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Characterization of the Clay Collected in the Locality of Dolisie in Congo-Brazzaville

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Abstract

This work aims at the characterization of the clay of the locality of Dolisie for its valorization. The mineralogical analysis was determined by the following techniques (DRX, IR, ATG and ATD), chemical analysis was determined by ICP-AES, CEC was assessed by the Metson method. The geothermal properties were determined by the granulometric analysis of the clay soil and allowed us to position the Dolisie clay in the texture triangle, the landings limits obtained allowed to place the Dolisie clay in the abacus of Casagrande and on the workability map of Bain and Highy. Chemical analysis showed that silica alumina as well as iron oxides are the major constituents in Dolisie clay The mineralogical balance showed that kaolinite and illite have similar percentages which are (20.51%) kaolinite, (28.08) illites. This leads us to believe that kaolinite is not the dominant mineral and the IR spectrum shows that kaolinite is poorly crystallized.

Keywords

Clay, Characterization, Mineralogy, Valuation

1. Introduction

Clays are natural minerals that have been used by humans for millennium due to their abundance. In recent years, particular interest has been given to the study of clays by numerous laboratories around the world, justified by the importance of the specific surface developed by this material, the presence of electric charges

on the surface, the possibility of cation exchange and their remarkable properties which make them suitable for multiple applications [1]: molds for metallurgy, oil extraction, cement manufacturing, agriculture, animal and human food, cosmetic health, stationery, textiles, plastics, construction materials and pharmaceuticals [2]. In the Republic of the Congo, clay is used by the Congolese populations in an artisanal way serving for a large part in the making of pottery, in the realization of various art objects, in the construction of houses, for consumption by pregnant women and other women to eliminate nausea and local people use the clay alone or in combination with plants or leaves to cure certain ailments. The Niari region has enormous unidentified clay potential. Only the characterization of a clay could allow its recovery. It is with this in mind that a research program has been set up for the characterization of various clay materials present in the Republic of Congo, with a view to their valorization. Several Congolese localities have already been characterized in particular Loutété, Loukoléla, mouyoundzi, Makonongo and londélakayes and many others [3] [4] [5] [6] [7]. Orient Loukoléla in the environment for adsorption of heavy metals. Mouyoundzi, Makonongo and londé-lakayes could be used in ceramics. It is with this in mind that we have chosen to mineralogically and physically characterize the clay soil sampled in the Niari region in order to predict its future use and also to understand why this soil is used by the local population for consumption.

2. Materials and Methods

2.1. Location of the Sampling Site

The clay soil sample comes from the town of Dolisie, located in the south of the Republic of Congo in the department of N2. Materials and methods.

2.2. Location of the Sampling Site

The clay soil sample comes from the town of Dolisie, located in the south of the Republic of Congo in the department of Niari (**Figure 1**).

2.3. Experimental Study

The collected sample was dried for a week in the laboratory at room temperature. It was then ground in a porcelain mortar and sifted through a sieve.

2.3.1. X-Ray Diffraction X-Ray Diffraction Was Used for the Analysis of the Compounds

The measurements were recorded on the raw Dolisie sample using a philips brand diffractometer using a copper anticathode (λ = 1.54054 Å) at the Bordeaux Condensed Matter Chemistry Institute (ICMCB). The X spectra were obtained under the following operating conditions:

- Temperature: 25˚C
- Angular range: 5˚C < 2θ < 80˚C
- The diffractogram was drawn using the full prof software

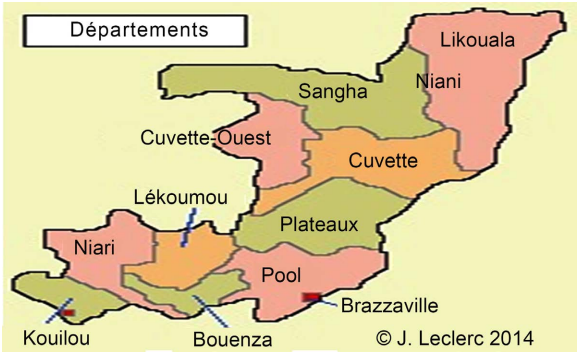

Figure 1. The location map of the sampling site.

