Original Research Article

Characterization and potential use of local clay materials: Case of clay material from Kombé, Republic of Congo.

ABSTRACT:

This work aims to evaluate the physicochemical and mineralogical properties of clay materials from Kombé in the Republic of Congo. Clays are recognized for their important characteristics, such as plasticity, high cation exchange capacity (CEC), and large specific surface area, which make them highly useful in various applications, such as ceramic manufacturing, water purification, soil decontamination, etc. This study completes missing data on Congolese clays by focusing on samples from Kombé. To assess the potential use of these clays, the following analyses were performed: particle size and Atterberg limits, X-ray diffraction (XRD), chemical analysis, cation exchange capacity (CEC) and specific surface area (SS), as well as chromate and nitrate ion adsorption tests. The results obtained show that the Kombé clay material is plastic and composed of 52% clay, 18% silt, and 30% sand, with a predominance of kaolinite and a significant amount of quartz. It has a CEC of 3 meq/100g and a SS of 16 m^2/g. In light of these results, this study reveals that the Kombé clay material can be used in traditional and structural ceramic applications (such as bricks, tiles, etc.), as well as in the treatment of polluted water as an adsorbent.

Keywords: Clay material, Kombé, characterization, adsorption, physicochemical and mineralogical properties.

1. INTRODUCTION

Clay materials, as a soil category, possess remarkable physicochemical properties, such as cation exchange capacity, high specific surface area, and notable plasticity [1]. These characteristics give clays significant adsorption and retention capacity for several chemical species, making them useful in many applications, including water purification, decontamination of polluted soils [2], ceramics [3], etc. In the Republic of Congo, several studies have already demonstrated the interesting properties of clay materials from certain localities, showing their potential in fields such as ceramics, civil engineering [3-6], and the environment [7-9]. However, there is a lack of data on clay materials present in several other localities, which limits the comprehensive assessment of their properties and potential applications. With this in mind, this research study aims to characterize the physicochemical and mineralogical properties of Kombé clay materials. To this end, techniques such as particle size analysis, Atterberg limit analysis, X-ray diffraction (XRD), chemical analysis, cation exchange capacity (CEC) measurement, and specific surface area analysis were used.

In addition, chromate and nitrate ion adsorption tests were conducted on these materials to better understand their environmental properties and thus assess their potential for practical applications.

2. MATERIALS AND METHODS

2.1. Location of the Sampling Site

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The materials used in this study were collected in Kombé, a neighborhood in Madibou District 8, Brazzaville, Republic of Congo. The geographic coordinates taken using a Sportiva 2/Two Nav GPS are: Longitude: 15.1682°E and Latitude: 4.3504°S. The following figure 1 illustrates the sampling area.

