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Study of Clay Sites by the Method of Physico-Chemical and Thermal Characterisation of Clays: Case of the DIBANG -CAMEROON Site

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Abstract— *The objective of this work is the identification of a natural clay from the Central Cameroon region: "Dibang". We made a descent in the locality. We extracted the clay then made an artisanal and effective mechanical treatment before subjecting it to physical, chemical then thermal characterizations in accordance with the various standards. The result of the mechanical treatment allowed us to have pure clay. The results of the physical characterizations gave a density between 1,21 and 2,09 g/cm3, a water content of 48,25% and a porosity of 54,49%; the TG and DSC curves showed the presence of Kaolinite, Quartz and illite in important proportion affirming that this clay is of the silty type with a strong rate of silts. The FTIR showed the presence of -Si groups of silicon, -C-O of amorphous silicas, indicating that it has a majority of three (3) oxides namely silica (SiO2), alumina (Al2O3) and hydrogen nitrates (NH2). These results affirm that this clay can be used as a binder in the construction of buildings and also for coatings. Finally, the results of physical analysis such as plasticity, thermal analysis and chemical analysis, reassure the ideal advantages of Dibang clay in the production of tiles and stabilized fired bricks because it has a good thermal stability at low temperature, which proves a good stability to degradation.*

Keywords— *ATG/DSC of clays, Basic physical properties of clay, FTIR of clays,* ***,*** *Mechanical processing of clays.*

I. Introduction

The clays occupy a dominating place in the whole of the sedimentary rocks. Clayey rocks are formed by mixtures of clay minerals, to which are associated allogenic minerals (Quartz, feldspars, micas, heavy minerals) or authigenic minerals (anatase, sulfates, etc.). In reference works, different definitions of clay are proposed. For example, Eslinger define clays as a mineral that dominates in the fine fraction < 2 microns of rocks and soils [1]. In contrast, Weaver groups all phyllosilicate minerals without any size connotation and proposes to use the term "physils" to avoid confusion [2]. The mineralogical and physico-chemical properties of clays are of particular interest in many applications including water treatment, paint, construction, barrier for pollutants, adsorbent, catalyst [3] [4] [5]. The interest given in recent years to the study of clays by many laboratories in the world is justified by their abundance in nature, their low cost, and the presence of electrical charges on this surface and especially the exchangeability of inter-foliar cations. The behavior of clays in the context of clay/pollutant interaction explains the numerous works concerning the exchange reactions in the clay-water system [6] [7] [8] [9]. Clay has widely demonstrated its beneficial contribution within the home, as its virtues are numerous (decoration, availability, and cost). However, its use as a raw material in construction has a number of drawbacks such as its weight, which makes it neglected in favor of other raw materials (sand, laterite). Unfortunately, this technique with its numerous ecological, thermal and economic advantages is being supplanted in many countries by concrete construction, a type of architecture that does not seem to be adapted to all environmental and social contexts. Presenting a negative connotation of poor quality housing. Indeed, a number of difficulties related to the use of clay in construction arise, including the variation in volume (shrinkage and swelling) during the drying of the material, causing many cracks that reduce the mechanical performance of the latter. This problem is usually addressed by adding stabilizers to the clay with the most commonly used stabilizers such as cement, lime and sand [10]. Clay is also used to renovate old houses and half-timberings. A number of observations by traveling through areas where the penetration of water in the soil takes enough time, added to the fact that the roads are muddy and slippery (case of the roads of the impossible as in Katanga in DRC, in the west and east of Cameroon, in Tanzania and many other roads). We get from previous work on clays that the percentage of clays in the land is high [11]. These regions should have a great number of advantages because these clays would turn into a very specific clay ore deposit. The difficulties contained in this type of reflection consist, at the time of its extraction, of the dirt that the clay possesses. Therefore, in order to obtain a specific quality of ceramic materials of high level from these clays, it would be desirable to proceed to a mechanical treatment whose result will allow to identify the type of clay that exists in the area and then the characterization of the obtained clay in order to list all the works (tiles, bricks, canaries... etc.) that can be produced in the concerned area. Studies conducted in the world in general show that many muddy soils have clay and can be used in civil, mechanical and electrical engineering constructions provided that they undergo a mechanical treatment. The world in general and Cameroon in particular has large deposits of clay soil [12] [13] and these deposits are therefore sufficient to be used as material. It is in this optics that for the account of this work; we are going to extract the clay of the commune of Dibang in the region of the center of Cameroon then to proceed to a mechanical treatment artisanal in order to obtain the clay to make physical, chemical and thermal characteristics, then to determine the behavior in the constructions of the buildings.

II. Materials and Experimental Methods

A. Materials

a. Location of the site: Dibang.

