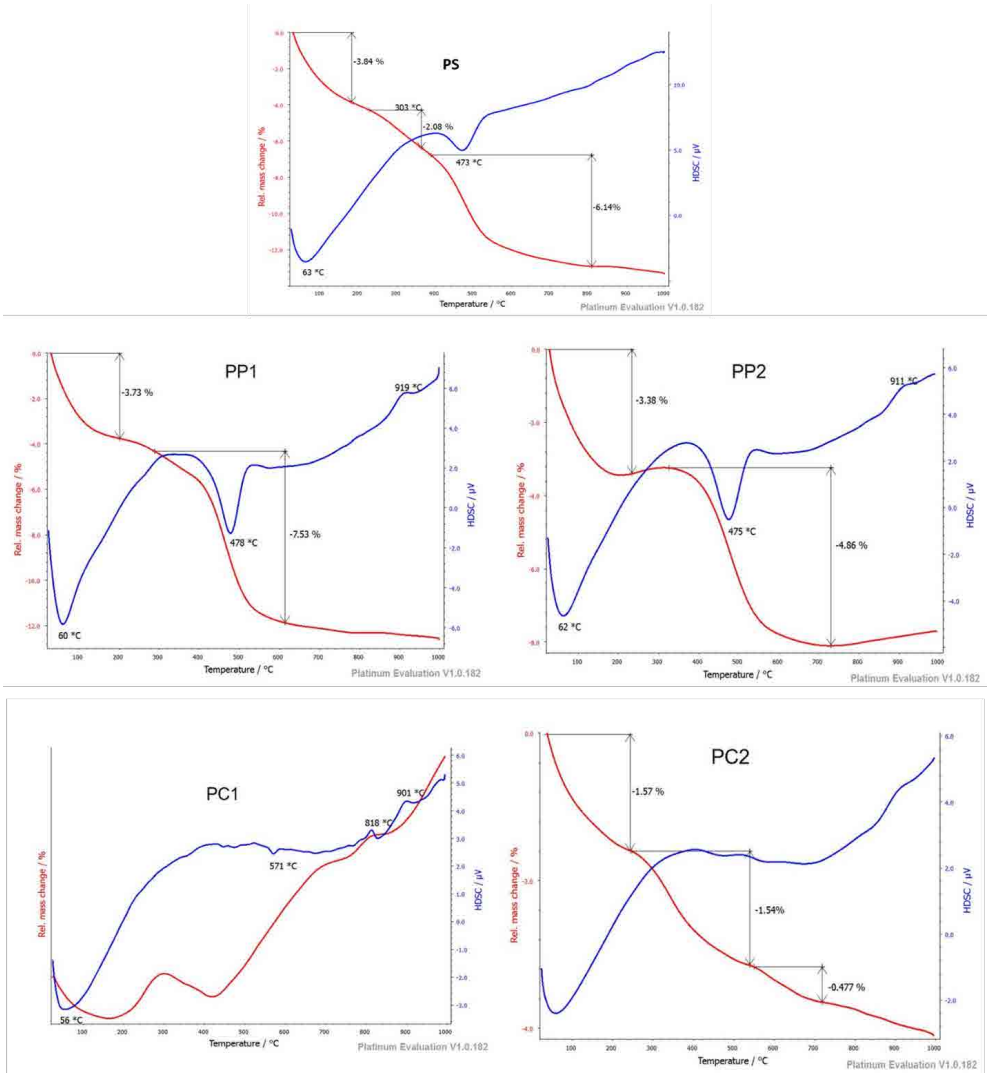

Figure 5. Thermograms of the samples PS, PP1, PP2 and PC1, PC2.

FTIR ANALYSIS

The FT- IR spectra of the samples are presented in **Figure 6** and Table 4.

FT-IR spectra show in all the samples, weak peaks around 798cm^{-1} and 698 - 693 cm^{-1} which correspond to the Si-O bending of quartz and 773 cm^{-1} which correspond to the Si-O-Si stretching vibration of quartz (SATHYA and VELRAJ, 2011: 119; SARAVANAN et al., 2013: 30). The weak peaks between 700 - 600cm^{-1} correspond to the feldspar minerals.

