

Chemothermal and physical characterization of dura palm kernel shells as load and reinforcement for the production of clay pieces from clay soils (case of the Nomayos-Cameroon soil).

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ABSTRACT: We present in this work that clay soils are a clay deposit capable of being used for the manufacture of several materials and that it can admit plant fillers. Thus, we show here that palm kernel shells can be used as filler/reinforcer for the production of clay matrix composites. We have taken the palm kernel shells and the clay soil from Nomayos-Yaounde-Cameroon, we have made the respective mechanical treatments and have characterized them physically, thermally and chemically. We made a mixture of the treated clay with 30% of the shell powder and made the thermal and chemical characterization. For the physical properties, we used standard tools in accordance with the standards in force. For the thermal properties, we used a LENSEI apparatus to obtain the ATG, DSC and DTG. For the chemical properties, we used an Alpha spectrometer of the Bruker firm analyzed by the ATR technique on a diamond crystal to obtain the IRTF. We used a USB digital microscope with HD color CMOS sensor, high speed DSP (dricer free available), 24bit DSP, optimal resolution 640x480, 5x digital zoom to obtain the shots at a large scale the clay-powder mixture of palm kernel shell. We obtained that: the clay soil of Nomayos has a bulk density of 1.85 g/cm³, a porosity of 28.60%, a percentage of silt of 11% and clay of 58.4%, the limit of atterberg presenting 55% of liquidity limit, 26.6% of

plasticity and 28.4% of plasticity index; the hull powder has 0.681 g/cm³ as bulk density, 18.04% as water content The TGA, DSC and DTG showed: for the clay, the presence of free water, Kaolinite, illite and Quartz in important proportion affirming that this clay is of kaolinite type. For the shells, the presence of free water, cellulose, lignin; For the mixture, the presence of both free water, Kaolinite, illite, Quartz, cellulose, and lignin showing the presence of both clay and shells in the mixture. FTIR showed: for clay, the presence of adsorption bands at 2931 cm⁻¹ and 2865 cm⁻¹, absorption peaks at 1554 cm⁻¹, 1494 cm⁻¹ and 693 cm⁻¹ and a peak near 1307 cm⁻¹. For the shells, the presence of intensity bands at 2924.39 cm⁻¹, average intensity of the fine band between 1509.08 cm⁻¹ and 1606.26 cm⁻¹, intensity peaks between 1372.10 cm⁻¹ and 1317.91 then 1239.65-1030.05 cm⁻¹. For the clay-shell mixture, the presence of adsorption bands at 2931 cm⁻¹ and 2865 cm⁻¹ corresponding to asymmetric and symmetric elongation vibrations of the -CH₂ groups showing the presence of the clay silane. The intensity peaks between 1239.65 and 1030.05 cm⁻¹ can be attributed to the -C-O groups of alcohols, Esther, ether, amorphous silica or -C-O bonds of cellulose and lignins showing the presence of shells in the mixture. Finally, the microscopic observations showed the presence of both clay and hull powder.

KEYWORDS: Mechanical processing of clay soils; Mechanical processing of palm kernel shells Dura; ATG; DSC; RTIF.

I. INTRODUCTION

For thousands of years, earth materials have been used in the building industry. A long time ago, man used it in the construction of bricks of dried earth and then in terra cotta. The actors of the field do not cease innovating and going forward so that this art several times millennium can be compatible with the requirements of our time that it is in energy saving, materials or environmental protection. These advances are all the more necessary as the basic material, namely clay, is becoming increasingly scarce with the advent of more advanced techniques that allow for faster production rates and a renewed interest from consumers. Nowadays, many and varied techniques that can be an alternative to the exploitation of clay deposits are neglected yet they can be very beneficial in the field of building construction, roof tile manufacturing, for decoration. Clay has proven to be very beneficial in a house because of its numerous virtues. However, its use as a raw material in construction leads to a certain number of defects which makes it neglected in favor of other raw materials (sand, laterite). Indeed, a certain number of difficulties related to the use of clay in construction arise. These include the variation in volume (shrinkage and swelling) during the drying of the material causing many cracks thus reducing the mechanical performance of the latter. This problem is generally treated by adding stabilizers to the earth. The most commonly used stabilizers are cement, lime and sand [1]. The production of these stabilizers requires quite a lot of resources. Moreover, their production pollutes the environment by producing CO₂ in the atmosphere. On the other hand, natural stabilizers based on plants such as palm kernel shells are a way to fight against environmental pollution. This natural stabilizer, when mixed with clay, will oppose the shrinkage and swelling of the manufactured blocks, thus preventing the numerous cracks responsible for the low mechanical resistance of clay-based blocks [2]. Clay is also used to renovate old houses and half-timberings. Indeed, it allows to preserve them, by absorbing moisture so that it does not penetrate the wood material. Studies carried out in Cameroon show that there is a large deposit of clay throughout the country [3,4]. This clay is therefore sufficient to be used as materials in the construction of our habitats. In addition, the use of base extracted from plant ashes for the stabilization of clay blocks,

which is the ancestral technique, is more and more used. Indeed, the ash of the plants mixed with clay allowed a stabilization of the blocks of clay for the construction of the houses [1]. Based on this observation, it would be interesting to consider the possibility of using palm kernel shells for the stabilization of construction materials instead of the binders commonly used. This technique will allow to fight against the environmental pollution by the elimination of CO₂ in the atmosphere during the production of cement and moreover to make lower the cost of the building materials. Hence the objective of this study which is to make blocks of clay and stabilized with palm nuts shells (natural stabilizer). Thus, it aims to highlight and confirm that the shells of palm kernel dura is a stabilizer that improves the mechanical and physical characteristics of clay blocks. Today, clay constructions allow the construction of buildings on several levels. With the addition of stabilizers, these clay constructions will be able to increase their bearing capacity as well as their durability. In view of these advantages, why are they so little used?

Unfortunately, this technique with its numerous ecological, thermal and economic advantages is supplanted in many countries by concrete construction, a type of architecture that does not seem to be adapted to all environmental and social contexts. Indeed, it seems that raw earth construction has acquired a negative connotation (poor housing, poor quality, etc.). As a result, as soon as a person's financial capital rises, he rejects earth as a building material and turns to concrete and sheet metal. Indeed, these two materials, although they do not contribute to the local economy and often do not meet the requirements of the climate, refer to the image of European development, wealth, solidity and durability. Indeed, several works in the field of vegetable fillers have been the subject of research and theses, particularly in sugar cane fibers, hemp fibers [5] etc. The results have been verified and found satisfactory both in the properties of the material. As a result, the fillers usually used in the world of mud brick construction (raw and fired) are vegetable fillers. Observations show that the degradation of earth bricks, in association with these vegetable fibers is faster. Given the importance and rigor in the field of building construction, this problem becomes a danger to the lives of people (cracks, collapses ...). Research has come to the conclusion that it is necessary to find solutions to solve this problem. In recent years, experiments are becoming more and more favorable in the use of vegetable fillers for earth

materials (raw and baked). Palm kernel shells have been studied for their use as fuel [6], for the production of activated carbon [7, 8]. Vegetable fillers are in full success in the development of polymer matrix composites [9, 10]. The exploitation of palm kernel shells as filler for soil material leads to the valorization of this agricultural sector of the oil palm.