2.3.2. Infrared Spectroscopy Measurements

The infrared spectroscopy measurements were carried out on a device called Nicolet is 10 Smart FT-IR. Samples were characterized via Attenuated Total Reflection (ATR) by mounting an ATR unit in the spectrometer control compartment. Spectra were recorded in a range between 500 cm^{-1} and 2500 cm^{-1} with a 0.5 cm^{-1} resolution spectrum at the University (UQTR), Duonglab. The IR spectrum was plotted using the origin software.

2.3.3. Differential Thermal and Gravimetric Analysis

Thermogravimetric analysis (TGA) of the Dolisie sample was performed by a thermogravimetric analyzer device named Diamond pyris 6000 from perkin-elmer. The measurements were carried out in the temperature range of 30 to 850˚C using a heating rate of 10˚C·Min^{-1}. Thermal analysis was performed in alumina crucibles under a nitrogen flow rate of 20.0 ml·min^{-1} at the University (UQTR), Duonglab. The ATG/ATD curve was obtained using the origin software.

2.3.4. Chemical Analysis

The percentage of the elements in oxide was determined by ICP-AES at the petrographic and geochemical research center of Nancy (France) [8].

1) The determination of the CEC

The CEC was determined by the Metson method at the Arras Laboratory in France, consisting of the following steps:

- The sample is first saturated with ammonium ions (NH_4^+) by successive percolations of a 1 mol/l solution of ammonium acetate ($CH_3CO_2NH_4$).
- The buffering capacity of the latter makes it possible to bring the pH of the medium to around 7, which constitutes one of the essential characteristics of this method. After removing the excess ammonium ions by percolating them with ethyl alcohol, their exchange is then carried out with a 1 mol/l solution of sodium chloride. The displaced ammonium ions are assayed by spectrocolometry on the previous solution, once filtered. The concentrations found are converted to $cmol^+/kg$ (centimoles of positive parkilogram charges of soil). The procedure used is described in the AFNOR NFX31-130 [9] standard. The test is 2.5 g of ground salt at 2 mm.

2) Organic material

The percentage of carbon and the percentage of nitrogen which represent the organic matter were determined by the following method: weigh a mass greater than 5 mg of material to be analyzed, so that it is representative, homogenized and steamed for 24 hours at 90˚C.

The resulting sample is introduced into the device using a sample tube port. The helium stream is automatically enriched with a determined amount of pure oxygen, causing flash combustion of the capsule and sample. The combustion gases entrained by the helium stream pass over an oxidation catalyst which transforms them into CO_2, H_2O, SO_2, SO_3, NxOy…. These gases then pass over a second catalyst (reduced copper) which will reduce the nitrogen oxides to elemental nitrogen, the SO_3 to SO_2 and trap the excess oxygen. At the exit of the tube, we find in addition to the helium carrier gas, the gases N_2, SO_2, CO_2 and H_2O. Non-dosed products are trapped. The gases obtained are then separated in a chromatography column and quantified by a thermal conductivity detector. The signal obtained is amplified and then processed by computers. This method is described by NF ISO 10694 and 13878 at the Aras Soils Laboratory in France [10].

2.3.5. Particle Size Analysis

The particle size distribution was determined at the Arras Soil Laboratory (LAS) in France according to standard NF X 31-107 [11]. The finest fractions (<50 μm) are determined by means of 3 successive samples (with the so-called Robinson pipette) in a soil suspension during sedimentation. The fine sand fraction is separated by passing through a 50 μm sieve and under a stream of water from the suspension after sampling of the fine fractions. The samples and sieving are carried out after destruction of the organic matter by hydrogen peroxide (H_2O_2) on a test sample of approximately 10 g. The final dispersion is achieved by a short passage to the ultrasons après addition de dispersant [$(NaPO_3)_6$ + Na_2CO_3] and after having previously separated the coarse sands (>0.200 mm) by sieving. The weighings after evaporation and drying of the fractions taken with a pipette made it possible to determine the proportions of the different particle size classes.