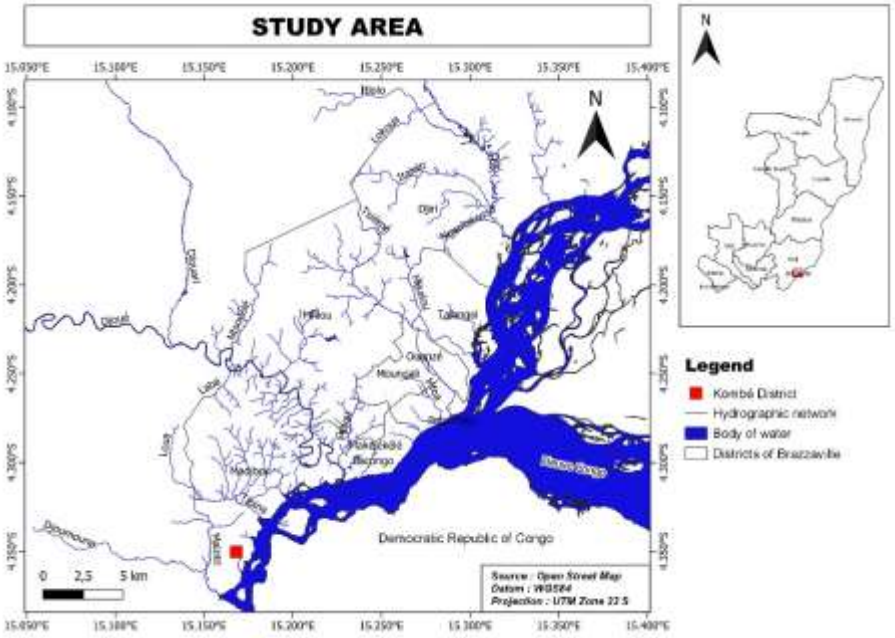

Figure 1: Map of the Sampling Site

2.2. Sample Preparation:

2.2.1. Description

The collected materials were dried, ground, and sieved using a series of sieves from 2 mm to 0.08 mm and a 56 µm sieve, followed by extraction of the fine fraction (fraction < 2 µm) by sedimentometry. The different samples were named K-B, K-56, and K-F, respectively, for the raw, unsieved Kombé clay material, the 56 µm sieve, and the fine fraction.

2.2.2. Equipment Used

The following equipment was used for grinding and sieving:

• A porcelain mortar and pestle;

• A series of six (6) sieves from 2 mm to 0.08 mm;

• A 56 µm sieve.

The following equipment was used for sedimentometry:

• A Bouyoucos-type torpedo-shaped hydrometer, graduated from 0.995 to 1.030 g/cm3;

• Two 2-liter test tubes;

• A mechanical immersion stirrer;

• A manual stirrer for homogenizing the suspension before testing;

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• A graduated thermometer;

• A stopwatch.

2.2.3. Reagents Used

• Sodium hexametaphosphate;

• Distilled water.

• Potassium chromate,

• Sodium nitrate,

• Hydrochloric acid solution,

• Sodium hydroxide solution.

2.3. Characterization Methods

2.3.1. Particle Size Analysis

Particle size analysis of Kombé clay materials was carried out at the Building and Public Works Control Office (BCBTP) in Brazzaville using the method described in standards NF P94 056 and NF P94 057 [10].

2.3.2. Atterberg Limits

Atterberg limits was Carried out at the Building and Public Works Inspection Office (BCBTP) in Brazzaville in accordance with standards NF P94 051 (1992) and NF P94 051 (1993) [11]

- ***Determination of the liquid limit:***

The liquid limit ($\mathbf{W_L}$) was carried out using the CASAGRANDE apparatus and was calculated using the following formula (I)

$$\mathbf{W_L} = \mathbf{W} \times \frac{\mathbf{N}^{0,121}}{\mathbf{25}} \qquad (I)$$

Where: N = the number of blows and W = the water content

- ***Determination of the plastic limit:***

The plastic limit (Wp) was determined using the following equation II:

$Wp = (Wp1+Wp2+Wp3)/3$ (II) Use the equation form

Wp_1, Wp_2, and Wp_3 are the plastic limits over the course of three tests.

- ***Plasticity Index (IP)***

The plasticity index was calculated by the difference between the liquid limit and the plastic limit according to the following relation (III):

$IP = W_L - W_p$ (III)

2.3.3. X-ray Diffraction

X-ray diffraction was performed at the Darmstadt University of Technology (TU Darmstadt - Joint Research Group Laboratory of Materials Design by Synthesis) in Germany, using a Bruker D8 diffractometer equipped with a copper (CuKa) anticathode. The angular analysis range was 5° to 90°.

2.3.4. Chemical Analysis

Chemical analysis was performed using an Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-AES) at the Centre for Petrographic and Geochemical Research (C.R.P.G.) in Nancy, France.

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2.3.5. Cation Exchange Capacity

CEC was determined using the METSON method. The procedure used is that described in AFNOR standard NFX31-130 [12]; the test sample was 2.5 g of clay material ground to 2 mm.

2.3.6. Specific Surface Area

The specific surface area of the Kombé material was determined using the B.E.T. method [13] in the laboratory of the Technical University of Darmstadt (TU Darmstadt - Joint Research Group Laboratory of Materials Design by Synthesis) in Germany.

