



X RAY AND INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROSCOPY ANALYSIS OF CRISTALLOGRAPHIC STRUCTURE AND COMPOSITION OF PAVEMENT BASED CLAY MATERIALS

Hilaire Elenga¹, Timothée Nsongo², Bernard Mabiala³, David Bilembi³ and Ferland Ngoro Elenga²

¹Faculté des Sciences et Techniques, Université Marien Ngouabi, Brazzaville, Congo/Centre de Recherches Géologiques et Minières

²Faculté des Sciences et Techniques, Université Marien Ngouabi, Brazzaville, Congo/Centre de Recherches Géologiques et Minières/ Groupe de Recherche sur les propriétés physico-chimiques et minérales des Matériaux

³Laboratoire Mécanique, Energétique et Ingénierie, Ecole Nationale Supérieure Polytechnique, Université Marien Ngouabi

E-Mail: nsongo@yahoo.com

ABSTRACT

In order to predict technological properties of local clay based materials mixed to wood waste and to prevent human health and environment, experimental mineral structure and composition study were conducted. The influence of wood waste on the structural properties of clay samples were also investigated. Non and stabilized clay sample at 4% of cement were made at different conditions and waste wood at different content have been incorporated. Mineralogical X-ray analysis was carried out using X- ray diffractometer with Geiger counter using cobalt $K\alpha$ radiation with wavelength $\lambda=1,789 \text{ \AA}$. inductively coupled plasma atomic emission spectroscopy (ICP/AES) and inductively coupled plasma optical emission spectroscopy (ICP/OES) were used respectively to determine major, Minor and trace elements. The results showed that incorporation of wood waste has a strong effect on the crystallographic structure, making partially amorphous clay structure. It is found that the waste wood content doesn't influence the lattice constants of the components of the clay. Chemical analysis of clay sample indicates kaolinite and SiO_2 as a dominant clay minerals. The results has shown some trace and heavy metal contamination.

Keywords: clay, cement, waste, wood, mineral structures, structure, lattice parameter, x-ray diffraction.

INTRODUCTION

With the development of the wood industry, the abundance of waste wood generate an important environment pollution. A wide range of studies has been reported to recycle wood waste in clay materials in order to improve technological properties[1,2,4]. Most of studies focused on mechanical properties and have shown that the addition of wood waste in clay considerably reduced the mechanical strength of the materials[5,6,7,8,9]. Knowledge of the microscopic properties such as structure and composition of clay materials used in construction may have technological interest[3,10]. This work studies the composition of the clay and the influence of waste wood on the structure in order to improve technological properties

Apparatus and Experimental conditions

A diffractometer automated X type Siemens D5000 equipped with a cobalt anticathode was used the $K \alpha$ ray of cobalt with $\lambda = 1,79026 \cdot 10^{-10} \text{ m}$ wavelength. The diffracted rays following the Bragg angle were recorded by a counter type GEIGER MULLER. This counter is placed in order to receive the rays diffracted by the sample in terms .The BRAGG relationship $2d_{hkl} \sin \theta = \lambda$ was used to determine the inter-reticular distances. The crystal lattice can be deduced from the expressions (6) and (7). The scanning angle varied between 4 to $84^\circ 2\theta$, the scanning speed was about $0.02^\circ 2\theta/\text{second}$.

X ray mineral powder analysis

For carrying out the analyzes, the samples were previously dried and ground into powder particle size less than $80 \mu\text{m}$. The counting time for this test was about 5 seconds when the sample was turning from 4 to $84^\circ 2\theta$. The crystallized fraction of the samples was determined by X-ray diffractometry from their powder diffractogram. This technique is mainly qualitative and can only give a semi quantitative result. The detection limit is about 5% and can vary widely depending on the nature of the different phases.