The sampling site of the clay of the study is DIBANG in the river Mbanda located between 500 to 700 meters (m) of altitude. Administratively, DIBANG is located in the central region, department of Nyong-et-Kelle. The demographic and geographic situation is presented in Table 1 and Figure 1[14] shows the geography on the map of Cameroon according to data from en.climate-data.org.

TABLE 1: Geographic and demographic location of the study area

Country	Cameroon
Region	Center
Department	NYONG-et-Kelle
Demographic	
Population	906 inhabitants (2005)
Geographic	
Coordinates	3° 59′ 56″ North, 10° 43′ 7″ East

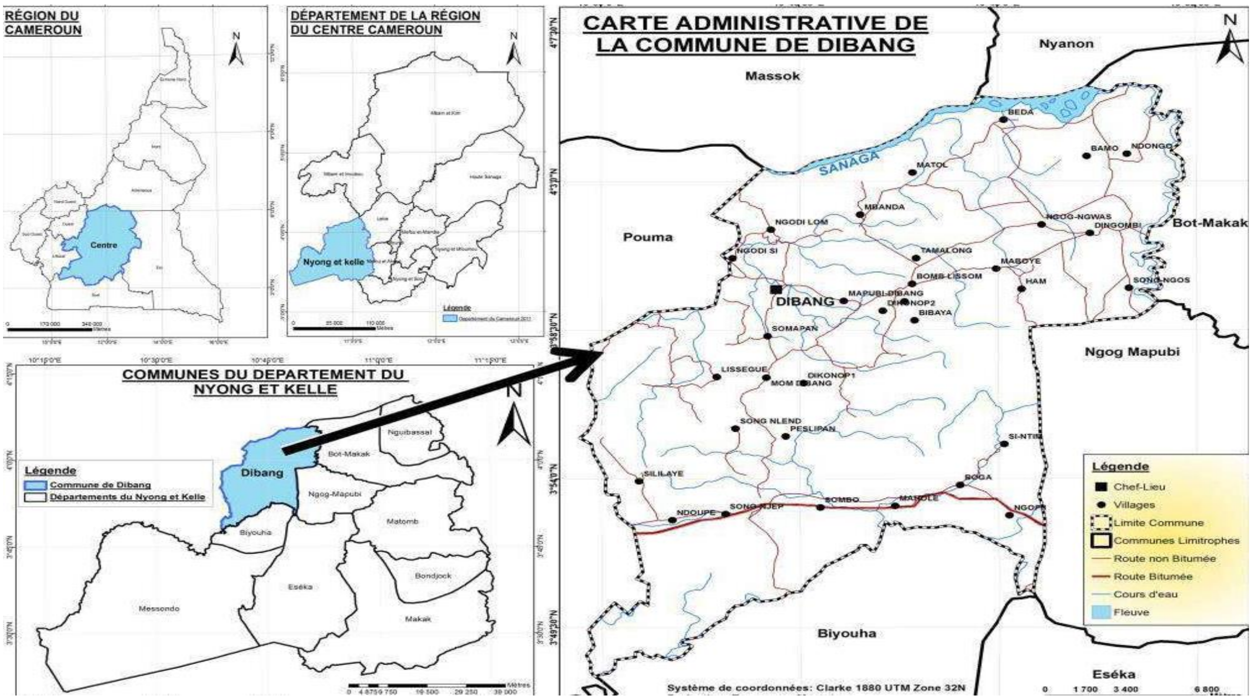

Figure 1 : Geolocation of the commune of Dibang on the map of Cameroon

The commune of DIBANG covers an area of 475 km2 and is located 119 km from Yaoundé and 63 km from Eséka. It is limited to the North by the Sanaga River and the Massok and Nyanon districts, to the East by the Djuel River and the Ngog Mapubi district, to the North-East by the Bot-Makak district, to the West by the Pouma district, and to the South by the Kéllé River and the Biyouh district. At the time of the 2005 census, the number of people living there was 906. The average annual temperature in Dibang is 23.5°C and rainfall is 831.7 mm.

b. Materials for mechanical processing and physical properties.

Standard materials for mechanical processing of clays: A container of at least 10 liters (L); a shovel; plastic bags and packaging. Several sieves for granulometric analysis with mesh sizes ranging from 0.1; 0.2; 0.4; 1.2; 4; 6.5 and 10 (mm), a MIHEE (Ming Heng Electronic Digital Scale) scale at 1/100^{e}, a digital camera with optimal resolution, 4x digital zoom; an oven, a pycnometer and SCALTEC scale at 1/1000^{e} .

c. Materials for chemical properties.