II. MATERIALS AND METHODS

A Materials

A-1) Study materials.

We study a clay matrix composite material from clay soil loaded (or reinforced) with palm kernel shell powder.

A-1-1) raw clay soil

The clayey soil of the study comes from Nomayos, city of Yaounde, Province of the center in Cameroon.



Fig 1: Geolocation of Nomayos on the map of Cameroon

Nomayos is a village in the Centre Region, located in the commune of Mbankomo in the Mefou-et-Akono department, city of Yaounde in Cameroon. Nomayos is home to a Lafarge Group cement plant with a capacity of 500,000 tons.

Figure 2 shows the annual rainfall of the city of Yaoundé and figure 3 shows the annual climatic situations of Nomayos according to the data recorded at the Ministry of Statistics.

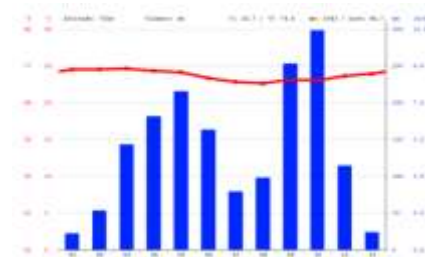


Fig 2 : Umbrothermal diagram of Yaoundé



Fig3: Climatic situation of the city of Nomayos

From figures 2 and 3, we obtain that January is the driest month, with an average of 22 mm of rainfall, while October is the month with the highest rate of rainfall with 298 mm, making it the month with the highest rainfall rate in Yaounde. Similarly, March is the hottest month of the year, with an average temperature of 24.6°C, while August has the lowest temperature with an average of 22.6°C, making it the coldest month of the year in Yaounde.

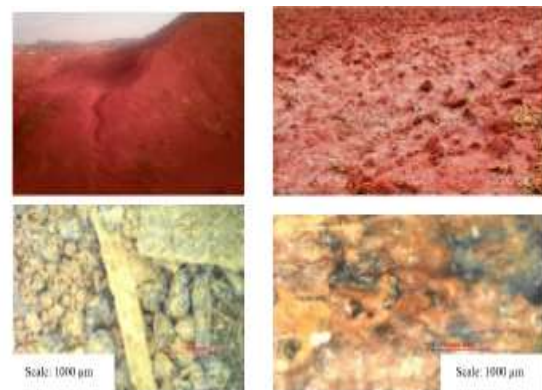


Fig4 : clay soil of the city of Nomayos (click DC001 of 12/01/2021)

Fig. 4 presents the images of the clay soil of Nomayos filmed in January 2021. We obtain that this earth has aggregates of all kinds. The microscopic observations show the necessity of making a mechanical treatment of this clayey ground in order to obtain the clay necessary to the manufacture of the earth materials.

In summary: clay soils are slippery soils making roads impossible. These soils are found in countries like Gabon, Botswana, Congo, Zimbabwe, Africa, Brazil, Argentina...

We find that the rainfall of these localities is similar and the territorial areas are quite large.

Thus, we demonstrate in this article that however we meet the slippery lands, it is the meaning of the presence of a strong quantity of clay that deserves to be exploited.

A-1-2) raw shell.

Raw dura palm kernel shells are obtained by crushing the palm kernel and removing the nut called fine. Several studies have been carried out on palm kernel shells, including those by Epesse missé and al in 2013, Djomi and al in 2018, Ernesto de la Tore in 2015..

Fig 5 (a) shows a cluster of palm kernel shells taken randomly from a bag and then Fig 5 (b) shows the morphology of the shells. These figures show the need to make a mechanical treatment of the shells before the manufacture of materials.

(a) (b)

Fig 5 : Morphology of raw palm kernel shells (click DC002 of 20/11/2020)

A-2) materials

A-2-1) for mechanical treatments : Crusher ; Oven ; Pycnometer ; Seditech 1/1000 scale ; Afnor sieve for granulometric analysis ; 1/1000 scale ; Erleu meyer.

A-2-2) for microscopic observations : USB digital microscope

A-2-3) for the thermal properties : A LENSEIS instrument with an alumina oxide crucible and a capacity of 150 mg. The heating rate is 10°/min. The gas used is oxygen and nitrogen.

The water in this basin is left to settle for 24 hours at room temperature. We release the water and the clay remains at the bottom of the basin (Figure 6d) which will be stored directly in a porous bag for dehumidification for 45 days at room temperature.

From palm kernel shell material : The process is as follows:

Figs 5a and 5b show the morphology of the hulls as they are collected from mining sites in the coastal region of Cameroon, in the Mounjo department. It is difficult to ensure proper mixing of clay and hulls to produce a good quality earth material. Thus, the laboratory centripetal mill described in the 2018 article by Djomi et al [11] is used. To obtain the desired particle size.

A-2-4) for Chemical Properties : An Alpha spectrometer from Bruker analyzed by the ATR (Attenuated Total Reflection) technique on a diamond crystal. The resolution during the collection of the spectra is fixed at 4 cm⁻¹, from the laboratory of physical and analytical chemistry of the University of Yaounde 1

A-2-5) for the mixture : An automatic mixer of table equipped with an agitator of mark Maurice PERRIER and Cie, 20 street Marie Debos 92120. MONTRouGE (France), Type 32, N0 651 of the laboratory of geotechnics of the national superior polytechnic school of Yaounde.

B) Experimental methodology

B-1) Mechanical treatment of the study materials.

Earth material : The purpose of this treatment is to remove aggregates and waste of any kind. It consists of extracting the soil in the study area (Figure 6a), stirring with water and an aluminum ladle for about 1 hour so that the soil is well mixed (Figure 6b). The slurry obtained is poured into a basin through a 500 micron filter cloth (figure 6c) in order to remove impurities (roots, stones, gravel...)