2.3.6. The Limits of ATTERBERG

ATTERBERG limits were determined at the building and public works control office (BCBTP) in CONGO Brazzaville. These limits are calculated by the following formulas:

- Determining the liquidity limit $W_L = \frac{Ph - PS}{(\text{Pnetsec})}$ [12]
- The determination of the limit of plasticity WP
- The deduction of the plasticity index $I_P = W_L - W_P$

3. Results and Discussion

3.1. Diffractograms of the Raw Dolisia Sample

Figure 2 shows the diffractogram of Dolisie clay.

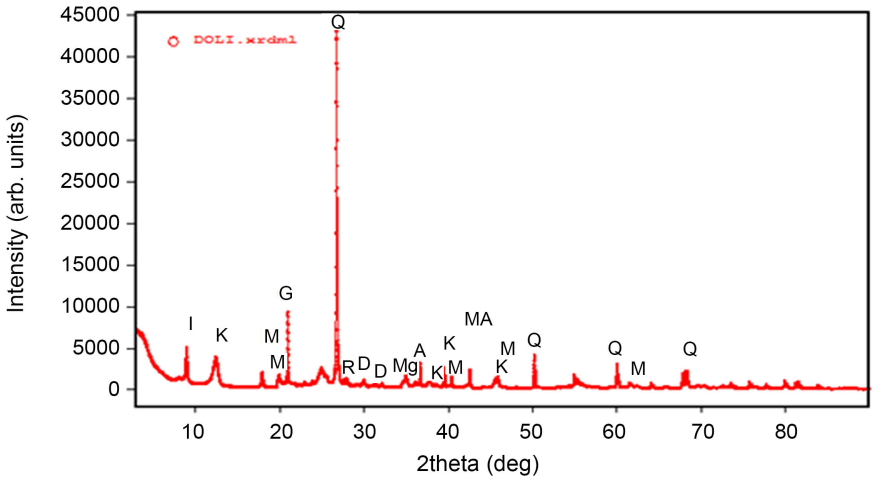

Figure 2. Diffractogram of raw Dolisie clay.

The lattice distances observed in the spectrum of the Dolisie sample are compared to those of kaolinite and quartz. From this comparison, it emerges that the reflections of kaolinite (mineral T-O) are present (7.16; 4.46; 3.57).

Analysis of this spectrum identifies the following species:

- Illite (4.98 Å) and muscovite
- Kaolinite (7.16 Å), (4.46 Å) (3.57 Å)
- Iron oxides (goethite, magnesite and maghemite) (4.18 Å)
- Anatase and feldspars (3.16 Å)
- Rutile (3.2 Å), dolomite (2.82 Å)
- Quartz (3.34 Å), (1.98 Å)

The comparison of the lattice distances of the main peaks of illite and kaolinite leads us not to consider kaolinite as the most abandoned clay mineral.

The considerable abundance of muscovite translates into the importance of the potassium content as reported by chemical analysis. The SiO_2 content between 60% and 80%, that of Al_2O_3 between 10% and 35% and the sum (Cao + TiO_2 + Fe_2O_3 + K_2O + MgO + Na_2O) between 5% and 30% allow us to consider that this clay has a low porosity or can be used in the manufacture of sandstone tiles [13]. The high content of K_2O corresponding to the presence of illites and possibly feldspars associated with that of Fe_2O_3, indicates that this clay may, during firing, produce a large quantity of fluxes. The need for fluxes to produce phase change reactions when firing or inducing vitrification is provided by clays containing alkalis or to a lesser extent, rare earths and ferrous ions, and is usually satisfied by the illitic species [14].