Nicolet iS5 IR spectrometer [15] of the Laboratory of Research in Chemistry of Nuisances and Environmental Engineering (URCHINGE) of the University of Dschang. Dschang - Cameroon.

d. Materials for thermal properties

Thermal analyzer LINSEIS STAPT-1000 C of type Platinum Evaluation V1.0.182, coupled to a computer and

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programmed for this purpose, of the Laboratory of Physical Chemistry of Materials of the Faculty of Sciences of the University of Yaoundé 1. Yaoundé - Cameroon.

B. *Experimental Methods*

Mechanical treatment of the clay: artisanal method.

The purpose of this treatment is to remove the aggregates and the waste of any kind which is on the clay. The principle consists of extracting the clay in the study area (figures 2a and 2b), stirring it with water and a wooden ladle for about 25 min so that the clay is well mixed (figure 2c). The resulting slurry is poured through a 500 µm (micron) white filter cloth into a basin to remove any impurities or particles that may be in the clay (roots, stones, gravel, etc.) (Figure 2d). The water in this basin is left to settle for 24 hours at room temperature. Then we release the water and the clay remains at the bottom of the basin which will be kept directly in a porous bag for dehumidification for 45 days at room temperature (figure 2e). This method is called mechanical treatment of clays.

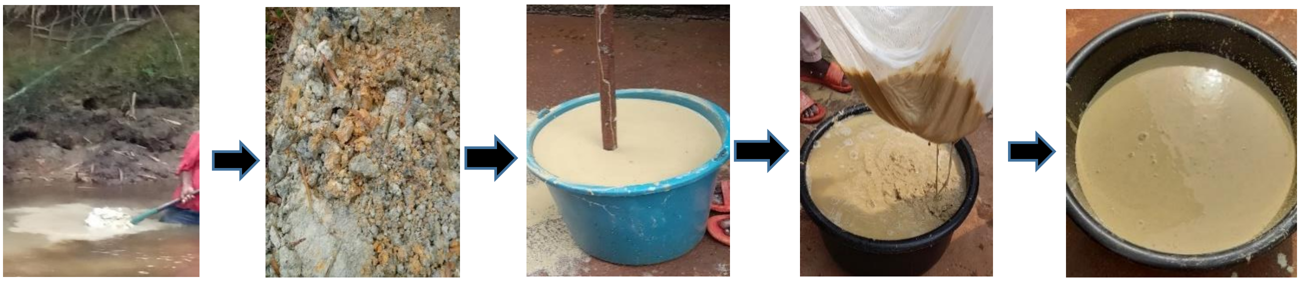

Figure 2a figure 2b figure 2c figure 2d figure 2e

Figure 2 : Process of mechanical treatment of clays (case of Dibang (Photo DC001 of 28/02/2022))

Characterization of the clay

a) Physical characterizations

- Particle size analysis by dry sieving after washing: this test is carried out in the MIPROMALO laboratories (Mission for the Promotion of Local Materials) [16] according to the NF P 94-056 standard.
- Particle size analysis by Sedimentometry: this test is carried out in the laboratories of MIPROMALO according to the standard NF P 94-057.
- Limits of Atterberg: this test is carried out in the MIPROMALO laboratories according to the NF P 94-051 standard.
- Water content: this test is carried out in the laboratories of MIPROMALO, it is necessary to weigh the tare with vacuum beforehand cleaned and dried with the oven; to put a quantity of clay in the tare and weighed the wet mass then to put that with the oven regulated with 105°C during 24 hours; after these 24 hours weighed the dry mass of the clay Finally applied the relation (b.1) to determine the water content of the treated clay.

$$TE\ (\%) = ((Mh-Ms)) / Ms\ X100 \qquad \text{(b.1)}$$

TE: Water content

Mh: Wet mass

Ms : Dry mass

- Methylene blue test: this test is carried out in the MIPROMALO laboratories on clay samples according to the NF P 94-068 standard.
- Real density: this test is carried out in the laboratories of MIPROMALO, following the steps which are : first weighed the pycnometer with vacuum and weighed the pycnometer with distilled water filled up to the gauge line, secondly decreased a good quantity of distilled water (more than half) and poured 20g of the clay in the pycnometer, thirdly placed on a hot plate and allowed to heat until boiling then removed and allowed to cool the mixture in the open air and then in the water bath for a total cooling, the fourth step is to add water up to the gauge line and then weighed the mixture. After finishing apply the relation (b.2) to determine the real density of the treated clay.