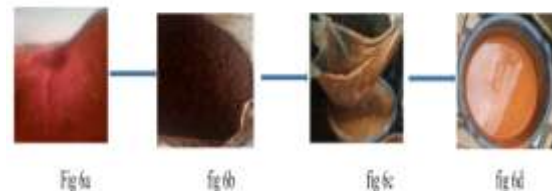


Fig6: process of the mechanical treatment of the earth of Nomayos (click DC003 of 20/11/2020)

B-2) characterization of the study materials

B-2-1) earth material: clay

Physical properties

Some physical properties were carried out in a company specialized in the Mission of Promotion of Local Materials (MIPROMALO) Yaounde-Cameroon according to the standards: Particle size analysis by dry sieving after washing (standard NF P 94-056), Particle size analysis by sedimentometry (standard NF P 94-057), Atterberg limits (standard NF P 94-051), Water content (standard), Methylene blue tests (standard NF P 94-068), Real density (standard NF P 94-068). Several works aimed at determining the properties of clays and aggregate materials have used the same standards [12, 13, 14, 15].

Thermal properties

According to the work of Djomi et al in 2018 and then Olembe et al in 2021 the methodology applied for the determination of thermal properties of clays is the one applied in their work [11, 16].

Chemical properties

An Alpha spectrometer from Bruker analyzed by the ATR (Attenuated Total Reflection) technique on a diamond crystal. The resolution during the collection of the spectra is fixed at 4 cm⁻¹, from the laboratory of physical and analytical chemistry of the University of Yaounde1. The method used is by attenuated total reflectance (ATR) whose methodology is standard. The data acquisition is done with the help of a software incorporated in the apparatus which allows the recording and the plot of the spectra automatically on a computer. We are provided with recording data and FTIR spectra [16].

B-2-2) Shell materials

It is the powder of palm kernel shells previously crushed.

Physical properties

The physical properties such as permanent moisture content, bulk density or poured density, wetted density, water absorption rate were calculated in accordance with the method used by Djomi and his team in 2018 when they used palm kernel shells as a filler for polymers in the manufacture of plastic materials [11].

Thermal properties

According to the work of Djomi et al in 2018 and then Olembe et al in 2021 the methodology applied for the determination of thermal properties of the shells is the one applied in their work [11, 16].

Chemical properties

The machine of the test is an Alpha spectrometer of the Bruker firm analyzed by the ATR technique (Attenuated Total Reflection) on a diamond crystal. The resolution during the collection of the spectra is fixed at 4 cm⁻¹. The methodology is as described above.

B-2-3) Routine test (Mixture of clay and cockle powder).

Schematic of the mixing process.

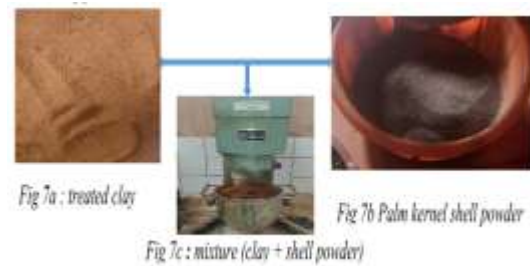


Fig 7: Mixing process of clay and palm kernel shell powder

Laws of mixtures:

According to the work of Huisken and al in 2022, the Laws of Permanent Mixtures are calculated according to the relation (b.1) and applied in this work [17]

$$\begin{cases} M_f = \frac{\rho_f}{\rho_c} V_f \Rightarrow M_f = \frac{\rho_f}{\rho_f V_f + \rho_m (1 - V_f)} V_f \\ M_m = \frac{\rho_m}{\rho_c} V_m \Rightarrow M_m = \frac{\rho_m}{\rho_f V_f + \rho_m (1 - V_f)} V_m \end{cases} \quad (b.1)$$

Mixing

The masses of the clay are calculated (Figure 7a) and poured into the mixer (Figure 7c). The masses of the palm kernel shell powder (Figure 7b) are calculated and poured into the mixer (Figure 7c). The mixing is carried out (figure 7c) in the laboratories of geotechnics and materials of the National Polytechnic School of Yaounde (ENSPY), using an automatic mixer of table of mark Maurice PERRIER and Co, 20 rue Marie Debos 92120. MONTRouGE (France), Type 32, N0 651. it is the total homogenization of the paste ready for the production of bricks. The mortar of clay and palm kernel shells is mixed and kneaded, this to get as close as possible to the model of a homogeneous and isotropic material [18].

Characterization of the mixture

Two intrinsic properties of the mixture are necessary to ensure the quality of the products to be made with the mixture of nomayos clay and palm kernel shells and to obtain a product that complies with the standard. These are the thermal properties and the chemical properties.

Thermal property of the mixture

The machine and methodology are those described in the thermal analysis of clays.

Chemical property of the mixture

The machine and the methodology are those described for the chemical analysis of clays.

III. RESULTS AND DISCUSSION

A-1) Result of the clay

A-1-1) Results of the mechanical treatment of the clay soil of Nomayos.



Fig 8a : Treated and dehumidified clay.



Fig 8b : Treated and dehumidified clay seen with a USB digital microscope
 Fig 8 : result of the Nomayos clay treatment

Figure 8 shows on the left (fig. 8a) the aspect of the clay after dehumidification, then on the right (fig. 8b) the aspect of the clay seen under a microscope of 1000 scale. This aspect allows us to observe the granulometry of the clay which will receive the shell powder during the mixing.

A-1-2) Results of the physical characterization of the nomayos clay

Table 2: Results of the physical characterization of the Nomayos clay

Granulometric analysis					
Ref	Color	% of gravel $\Phi > 2$ mm	% of sand $2 > \Phi > 0.02$ mm	% of silt $0.02 > \Phi > 0.002$ mm	% of clay $\Phi < 0.002$ mm
AR	Reddish	7.0	23.6	11.0	58.4
Limit of Atterberg					
AR	Liquidity limit (%)	Plasticity limit (%)	Plasticity index (%)		
AR	55.0	26.6	28.4		
Water content, density, Vbs and porosite					
Water Content (%)	VBS	Apparent Density (g/cm^3)	Real Density (g/cm^3)	Porosity (η) (%)	
29.1	1.43	1.85	2.06	28.60	

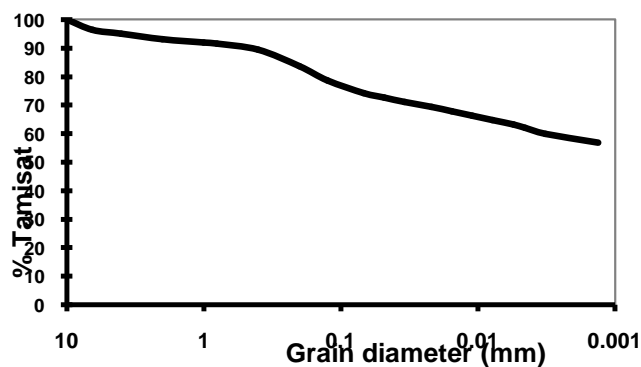


Fig 9 : Nomayos clay granulometric curve

The table 2 and the fig 9 coupled present the different results obtained from the physical characterizations of the clay obtained after the mechanical treatment of the clayey ground of the region of Nomayos. It appears: To the granulometric analysis that the clay is reddish in color, with sand and 7% gravel.

