INTRODUCTION

Agricultural waste disposal is continuously increasing in developing and Third World countries. Indeed, world and national authorities are establishing strict regulations for the recovery of these wastes regarding environmental issues. Besides, some authors have pointed out the potential use of such wastes for manufacturing various materials.1,2 In recent
years, the incorporation of agricultural or plant wastes has been investigated particularly regarding the manufacturing of ceramic products.\textsuperscript{3–15} Obviously, the use of plant wastes would promote pore development within ceramics during the firing or sintering process, due to their chemical degradation and related evolved gases. Although porosity in some ceramic products has been considered problematic for a long time, the potential offered by porous ceramic attracts much more attention today in comparison with the considerations applied a few years ago.\textsuperscript{16}

Porous materials appeared of great interest both in everyday life (for example, with the filtration of drinking water) and in the industrial field with the elimination of dust by granular filters in industrial processes requiring high purity of the fluids (liquids or gases). In general, these porous ceramics are suitable candidates for overcoming some environmental and energy issues,\textsuperscript{17,18} in the fields of heterogeneous catalysis and chemical separation. The use of organic residues in brick production to obtain porous ceramic bodies with enhanced insulating properties was studied by several authors.\textsuperscript{4,5,9,11,13,19} Clays, the basic raw material in the manufacture of such ceramic products, exhibited various chemical and mineralogical characteristics that would significantly influence the final properties of use at the industrial scale. In the Central African Republic, the State's General of Mines in 2003 reported that this country abounds in more than 400 mineral substances, among which clay materials were predominantly identified.\textsuperscript{20}

Moreover, Central African Republic is facing a drastic housing crisis combined to the drinking water crisis, as in most African countries, which can ruin the lives of the population. The use of these local clay deposits could be promoted for producing porous clay-based ceramics for water purification or building purposes. The local agricultural production and the development of agribusiness in many countries of the world have led to great quantities of agricultural waste, which are not properly managed or reused. The incorporation of these organic waste should be thoroughly considered within clay materials, aiming at promoting tunable microstructures and final properties of the related fired ceramics. The development of local natural resources (clays and plant waste) for the production of silicate materials is therefore of great interest and constitutes one of the major assets of the sustainable development process, therefore, very promising for environmental and economical issues in Central African Republic and in most countries in the tropical region.

For clay-based fired bricks, the reduction of their thermal conductivity values could result from the creation of cavities thanks to the incorporation of some porogenic additives (increase of the open porosity). Indeed, during the subsequent heat treatments (drying and firing), the added porogens are burnt out, leaving voids due to their breakdown.\textsuperscript{22} The formation of these pores has a significant impact on the mechanical and thermal properties of such clay bricks. For example, Raut and al conducted a study on the “Development of thermally efficient fibre-based eco-friendly brick reusing locally available waste materials.” They concluded that the use of oil palm fibers (OPF) was beneficial in lowering the thermal conductivity of bricks. For the incorporation of 1% wt of OPF, a compressive strength of 7.21 MPa was obtained, and a decrease of the thermal conductivity from 0.58 to 0.39 W/mK was noted. Such product was proposed as an alternative to non–eco-friendly commercial common bricks.\textsuperscript{23}

The aim of this study is to promote the use of mixtures based on plantain skins (“Musa paradisiaca”)\textsuperscript{21} and local clay (Central African Republic), for the manufacture of porous ceramics (with open porosity >35%). The clay raw material used, noted NZ2, was subjected to physical and chemical analyses, followed by the investigation of their sintering behavior with and without the addition of plantain skins (noted MP). Finally, the mechanical properties (compressive strength) and the thermal conductivity values of the manufactured products were characterized in relation to the effect of the addition of plantain skin on the microstructure. The characteristics of the ceramics obtained may highlight the role of this waste regarding application as insulating clay-based ceramics.

## 2 | MATERIALS AND METHODS

### 2.1 | Raw materials

Among the different clay deposits in Central African Republic the Nzila deposit (noted NZ2), located at 9 km from the capital (Bangui), were selected for this study. This clay material was used previously for the local production of pottery and bricks in Bangui. In the case of agricultural waste, we were interested by the plantain skins, issued from \textit{M. paradisiaca} plant noted MP, and collected in Central African Republic (wild tips) and in France (household waste). These wastes were used in the past for the traditional manufacture of culinary salt thanks to their potassium content. These practices had gradually disappeared due to the preferred use of conventional sodium-based salts. Therefore, these skins resulting from the transformation of the \textit{M. paradisiaca} fruit would constitute a source of environmental pollution. A commercial kaolin (kaolin BIP, Imerys Ceramics)\textsuperscript{24} served as the reference raw clay to investigate the MP waste influence over the sintering behavior.