The FT-IR vibration of samples PS, PP1 and PP2 are similar**,** they show weak bands at 3693 cm^{-1}, around 3648 cm^{-1} and 3618-3620 cm^{-1} which corresponds to O-H stretching vibration, crystalline hydroxyl (VIJAYARAGAVAN et al., 2013: 64; SINGH and SINGH, 2016: 119; SIDDIG et al., 2018: 5). The band around 3400cm^{-1} is due to O-H stretching of absorbed water and the 1653cm^{-1} corresponds to the H-O-H bending water (SINGH and SHARMA, 2016: 559; SIDDIG et al., 2018: 5). The very broad bands around 1027 cm^{-1} the bands around 1003cm^{-1} and the bands at 912 cm^{-1} correspond to the Si-O-Si stretching and Al(OH) vibration kaolinite

(SIDDIG et al., 2018: 5; VIJAYARAGAVAN et al., 2013: 67; SINGH and SINGH, 2016: 119; MANOHARAN et al., 2012: 416). The bands around 757cm^{-1} correspond to the Al-O-Si stretching mode of mica (VIJAYARAGAVAN et al., 2013: 67; SINGH and SINGH, 2016: 121).

The samples PC1 and PC2 have also a similar FT-IR vibration behaviour. They present a broad band around 1045 cm^{-1} which corresponds to the Si-O stretching of mica (Seetha and Velraj 2016: 348) and very weak bands between 700 and 600 cm^{-1} which to vibration bands of quartz and feldspar (SATHYA and VELRAJ, 2011: 5) (Table 4).

The absence of mica in the XRD patterns could be explained by his worse crystal state in all the samples.

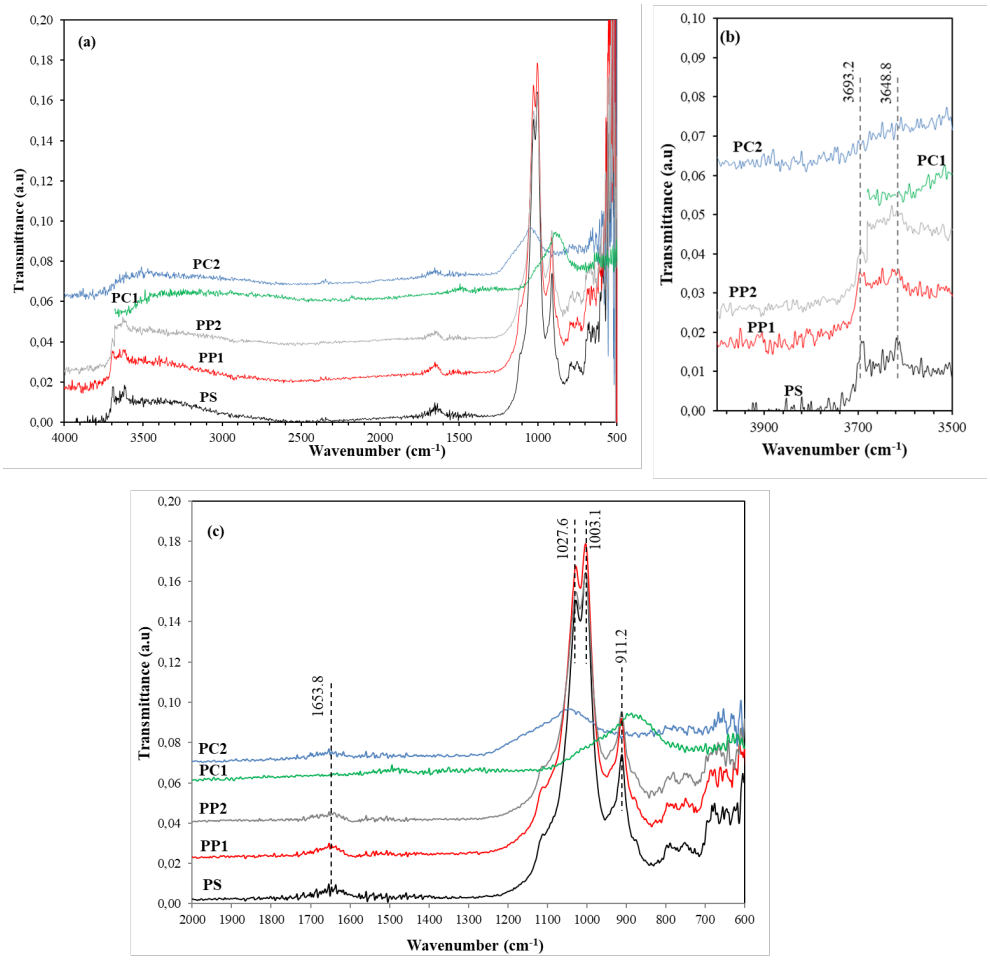

Figure 6 (a) FT-IR Complete spectra of the samples, (b) 4000-3500 cm^{-1} domain and (c) 2000-600 cm^{-1} domain.