$$dr = \frac{(M2-M1)}{(M2-M1)-(M3-M4)} \qquad \text{(b.2)}$$

M_1 =empty Pycnometer mass,

M_2 = Pycnometer mass with sample

M_3 = mass of Pycnometer, sample and water

M_4 = mass of Pycnometer with water

dr= Actual density

b) Chemical characterizations

Fourier transform infrared spectroscopy (FT-IR) was performed using the Nicolet iS5 IR spectrometer. We recorded the IR spectra in ATR (attenuated total reflectance) mode on a germanium crystal [15]. FTIR spectra are obtained from the data records and we can plot our data in two parts: from 4000 - 500cm-1 and from 500 - 400 cm. The samples are primed as a fine powder, we will take a quantity of about 26 g to perform the analysis. The test conditions and ranges are governed by the standard detailed in the manufacturer's catalog [15]. This analysis was performed at the Laboratory of Research in Chemistry of Nuisances and Environmental Engineering (URCHINGE) of the University of Dschang. Dschang-Cameroon in the same way that **Djomi** and his team described in his work "*Physicochemical and Thermal Characterization of Dura Palm Kernel Powder as a Load for Polymers: Case of Polyvinyl Chloride*" in 2018 [17]

c) Thermal characterizations

The machine is a LINSEIS STAPT-1000 C thermal analyzer of type Platinum Evaluation V1.0.182, coupled to a computer and programmed for this purpose. The thermal treatment of the device ranges from room temperature (20°C - 35°C) to 1000°C. The heating rate varies between 1°C and 100°C. The crucible is made of alumina oxide with a capacity of 150mg and a control crucible of alumina. The mass of the charge is between 100mg and 125mg. The initial heating temperature depends on the ambient temperature at the time of the measurement. The heating rate according to the literature is 10°/min. The computer plots the ATG/DSC thermograms by recording the data. This analysis was carried out in the Laboratories of Physical Chemistry of Materials of the Faculty of Sciences of the University of Yaoundé 1. Yaoundé - Cameroon in the same way as Roland Olembe and his team in their work on "*Investigation of the Physical, Mechanical and Chemical Properties of the Marrow of Raffia Hookeri*" in 2021 [18] then Djomi and his team described in his work [17].

III. Results and Discussions

A. Results

Result of the mechanical treatment of the clay

The obtained clay is muddy and pasty after decantation and dehumidification (figure 3a), Then a conservation of 45 days in a packaging at room temperature is necessary to have a workable clay (figure 3b).

(3a) (3b)

Figure 3 : Result of the mechanical treatment of the Dibang clay

TABLE 2: Results of the physical characterization of the Dibang clay

Particle size analysis					
Reference	**Color**	**% of gravel Φ>2 mm**	**% of sand 2>Φ>0.02 mm**	**% of silt 0.02>Φ>0.002 mm**	**% of clay Φ<0.002 mm**
Ech Clay	**Semi-white**	**2,5**	**32,5**	**38,4**	**36,6**
Limit of Atterberg and plasticity Index					
Reference	Liquidity limit (%)	Plasticity limit (%)	Plasticity index (%)		
Ech Clay	**55.43**	**47.15**	**8.28**		
Water content, bulk and true density, VBS and porosity					
Water content (%)	VBS	Apparent density (g/cm^3)	Real density (g/cm^3)	Porosity (ŋ) (%)	Actual density (dr)
48.25	**3.27**	**1.21**	**2.09**	**54.49**	**2.09**

The practical observations after this mechanical treatment of the clay show that the quantity of aggregates, namely aggregates and other waste, occupy a very low proportion by volume. This result is justified by the quality of the clay taken in the middle of the rainy season, add to that the quality of the road permanently slippery in the rainy season and the habitats noted in the region built in rammed earth. This wanting to add to this above, the volume of aggregates and waste of any kind very small at the surface at the time of the samples show that in depth, the titration of the clay will be too high. Thus; the mechanical treatment will not be any more a great necessity in the shaping of certain objects even of certain works.

Results of the clay characterizations.

Results of physical characteristics.

Table 2 gives the results of the physical identification tests on Dibang clay. After having applied the standard NF P 94-056 to the granulometric analysis by dry sieving and the standard NF P 94-057 to the granulometric analysis by Sedimentometry on the clay obtained after mechanical treatment, they reveal that the color of the clay is Semi-white, with percentages thus the gravel is of 2,5%, the sand is of 32,5%, the silts is of 38,4% and finally the clay is of 36,6%. Similar results were obtained by Désiré Tsozué on the alluvial clays of Maroua [19], Dominique Westshondo on the clays of Kinshasa [20] and by Nevila Jozja on the Albanian alluvial clays [21]. By applying the standard NF P 94-051 to the Limit of Atterberg, we obtain that the liquidity limit has a percentage of 55.43%, the plasticity limit is 47.15% and the plasticity index is 8.28%. Similarly, the standard NF P 94-068, to the test with methylene blue gives us a value of VBS of 3.27 with the percentage of water content of 48.25% and a Porosity (ŋ) of 54.49%; the apparent and real density of the clay is between 1.21 and 2.09 g/cm3 and finally the real density is 2.09.