At the Limit of Atterberg that the liquidity limit is 55%, the plasticity limit is 26% and the plasticity index is 28.4. At the real density it is 2.06g/cm³ with a bulk density of 1.85, a porosity rate of 28.60%, a water content of 29.1% with a VBS of 1.43. These compositions show that with the high rate of clay, the high rate of plasticity and liquidity and

that of silt, we reassure that this clay soil is perfect for the creation of industries in the fields of pottery, decoration, construction of mud houses, in the manufacture of roofing tiles, in the production of handmade bricks and stabilized bricks for construction and decoration of buildings

With the rate of VBS and gravel coupled with sand, we are reassured of their perfect use in the pavement of slippery roads as pavement provided that it is baked.

**A-1-3) Results of gravimetric and differential thermal characterizations of the treated clay.
 Results of the thermogravimetric and differential analysis of the treated clay**

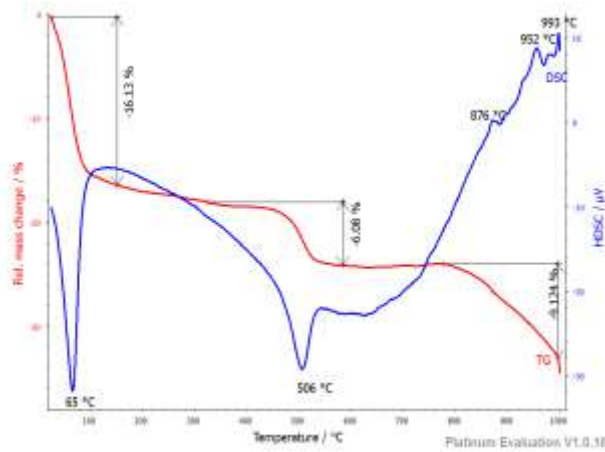


Fig 10 :TG and DSC thermogram of palm kernel shell powder.

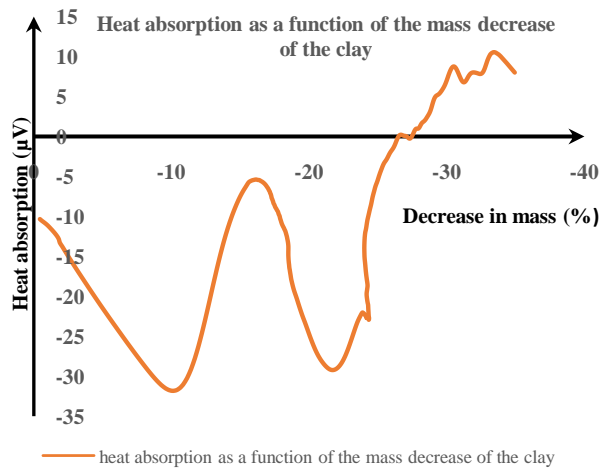


Fig 11 : heat absorption as a function of the mass decrease of the clay.

Fig. 10 is the thermogram of the Nomayos clay. We obtain in red the thermogram TG and in blue the thermogram DSC.

- At 65 °C, we have an endothermic dip at the DSC which corresponds to the departure of hygroscopic water and zeolitic water from illite; the percentage of mass loss determined from the ATG and is about 16.13 %;
- At 506 °C a second endothermic trough marks the departure of the water of constitution of

Kaolinite and its transformation into metakaolinite; here the loss of mass is estimated at 6.08 %.

- At 565 °C; we have an endothermic peak which highlights the presence of quartz. This temperature marks the allotropic change of quartz. It passes from quartz α to quartz β . This reaction does not involve any loss of mass.

- At 952°C, we have an exothermic peak which indicates the formation of mullite from metakaolinite.

Going through the literature on clays and their use in life around us, we get similar results to some works like those of Argin et al, 2021 in their work on "Pozzolanic activity of calcined halloysite-rich kaolinitic clays" [19], Ouedraogo, K. A. J in 2019 in their work on "Synthesis and characterization of geopolymeric binders based on local materials of Burkina Faso for stabilization of Compressed Earth Bricks (CEB)" [20].

Similarly to Figure 10, Figure 11 shows that in the vicinity of 30μv, we obtain an endothermic peak with a mass decrease close to 10% corresponding to the disappearance of free water which is obtained in the vicinity of 65°C at DSC (Figure 10). Also, during the thermal degradation of our clay, we obtain in the vicinity of

506°C a decrease in mass estimated at 6.08%. This mass decrease is obtained with a heat discharge up to a neighborhood of 5 μv. Again the rest of the degradation presents a heat absorption equivalent to 28 μv by losing on average 22% of its mass (Figure 10). Finally, the clay behaves as if they release heat as the temperature increases without, however, decreasing in mass significantly. At times, abrupt degradations arise marking the presence of the transition from quartz α to quartz β, this is the case of the peaks in the vicinity of: -22 μv, -0.1 μv as we obtained at 565°C has the DSC as Laibi in 2017 in his work [21].

This phenomenon of heat absorption as a function of the decrease in mass highlights the phenomenon of the degradation of our materials obtained from Nomayos clay. This phenomenon was observed during the study of the physical characteristics of the Nomayos clay.

A-1-4) Results of chemical characterizations by infrared transform of the treated clay.

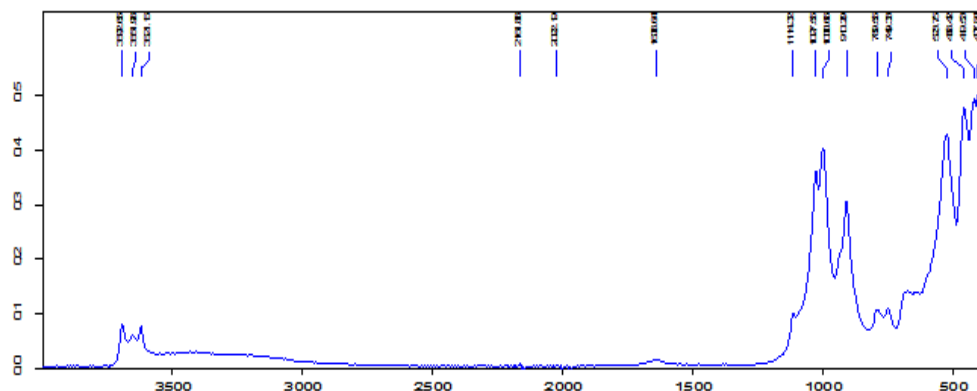


Fig12 : infrared spectral analysis of the clay

The infrared transform spectra of the clays show an intense peak at 910.29 cm⁻¹ and a band at 523.75cm⁻¹ related to the elongation and torsion vibrations of Si-O bonds respectively. The broad bands at 3651.16 cm⁻¹ and 1635cm⁻¹ are attributable to elongation and deformation vibrations of -OH and water molecules absorbed in the interfoliar space of the clay sheets [22]. These results highlight the presence of two new adsorption bands at 2931 cm⁻¹ and 2865 cm⁻¹ corresponding to asymmetric and symmetric

elongation vibrations of the -CH₂ groups, which shows the presence of silane in the clays. Similarly on the elongation and deformation vibrations, then on the asymmetric and symmetric elongations of the groups [23].