3.2. Infrared Spectroscopies

Figure 3 represents the infrared spectrum of Dolisie.

The XRD results showed that kaolinite and illite have similar percentages, quartz is associated with them. Kaolinite spectra are generally divided into three domains: the range from 3700 cm^{-1} to 3100 cm^{-1}, the range from 3100 cm^{-1} to 1200 cm^{-1} and the range from 1200 cm^{-1} to 600 cm^{-1} [5]. The IR spectrum of

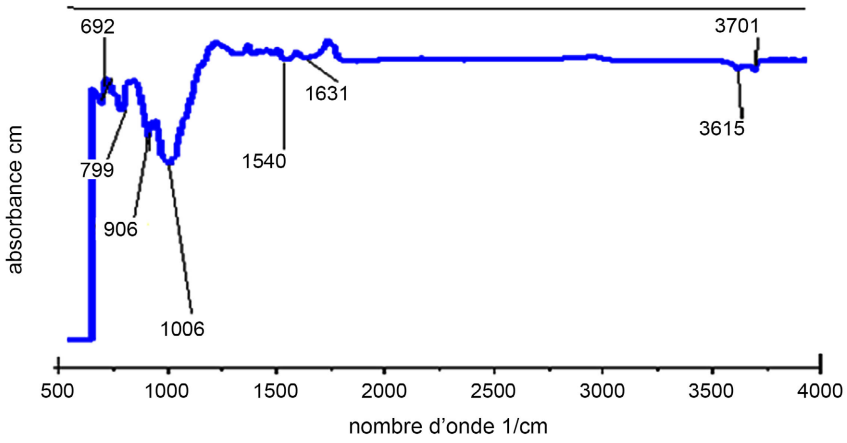

Figure 3. Raw Dolisie infrared spectrum.

Dolisie has two bands (3701 cm^{-1} and 3615 cm^{-1}), the band at 3701 cm^{-1} could correspond to the OH elongation modes. Ray. L. FROST and Philippe De Donato et al by studying the spectrum of kaolinite attributed the frequencies at 3630 cm^{-1} to the elongation or valence modes of AlO-H [13]. Let us think that the band at 3615 cm^{-1} corresponds to the vibrations of valence Al-OH.

The presence of quartz in kaolinite is manifested by the Si-O elongation band at 798 cm^{-1} and by the Si-O deformation band at 696 cm^{-1} [5]. The band at 799 cm^{-1} could match the Si-O elongation band and the 692 cm^{-1} band by the Si-O strain band. The band at 906 cm^{-1} would correspond to vibrations of Al-OH valence, the band at 1540 cm^{-1} could correspond to muscovite and the band at 1631 cm^{-1} corresponds to adsorbed water [14]. Normally in the kaolinite spectra we observe four bands relating to four hydroxide groups when the kaolinite is well crystallized and when the Kaolinite is poorly crystallized this reduces to three bands [5] (**Figure 4**).

By comparing the spectrum of Dolisie with that of mouyoudzi whose kaolinite is the dominant mineral in the range from 3100 cm^{-1} to 3700 cm^{-1} (**Figure 5** and **Figure 6**), we find that our Dolisie spectrum shows only two bands of kaolinite instead of four bands we can say that the kaolinite of Dolisie clay is not very crystallized because for a well crystallized kaolinite we have the presence of four (4) bands [5].

3.3. Differential Thermal and Gravimetric Analysis

- From ambient to 100˚C, a peak is observed corresponding to a mass loss of 0.9% caused by the departure of water from the surface of the clay soil of Dolisie.
- From 100˚C to 300˚C start of the water adsorbed in the interfoliar space, arranged in 1 or 2 layers corresponding to a mass loss of 1.1% [15].
- From 300˚C to 500˚C corresponds to the loss of mass of 1.5% of carbonates.
- From 500˚C to 800˚C with a mass loss of 3% corresponds to a departure of water from the kaolinite. This translates to dehydroxylation and leads to the formation of metakaolinite [16].