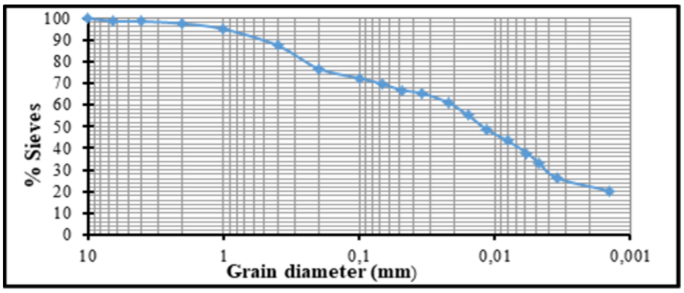

Figure 4 : Particle size distribution of the Dibang clay

In sum, Figure 4 presents the particle size distribution of Dibang clay, it shows that the mechanical treatment gives the clay a smoothing curve fairly continuous and decreasing which promotes a good compactness to increase the mechanical strength and even durability. Similarly, this character of the clay proves that the manufacture of composites with ceramic matrix as for example the stabilized clay bricks loaded or reinforced with various substrates will give competitive mechanical results and a good fire resistance appreciable.

Results of the chemical characterization

The Fourier Transform Infrared spectrum of the Dibang clays show an intense peak at 1003.59 cm-1 and a band at 686.70 cm-1 relating to the elongation and torsional vibrations of Si-O bonds respectively. The broad bands at 3090.25 cm-1 and 1990.01 cm-1 are attributable to elongation and deformation vibrations of -OH and water molecule absorbed in the inter-leaf space of the clay sheets, the results obtained highlighting the presence of two new adsorption bands at 3403.21 cm-1 and we have at 3090.25cm-1 corresponding to asymmetric and symmetric elongation vibrations of -CH2 groups, which shows the presence of silicon in the clays. The presence of new absorption peaks at 1113.54 cm-1, 873.77 cm-1 and 790.62 cm-1 corresponding successively to deformation vibrations of -NH2 and -CH2 and out-of-plane deformation of -CH. The peak at 3750.61 cm-1 is attributed to an elongation vibration of C-N. These results are very close to those obtained by some authors in their research work including Shanmugharaj and his team in 2007, Benbayer and his team and then Dhakal and his team in 2014 [22], [23], [24].