Table 4. The characteristics of infrared frequencies of the ceramics and tentative vibrational assignments.

Frequency with relative Intensity (Cm^{-1})					
PS	**PP1**	**PP2**	**PC1**	**PC2**	**Tentative vibrational assignments**
3693.21 w	3696.66 w	3695.32 w			O-H stretching,crystalline hydroxyl
3648.88 w	3648.88 w	3648.88 w			O-H stretching of inner surface hydroxyl group
3619.01 w	3620.03 w	3628.92 w			O-H stretching vibration inner hydroxyl
	3486.05 vw	3460.48 vw			Al-OH stretching
3400 vw	3399.73 vw	3401.6 vw			H2O vibration
1653.83 w	1653.31 w	1653.34 w			H-O-H water bending
			1046.45 s	1047.19 s	Si-O stretching of Mica
1027.62vs	1027.98 vs	1027.34vs			Si-O-Si stretching of kaolinite
1003.18vs	1003.43 vs	1003.34 vs			Si-O stretching of kaolinite
911.2 s	912.42 s	912.05 s			Al-(OH) kaolinite
798w	790w	792w	798w	798w	Si-O bending of quartz
773w	773.7w	780.45w	773w	773w	Si-O stretching of quartz
752.17w	749.78w	750.03w	759w	750 w	Al-O-Si mica strechting
698 w	693 w	693 w	698 w	698 w	Si-O bending of quartz
655.77vw	646.72 vw	646.34 vw	648.94 vw	653.96 vw	Al-O-Si stretching of Felspar

Indications: vw=very weak, w=weak, s=strong, vs= very strong.

DISCUSSION ON DRYING AND FIRING TEMPERATURE OF THE POTTERY

The mineralogical phases obtained by XRD, DTG/TG and FT-IR are in accordance with all the methods. The estimation of firing temperature or equivalent firing temperature (EFT) (MAGGETTI et al., 2011: 7) can be deduced by the mineralogical changes from clay to the fired clay. Therefore, Xray diffraction are commonly used in archaeometry to deduced the EFT, the combination of X-ray Diffraction, DSC/TG and FT-IR gives more precisions. Quartz and K-feldspar are stable up to higher temperature. According to the literature, quartz can be stable up to 1200°C and the K-feldspar up to 1000°C. Only the clay minerals are mostly used for the deduction of the firing temperature because they can destroy their octahedral sheet at temperature around 500-650°C (SINGH, 2016: 558; MAGGETTI et al., 2011: 7).

The FT IR spectra of the three pottery sherds PS, PP1 and PP2 show the water bands at 3700 and 3620 cm^{-1} which reveal that they were dried or pre-fired below 450°C (SARAVANAN et al., 2013: 31). This result is confirmed by the presence of the peaks of the absorbed water around 3400 cm-1, 1653 cm^{-1} and the kaolinite peaks at 1027, 1003 and 912 cm^{-1}. The decomposition of kaolinite occurring between 478-650°C with weight loss (DSC/TG) indicates a EFT below this range of temperature. However, the sample PS was submitted at lower temperature than the samples PP1 and PP2 which were dried in the firing system whereas the sample PS was dried in the sun (Figure 2: a, b). This difference of temperature is observed by the presence of weight loss at 301°C (TG, -2.08%) in the sample PS

and its absence in the samples PP1 and PP2. This result reveals that the sample PS contained more water than the two others.

The absence of the important endothermic peak around 450-650°C in the DSC/TG curves of the samples PC1 and PC2 suggests a firing temperature above 650°C. This result is confirmed by the absence of the vibration bands between 3700 to 3400 cm-1 and 1653cm-1 (water vibration), 1027 and 1003 cm^{-1} (kaolinite vibrations). The absence of these vibrations shows that these minerals were destroy during the firing. Annamalai et al. (ANNAMALAI et al., 2014: 29) suggests a temperature over 500°C due to the absence of the water bands at 3700°C and 3620 cm^{-1}. It is also confirmed by the absence of the vibration at 912 cm^{-1}, according to Ravisankar et al. (RAVISANKAR et al., 2011: 373), this vibration begins to disappear at 500°C. The only vibration observed around 1050°C, corresponding to the mica vibration, means that the firing temperature was not high enough to decompose the mica. Therefore, the estimated firing temperature of these samples is above 650°C and below 800 or 900°C because the decomposition of mica starts around 800°C or between 900-1000°C depending on the type of clay (EPOSSI NTAH et al., 2017: 420)