Figure 4. Amplification of the Dolisie infrared spectrum.

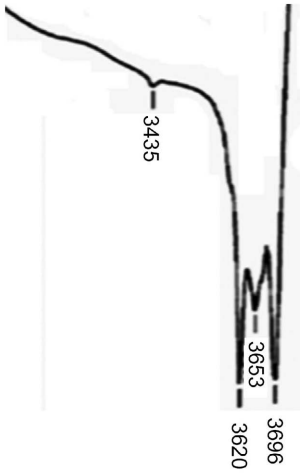

Figure 5. Amplification of the infrared spectrum of Mouyondzi [5].

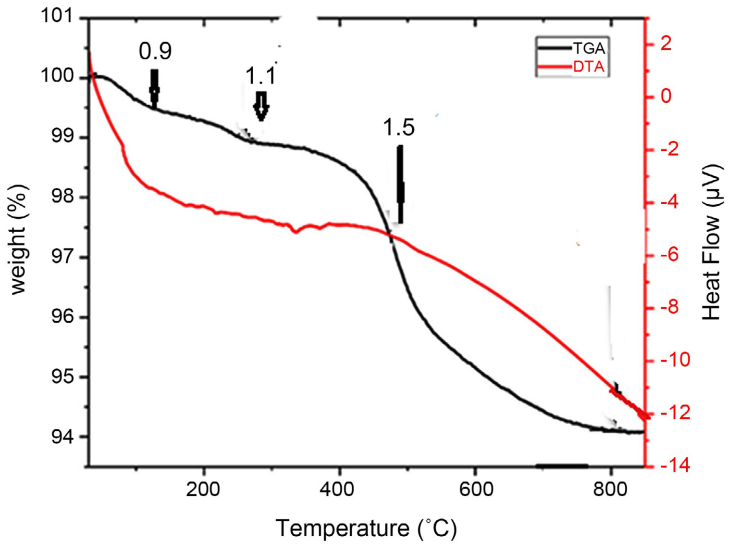

Figure 6. ATG/ATD curve.

3.4. Chemical Analysis

Silica and alumina as well as iron oxides are the major constituents in Dolisie clay with levels of 64.72% and 18.67% respectively, ie a SiO_2/Al_2O_3 ratio equal to 3.46. This value is generally observed in minerals of the kaolinite, illite and montmorillonite type [17]. Other elements (Mn, Ca and Na) also appear, but in very small quantities. Dolisie soil has a high K_2O content (3.37%), which indicates

that this clay is probably very rich in illite because the interfoliar space of illites is very rich in potassium [18]. The Fe_2O_3 content (3.28), Dolisie's XRD spectrum showed the peaks corresponding to Iron. The K_2O content is (3.37), the presence of potassium can be justified by feldspars and/or muscovite, the examination of the Dolisie diffractograms made it possible to observe the lines of calcite, magnesite and dolomite (Table 1).

The cation exchange capacity is equal to 3.78 meq/100g. Recall that the CEC of kaolinites is between 3 and 5 meq/100g and that of illites is between 10 and 40 meq/100g, the CEC of Dolisie clay is low compared to those of illite and kaolinite (Table 2).

3.5. Mineralogical Assessment of Dolisie Clay

The percentages of kaolinite, quartz and dolomite in the Dolisie sample are calculated using the following relationship:

$$(a)=\sum_{i=1}^{n}\left(M_i \cdot P_i(a)\right) \quad [19]$$

Avec:

T_a: content (in%) of element "*a*" in the material; T_a is given by chemical analysis;

M_i: content (in%) of mineral "*i*" in the material and containing the element "*a*"; $P_i(a)$: proportion of the element "*a*" in the mineral "*i*".

The procedure for calculating the mineralogical balance is such that:

1) Illite is determined from the K_2O content given by chemical analysis.