### 2.2 | Experimental procedure

The plantain skins collected for the present study were dried within an oven (40°C) for at least 72 hours (or until constant mass is reached). They were subjected to grinding using a
planetary ball mill. The container (volume 250 mL) and the grinding media were constituted of alumina. The milling was conducted at 200 rpm for 1 hour, followed by a sieving stage (full passage <200 µm). The raw materials (clay + waste) were thoroughly mixed within a porcelain mortar then send to a pelletizer. During the rotation of the pelletizer, the powder was continuously mixed while spraying progressively the appropriate water content. The as-obtained granules were used to manufacture the studied compacts (25 mm in diameter), shaped by unidirectional pressing under a pressure of 10 MPa. Firstly, various amount of the MP (0, 5, 10, 15, 20, or 30 mass%) were mixed with kaolin BIP and sintered at 1000 and 1200°C with a soaking time of 1 hour. Secondly, the sintering behavior of NZ2 clay was considered from previous experiments and used to manufacture samples containing 10 and 15 mass% of the MP waste. In the latter case, sintering was performed at 1100°C with soaking times of 1 and 2 hours.

The particle size distribution of clay powder sieved at 200 µm was determined by laser diffraction using laser scattering equipment, Horiba LA-950 V2 through the liquid route. Aqueous suspensions of the studied clays were prepared by mixing the sieved powder with deionized water containing 1/1000 by mass of sodium hexametaphosphate (Na₅P₂O₁₀). Prior to the measurement, each suspension was stirred for five (05) minutes under sequential ultrasonication. The Atterberg limits (plasticity of clay) were determined in agreement with NF P 94-051 standards; the plasticity index Iₚ of NZ2 clay was determined by relation (1).

\[ I_p = W_L - W_P \]  

(1)

With \( W_L \) the liquidity limit and \( W_P \) the plasticity limit.

The specific surface was determined by the BET method of nitrogen adsorption using Micromeritics ASAP 2020 equipment (Micromeritics) on samples degassed at 200°C under nitrogen for 10 hours. The density of the clay powders was measured by a helium pycnometer (Micromeritics AccuPyc II 1340 from Micromeritics). The open porosity was determined by the Archimede’s method (buoyancy test) using water as the impregnation liquid. Relation (2) served to determine the open porosity \( P \) in percentage.

\[ P (%) = \left( \frac{M_3 - M_1}{M_3 - M_2} \right) \times 100 \]  

(2)

with \( M_1 \) (g): mass of dry sample. \( M_2 \) (g): apparent mass determined by hydrostatic weighing. \( M_3 \) (g): mass in air when the sample is still impregnated with the liquid. All experiments were repeated at least five times with similar conditions.

The chemical analysis was carried out using an X-ray fluorescence spectrometer of the Panalytical Zetium 1KW type (Malvern Panalytical). The mineralogical analysis by X-ray diffraction (XRD) was carried out using a D8 ADVANCE BRUKER (Bruker) diffractometer, and the data processing was carried out using EVA software (Bruker AXS) as well as the International Center for Crystallographic Data database with the help of Joint Committee on Powder Diffraction Standards (JCPDS) files for inorganic compounds. The measurements were carried out using a 2θ angular scanning mode in the range 5 ≤ 2θ ≤ 60°, with a step of 0.02° and a scanning time of 1.1 seconds per step. A scanning electron microscope IT300LV (supplied by JEOL) coupled with energy dispersive spectroscopy (EDS) analysis was used to observe the pore size and morphology of the materials produced and to identify the chemical elements that constituted the MP waste.

A Setsys Evolution TMA vertical dilatometer (Setaram) was used to monitor the expansion and/or shrinkage of the studied samples upon the heat treatment. For the thermolatometery analyses performed under dry air, a heating rate of 5°C/min was used from room temperature (30°C) up to 1250°C with a dwelling of 1 hour at 1250°C, followed by a cooling at 10°C/min down to room temperature (30°C). Differential thermal (DTA) and thermogravimetric (TGA) analyses were carried out using a coupled assembly of DTA-TGA, namely the Setsys TG-DSC Evolution (Setaram). The analyzed samples were the 200 µm fraction previously dried at 105°C for 24 hours. The analyses were carried out under dry air with heating rate of 5°C/min up to a temperature of 1300°C and a cooling rate of 20°C/min. Alumina previously calcined at 1500°C served as the reference material and platinum crucibles were used for both sample and reference powders.