DISCUSSION ON ETHNOGRAPHIC AND ARCHAEOLOGICAL IMPLICATIONS

The first work on the ethnographic firing from Cameroon (Mbam region) was done by Gosselain (GOSSELAIN, 1992: 257). He did firing experiments with the potters in several villages and used thermometric data to study the range of temperature in each firing system. He placed thermocouples directly to the inner and outer wall of the pottery. He concluded from his experiments that two thirds of the flame data were in the range 300–900 °C for surface bonfires, 670–870 °C for surface firings with broken ceramics covering the pottery, 620–870 °C for pit firings and 770–870 °C for pit firings with broken ceramics covering the pottery (MAGGETTI et al., 2011: 7). These results are in agreement with the conclusion obtained in this work by using a mineralogical approach, the range of firing temperature is between 300 - 900°C in a simple bonfire i.e. firing on the surface and the pottery covered with the plants/herbs (Figure 2: c, d).

In pottery analysis, colour is one of the properties which tells us about the raw materials used and the way it was fired (RICE, 1987: 331). The colour of the cross section or fracture section of a fresh cut of the pottery sherd is a key factor in archaeology for the estimation of firing temperature and atmosphere whereas the colour of external and internal surfaces is not concerned because they can be changed due to the use of the vessel or their burial stage. Thus, the samples studied in this work present different colour of their fracture section at drying, pre-firing and firing stages (Table 1). Sample PS collected in the drying stage is yellowish whereas the samples PP1 and PP2 from pre-firing stage are greyish. These two colours reveal that the clay material used to produce these samples

were yellowish for PS and greyish for PP1 and PP2 because drying and pre-firing do not affect the original clay colour due to the low temperature. They produce physical transformations due to the water loss and keep the colour of some mineral in the natural state, e.g. Some hydrous ferric oxides as goethite and limonite are yellowish (RICE, 1987: 334-335).

On firing, these minerals lose their water and change to ferric oxide yielding a red colour (or reddish colour). Thus, the sample PC1 presents a sandwich structure of the fracture surface (margins red with a black core) and PC2 a red or reddish colour. These colours can be explained by the presence of iron oxide. However, the presence of organic matter and the amount of iron oxide can also play a role. Organic matter in a raw material makes it gray, black or dark brown depending on the amount present (RICE, 1987: 333). Thus, the sample PC1 with a black core can be explained by the presence of organic matter on one side. On the other side, a sandwich structure of a fresh cut can be explained by a short duration of firing or a low fired clay (ARTIOLI, 2010: 240) or an incomplete oxidation atmosphere (MARTINEAU and PETREQUIN, 2000: 353)**.** However, the thermal analyses and the infrared spectroscopy vibrations do not show the presence of the organic matter in this sample. On the contrary, sample PC2 with a full red fracture section is a result of a full oxidation due to the long firing time, therefore we can assume that the sample PC1 was fired at lowest temperature than the sample PC2 but both samples are firing in the range of temperature 650 to 800°C or 900°C due to the presence of mica in their paste.

CONCLUSION

The investigation of dried, pre-fired and fired ethnographic pottery from Bankim (Tikar area, Adamawa) by using X-ray Diffraction, Thermal Analyses and FT-Infrared Spectroscopy shows a good correlation between the methods. The mineralogical phases observed in all the pottery are quartz, mica and feldspar. Kaolinite was observed in dried and pre-fired pottery. Due to the mineralogical changes observed, it has been deduced that the drying and pre-firing temperature of the pottery was below 450°C but the pre-firing temperature was superior to the drying one. The firing temperature of the pottery in a simple bonfire was in the range 650 - 800°C. However, due to the fact that pottery firing strategies are complex and there is not a direct correlation between firing structures, temperatures, duration and fuels, hence, it is difficult to differentiate these parameters with the mineralogical approach using the analytical methods. Therefore, the analysis of the colours of the fracture surface of the pottery has been applied to give complementary information concerning the firing atmosphere, duration and temperature. The dried and pre-fired samples have the colour of their original clay whereas the two fired samples could have been fired in the oxidation atmosphere at different duration and temperature.

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