2) Kaolinite is determined by the difference between the percentage of Al_2O_3 given by chemical analysis and the proportion of Al_2O_3 corresponding to illite.

3) Quartz is determined by the difference between the SiO_2 content of chemical analysis and the proportions of SiO_2 corresponding to kaolinite and illite.

4) Magnesite ($MgCO_3$) is calculated from the percentage of MgO given by the analysis (Table 3).

Table 1. Chemical analysis of Dolisie clay.

échantillon	SiO_2	Al_2O_3	Fe_2O_3	MnO	MO	CaO	NaO	K_2O	TiO_2	P_2O_5	PF	Total
dolisie	64.72	18.67	3.28	0.00	1.11	<LD	0.05	3.37	1.07	0.10	6.63	99.00

Table 2. Chemical property.

échantillon	%C	%MO	CEC	Azote total
Dolisie	0.073	0.126	3.78	0.075

Table 3. Bilan minéralogique.

échantillon	kaolinite	illite	quartz	anatase	magnésite	hématite
Dolisie	20.51	28.08	42.44	1.07	3.83	3.28

The mineralogical composition shows that the Dolisie clay consists mainly of quartz (42.44%) followed by illite (28.08%) and Kaolinite (20.51%). The presence of kaolinite and illite in Dolisie's clay allows the latter to be effectively used as a protectant, anti-diarrheal and gastrointestinal [20].

3.6. Particle Size Analysis

The Dolisie sample has the following particle size composition: 24.4% fraction less than 2 µm, 72.7% fraction between 2 µm and 50 µm and 2.9% fraction between 50 µm and 2 mm. The fraction of particles between 2 and 50 µm in size represents 72.2% of the sample. These percentages are close to those found in the MY41g, MY45g clays studied by Ms. Djangang [21]. The latter concluded that clays with this particle size could be used in the manufacture of refractory materials. The percentage of particles smaller than 2 µm in the Dolisie sample is greater than those of the MY41g and MY45g clays. The rate of sand particles is very low (2.9%) but fine sand represents 2.5%. Then the sieving of the coarse fraction will not be efficient. The 24.4% clay, 72.7% silt and 2.9% sandy fraction allow us to attribute the clay-silty texture to Dolisie (**Table 4**, **Figure 7**).

According to this triangle, Dolisie is found in the zone of soils having a clayey-silty texture, which is in agreement with the particle rates given by the particle size analysis.

Table 4. Shows the Dolisie particle size analysis.

Echantillon	Argiles <2 µm	Limons fins (2 - 20 µm)	Limons grossiers (20 - 50 µm)	Sables fins (50 - 200 µm)	Sables grossiers (200 - 2000 µm)
Dolisie	24.4%	62.5%	10.2%	2.5%	0.4%

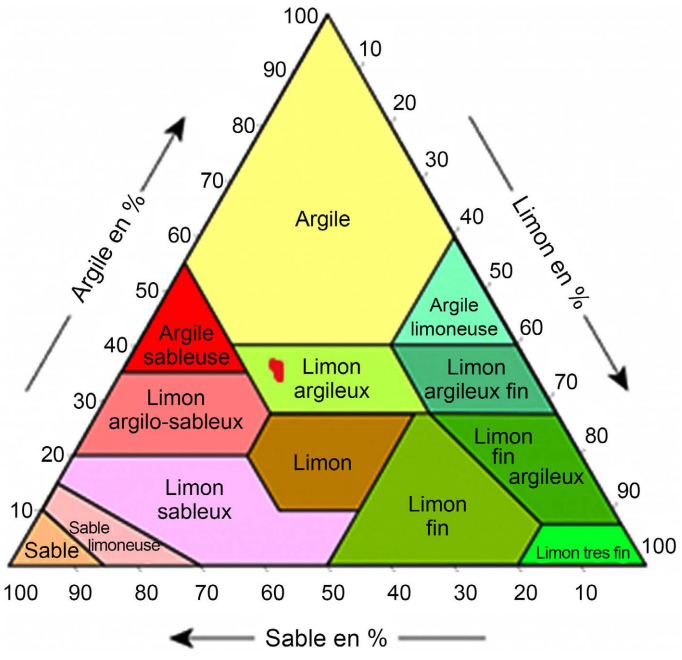

Figure 7. Positioning of Dolisie in the texture triangle.

