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Research Article Mineralogical and Physicochemical Characterization of the Clay Soil in the Locality of Loukolela (Congo)

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Abstract: The objective of this study is to characterize the clay of the locality of Loukolela. The mineralogical analysis is given by using different techniques (DRX, IR, DTA, TGA, DTG). Among the physico-chemical properties, the chemical analysis is given by using the procedure of Carignan by proportioning each oxide by ICP-AES, the CEC is evaluated by the method of Metson. The properties of surface and the mesoporosity are deduced from the isotherm of adsorption/desorption of nitrogen on LOUK2 sample. The geotechnical properties of this soil are highlighted by determining the particle-size distribution of the soil by the method of sedimentation and sifting and the determination of the Atterberg limits. The potential of swelling is estimated by empirical methods. The mineralogical composition obtained and the physicochemical properties have revealed the predisposition factors of the shrinkage-swelling phenomenon. The estimated swelling potential allowed to classify the soil of the bank of the Loukolela as a swelling clay.

Keywords: Chemical properties, clay soil, geotechnical properties, mineralogical composition, specific surface area, swelling potential

INTRODUCTION

Clays are primarily phyllosilicates, consisted a stacking of layers. They are used in fields as various as ceramics, oil drilling, the building, medicine, clean-up... (Decarreau, 1990). The knowledge of the structural properties and the composition of clay minerals are basically significant to define their uses (Manea *et al.*, 2006). Therefore, a research program was set up for the characterization of various clay materials present in the Republic of Congo, with the view to their valorization (Moutou *et al.*, 2012).

In the locality of Loukolela located at the bank of Oubangui River, we observed more or less important cracks on house built not far from the shore. In many identical phenomena countries. are observed (Nowamooz, 2007). Studies carried out in these countries considered that these disorders affecting mainly the individual buildings were caused by the shrinkage and swelling phenomena occurring on certain geological formations (Odent and Le Roy, 2007). The clay soils containing of expansive minerals fostered these phenomena (Djedid et al., 2001). These minerals belong to the group of the smectites (montmorillonite, beidellite, nontronite and saponite). Furthermore,

Youdjari (2010) showed that the variation of the water content in the soil due to alternation dry season/rainy season causes volume changes of the potentially swelling clay soil. In spite of the disadvantages, certain properties of expansive clays make them very interesting materials in many applications (Jordan, 1949). Smectites are currently used as adsorbents in the processes of clean-up, discoloration of oils, refining (Bouras, 2003), or antibacterial agents (Otto and Haydel, 2013).

The bentonites develop the adhesive properties when they are mixed with only one small quantity of water (Jordan, 1949).

The sticky consistency of the clay soil from the bank and the observations of the cracks on the houses had led us to consider the presence of smectites in this soil.

This study aims at the mineralogical, physicochemical and geotechnical characterization of soil collected on the shore at Loukolela.

The spectroscopic techniques (X-rays diffraction and Infra-red spectroscopy) will be used to identify the mineral species present in the soil collected. The chemical composition and the chemical properties (Tamm iron and Mehra-Jackson iron, organic matter,

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Fig. 1: Localisation of loukoléla

cationic exchange capacity) will be carried out as well as the differential thermal and thermo gravimetric analyses. The properties of surface (specific surface and porosity) will be considered in this study. The geotechnical properties will be given (grain-size distribution and Atterberg limits) in order to estimate the extent of the shrinkage-swelling phenomenon.

These analyses will enable us to propose an explanation to the phenomenon of cracks observed.

MATERIALS AND METHODS

Localization and description of the site: Loukolela is located in the East of the department of the Central Basin on banks of the Oubangui River, an affluent of the Congo River (Fig. 1) The vegetation consists of flooded forests. Loukolela has a soil of the hydromorphic type (Convention des Nations Unies sur la Lutte Contre la Desertification, 2006). The site of taking away (located at a few meters of the bank) is flooded during the rainy season. UTM co-ordinates of this site are: Altitude 288 m, Longitude 0734988, Latitude 9885026.