2.3.7. Chromate and Nitrate Adsorption Tests

The chromate and nitrate adsorption tests were performed using the batch method, and the data obtained allowed us to construct adsorption isotherms based on the following equation:

Qads = (C0 - Ce). V / m [14] (IV) Use the equation form

Where:

C_0: initial concentration of the solute or adsorbate (mg/L);

Ce: equilibrium concentration of the solute in the solution (mg/L);

m: mass of the adsorbent (g);

Qads: amount of solute adsorbed per unit mass of the adsorbent (mg/g);

V: volume of the solution (L).

3. RESULTS AND DISCUSSION

Table 1: Sieving results of the Kombé sample

Sieve diameter	1mm	0,4mm	0,2mm	0,1mm	0,08mm
mR (en g)	0	4.1	22.8	58.2	69.9
%R	0	1.36	7.6	19.4	23.3
100-%R	100	99	92	81	77

Table 2: Results of sedimentometry of the Kombé sample

Grain diameter (mm)	0,105	0,075	0,055	0,038	0,025	0,017	0,012	0,0085	0,006	0,005	0,0016	0,0014
% of grains (d)	77	75	71	68	64	64	60	60	57	57	52	52

With:

mR: cumulative mass of rejects;

%R: percentage of cumulative rejects;

100-%R: percentage of passing particles;

% of grains d: percentage of grains with a diameter d.

Using these tables allowed us to plot the particle size curve for the Kombé sample (Figure 2), with the particle size (in millimeters) on the x-axis and the weight percentage on the y-axis.

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Coarse sand	Fine sand	Silt	Clay

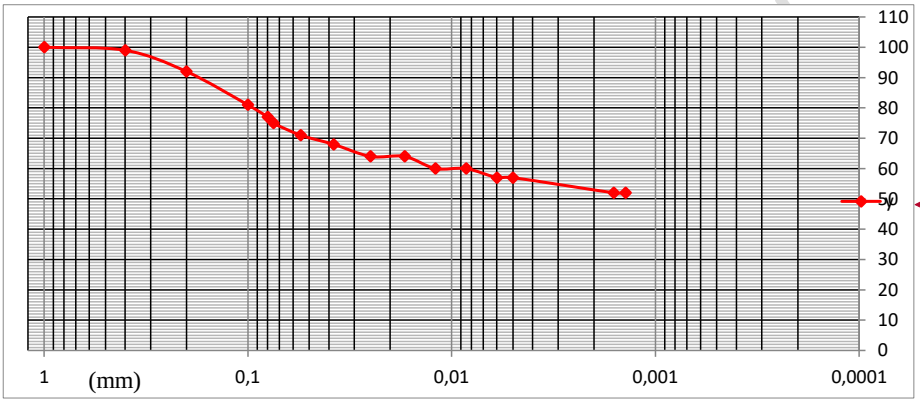

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Figure 2: Granulometric analysis curve of the Kombé sample

Analysis of this granulometric curve reveals the following granulometric composition: 52% clay, 18% silt and 30% sand. This result made it possible to position this Kombé sample in the texture triangle of Figure 3 below.

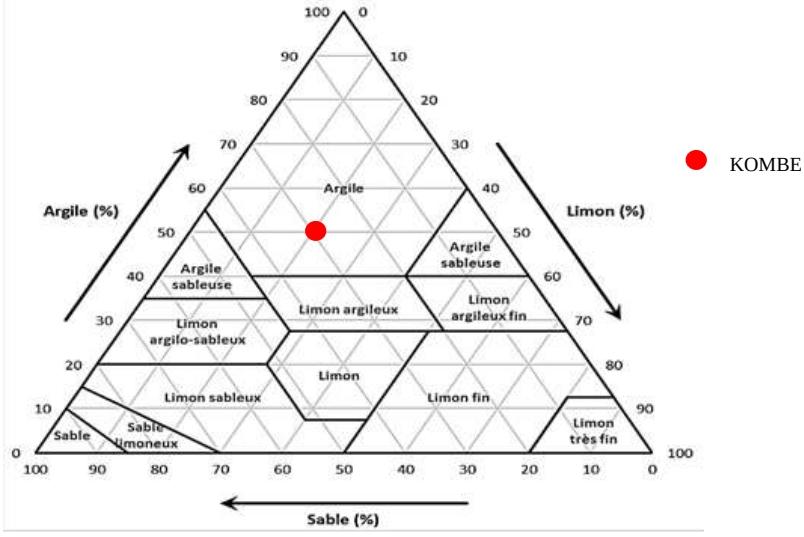

Figure 3: Sample positioning in the texture triangle "Soil Survey Manual"

Given the location of this sample within this texture triangle, the Kombé material has a clayey texture. This same observation was made by Banzouzi Samba et al. in a study of the material extracted at Ntokou. [5].