X ray mineral analysis using oriented blade

The counting time for this test was about 2 seconds in the rotating sample from 4 to $36^\circ 2\theta$. The phylliteuse fraction of the samples was determined by diffraction from normal oriented blades glycollées for 12 hours in steam pressure and heated up to 490°C for 4 hours. The composing proportions were estimated from peak areas

Method of identifying crystal phases



Generally the identification of a crystallized phase is made by comparison of the experimental with the theoretical diffractogram. The content of mineral components is estimated from peak areas. Match 2 software FotoMix!, XRD2DScan were used to analyze the diffraction patterns. The crystal lattice of α quartz is hexagonal. The values of



the lattice constants are obtained after indexing peaks using the expression linking to the interplanar spacing d_{hkl} . Kaolinite crystallizes in the triclinic system. Montmorillonite crystallizes in the monoclinic system. Chlorite also crystallizes in the monoclinic system.

Spectroscopy analysis

Inductively coupled plasma atomic emission spectroscopy (ICP/AES) and inductively coupled plasma optical emission spectroscopy (ICP/OES) were used respectively to determine major, minor and trace elements. The ICP-MS X7 and ICP OES Icap 6500 equipment was used with the following:

RF power ~1200 W, Plasma argon gas ~ 15 l min⁻¹, Auxiliary gas ~ 0.4 l min⁻¹, Sample gas ~ 0.9 l min⁻¹, Dwell time ~100 ms

RESULTS AND DISCUSSIONS

1X ray analysis of clay sample

Raw clay sample structure

The X ray pattern of analyzed clay sample before incorporating wood waste are shown in the Figure-1.

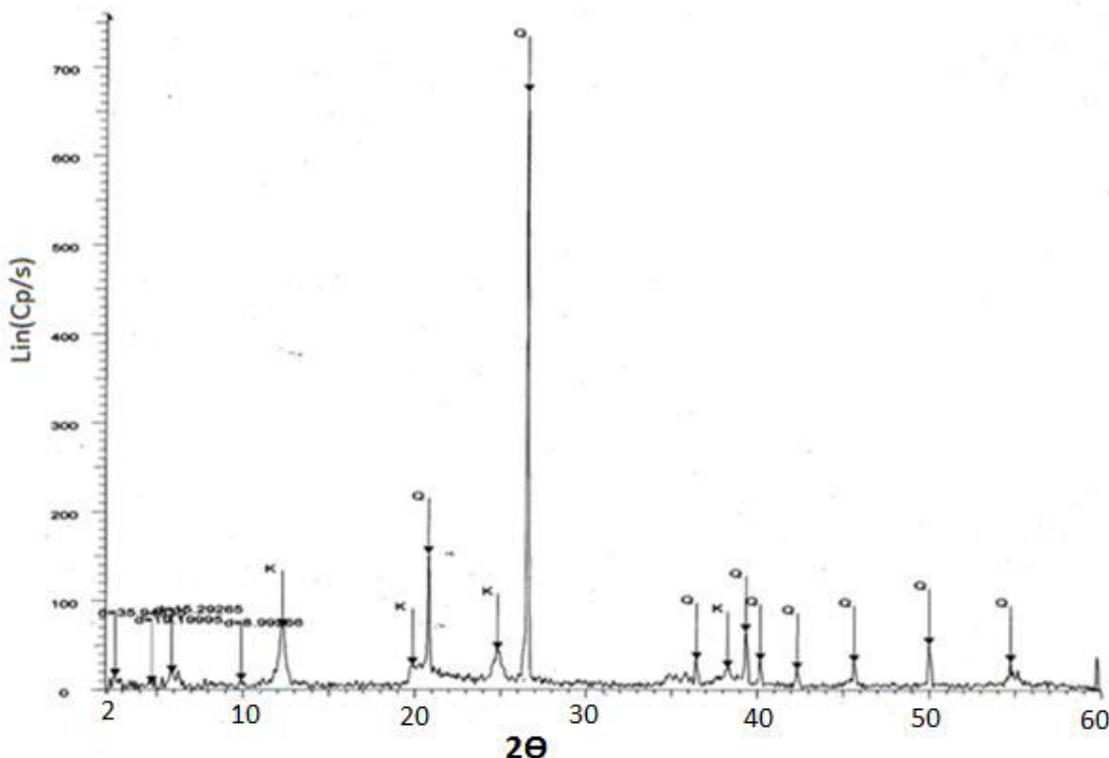


Figure-1. X-ray pattern of raw clay sample.

