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Mineralogical and Chemical Characterizations of Natural Clays from NW Cameroon

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INTRODUCTION

Clay is usually defined as any very fine grained, naturally occurred material that becomes plastic when mixed with a small amount of water and hardens when dried or fired (Guggenheim et al., 1995). Clays and clay minerals are very important in process industries, construction, agriculture, geology, engineering, environmental and miscellaneous applications (Ngun et al., 2011). They are widely applied in ceramics products, decolorization and stabilization of vegetable oils, the paper industry as well for cleaning and as detergents (Nguetnkam et al., 2011).

The main objective of this study is to investigate clay samples taken from the Sabga area (NW Cameroon), in order to determine the clay type mineralogy, chemical composition, thermal behavior, surface properties and morphological characteristics.

MATERIALS AND METHODS

Seven clay samples named: (S313, S314, S412, S413, S414, S424 and S425), were collected from Sabga (Cameroon). The samples were air dried at room temperature and ground to pass through a < 250µm size.

The XRD diffractograms were obtained with a D8 Advance Bruker diffractometer, using CuKα radiation (λ=1.5406Å) at 40kV and 40mA, in the range 2-45° 2θ, scanned with a step of 0.02°2θ. For further characterization of the swelling clay, the Greene-Kelly test was performed in view to differentiate smectite with tetrahedral substitution (beidellite) to that with octahedral substitution (montmorillonite). To this end, the clay fraction was solvated with glycerol.

Differential Scanning Calorimetric and thermo-gravimetric (DSC-TG) analysis were carried out by heating the samples from 25 to 700°C at 5°C min-1 using a SETARAM TG-DSC 111 analyzer.

Major elemental analyses were carried out by energy dispersive X-ray spectrometry (EDX).

The microscopic structure of the samples was investigated by a FEG-ESEM FEI XL30 scanning electron microscope (SEM). The particle size distribution (PSD) of the samples was carried out by the wet sieving, followed by the sedimentation test.

Cation Exchange capacity (CEC) were measured by saturating the clay fraction(<63µm) with ammonium acetate (1M, pH=7) as an exchangeable ion (Meunier, 2002), the amount of ammonium fixed by the solid phase was determined by Kjeldahl titration.

The Specific Surface Area of the clay samples were determined by the nitrogen adsorption-desorption isotherm at 77K using a Carlo Erba Sorptomatic 1990 volumetric device, the final results were given by the BET equation.

RESULTS AND DISCUSSION

XRD Analysis

The XRD patterns of the seven samples are illustrated in Figs. 1a and b. All samples show that the main clay minerals are smectites with a small amount of kaolinite; the non clay minerals are cristobalite, K-feldspars, plagioclase, ilmenite and quartz.

The irreversible collapse of an expanding mineral to 9.5 Å after saturation with Li + and heating at 300°C was the criterion for identify the mineral as montmorillonite.

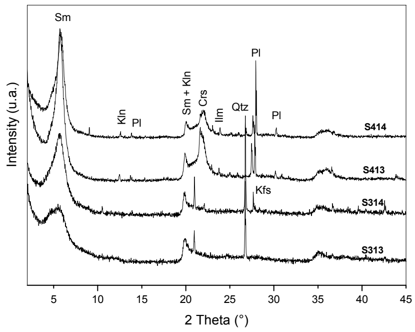

fig 1a. XRD patterns of the natural samples.

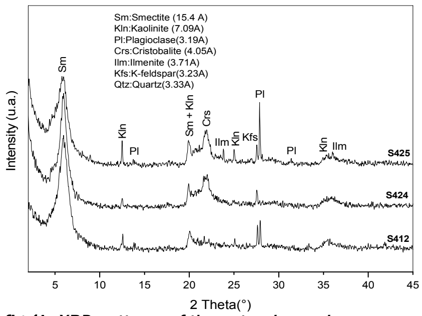

fig 1b. XRD patterns of the natural samples

DSC-TG

The results of the DSC analysis of samples are presented in (Fig 2).

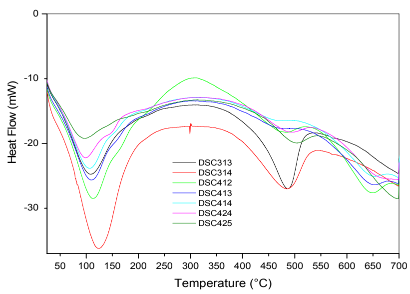

fig 2. Differential scanning calorimetry (DSC) curves of Sabga clays

palabras clave: Cameroon, Arcillas, Caracterización fisicoquímica, Mineralogía

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Four samples (S412, S413, S414 and S424) shows three endothermic peaks and S313, S314 and S425 shows two endothermic peaks.