Atterberg limits of the Kombé sample

Table 3: Results of the Atterberg limits of the Kombé sample

Number of strokes	Liquid limit					Plastic limit		
	15	19	23	27	31			
Tare number	23	48	370	730	D	A	51	128
Water content	50,6	49,3	48,1	47,6	46,6	22,5	23,1	19,0
PI=27%	**W_L= 48,1%**					**W_P= 21,5%**		

The results of the Atterberg limits of this sample were positioned in the Casagrande abacus below:

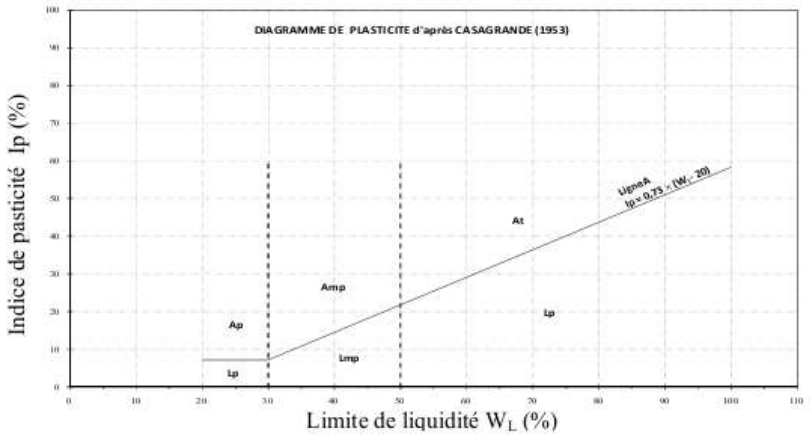

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Figure 4: Positioning of the Kombé sample in the Casagrande abacus

The liquid limit and plasticity index of the Kombé material were plotted on the Casagrande diagram. Given its position in this diagram, the Kombé material is a plastic clay material. The plasticity of the Kombé sample can be explained by its clay content, which is at the acceptable limit according to the results of the particle size analysis.

With this plasticity index (PI) value of 27%, this material can be used in the field of traditional ceramics, as demonstrated by Chedlia Ounissi et al. in the study and ceramic application of clays from southeastern Tunisia [15].

X-ray diffractogram of the Kombé sample

The diffractogram in Figure 5 below presents the results of X-ray diffraction on the Kombé material sample.

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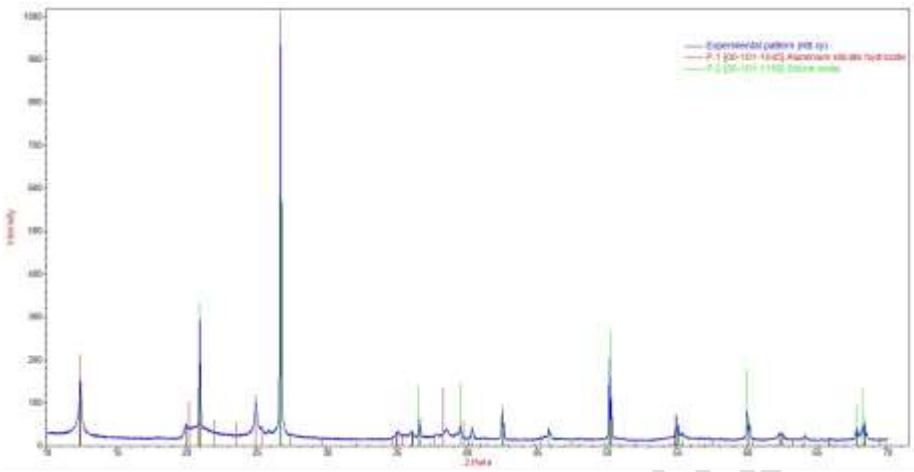

Figure 5: Diffractogram of the Kombé sample (K-B)

Analysis of the diffractogram of the Kombé material reveals the presence of clay minerals and crystalline phases, primarily in the form of tectosilicates.