The presence of new absorption peaks at 1554cm⁻¹, 1494cm⁻¹, and 693cm⁻¹ corresponding successively to deformation vibrations of -NH₂ and -CH₂ and out of plane deformation of -CH. The peak at 1307cm⁻¹ is attributed to an elongation vibration of C-N [19, 21, 24]

A-2) Results of the palm kernel shells

A-2-1) Results of mechanical processing of palm kernel shells.

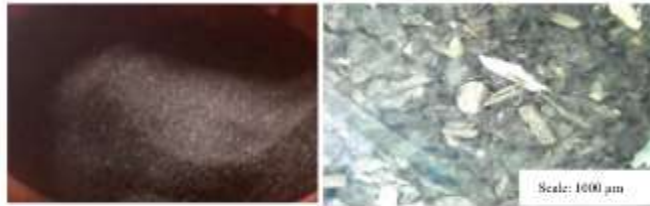


Fig 13a: Palm kernel shell powder obtained at a particle size of 500 μm

Fig 13b: Palm kernel shell powder seen with a USB digital microscope

Fig 13 : shell treatment result

Figure 13 shows on the left (fig.13a) the aspect of the palm nut shell powder and the (fig.13b) the aspect of the palm nut shell powder seen under a microscope of 1000 scale. This aspect allows us to evaluate the granulometry of the hull powder to fill the gaps (void) between the grains of the clay after shaping

A-2-2) Results of the physical characterization of palm kernel shell powder

Result of the moisture content of the powder

The records obtained during the test for the determination of the moisture content of the cockle powder give us the value of the average moisture content of :

$$T_m = 1.78\% \mp 0.19$$

Density result

a. Result of bulk density.

From the records obtained after the tests, the average bulk density of the hull powder is :

$$\rho_a = 0.599 \mp 0,03 \text{ g/cm}^3.$$

b. Wet Density Result.

From the record obtained the average wetted density of the hull powder is :

$$\rho_m = 0.500 \mp 0,01 \text{ g/cm}^3 .$$

Discussion : We summarize that the actual density of the hull powder ρ_r is between

$$0.500\text{g/cm}^3 \leq \rho_r \leq 0.599\text{g/cm}^3$$

It appears that the poured density tells us about the quantity of the powder during the mixing. The wetted density tells us about the density of the manufactured materials (mud bricks, roof tiles...). So, we get that both values are relatively lo

This low value of the powder density offers an advantage to the manufactured materials because the mass will be relatively low. This is a particular advantage when using palm kernel shells as filler in the manufacture of materials for the construction of high rise buildings

.Dry particle size analysis results

The starting dry mass is $M= 7.21\text{Kg}$

Result of the water absorption rate

The experimental results for the calculation of the water absorption rate of hull powders obtained are recorded in Table 4.

Table 4: Results of water absorption tests on dura palm kernel shells.

	Experiment 1	Experiment 2	Experiment 3
Initial mass M in kg	0.750	0.800	0.500
Mass M' after 24h in kg	0.879	0.927	0.605
Absorption rate Tai in %.	17.200	15.900	21.02
Average absorption rate Ta in %.	18.04		
Standard deviation of the absorption rates	2.661135304		

The absorption rate is 18.04%. Compared to the absorption rates of some works [25] we note that the results are very close.

We summarize By saying on the physical properties of the shells that, as we said in the methodology applied experimentally, we obtained results substantially identical to those obtained by

Djomi and his team in 2018 [11] during his work that required him to physically characterize the shells of palm kernel. Also by checking the works cited [7 ,8] and comparing our results to all of them, we obtain that the results are very close reassuring the quality of our experiments.

A-2-3) Results of gravimetric and differential thermal characterizations
Thermogram TG and DSC of thermogravimetric and differential analysis.

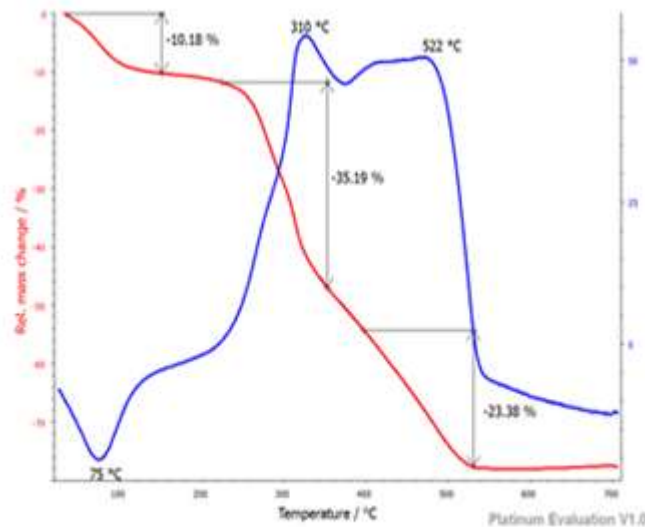


Fig 15: Thermogram TG (in red) and DSC (in blue) of palm kernel shell powder

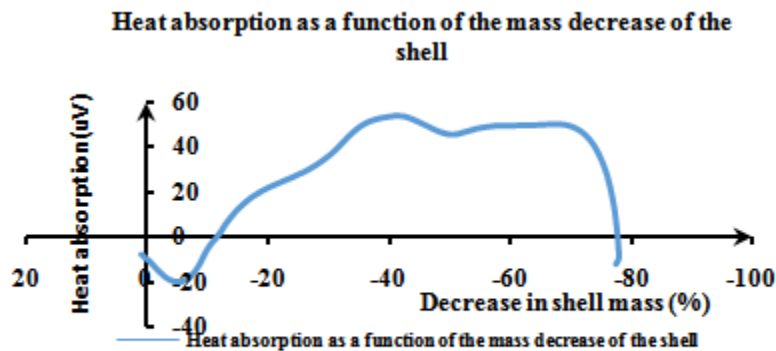


Fig 16: Heat absorption as a function of decreasing hull masses

Figure 15 is the thermogravimetry in red and the thermodifferential in blue of the hull powder. It allows us to see figure 16, the curve of the heat absorption of the hulls as a function of the decrease of its mass during its rise in temperature.