The peak between 100-150°C is attributed to elimination of adsorbed (or absorbed) water of interlayer, and the second endothermic peak, which appeared at a temperature of about 500°C, is due to the liberation of water caused by dehydroxylation of coordinated and structural water molecule. The other peak occurred nearby 650°C correspond to the removal of OH groups of smectite.

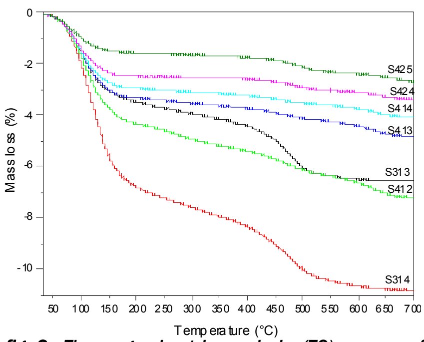

fig 3. Thermogravimetric analysis (TG) curves of Sabga clays

The TG curves (Fig. 3) of the natural clays show two well-defined mass loss regions. The first mass loss (3-8.5%) between 25 and 250°C is due to the dehydration of interparticle water, adsorbed water and interlayer water. The second mass loss (1-3%) between 400 and 650°C is due to the dehydroxylation of coordinated and structural water.

SEM

The SEM of S413 clay sample is presented in (Fig. 4), showing the morphological features.

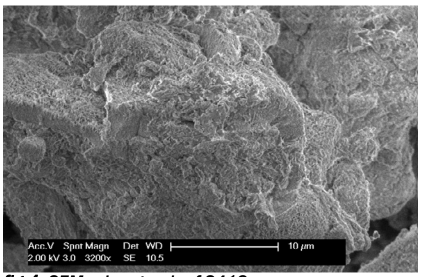

fig 4. SEM micrograph of S413.

Particle size distribution

The particle size distribution analysis quantifies particle size categories of clay samples collected from the field (Table 1.). These particles are made up of clay (< 2µm), silt (fine and coarse), sand (fine and coarse) and very insignificant quantity of nodular fraction. The result of the analysis shows the clay, silt and sand fractions in percentages found in each of the clay material.

	Clay	Silt		Sand		Gravel	
Sample	<2 µm	Fine 2-20 µm	Coarse 20-50 µm	Fine 50-200 µm	Coarse 200-2000 µm	>2000 µm	Total
S313	3	29	30	24	14	0	100
S314	6	14	25	42	12	1	100
S413	29	29	8	20	12	2	100
S414	21	25	15	27	11	1	100

Tabla 1. Particle size distribution of clay samples (S313, S314, S413 and S414).

CEC, pH and Specific Surface Area

The clay samples have presented a Cation Exchange Capacity (CEC) between 24.2 and 62 meq/100 g (Table 2). The CEC corresponds only to the smectite phase. The low CEC obtained is due by the presence of non-clay minerals associated with the clay minerals for all samples.

Samples	CEC (meq/100g)	pH
S313	44.0	4.8
S314	61.0	4.8
S412	62.0	4.9
S413	44.5	5.1
S414	44.5	4.4
S424	41.0	5.2
S425	24.2	5.1

Table 2. Cation Exchange Capacity and pH.

The pH measured of all clay samples are ranged between 4.1 and 5.1 (Table 2), indicating an acid character of clays.

The specific surface area (S_{sp}) of the natural clays taken from Sabga ranged from 58 to 123 m^2/g. These values are not conforming to those observed for the pure montmorillonite.

Chemical Analysis

Clay samples from NW Cameroon were analysed by EDX for their major elements, as given as example in Table 2 and Fig. 5. The chemical composition of the clays is dominated by SiO_2, Al_2O_3 and Fe_2O_3 whereas MgO and Na_2O are present only in small quantities.

Elements	Mass (%)	At (%)
Ok	36.78	53.63
Nak	0.97	0.98
Mgk	0.82	0.79
Alk	12.37	10.70
Sik	32.46	26.96
Fek	16.60	6.94
Total	100.00	100.00

Table 2. Chemical composition (EDX) of a particle S314.

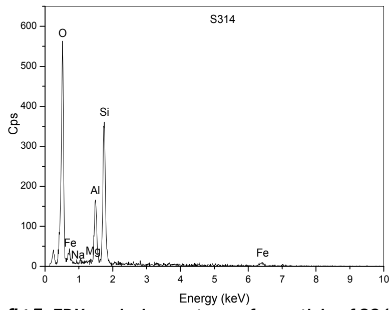

fig 5. EDX analysis spectrum of a particle of S314.

CONCLUSION

The results of this investigation show that the clay samples from Sabga (NW Cameroon), mainly consist of smectite with varying amounts of kaolinite, cristobalite, k-feldspar, plagioclase and ilmenite. The Greene-Kelly test show that the smectite are montmorillonites.

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