The mechanical strength of each sample was assessed by the diametrical compressive test also called the Brazilian testing technique. It was performed using an Instron traction and compression press (Instron). The cylindrical specimens had dimensions around 24.5 × 13 mm (diameter and thickness). All experiments were repeated at least five times with similar conditions. If \( F \) (in N) is the load provided at the time of the rupture, \( D \) (in mm) the diameter of the disk-like sample and \( H \) (in mm) its height, the compressive strength through diametrical compressive test in MPa is determined using relation (3).

\[ \sigma = \frac{2F}{\pi DH} \]  

(3)

The Hot-disk method was used to determine the thermal conductivity of our samples. The equipment was a TPS 2500 S model (Hot Disk), including a transient plane sensor which was constituted of an electrically conductive pattern in the form of a double spiral engraved on a thin sheet of metal (Nickel). The specimens (shaped by unidirectional pressing) and the probe (kapton type disk) diameters were 24 and...
For each type of mixture, the thermal conductivity value provided results from mean value of five measurements of each sample.

### 3 | RESULTS AND DISCUSSION

#### 3.1 | Characteristics of raw materials

NZ2 clay and MP plantain skin powder were characterized before sintering in order to assess their chemical and mineralogical compositions. The chemical compositions listed in Table 1 showed major oxides as: SiO₂ (74.61%) followed by Al₂O₃ (12.57%) for NZ2, and the presence of potassium oxide (flux oxide) as main inorganic constituent in MP. The physical and technological (plasticity index, \( I_p \)) properties of NZ2 and MP are summarized in Table 2. The specific surface area of NZ2 (\( 10 \leq S_{BET} \leq 30 \text{ m}^2/\text{g} \)) and the plasticity index \( I_p \) (\( 5 \leq I_p \leq 10 \)) showed that NZ2 was a silica-rich clay and exhibited low plasticity. The density of NZ2 (Table 2) was close to 2.67 g/cm\(^3\), which was consistent with the values provided by literature. The density of NZ2 was between 2.3 and 2.7 g/cm\(^3\). The presence of iron oxide Fe₂O₃ (3.50%) and titanium oxide TiO₂ (2.74%) was noted in significant amount as expected, in relation with the reddish color of the local NZ2-based potteries found in CAR. The flux oxides content of NZ2 appeared very low (K₂O, CaO, Na₂O, and MgO close to 1.47%) and could justify the densification and consolidation problems reported by some craftsmen in Central African Republic. The NZ2 clay exhibited bimodal grain size distribution with characteristic grain sizes (Table 2) of several micrometers due to the presence of coarse silica grains. The analysis of the XRD diagram (Figure 1) indicated that the main mineralogical phases were kaolinite (JCPDS 14-164) and illite (JCPDS 26-911) associated with secondary phases as quartz (JCPDS 46-1045), goethite (JCPDS 29-713), rutile (JCPDS 21-1276), and anatase (JCPDS 21-1272). This result confirmed the trend observed in Table 1 for the high SiO₂/Al₂O₃ mass ratio, which was 5.94%.

The DTA/TG curves and the thermodilatometry (Figure 2A,B) illustrated the behavior of NZ2 and mixture NZ2 with 10 mass% of MP (NZ2-10%MP) upon firing. Between 31 and 400°C, the dehydration of the sample, the dehydroxylation of goethite and the decomposition of organic residues corresponded to the consecutive endothermic and exothermic peaks (observed in region (1)). The dehydroxylation of kaolinite and illite detected in NZ2 was noted in the range 400-600°C (endothermic peak region 2) and their subsequent structural reorganization occurred between 900°C and 960°C (exothermic peak due to metakaolinite crystallization in spinel or mullite, in region 4). The allotropic transition of quartz was illustrated in region (3) by the sharp endothermic peak close to 573°C. The shrinkage recorded after sintering at 1200°C was 3.42% and 2.46% for NZ2 and NZ2-10%MP samples, respectively. Moreover, the consolidation appeared to start at 1017 and 1010°C for NZ2 and NZ2-10%MP, respectively (derivative curves are presented in Figure S1). Indeed, the enhancement of consolidation onset point was not achieved when using NZ2 with 10 mass% MP, while the overall shrinkage at 1200°C was reduced by 30%, which could be related to microstructure changes due to MP degradation (increased porosity; Figure 3).