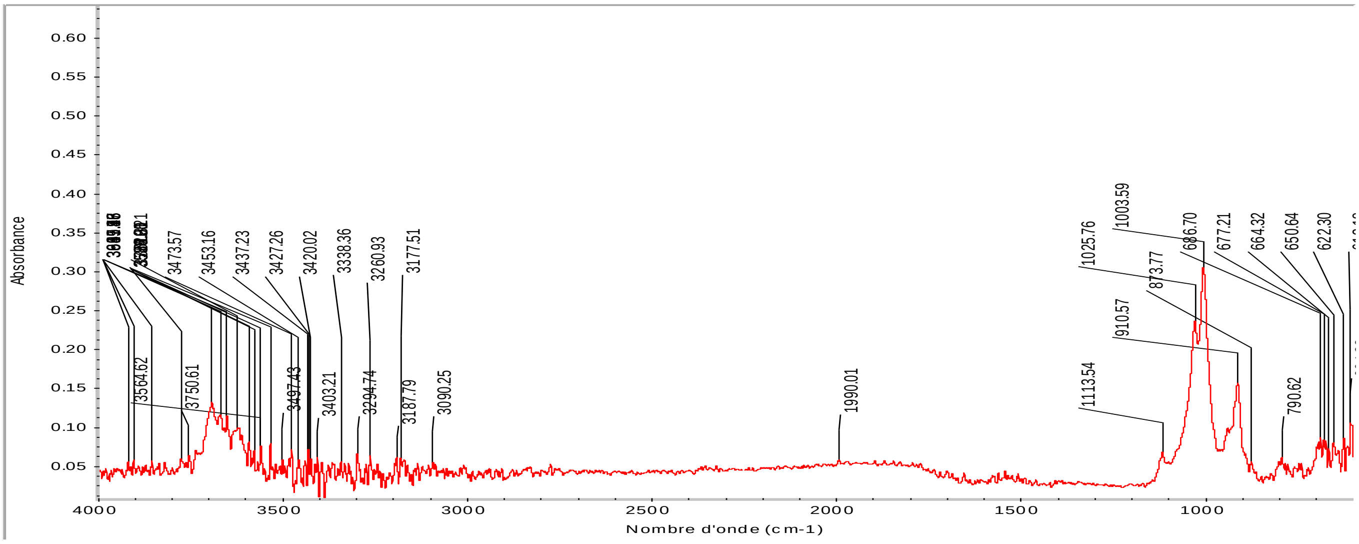

Figure 5 : Fourier transform infrared spectrum of Dibang clay

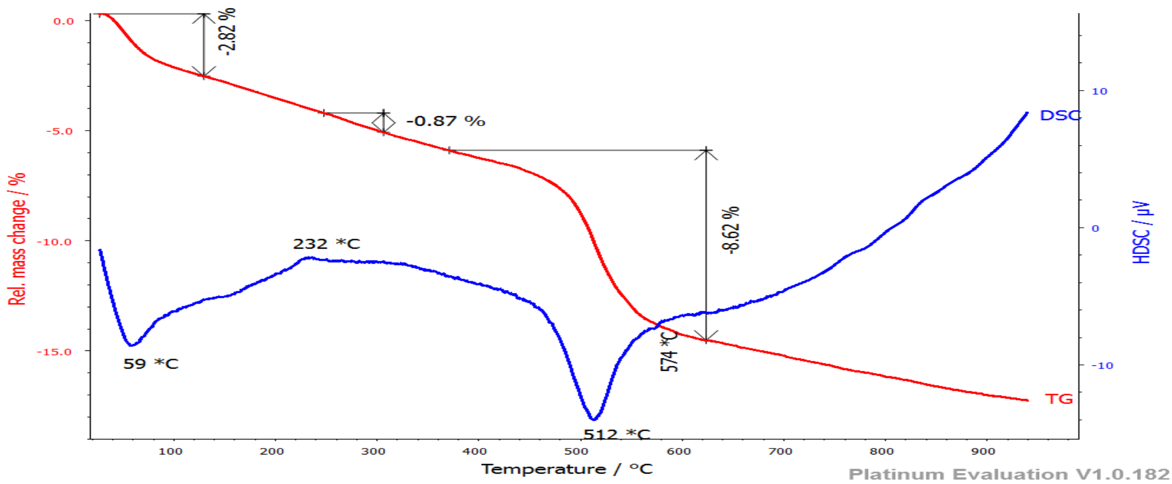

Figure 6 : TG and DSC thermograms of Dibang clay

Results of the thermal characterizations

Figure 6 shows the TG and DSC thermograms of the Dibang clay. They allowed to bring out 3 figures namely the thermogravimetric curve (figure 7), the thermo differential curve (figure 8) and the curve of the heat absorption of the clay according to the decrease of its mass during its rise in temperature (figure 9). All these figures make it possible to understand the phenomenon of the degradation as well as the fire resistance of the Dibang clay according to the temperature at the time of the firing and during the fires.