The analyses are done in order to understand the phenomenon of palm kernel shell

degradation as a function of temperature. The DSC thermogram in Figure 15 in blue shows an endothermic heat peak at 75 °C. This is the dehydration of the powder presented by the TG figure 15 in red with a mass loss of 10.18%. The DSC shows in addition 2 representative and sensitive peaks of the degradation. These are the

peaks at 310 °C and 522 °C. The peak at 310 °C is the peak representing the transition between cellulose and lignin. The 2nd peak (522 °C) shows the beginning of the total degradation of the powder, it is the transition from lignin to ash. The passage from 75 °C to 310 °C shows on the DSC level that a very high energy is needed to make the cellulose leave, which represents 35.19 % of the initial mass of the hull powder. Similarly, the peak at 310 °C and 522 °C show that the degradation of lignin does not require high energy because lignin represents only 23.38% of the mass of the hulls present. The ash is found between 560 °C and 600 °C where the remaining material does not absorb or release heat. Note in passing that the shells of palm kernel do not have hemicellulose. For this reason, we note that the shells of palm kernel do not rot in the face of moisture or water. It should be noted that several works in the literature have

brought the same results and the same observations [7,11].

Similarly to Fig. 15, Fig. 16 shows the behavior of the hull powder when it absorbs heat. It shows that to release free water, the hulls absorb a maximum of heat to lose an average of 10.18% of its mass, after it gradually releases heat until an average of 50µv, decreasing about 39% of its mass. This is due to the total absence of hemicellulose and the direct passage of free water to the lignin requires a very high energy. Let us note that after having released this strong heat, the hulls maintain this heat constant for a long time but by decreasing considerably the mass until its total degradation. These results are close to those obtained by Djomi and his team in June 2018 in his research work on Physicochemical and thermal characterization of Dura palm kernel powder as filler for polymers: Case of polyvinyl chloride [11].

A-2-4) Results of chemical characterizations by infrared transform.

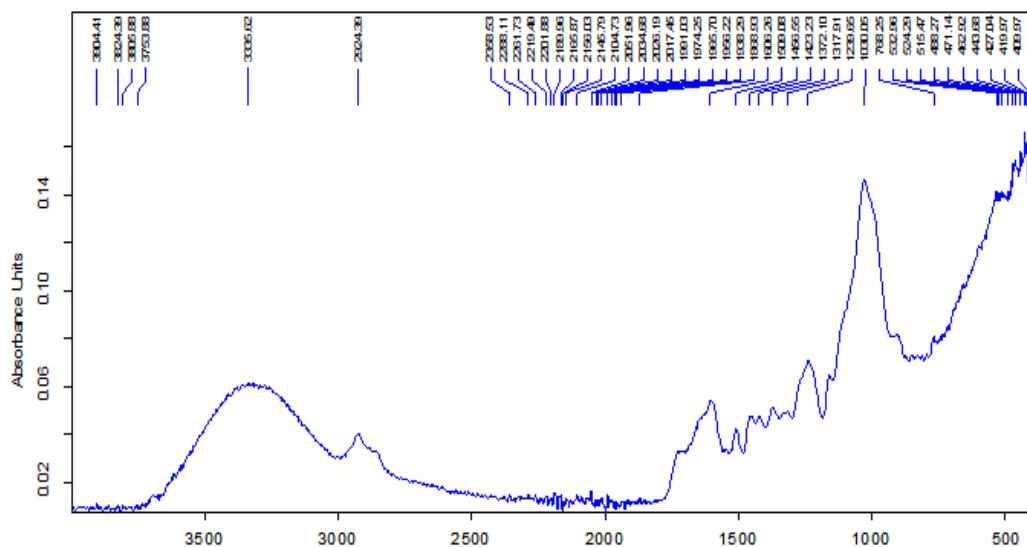


Fig 17: Fourier transform infrared spectrum of hull powder.

The FTIR spectrum of palm kernel shell powder in Figure 17 shows different intensity peaks at ;3335.62 ;2924.39 ;2017.45 ;1606.26 ;1509.08, 1239.65 and 1030.05 cm-1; The broad intensity peak at 3335.62cm-1 is the elongation vibration of the O-H groups of alcohols (cellulose contained in the shell powder). The intensity at 2924.39 cm-1 is the elongation vibration attributed to the -C-H groups of alkanes or the vibrations of methoxyls of the lignin group. The average intensities of the fine band between 1509.08 and 1606.26 cm-1 are deformations that can be attributed to C=C groups in an aromatic ring or carboxyl groups of the lignin. The peaks of average

intensities of the fine band between 1372.10 cm-1-1317.91 are -C-H vibrations of symmetrical deformation and other peaks between 1239.65-1030.05 cm-1 can be attributed to -C-O groups of alcohols, esters, ethers, amorphous silica or -C-O bonds of cellulose and lignins. Going through the works done on infrared spectral analysis in the literature, we obtain the very close results as especially those of Zhang and al in 2012 [26], Kundu and al in 2015 [27], Hidayu and al in 2016 [8], djomi and al in 2018 [11] and Ernesto de la tore in 2015 [7].

A-3) Results of the mixture of clay and palm kernel shells

A-3-1) Results of the law of mixtures

We have chosen a mixture of 30% of palm kernel shell powder in order to make feel the mutual

behavior of clays and shell powder during their study. We obtained the table 5:

Expense rates	Mass of shells (g)	Mass of clay(g)	Mass of the mixture (clay + hull powder)
0%	0	527.36	527.36
30%	56.1408	369.52	425.2928

Table 5: Laws of material mixtures

From table 5, we obtain that we took a mass of clay of 527.36g for the mixing at 0% of load with the hull powder. Similarly, we took 369.152g of clay and 56.1408g of the hull powder for a mass of mixture of 425.2928g representing a dosage of 30% hulls in the clay, the whole being mixed, we take samples for analysis before the shaping

We obtained figure 18 pictures representing the clay mixed at 30% with the hull powder, seen with a USB digital microscope with HD color CMOS sensor, high speed DSP (dricer free available), 24bit DSP, optimal resolution 640x480, digital zoom 5x .