Results showed on Figure 3 were obtained from the diametrical compressive strength \( \sigma \), the thermal conductivity \( \lambda \) and the open porosity \( P \) measurements for the samples produced using NZ2 clay without modification and fired at 1000, 1100, and 1200°C with a dwelling time of 1 hour (at the maximal temperature). \( \sigma \) was not significantly changed (close to 3 MPa) with increasing sintering temperature; however, the porosity \( P \) was slightly decreased from 31% (at 1000°C) to 25% (at 1200°C). The thermal conductivity \( \lambda \) was increased from 0.58 (at 1000°C) to 0.88 (at 1200°C) ±0.03 W/m/K. It was shown that the increase in temperature led to the open porosity decreased from 31% to 25%, this decrease is probably related to some open pores that were closed during heating. The decreasing of the open porosity may also contribute to the densification of the material. The higher increase in thermal conductivity compared to the increase observed for the compressive strength in the range 1000 to 1200°C for NZ2 could be justified by the occurrence of crystalline phases with higher intrinsic thermal conductivity upon firing like mullite.

The chemical analysis of MP indicated that the chemical elements present in this waste, apart from carbon and oxygen 58.4 and 37.6 mass%, respectively (Figure 4), were potassium and low amount of silicon, sodium and aluminum oxides (Table 1). These results verified that MP was mainly constituted with organic materials in agreement with the great loss on ignition (≈96 mass%) recorded at 1050°C. The DTA/TG curves (Figure 5A) of MP highlighted the

| Oxide | SiO₂ | Al₂O₃ | Fe₂O₃ | TiO₂ | K₂O | Na₂O | MgO | CaO | P₂O₅ | LOI (1050°C) | SiO₂/Al₂O₃ |
|-------|------|-------|-------|------|-----|------|-----|-----|------|-------------|------------|
| NZ2   | 74.61| 12.57 | 3.50  | 2.74 | 1.08| 0.008| 0.29| 0.09| 0.07 | 4.80        | 5.94       |
| MP    | 0.31 | 0.08  |      |      |     | 6.46 |     |     |      | 95.8        | 3.88       |

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**Note:** Table 1: Chemical composition (mass%) of clay NZ2 and waste MP.
TABLE 2  Density, specific surface, and characteristic sizes of NZ2 and MP grains

| Sample | Density (± 10^{-3} g/cm^3) | BET specific surface area (m^2/g) | I_p | Mode 1 | Mode 2 | d_{10} | d_{50} | d_{90} |
|--------|-----------------------------|----------------------------------|-----|--------|--------|--------|--------|--------|
| NZ2    | 2.67                        | 20.24 ± 0.1                      | 8.3 | 1.98   | 51.47  | 1.01   | 2.01   | 61.45  |
| MP     | 1.54                        | 0.65 ± 0.03                      |     |        |        |        |        |        |

FIGURE 1  X-ray diffraction spectra of the NZ2 sample

FIGURE 2  Differential thermal (DTA)/TG curves and the thermodilatometry. A, DTA/TG of NZ2 clay and B, Dilatometry of NZ2 clay and NZ2-10%MP

corresponding degradation stages of the lignocellulosic components in the temperature range of 200-660°C (several exothermic peaks and overall mass loss of 96.6% at 1090°C).

These lignocellulosic components were also detected on the XRD pattern of MP (Figure 5B), corresponding characteristic peaks between 22 and 40 ° (2θ).43
Effect of the waste MP onto the final properties and the microstructure

For the same dwelling time while firing at 1100°C, the incorporation of 0%-15% by mass of MP in NZ2 clay favored the decrease of the compressive strength (≈60%) and of the thermal conductivity together with a significant increase in porosity (about 40%) compared to the initial values without MP waste (Figure 6).

The open porosity values obtained for kaolin BIP were correlated to the compressive strength values regarding MP content (Figure 7). Increasing the content of MP waste from 0 to 30 mass% within kaolin BIP samples led to an increase of the total porosity after sintering at 1000 and 1200°C, respectively.
A significant reduction (90%) of the compressive strength and an increase in open porosity values (∼50%) was obtained after addition of 30 mass% MP to Kaolin BIP and firing at 1000°C (dwelling time = 1 hour). In the case of the firing of the latter specimen at 1200°C (dwelling time = 1 hour), a more important decrease of the compressive strength (85%) and increase in open porosity (∼153%) was recorded. The porogen effect of MP was evidenced and correlated with the compressive strength, which offered the possibility of tuning the final characteristics of fired silicate ceramics accordingly.