Two main mineralogical phases were identified in this sample: quartz and kaolinite.

The set of diffraction peaks at 4.434; 4.225; 3.347; 2.229; 2.2332; 1.670; 1.540; 1.371; and 1.2554 Å confirms the presence of quartz as a very common tectosilicate. This suggests that quartz is a major component of this sample. Furthermore, the characteristic peaks of kaolinite (7.072; 3.557; 2.558; 2.495; 2.373; 2.335; 2.279; 2.2332; 2.124; 1.667; 1.488; 1.4333; 1.4165 and 1.381 Å) demonstrate that kaolinite is the predominant clay mineral in this Kombé sample.

It is noteworthy that the quartz peaks are significantly more intense than those of kaolinite. This not only means that the proportion of quartz is higher in the sample, but also indicates a higher degree of crystallinity for quartz compared to kaolinite. In other words, the quartz crystal lattice is more ordered and better formed than that of kaolinite in this sample.[16].

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Chemical Composition of the Kombé Sample

Table 4 below gives the results of chemical analysis of major elements (Si, Al, Fe, Mn, Mg, Ca, Na, K, Ti, and P) as a percentage of the most stable oxide.

Table 4: Results of the Chemical Analysis of Kombé

Sample	SiO_2 %	Al_2O_3 %	Fe_2O_3 %	MnO %	MgO %	CaO %	Na_2O %	K_2O %	TiO_2 %	P_2O_5 %	PF %	Total %
Kombé	73,04	15,29	1,92	<L.D.	0,24	<L.D.	<L.D.	0,42	0,88	<L.D.	8,06	99,84

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With: LD: detection limit and PF: loss on ignition at 1000°C

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Chemical analysis revealed that the Kombé clay is remarkably rich in silica (SiO_2), with a content of 73.04%. This high silica content is consistent with the results of the grain size analysis, which indicate a significant proportion of quartz (SiO_2). Silica is also a major component of kaolinite with the formula $Al_2Si_2O_5(OH)_4$, a predominant clay mineral species often associated with this type of deposit.

The calcium oxide (CaO) and sodium oxide (Na_2O) contents are very low, suggesting significant chemical alteration of the parent material. This low concentration of alkali and alkaline-earth elements indicates a near absence of minerals such as plagioclase (calcium-sodium feldspars), granite, and gneiss, which are often the main sources of these oxides. This phenomenon is consistent with the kaolinization process, where feldspars are decomposed into kaolinite, releasing alkali and alkaline-earth elements.

The loss on ignition (PF), measured at 8.06%, corresponds to the thermal decomposition of clay minerals, mainly kaolinite, which releases water of constitution at high temperatures. This value is typical of kaolinitic clays.

The SiO_2/Al_2O_3 molar ratio is an essential indicator for classifying and evaluating the refractory potential of clays. This ratio, being 4.77 for the Kombé sample, places this clay in the category of siliceous refractory clays, whose ratio is typically between 3.5 and 6.7 in the classification of refractory clays according to ISO10081-1, 2003 [17].

This value confirms the potential of Kombé clay for applications requiring high thermal resistance, such as refractory bricks or specialty cements. This result is consistent with the work of POUNTOUENCHI Amadou (2020) [18] on clays of similar compositions, which demonstrated their successful use in the manufacture of refractory ceramic materials.

The kaolinite content and quartz content in the Kombé sample were determined using the following formulas: Indicate the source of these formulas.

$$\%\text{Kaolinite} = \frac{\%Al_2O_3}{102} \text{ x } 258 \quad \text{(V)}$$

$$\%\text{Quartz} = \%SiO_2 - \%\text{Kaolinite x}\frac{60 \text{ x } 2}{258} \quad \text{(VI)}$$

The mineralogical balance of this material is 38.67% kaolinite and 55.05% quartz. Analysis of this balance confirms the significant amount of silica in the Kombé sample, given the relatively high percentage of quartz. This is consistent with the results of the grain size analysis, which showed a relatively high percentage of sand.