Sampling has been carried out at 50 cm depth. After air drying ambient of the laboratory, the sample is subjected to a series of analyses.

Methods: The XRD of sample powder was performed using a Panalytical X' PERT PRO diffractometer operating by reflection under the Cu-K α radiation for



Fig. 2: X diffraction pattern of LOUK2

 2θ angle ranging from 3° to 90°. Diffuse Reflectance Infra-red Fourier Transform Spectroscopy (DRIFTS) was performed over a wave number domain between 600 and 4000 per cm using a Bruker IFS 55 spectrometer equipped with a broad band detector of MCT type (Mercury and Cadmium Tellurium) cooled at 77K and with an accessory of diffuse reflection (Harrick Corporation). The powdered sample was diluted in KBr (50 mg of sample in 350 mg of KBr). The spectra were recorded by accumulating 200 scans at a 2.0 per cm resolution.

The Differential Thermal Analysis (DTA) and Thermo Gravimetric Analysis (TGA) were carried out with a SETARAM TAG 2400 device coupled with a thermobalance. The speed of heating was 10°C/mn.

The chemical analysis of the major elements was carried out according to the procedure of Carignan *et al.* (2001). The limits of Atterberg were measured according the norms NF P94-051

The particle-size analysis was performed according to standard NF X 31-107

The extraction of iron is done according to two methods:

- Mehra Jackson method: (Mehra and Jackson, 1960; Jeanroy, 1983)
- Tamm method (Duchaufour and Souchier, 1966)

The iron content, extracted from these two techniques was carried out by Inductively Coupled plasma-Atomic Emission Spectrometry (ICP-AES)

The isotherm of N_2 adsorption/desorption was recorded with an automatic volumetric device of adsorption of gas, developed in the Laboratory

Environment and Mineralurgy (LEM) actually integrated in the Interdisciplinary Laboratory of Continental Environments (Lorraine University). The determination of the CEC is made according to the method of Metson described in the norm NFX 31-130.

RESULTS AND DISCUSSION

XRD pattern of the clay of Loukolela is given in Fig. 2.

The analysis of this XRD pattern was enabled to identify the following species (Brindley and Brown, 1980):

- Smectites by the peak at 15.00Å
- Kaolinite with its principal peaks at 7.15Å and 3.57Å
- Quartz (4.28Å, 3.35Å) and the anatase whose the peak at 3.51Å is not hidden by the peak 3.57Å of kaolinite as that often happens when kaolinite is very abundant.

The comparison of the height of the main peaks of the clay species led to consider the smectite as the most abundant clay mineral.

The results of the chemical analysis are consigned in Table 1.

The ratio SiO_2/Al_2O_3 is equal to 2.89. In some relatively pure montmorillonites this ratio is 3.21 (Wyoming Montmorillonite), 3.431 (Arizona Montmorillonite) and 4.381 (Texas Montmorillonite) (Brindley and Brown, 1980). This relatively low ratio in LOUK2 suggests the presence of minerals with an important rate of Al₂O₃ like the kaolinite whose this ratio is 1.14 (slightly crystallized kaolinite of Georgia) or 1.11 (well crystallized kaolinite of Georgia KGa-1) (Brindley and Brown, 1980). This observation may be explained by the presence of the kaolinite noted in the XRD pattern of LOUK2.

The Fe₂O₃ content (5.38%) is quite high. In spite of this Fe₂O₃ abundance, no peak corresponding to iron oxides (hematite and magnetite) and hydroxides (goethite) has been observed on XRD pattern of LOUK2 sample.