From these two tests (kaolin and NZ2 series), we found that the addition of waste tended to decrease the compressive strength of the materials that was decreasing significantly with the increase in the waste content. Moreover, the thermal conductivity of the NZ2 samples decreased with the addition of 10 or 15 mass% of waste upon firing at 1100°C from 0.78 to 0.52 ± 0.03 W/m/K. This is due to the increased presence of pores in the structure of the materials produced. Indeed, the NZ2 sample has a very high rate of SiO₂ (74.61%) and low rate of Al₂O₃ (12.57%) and the SiO₂/Al₂O₃ ratio of 5.94 highlights the presence of free quartz in this sample. Furthermore, with the mixture of NZ2 and MP (10 or 15 mass%), the change in the dwelling time at 1100°C from 1 to 2 hour did not significantly affect their thermal conductivity values.

An important parameter for the production of ceramic materials based on clay is plasticity, but the NZ2 clay used for this
**FIGURE 7** Evolution of the open porosity and compressive strength of kaolin BIP-based samples sintered at 1000 and 1200°C for 1 h regarding the MP waste content.

**FIGURE 8** Scanning electron microscopy images of NZ2 samples.
study has low plasticity ($5 < I_p < 10$), which is at the origin of its weak consolidation even at 1200°C. This low plasticity is in line with the coarse grains that are included in the starting clay material and the relative high silica (quartz) content of this clay. Consequently, the addition of an organic material, which was entirely burnt during sintering led to the production of gases (carbon dioxide, water vapor, etc), giving rise to porous materials, lighter and less resistant if the sintering temperature could not allow the pores removal. Therefore, the addition of MP to NZ2 clay and the subsequent sintering at 1100°C only increased the porosity without improving the consolidation of the final product. In order to confirm these microstructure changes, we proceeded to the observation of several fired materials. Scanning electron microscopy was used to examine the surface of the materials produced and thus observe the morphological changes that occurred after adding the waste and sintering at different temperatures (Figures 8 and 9). We noted that the pores were heterogeneous both in size and in morphology while increasing the waste content. In the case of the kaolin BIP, the increase in porosity was well highlighted on Figure 9 according to the increase in MP. Besides, the NZ2 samples presented on Figure 8 exhibited a complex microstructure, dominated by the presence of large quartz grains. Therefore, it was difficult to assess the evolution in pore morphology when adding MP to NZ2.

4 | CONCLUSION

The main results obtained during this study focused on the use of plantain fruit skin to produce porous clay-based ceramics are summarized hereafter.

The local clay from Central African republic (NZ2), which was a silica-rich clayey raw material, exhibited an open porosity in the range 31%-31% upon firing in the range 1000-1200°C, respectively, and a low shrinkage (2.73%). This trend justified the consolidation and densification problems reported by the Central African Republic craftsmen regarding NZ2. Accordingly, the thermal conductivity and the compressive strength were increased after firing at 1200°C by 50% and 20%, respectively, compared to the values obtained after firing at 1000°C. This trend was due to the simultaneous consolidation of the samples and the occurrence of mullite while increasing the firing temperature.

The mixture of plantain skin (MP) with NZ2 or Kaolin BIP modified the characteristics of the materials obtained by the creation of pores and the degradation of mechanical properties. In fact, all the fired materials obtained with the incorporation of MP waste exhibited a decrease in compressive strength values and an increase of the open porosity values with increasing MP content. The compressive strength obtained with the Kaolin BIP-MP mixtures was higher than those obtained for the NZ2-MP mixtures. This behavior was related to presence of higher amount of nonreactive silica (quartz) and low alumina content in NZ2 compared to the kaolin BIP. Nevertheless, MP was proved to enhance pore formation during firing of both clay materials. Besides, the thermal conductivity of NZ2 with 10 or 15 mass% of MP led to a significant decrease of the thermal conductivity values after firing at 1100°C (soaking tie of 1 or 2 hours). The use of 15 mass% of MP with NZ2, allowed reaching a porosity of 40% after firing at 1100°C.
(dwelling time = 1 hour). In the case of kaolin BIP, open porosity values >40% were obtained by using 10 mass% of MP or 30 mass% of MP after firing at 1000 or 1200°C, respectively (dwelling time = 1 hour).

This study demonstrated the feasibility of using plantain skins intended for rejection as a blowing agent for the preparation of porous ceramics (with open porosity >35%). Indeed, the combine valorization of local mineral raw materials and plant waste in Central African Republic may bring novel opportunities to tune the microstructure of clay-based ceramics (insulating building or filtration materials) in a sustainable route.

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**SUPPORTING INFORMATION**

Additional supporting information may be found online in the Supporting Information section.

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