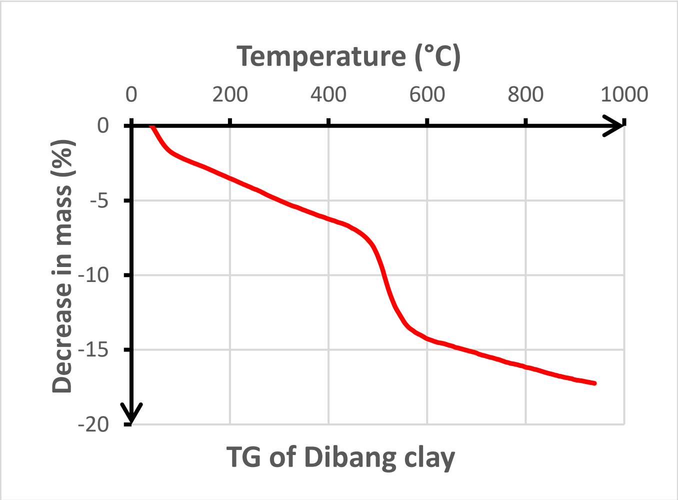

Figure 7 : Thermogravimetric analysis (TG) of Dibang clay

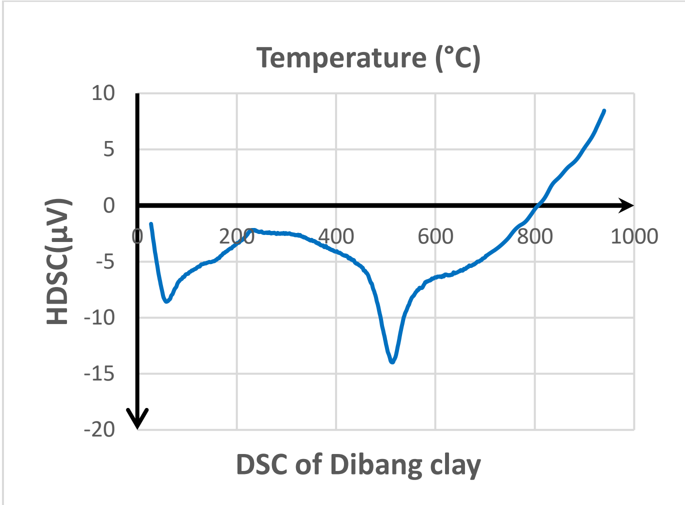

Figure 8 : Differential thermal analysis (DSC) of Dibang clay

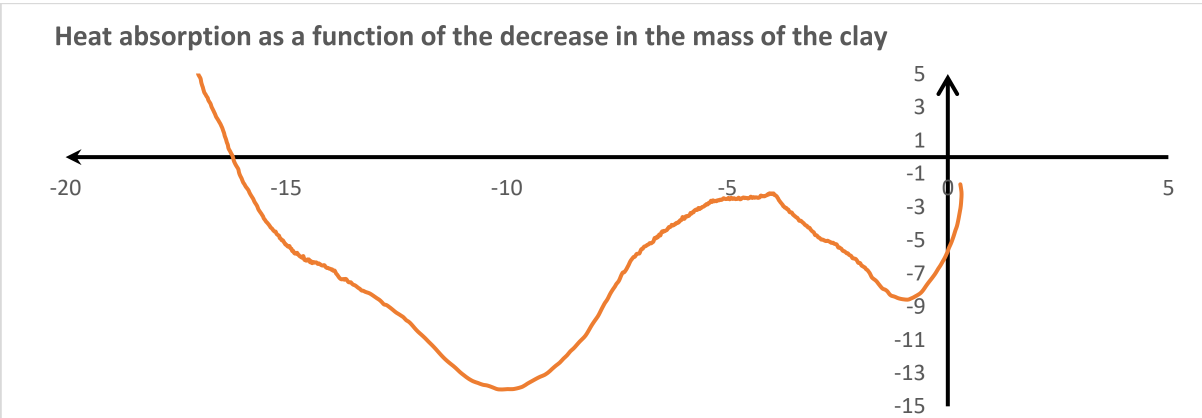

Figure 9 : Heat absorption as a function of the mass decrease of the Dibang clay

After observation of the different figures, it appears that the DSC (Figure 8) shows 5 representative and sensitive peaks of the degradation. These are the peaks at 59°C, 232°C, 512°C, 574°C and 805°C such as:

▪ At 59°C, we have a first endothermic peak that corresponds to the displacement of adsorbed water traditionally called free water or moisture that is associated with a mass loss of 2.82 wt% determined from the TG curve (Figure 7).

▪ At 232°C, we have an exothermic dip that corresponds to the dehydroxylation of goethite in hematite. This transformation is associated with a loss of 0.87 wt% determined from the TG curve (Figure 7).

▪ At 512 °C, we have a second endothermic peak that highlights the presence of quartz. This temperature marks the allotropic change of quartz. It changes from quartz α to quartz β. This reaction does not result in mass loss; [25].

▪ At 574°C, we have a third endothermic peak that corresponds to the dehydroxylation of kaolinite to metakaolinite [26]. This peak is the largest and is associated with a mass loss of 8.62 wt% determined from the TG curve (Figure 6a).

▪ At 805°C, we have exothermic peak that corresponds to the structural reorganization of the metakaolinite and primary illite phase.

These thermal analysis results show that the Dibang clay contains kaolinite, quartz and illite in large amounts. This confirms that the mineralogical analysis will show kaolinite, illite and quartz [25].

Similarly Figure 9 shows that in the vicinity of -20µv, an endothermic peak with a mass decrease close to 10% corresponding to the disappearance of free water in the clay and this decrease is obtained in the vicinity of 59°C present at DSC (Figure 8). Also, during the thermal degradation of this clay in the vicinity of 574°C is observed a decrease in mass with a heat discharge going to a vicinity of -5µv is estimated at a percentage of 8.62%. 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 value : -12µv, -3µv obtained at a temperature of 512°C and visible at DSC (Figure 8), result also reported and close to that of Laibi in his work in 2017 [27].

Thermal studies also show that when this clay is impregnated with water it dries out, shrinks and dissolves quickly on the other hand as it is raised in temperature it is softer and lighter that can be used for the manufacture of stabilized clay bricks and fully-fired roof tiles.