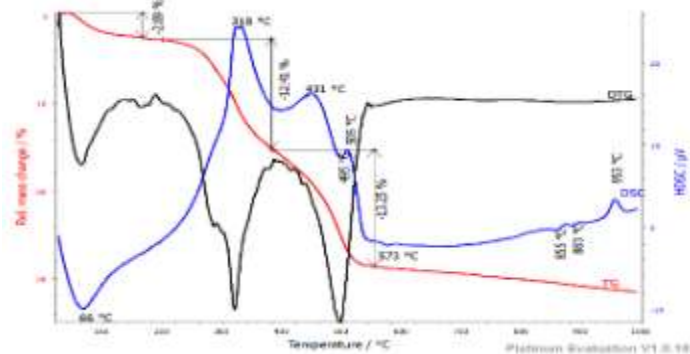


Fig 18: mixture of 70% treated clay and 30% palm kernel powder seen with a USB digital microscope.

From fig 18, we obtain that the mixing was homogeneous showing both treated clay of reddish coloring and palm kernel shell powder of black coloring this observation justifies the object

of our work namely the feasibility of the production of earth bricks loaded with palm kernel shell powder.

A-3-2) Results of gravimetric and differential thermal characterizations



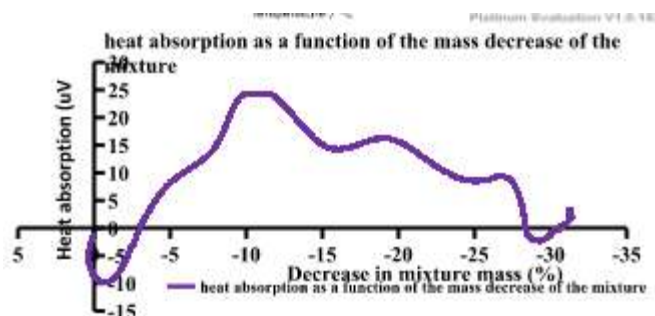


Fig. 19 is the thermogravimetric and differential of the mixture of the clay of the clay soil with 30% of the powder of palm kernel shells. It allows to bring out the fig 20 which is the curve of the absorption of the heat of the mixture according to the decrease of its mass during its rise in temperature. We note on the mixture of curves of DTG the presence of hulls and clay at the same time.

Thus, the first endothermic peak observed at 66°C of our mixture of clay + palm kernel powder in Figure 19 corresponds to the departure of the water retained on the surface of the clay and the shells. This justifies the presence of water in the clay and water in the hull powder so the dehydration temperatures are 65°C and 75°C. The percentage by weight according to the ATG curve is about 2.89%, this rate is mainly due to the percentage in this mixture of the fine fraction of the clay and the fine fraction of the hull powder Séraphin Agré djomo and his team in 2019 presented symmetrical results during their work on the stabilization of clay blocks with a natural base, potash [28].

The exothermic peak observed at 318°C is the peak representing the transition between

cellulose and lignin [29]. This transition shows at the DSC level the departure of cellulose which represents a loss of 12.41% of the initial mass of the hull powder. Similarly, the peaks from 318 °C to 505 °C show the degradation of lignin with a mass loss of 13.25 %. The ash is noticed between 573 °C and 600 °C where the remaining material does not absorb or release heat [30].

At 505°C; a second endothermic peak marks the departure of the water of constitution of Kaolinite and its transformation into metakaolinite.

At 573°C; we have an endothermic peak which highlights the presence of quartz. This temperature marks the allotropic change of quartz. This phase does not lead to a loss of mass.

At 953°C, we have an exothermic peak which indicates the formation of mullite from metakaolinite. At this phase we no longer have the presence of palm kernel shell powder. Here we see the presence of clay constituents such as: kaolinite, illite and quartz. These results of thermal analyses ATG, DTG and DSC show the presence in this mixture of the powder of the shells of palm kernel and the clay of the clayey ground of Nomayos.

A-2-4) Results of the chemical characterizations by infrared transform of the mixture.

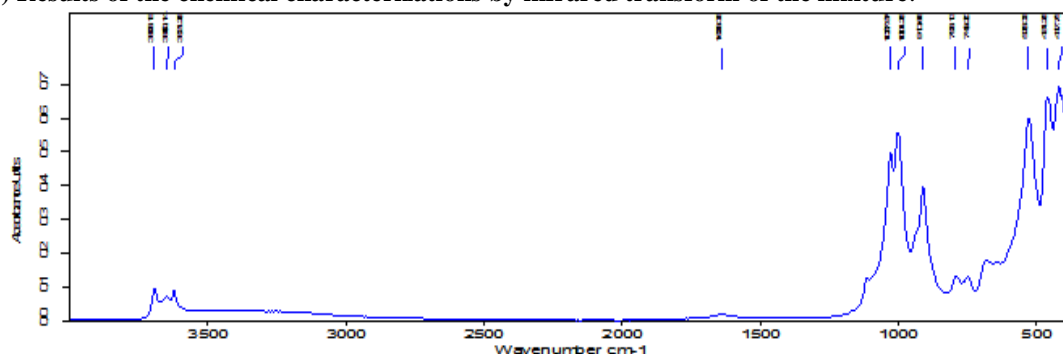


Fig 21: FTIR spectrum of the 30% mixture of palm kernel shell powder.

The infrared spectrum of the clays show an intense peak at 910.29 cm⁻¹ and a band at 523.75cm⁻¹ related to the elongation and torsion

vibrations of Si-O bonds respectively. These results are similar to those obtained in the literature [22]. The broad bands at 3651.16 cm⁻¹ and 1635cm⁻¹

are attributable to the elongation and deformation vibrations of -OH and water molecule absorbed in the interfoliar space of the clay sheets, the results obtained highlighting the presence of two new adsorption bands at 2931 cm⁻¹ and 2865 cm⁻¹ corresponding to asymmetric and symmetric elongation vibrations of -CH₂ groups, which shows the presence of the silane in clays. These results obtained on the elongation and strain vibrations and then on the asymmetric and symmetric elongations of the groups are close to the results of Risite, shanmugaraj and Hongping, in their works on clays in 2015, 2006 and 2005 respectively [19, 22,24].

The presence of new absorption peaks at 1554cm⁻¹, 1494cm⁻¹, and 693cm⁻¹ corresponding successively to deformation vibrations of -NH₂ and -CH₂ and out-of-plane deformation of -CH. The peak at 1307cm⁻¹ is attributed to an elongation vibration of C-N [21,24]. We find that the FTIR results of the clay are almost similar to those of the mixture of clay, palm kernel shell powder loaded at 30%. In view of this finding and based on the identical result of Belver et al [31] we can say that the palm kernel shell powder did not destroy or penetrate the crystalline structure of kaolinite, so does not influence chemically during the mixing.

B) Discussions générales.