The main peaks of carbonates (calcite, magnesite and dolomite) have not been detected on the XRD pattern of LOUK2. The magnesium could come mainly from the montmorillonite. Indeed, as shown in the general formula of the montmorillonites, magnesium is present in part in the octahedral layers (Brindley and Brown, 1980). Calcium would be thus an exchangeable

Table 1: Chemical analysis of LOUK 2

Oxydes	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MnO	MgO	CaO	Na ₂ O	K ₂ O	TiO ₂	P_2O_5	PF	Total
Content (%)	52,09	18,02	5,38	0,03	1,77	0,47	0,04	1,13	0,67	0,15	19,47	99,21

Organic matter	Organic carbon	Nitrogen total	C/N	Organic matter		
	0,99g/kg	0,238 g/kg	4,2	2,39 g/kg		
CEC	47,38 cmol+/kg					
Extractable Fe	Fe Tamm 0,069 g/100 g		Fe Mehra Jackso	on 0,469 g/100 g		
Extractable Al	Al Tamm 0,416 g/100 g		Al Mehra Jackso	on 0,416 g/100 g		

Table 2: Chemical properties of sample LOUK2



Fig. 3: XRD pattern of LOUK 2: angular range 3-11°(20)

cation in interlayer space of montmorillonite. The peak at approximately 15Å generally indicates the presence of a montmorillonite-Ca (Brindley and Brown, 1980)

The presence of potassium could indicate feldspars and/or muscovite.

Figure 3 gives us an amplification of the angular field going from 3 to $11^{\circ}(2\theta)$ of XRD pattern of LOUK2.

We can then observe the presence of a very weak peak at 8.90° (2 θ) corresponding to the 9.93Å d-spacing. From there, we can suppose the presence of muscovite. Table 2 gives us the chemical properties of sample LOUK 2.

The very small percentage of Fe (0.47%) obtained by Mehra-Jackson method indicates that the free iron, in other words engaged in oxides and hydroxides (hematite and goethite), bound to organic matter and present in amorphous matter (ferrrihydrite) (Segalen, 1971) is weak. Iron linked to the organic and amorphous matter is very weak (0.07%) (Jeanroy *et al.*, 2010). This result is in agreement with the organic matter rate (Table 2) in this soil (1.73 g/kg). These observations suggest that iron would be present in layer silicates, making the substitutions either in the octahedral sites or in the tetrahedral sites in montmorillonite or the illite (Brindley and Brown, 1980).

Figure 4 gives us the FTIR spectrum of LOUK2

In the interval of frequencies 3700-3500 cm⁻¹ kaolinite presents at least three bands (the 3695 cm⁻¹ most intense, 3650 cm⁻¹ and 3620 cm⁻¹) while montmorillonite expresses only one intense and widened band around 3628 cm⁻¹ (Van Olphen and Frippiat, 1979). The intense band to 3621 cm⁻¹ can be allotted to the stretching vibration AlO-H of montmorillonite (Van Olphen and Frippiat, 1979). The shoulder to 3695 cm⁻¹ would correspond to one of the elongation bands of kaolinite, the two others being masked by the peak of montmorillonite. As it was observed in XRD pattern, we can admit here that the smectites would be more abundant than kaolinite in considering the difference in intensity of their bands. The observation of an intense and broad band around 3413 cm⁻¹ and of a band to 1637 cm⁻¹ of relatively significant intensity would indicate to the presence of adsorbed water (Richard, 2006). Indeed by comparing the intensity of the band with 1637cm⁻¹ with that observed in the FTIR spectrum of clay collected in Loutete made up primarily of kaolinite (Moutou et al., 2012), we can deduce that the water retention capacity



Fig. 4: FTIR spectrum of LOUK2



Fig. 5: Comparison of spectra of LOUK2 and LOU

of LOUK2 is very high indicating the possibility of a phenomenon of rather significant swelling-shrinkage. In the Fig. 5 the FTIR spectra of LOUK2 and LOU (Moutou *et al.*, 2012) are compared in the range of frequencies going from 1200 cm^{-1} to 600 cm^{-1}

We can notice the presence of the additional bands on LOUK2 at 881 cm⁻¹ and 832 cm⁻¹. The substitution of Al by Fe (III) in the octahedral sites moves the Al-OH-Al band towards weaker frequencies (Van Olphen and Frippiat, 1979). Then the band at 881 cm⁻¹ is allotted to OH grouping linked both on Al and Fe (Van Olphen and Frippiat, 1979). This observation corroborates the fact that the percentage of free iron is negligible in LOUK2 sample and leads us to consider that iron is rather structural (Jeanroy, 1983). The band at 832 cm⁻¹ would correspond to the substitution of aluminium by magnesium giving thus AlMgOH modes (Van Olphen and Frippiat, 1979). The absence of these bands on LOU which is kaolinitic clay is justified by the fact that these substitutions are rare on the kaolinite.