The results of the particle size analysis allowed us to position Dol in the Winkler diagram (Figure 8, Figure 9).

3.7. The Limits of ATTERBERG

1: Hollow bricks with thin walls. 3-Bricks with vertical perforation.

2: Light block tiles. 4-Solid bricks.

2 - 4: Low frequency of use.

5 - 9: Average frequency of use.

10 - 24: High frequency of average use.

>24: Very high frequency of use.

The Dolisie sample is in the very low frequency of use area for structural ceramics.

- The ATTERBERG limits obtained allow Dolisie to be placed in the Casagrande abacus. Dolisie.

Depending on the ATTERBERG limits, and using the CASAGRANDE chart, the Dolisie sample exhibits average plasticity and can be considered as a loamy soil with low plasticity. This observation is in perfect agreement with the particle size composition of this sample (loamy soil-clay) (Table 5, Figure 10).

The Bain and Highly workability map allows us to indicate that the Dolisie sample would have moderate drying shrinkage and acceptable molding properties (Figure 11).

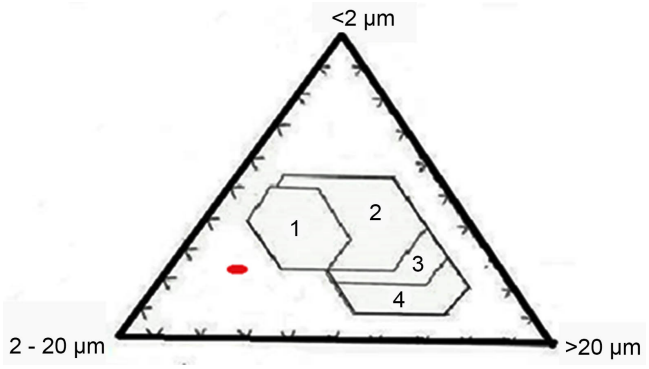

Figure 8. Positioning of Dol in the Winkler diagram.

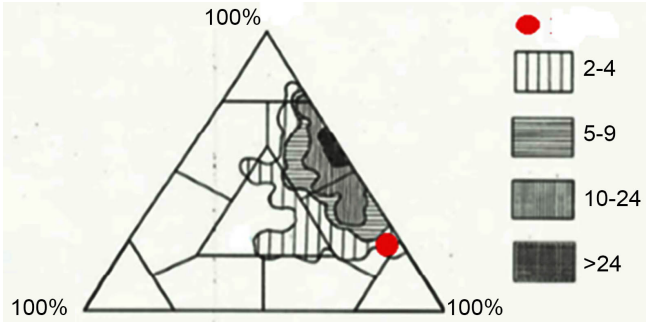

Figure 9. Relationship between frequency of use and texture frequency of use.

Table 5. ATTERBERG limit of Dolisie.

Limits samples	Liquidity limit	Plasticity limit	Plasticity index
Dolisie	41.4	24.4	17