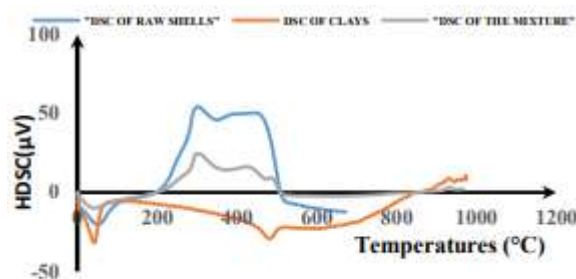


Fig 22a :

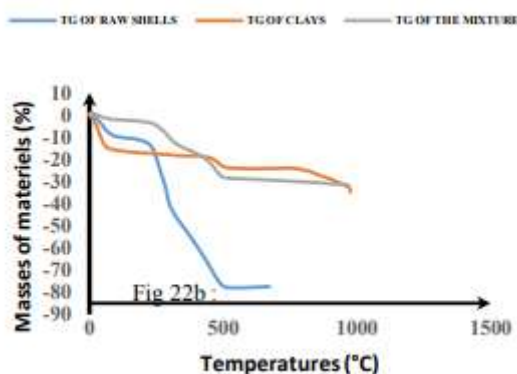


Fig 22b :

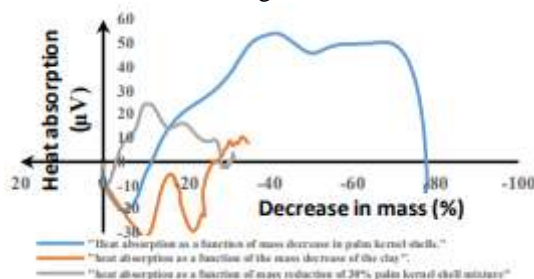


Fig 22c :

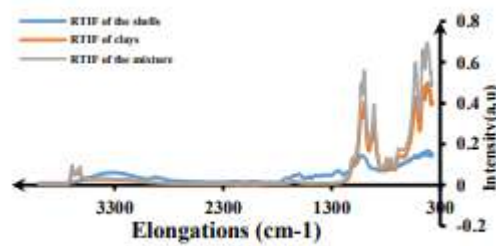


Fig 22d :

Fig 22: different thermograms from the machine recordings for the 3 materials: (Fig 22a: TG of clays, hull powder and mixture; fig 22b: DSC of clays, hull powder and mixture; fig 22c: heat absorption as a function of mass decrease of clays, hull powder and mixture; fig 22d: FTIR of clays, hull powder and mixture).

We obtained in the physical properties of the clay a rather large density compared to that of the hulls. This proves to us that the mixture of hull and clay will give a sufficiently light composite because the hull powder will lodge in the interstices left by the clay seeds. This behavior will help the composite to be compact because there will be no void. This advantage will be reflected in the quality of the results during the mechanical characterization of the material, thus, the greater the loading with the hull powder, the smaller the voids between the clay seeds and the better the quality of the composite. Added to that, the more the quantity of the load with the shell powder is big, the more the material is light because the density of the shell powder is 0.500g/cm³ against 2.18g/cm³ for the clay. This advantage particularly retained at the level of the physical properties makes this work rather considerable today that we seek the materials of constructions rather light, aesthetic and durable for the buildings at several levels (case of the stabilized bricks for the construction buildings). This phenomenon is remarkable in fig 22b where the mixture powder reassures us that as we heat the composite, our product loses enough mass, which will give an advantage to the firing of our mixture in its lightness. Moreover, the rate of water absorption of the clay is high compared to that of the shells, this proves that the mixture will provide a solution to the degradation of the composite. Also the granulometric analyses obtained on the clays and the density of the palm kernel shell powder show that the composite obtained from the clay-shell mixture will give a light material with very high mechanical resistances. This is a great advantage to the composite that we are studying at a time when we are looking for light materials in construction. Thus, fig 22a shows that this mixture can reach sufficiently high temperatures with the presence of kaolinite, illite and quartz showing its good attitude to be used for the manufacture of low temperature

clay bricks and light roof tiles. At the same time fig 22b shows that the mixture includes at low temperature (higher than 600°C) the simultaneous presence of kaolinite, illite and quartz coming from the clay and lignin coming from the powder of palm nut shell. These materials, namely kaolinite, illite, quartz and lignin, will provide good thermal resistance and stability to the material when fired.

Fig. 22c shows at the level of the shells that it gives off heat to lose free water, in the same way as the clay. This phenomenon is felt in the mixture. We observe that the hull powder abruptly absorbs heat as it rises in temperature, then retains it for a long time by greatly decreasing its mass. This heat retention phenomenon is felt on the mix. This is a considerable advantage for building constructions in regions where the temperature is very low (icy regions), for a high content of palm kernel shell powder in the clay, the heat retention in the building will last as long as the shell powder has absorbed heat.

Finally, Figure 22d shows that the vibrations around 4000 Cm⁻¹ characteristic of the presence of lignin from palm kernel shells are still found in Figure 22b when raised in temperature. Moreover, in the vibrations around 910.29 cm⁻¹, 523.75cm⁻¹, 1635cm⁻¹, 2931 cm⁻¹, 2865 cm⁻¹ observed in the mixture, shows the accentuated presence of carbon dihydrogen bonds (-CH₂). This shows that the clay will be present in the mixture and will bring great efficiency to the clay-shell cohesion of our material.

IV. CONCLUSION

The use of local materials requires the control of its physical, chemical and thermal properties. For this reason, samples of clay from the Nomayos earth and dura palm nut shells were subjected to a series of characterizations. The physical characterizations revealed that the apparent density of the clay is 1.85 g/cm³, a water content of 29.1% and a porosity of 28.60% and that

of the shell powder is 0.681 g/cm³ with a water content of 18.04%. Chemical analysis indicates that our clay has mainly three (3) oxides namely silica (SiO₂), alumina (Al₂O₃) and iron oxides (Fe₂O₃). Our palm kernel shell powder has 51% of carbon (C), 8% of hydrogen (H), 32% of oxygen (O), 0.1% of nitrogen (N), 3.9% of water (H₂O) and 5% of ashes (K₂CO₃) The thermal analyses allowed to identify and confirm the minerals contained in the samples of clays, shells and the mixture clay-shell. Thus, it appears that the clay contains kaolinite, illite, and quartz and the palm kernel shell powder contains mostly cellulose, hemicellulose and lignin. The mixture of clays and palm kernel shells shows that in regions with very low temperatures, the use of fired bricks is recommended for building construction. This will allow the conservation of heat in the building for a long time and will be a great advantage for the constructions of several levels which is the solution to the problem posed in the introduction. This will allow us to continue the work on the composite clay-shell of palm kernel in order to find a sustainable, economic and ecological solution for our constructions.

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