The weak band to 1413 cm⁻¹ suggests the presence of the carbonates traces (Van Olphen and Frippiat, 1979). The bands at 1108 and 999 cm⁻¹ correspond to Si-O-Si stretching bands (Qtaitat and Al-Trawneh, 2005). The bands at 1159 and 800 cm⁻¹ indicate the presence of quartz.

The thermal analysis curves (DTA, TGA and DTG), are given respectively in the Fig. 6 and 7.



Fig. 7: Curve DTG of LOUK2

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Fig. 9: Isotherm of adsorption /desorption of N₂ on LOUK 2 (Qads: Adsorbed quantity, P/P₀: relative pressure)

One can note the presence of four phenomena (three endothermic phenomena and one exothermic phenomenon):

- A first intense peak between the ordinary temperature and 200°C (the maximum being located at 132.46°C) (Fig. 6 and 7). An important weight loss (≈13%) is associated with this first phenomenon (Fig. 6). It would result from the physically adsorbed water desorption. The strong intensity of this peak is explained by the strong water retention capacity related to the presence of swelling clays (Van Olphen and Frippiat, 1979).
- A second endothermic peak between 400 and 600°C, relatively weak compared to that of the first (with maximum at approximately 530°C). We can assign this second phenomenon to the deshydroxylation of 1:1 clays (kaolinite) (Jouenne

2010; Bouaziz and Rollet, 1972). The low content of kaolinite indicated by the low height of its principal line (7,15Å) in spectrum X would justify the weak intensity of this phenomenon. The corresponding weight loss is equal to 3%

- By amplifying DTG curve (Fig. 8), we can distinguish a third phenomenon related to a very weak weight loss to 899°C. It would correspond to the expulsion of hydroxyls of illite (Bouaziz and Rollet, 1972)
- The fourth exothermic phenomenon (918°C) would be related to the structural reorganization of the métakaolinite (Pialy, 2009).

In attributing the second endothermic accident to the deshydroxylation of kaolinite, we can estimate the content of kaolinite starting from the corresponding weight loss. The 3% of lost water mass would lead to kaolinite content around 21.42%. The 15Å reticular distance generally appears in montmorillonite-Ca (Brindley and Brown, 1980). The significant loss on the ignition would correspond to the departure of the water of residual moisture, the water contained in montmorillonite, kaolinite and muscovite and possibly of the organic matter.

Figure 9 represents the N₂ adsorption/desorption isotherm on LOUK2 sample. From this isotherm the values of specific surface area were deduced (Table 3). This value is relatively close to the specific surface area for the smectites obtained by BET method (Rouquerol *et al.*, 2003). Higher specific surface areas ($\approx 100 \text{ m}^2/\text{g}$) were observed in clays containing a mixture of the smectites and kaolinite (Sagbo *et al.*, 2015). The value of the specific surface area is lower than those observed for bentonitic soils but widely higher than those of the kaolinitic soils. The specific surface area values for kaolinitic soils range between 5 and 15 m²/g (Konan *et al.*, 2010). This relatively high value would indicate an abundance of the smectites.