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B. General Discussion

The mechanical treatment of the Dibang-Cameroon clays gave a workable clay of semi-white color resulting from the original character of formation of minerals such as ferric oxide based on hematite after dehydroxylation and dehumidification of the latter. The granulometric curve (fig.4) of the studied clay shows a spread granularity which is constituted in quasi-totality of very high proportions in silt notably 38,4%, rather high in clay with 36,6% and moderate in average of sand of approximately 32,5% in the sight of its proportions we can say that our clay is of the silty type, moreover the limit of atterberg shows that these clays are of good quality provided that that undergoes a good mechanical treatment. This is justified by the results of plasticity index, liquidity limit and plasticity limit presented in table n°2. Also the apparent density of the sample is 1.21 g/cm3, a value slightly lower than that required for traditional ceramics shows that there is a significant amount of water in this clay which brings an advantage to subject the clay to heat treatments because the mobility of its seeds will increase with temperature by the appearance of the glassy phase [28]. By promoting the interaction between minerals creating the crystalline structure that promotes fusion at low temperature on clays as the results of CHUN with his team in 2017[29]. Then the degradation of Dibang clay in Cameroon as a function of temperature showing from its TG and DSC thermograms (fig.6), that the clay dehydrates at very low temperature (59°C) it is hygroscopic water and zeolitic water that disappear making the clay light at a percentage of nearly 2.82%. Thus, by going through the works on slippery earths as those in Burkina Faso of Sore Omar and his team [30] and Laibi [27] in his research work we get the manifestations of quartz (passage of quartz α to quartz β) then the departure of the water of constitution of Kaolinite and its transformation into metakaolinite which presents a good attitude in traditional ceramic materials. That this result gives a significant advantage for the production of materials that must undergo heating because the loss of water evolves to a higher temperature of the clay. Furthermore, the formation of illite from metakaolinite at low temperature shows that the clay sample contains kaolinite, illite and quartz. This confirms that the mineralogical analysis will show kaolinite, illite and quartz. Going through the results of the research on clays and clay soils [13], [19], [20] and [21] and then descending to the regions with clay soils the peculiarity is remarkable from the point of view of vegetation, roads, construction of local technical activities, The plasticity index shows that these soils, by undergoing a mechanical treatment of good precision, these regions with clay soil are predisposed for the production of roof tiles, clay bricks, canaries, dishes, coal stoves and many ceramic objects although the commercial results are not competitive enough. In view of these results, we confirm that Dibang clay is of plastic silty type with high silt content and medium content of oxides and fluxes. The granulometric analysis indicates a fine and spread texture whose essential elements are kaolinite, illite and quartz. Finally, the clays behave as if they release heat when the temperature increases without however decreasing mass considerably. This phenomenon of heat absorption according to the decrease of its mass highlights the phenomenon of the degradation of the clay, this by interpreting the results of the clay obtained from the mechanical treatment and by observing the results of the studies on the physical characterizations of the clay, we obtain that the parts so much mechanical, domestic as buildings obtained from these clays resulting from Dibang will be rather light and will rise in temperatures quickly before stabilizing as well. Finally, the chemical analysis of our clay sample indicates that it has a majority of three (3) oxides, namely silica (SiO2), alumina (Al2O3) and hydrogen nitrates (NH2). The majority of the minerals have a high content of illite, kaolinite and quartz in significant proportion, thus affirming that this clay can be used as a binder in buildings and also for coatings. At the initial stage, this clay from Dibang locality is not suitable for the production of cold stabilized products. Finally, from the results of physical analysis such as plasticity and then thermal results, we bring the ideal advantages of Dibang clay in the great advantage of tile production because this clay has good thermal stability at low temperature, which proves a good stability to degradation.

IV. CONCLUSION

The regular encounter of soils that let water infiltrate with difficulty after a rain and the observation made on the roads that are muddy and slippery in these areas. Research results show that these soils contain clay. They are commonly called clay soils. The use of clays requires the control of its physical, chemical and thermal properties. That is why the clay samples from Dibang - Cameroon were subjected to a series of characterizations. The physical characterizations revealed that the apparent density of the clay is 1.21 g/cm3, with a water content of 48.25% and a porosity of 54.49%. The chemical analysis indicates that it has mainly three (3) oxides namely silica (SiO2), alumina (Al2O3) and hydrogen nitrates (NH2). The thermal analyses allowed to identify and confirm the minerals contained in the clay. Thus, it appears from this that the clay contains kaolinite, illite, and quartz. The results of the thermogravimetric and differential analysis show that the clays decrease in density when they are elevated in temperature, favoring the use of fired bricks for the construction of multi-level buildings. The results of the granulometric analyses of the Dibang clays reassure us of the potential of the silty clays contained in these soils. On the other hand, its behavior will be very negative if we plan to carry out building construction without heating. That is why it will be rather judicious to raise it in temperature until the cooking in order to facilitate its resistance and its durability. This phenomenon confers a particular advantage that it will allow the conservation of the heat in the building for a long time what gives to this clay a very big advantage for the constructions buildings to several levels. Thus, we can allow ourselves to say following these results what can be the behavior of the clays facing several problems as the ecological constructions, and the use of these clays as matrix for the manufacture of objects in composite with ceramic matrix as well for the clays of Dibang as for the clays having the same physical, chemical characteristics as thermal as described above.

Conflict of Interest

No conflict of interest in this work

Contribution of the Authors:

All authors contributed equally to this work

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