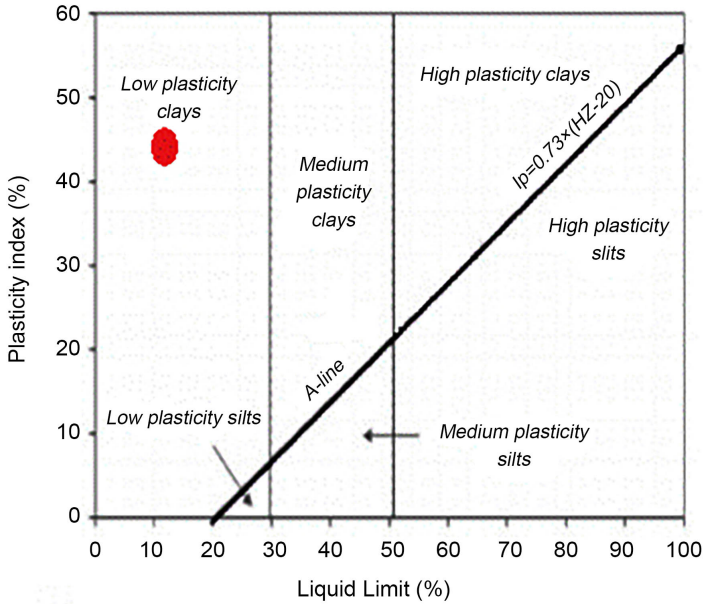

Figure 10. The Abacus of CASAGRANDE.

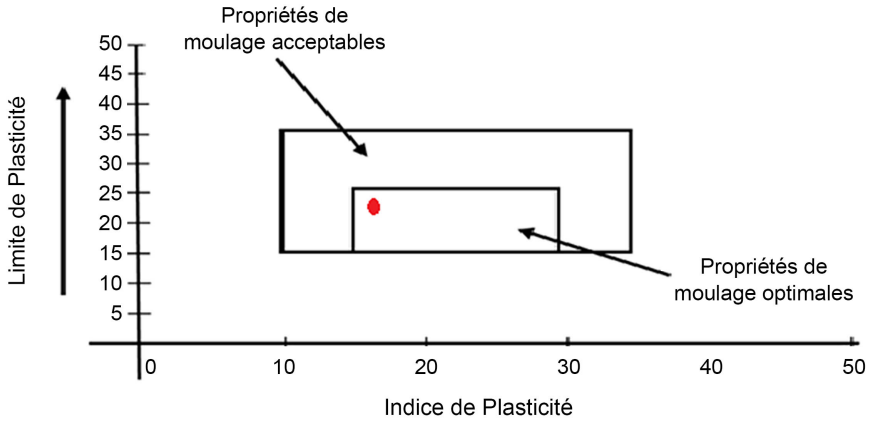

Figure 11. Bain and Highly's workability map.

4. Conclusions

The aim of this study was to characterize the clay of the locality of Dolisie in order to enhance it in several areas for this, it was a matter of doing the mineralogical analysis by (DRX, IR, ATG and ATD). Chemical analysis was determined by assaying for oxides by ICP-AES and CEC was determined by the Metson method.

- The geotechnical properties of this soil were determined by the granulometry and the limits of ATTERBERG. These properties made it possible to place this soil in the texture triangle, in the abacus of CASAGRANDE and in the map

of Bain and Highly. DRX showed the following species: kaolinite, illite, anathase, quartz, muscovite and others, IR showed kaolinite to be poorly crystallized. From ambient to 100˚C, a peak is observed corresponding to a mass loss of 0.9% caused by the departure of water from the surface of the clay soil of Dolisie.

- From 100˚C to 300˚C start of the water adsorbed in the interfoliar space, arranged in 1 or 2 layers corresponding to a mass loss of 1.1%.
- From 300˚C to 500˚C corresponds to the loss of mass of 1.5% of carbonates.

From 500˚C to 800˚C with a mass loss of 3% corresponds to a departure of water from the kaolinite. This translates to dehydroxylation and leads to the formation of metakaolinite. The CEC is 3.78, the granulometric analysis makes it possible to attribute to this soil a clayey-silty texture and the limits of ATTERBERG are: IL = 41.4, IP = 24.4 and IP = 17, the limits of ATTERBERG give this soil a low plasticity. The presence of kaolinite and illite in the clay allows the latter to be effectively used as protectors, anti-diarrheal and gastrointestinal. Analysis showed that silicon, alumina and iron oxides predominate in dolisia clay.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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