Micropores specific surface $(35.4 \text{ m}^2/\text{g})$ is relatively significant and corresponds at 40% of total specific surface area. The isotherm of adsorption is of

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BET specific surface	Micropores specific surface	Out micropores specific surface
88.2 m ² /g	35.4 m ² /g	58.5 m ² /g
oporosity		
	Phase of desorption	
Size of pores (nm)	Porous volume (cm ³ /g)	Size of pores (nm)
4.84	0.101	4.47
	BET specific surface 88.2 m ² /g porosity Size of pores (nm) 4 84	BET specific surface Micropores specific surface 88.2 m²/g 35.4 m²/g oporosity Phase of desorption Size of pores (nm) Porous volume (cm³/g) 4 84 0.101

Table 3: Specific surface area

Table 5: Limits of atterberg

	~8			
Sample	Liquid limit	Plastic limit	Index of plasticity	
LOUK2	80.3	40.7	39.6	

type IV according to the classification of Brunauer (Rouquerol *et al.*, 2003) the presence of a hysteresis indicates a mesoporosity in which capillary condensation is carried out during the adsorption of nitrogen.

The hysteresis of the H3 type would indicate a non rigid porosity through a texture made up of aggregates of the particles in the shape of plates or inflating layers (Rouquerol *et al.*, 2003). This observation is in agreement with the simultaneous presence of the kaolinite which is presented in the form of plates and of smectites having inflating layers.

The study of the mesoporosity gave the following results (Table 4). LOUK 2 sample has a heterogeneous porosity. However meso-porous volume is low. These results confirm the multi-scale structure of clay material (Rotenberg, 2007)

The CEC of measured LOUK2 is 45.8 meq/100 g. Indeed, the CEC of the smectites generally ranges between 80 and 150 meq/100 g whereas that of kaolinite between 5-15 meq/100 g (Saidi *et al.*, 2008). The considerable presence of clays such as kaolinite and muscovite and also of quartz attenuating the CEC coming from montmorillonite, would explain the value of the CEC observed.

The presence of the smectites highlighted by XRD and IR spectroscopy indicates a strong sensitivity to the phenomenon of shrinkage-swelling (Odent and Le Roy, 2007). Table 5 gives us the limits of Atterberg.

The Atterberg limits obtained enable us to place LOUK2 in the Casagrande's plasticity chart (Fig. 10). We deduce that LOUK2 is inorganic clay of high plasticity. These Atterberg limits are in agreement with the sticky consistency of LOUK2 by taking into account its position in the workability chart of Bain and Highly (1979) (Fig. 11).

The plasticity index of bentonites generally is much higher than that of LOUK2. Komine *et al.* (2009) noted values of plasticity index going from 90 to 517.8. The bentonites of Maghnia and M' zila respectively have plasticity index de 93 and 140 (Melki, 2012). The very strong values between 400 and 517 correspond to montmorillonite-Na whereas the value of 90 is observed in montmorillonite-Ca (Melki, 2012). The



Fig. 10: Positioning of LOUK2 in the Casagrande's plasticity chart



Fig. 11: Positioning of LOUK2 in the chart of Bain and Highly (1979) and Sagbo *et al.* (2015)

LOUK2 rate of sodium is so weak (0.04%). We are brought not to consider montmorillonite-Na. The value of the index of plasticity of LOUK2 (Ip = 40) is explained by the fact that the smectites coexist with kaolinite and quartz.

Indeed it is shown that the addition of sand to bentonites reduces the liquid limit and the index of

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			Coarse silts (20/50	Fine sands (50/200	Coarse sands
Sample	Clays <2 μm	Fine (2/20 µm)	μm)	μm)	(50/200 µm)
LOUK2	85.8%	8.3%	3.6%	1.5%	0.8%

Table 6: Grain-size distribution of LOUK2

 Table 7: Variation of the potential of swelling according to the properties of soil (Convention des Nations Unies sur la Lutte Contre la Desertification, 2006)

Potential of swelling	Very weak	Weak	Means	Extremely	Very extremely
Clay fraction	0-10%	10-15%	15-25%	25-35%	35-100%
Index of plasticity	0-10	10-15	15-25	25-35	<35

plasticity (Louafi and Bahar, 2012). A former study considered the behavior of the mixture bentonitekaolinite (Tammam and Hattab, 2008). Several bentonite-kaolinite mixtures had been realized (0, 5, 10, 15, 35, 50, 65, 75 and, respectively 100%). Classifications according to Dakshanamurphy and Raman on the hand and Biarez and Favre on the other hand based on the plasticity chart of Casagrande show that the various proportions are aligned on line A of the chart or on a parallel line near to line A (the line of Biarez) (Fig. 12).