The results reveal the presence of kaolinite with formula $Al_2H_4Si_2O_9$ and quartz α , formula SiO_2 in raw clay sample.

Estimates of the concentrations of mineral components obtained using the method of "Peak-height ratio" is as follows:

- Quartz alpha: 51%;
- Kaolinite: 43%.
- interstratified montmorillonite/chlorite: 6%

Clay sample at 4% content of cement after acajou waste wood incorporation

The phases identified are: alpha quartz (Q), formula SiO_2 ; kaolinite, formula

$Al_2(Si_2O_5)(OH)_4$; traces of calcite of formula $CaCO_3$ and anatase of formula TiO_2 . Traces of calcite and anatase are due to the presence of the cement used to stabilize the mixture clay- soil-cement-woodchips

- Quartz alpha: 58%;
- Kaolinite: 24%;
- Some traces of anatase: 8.88% and calcite: 8.31%.

By comparing the concentrations of mineral components of the crude clay and clay after incorporation of wood waste, we find that:

- The concentration of SiO_2 (quartz) in the crude clay (51%) is weaker than that with the clay after incorporating of mahogany wood waste (58%). Chemical



reactions between clay and cement with high levels (pozzolanic reactions) form of new products that contribute to the strength of a cement stabilized earth [Akpodje 1985]; [Bell 1996].

Clay paved samples were analyzed by X-ray, after incorporating wood waste. The obtained results are shown in Figure-2.

The results showed that incorporation of wood waste has a strong effect on the crystallographic structure, making partially amorphous clay structure.

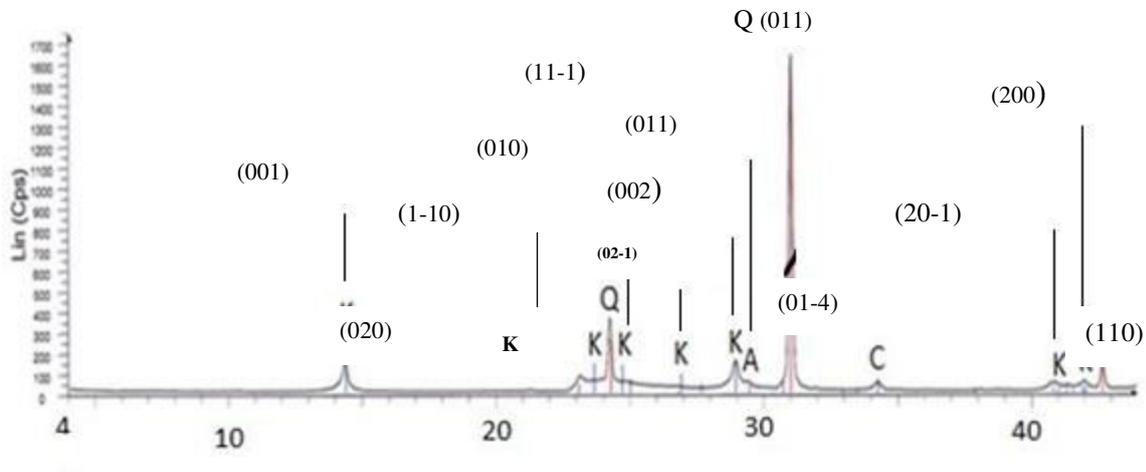


Figure-2. X Ray pattern after wood waste of limba incorporation.

Determination of lattice constants of clay components

Experimental lattice constants before and after waste wood incorporation was measured using d_{hkl} , the results are summarized in the Table-3.

Table-1. Theoretical experimental lattice constants before and after waste wood incorporation.