The position of LOUK2 seems to be aligned with the various mixtures and leads us to consider that the proportion of the smectite would be lower than 35%. By considering the height of the most significant peaks in XRD pattern, the smectite would have a higher abundance. The results of grain-size distribution are consigned in Table 6.

The grain-size distribution allows classifying LOUK2 as clay soil. The very strong fine fraction ($<2 \mu m$) responsible for plasticity justifies the relatively high values of the limits of Atterberg. The very significant proportion of the clay fraction (lower than 2 μm) indicates a strong susceptibility to the phenomenon shrinkage-swelling (Odent and Le Roy, 2007).

The most used approaches to determine the potential of swelling of a soil are based on the clay rate and/or the index of plasticity (Table 7).

With a clay proportion of 85.8% and a plasticity index (39.6) higher than 35, we can estimate that LOUK2 has a very high potential of swelling.

The LOUK2 activity according to Skempton (Tran, 2014) is equal to 0.46 and does not correspond to the presence of the smectites (active clays) revealed by XRD pattern. This value of the activity could be explained by the fact that we have a mixture of smectites, of kaolinite and quartz. Seed *et al.* (1962) proposed a method of classification of the expansive soils starting from the activity and the content of clayey fraction ($<2 \mu m$) (Fig. 13) (Tran, 2014). The activity according to Seed *et al.* (1962) is expressed in the following way:

Ac = Ip/(C2-5) Ip = plasticity index $C2 = fraction < 2 \ \mu m$



Fig. 12: Positioning of LOUK2 according to Biarez and Favre (Tran, 2014)



Fig. 13: Diagram of classification of the potential of swelling according to Seed *et al.* (1962)

The LOUK2 activity corrected is equal to 0.49. According to the Seed's classification diagram (Fig. 13), it is shown that LOUK2 has a high swelling potential. Collected soils in the town of Amenas (Algeria) whose activity is around to 0.5 were found expansible (Djedid *et al.*, 2001). Figure 14 gives the position of LOUK2 in the abacus of Williams and Donaldson (1980) which allows the estimate of the potential of swelling by using the correlation of the plasticity index and the content of clayey fraction.



Fig. 14: Potential of swelling according to Williams and Donaldson (1980)

The characteristics of LOUK2 place it in the zone of potential of high swelling.

CONCLUSION

This study had as a general objective the mineralogical, physicochemical and geotechnical characterization of the soil collected on the shore in the locality of Loukolela. The mineralogical composition was determinated by using different techniques (DRX, IR, DTA, TGA, DTG). The chemical analysis is given by using the procedure of Carignan *et al.* (2001) by proportioning each oxide by ICP-AES. The CEC is evaluated by the method of Metson. The properties of surface and the mesoporosity are deduced from the isotherm of adsorption/desorption of nitrogen on LOUK2 sample. The geotechnical properties (the particle-size distribution, the limits of Atterberg) were determinate. The potential of swelling was estimated by empirical methods.

The results of the mineralogical analysis showed the presence of three clay species (smectite, kaolinite and illite). The montmorillonte is the more abundant clay. Specific surface area is relatively high and is in agreement with the smectites. The CEC of measured LOUK2 is of 45.8 meq/100 g. The mesoporosity is weak while the microporosity is important (40% of specific surface area). The LOUK2 soil has a very fine grain-size distribution (85.8% of clay). This soil has a very high plasticity. The very fine particle size distribution and the presence of smectites in that soil are the factors of predisposition to the phenomenon of shrinkage-swelling. The evaluation of the potential of swelling of this clay has allowed classifying this soil among clays having a high potential.

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