	Quartz α		Kaolinite	
	Before incorporating waste	After incorporating waste	Before incorporating waste	After incorporating waste
Experimental lattice constants	a=b=4,92 c=5,66	a=b=5,52 c=5,68	a=5,14 b=8,86 c=7,50	a=5,59 b=9,68 c=7,31
Theoretical lattice constants	a=b=4,91 c=5,40	a=b=4,91 c=5,40	a=5,13 b=8,89 c=7,25	a=5,13 b=8,89 c=7,25

For the parameters a and b, the resulting values are identical to the results achieved by [13] in the case of α quartz, kaolinite and montmorillonite. There is no change on a and c lattice constant of chlorite conversely to lattice parameter a, c of quartz, kaolinite and montmorillonite. So then we can say that there has been an expansion of the crystal lattice; while in the case of chlorite this variation is observed in the parameter b, in this case we see a compression which results in a reduction of the volume of the crystal lattice.

Composition determination of clay sample

Chemical composition was determined by spectroscopy following the procedure NF P94-048. The results obtained are summarized in the Tables 1, 2 and 3.

The results indicated the presence of SiO_2 , Al_2O_3 and Fe_2O_3 as major oxides

And some traces of MnO, MgO, CaO, Na₂O, K₂O, TiO₂, P₂O₅ in the form of impurities

Table-1: values of trace elements.

**Table-2.** values of traceelements.

As	Ba	Be	Bi	Cd	Ce	Co	Cr
ppm							
0,8007	117,1714	0,7561	0,2981	0,0743	40,2884	4,26	70,5524
Cs	Cu	Dy	Er	Eu	Ga	Gd	Ge
ppm							
3,3022	11,9149	1,8503	0,9722	0,6891	35,7462	2,1793	2,1976
Hf	Ho	In	La	Lu	Mo	Nb	Nd
ppm							
7,1513	0,3574	0,0476	28,9338	0,1652	1,4584	18,1847	16,9186
Ni	Pb	Pr	Rb	Sc	Sb	Sm	Sn
ppm							
22,3332	18,9889	4,9762	15,2104	10,3	0,1421	2,9101	2,4378
Sr	Ta	Tb	Th	Tm	U	V	W
ppm							
33,4639	1,6413	0,3112	6,7255	0,146	2,1038	74,7965	2,4262
Y	Yb	Zn	c				
ppm	ppm	ppm	ppm				
8,6069	1,0443	56,3158	269,7383				

Forty four trace elements were determinate using ICP/AES; the obtained results are in agreement with those published by Jean Carignan *et al.* [11]. Among all these

impurities, Heavy metals such Sb, Cr, Cu, Pb, Zn Co and Ni has found to be toxic for human and environment[12].

The Table-3 presents the analysis results of major and minor elements, most of them like ceramics are very useful for industry applications.

Table-3. Values of major and minor constituents.

Eléments chimiques	SiO₂	Al₂O₃	Fe₂O₃	MnO	MgO	CaO	Na₂O	K₂O	TiO₂	P₂O₅	PF	Total
	%	%	%	%	%	%	%	%	%	%	%	%
	55,22	25,91	2,90	< L.D.	0,20	0,09	< L.D.	0,31	1,27	< L.D.	13,90	99,80

3. CONCLUSIONS

At the end of this study, these conclusions can be drawn: Makoua clay consists of the following minerals unevenly distributed: the quartz α , kaolinite, and interstratified chlorite/ montmorillonite which crystallize respectively in the hexagonal, triclinic and monoclinic system.

In this study, we have determined the mineralogical structure of the Makoua clay and the lattice constant of the components of this clay using the mineralogical analysis by X-ray powder and oriented blades.

The results showed that incorporation of wood waste has a strong effect on the crystallographic structure, making partially amorphous clay structure. It is found that the waste wood content doesn't influence the lattice constants of the components of the clay.

Chemical analysis of clay sample indicates kaolinite and SiO₂ as predominant component.

Some trace and heavy contamination has been identified to be toxic for some industry activities.



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