Cation exchange capacity of the Kombé sample

Analysis of the Kombé clay sample revealed a CEC of 3 meq/100g. This value is very low compared to other clays, but is entirely consistent with the mineralogical composition. The observed low CEC confirms the predominance of kaolinite in the sample.

Kaolinite is a 1:1 clay mineral. This crystal structure has very few isomorphic substitutions in its lattice, which explains its low surface charge and, consequently, its low CEC. CEC values for pure kaolinite typically range from 3 to 15 meq/100g [14]. The Kombé clay sample, with a value of 3 meq/100g, is in the lower part of this range, which confirms that the Kombé clay is a kaolinite. These results are consistent with the conclusions of several studies on the characterization of kaolinite deposits. For example, N.O. Pascal [19] demonstrated the effect of heat treatment on the CEC of kaolinitic clays. He observed that these clays reached CEC values of 13 and 15 meq/100g at 1000°C, which suggests that at low CECs, kaolinitic clays have high resistance to high temperatures, making them particularly suitable for the production of refractory materials. This result corroborates the potential use of Kombé clay in this field.

Specific Surface Area of the Kombé Sample

The specific surface area results for the Kombé sample is 16.2 **m^2/g**.

Analysis of the surface properties of Kombé clay highlights two key parameters: specific surface area (SS) and cation exchange capacity (CEC). The specific surface area of this sample is perfectly consistent with that of pure kaolinite, whose typical values range from 10 to 30 m^2/g [20]. This low

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Table 5: Mineralogical balance of the Kombé sample (in %)¶
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surface area is explained by the nature of kaolinite, a 1:1 phyllosilicate, where the layers are lightly charged and the surface charges are mainly located at the ends of the layers.

The low CEC, reported at 3 meq/100g, reinforces this conclusion. Such a low CEC is characteristic of kaolinite, as its crystalline structure undergoes few atomic substitutions, which reduces the number of negatively charged sites for cation exchange. The convergence of these two results, low specific surface area and low CEC, irrefutably confirms that kaolinite is the predominant mineral in the Kombé sample. Although the specific surface area and CEC values of Kombé clay are low, they are not negligible in terms of adsorption.

A study by Chen, M. et al. [21] showed that adsorption on kaolinite does not depend solely on specific surface area. Pore geometry and the specific interaction between cations and organic molecules also play a crucial role. These results suggest that Kombé clay, despite its low SS and CEC values, may have exploitable adsorption potential. These findings may lead Kombé clay to applications in water treatment and pollution remediation.

Adsorption tests of chromate and nitrate ions on Kombé clay

a) Adsorption of chromate ions on different fractions of Kombé clay materials

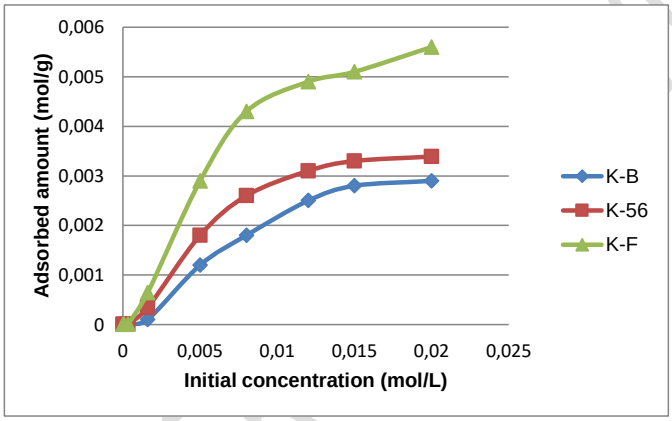

Figure 6: Adsorption isotherm for chromate ions on samples of K-B, K-56 and K-F

b) Adsorption of nitrate ions on different fractions of Kombé clay materials

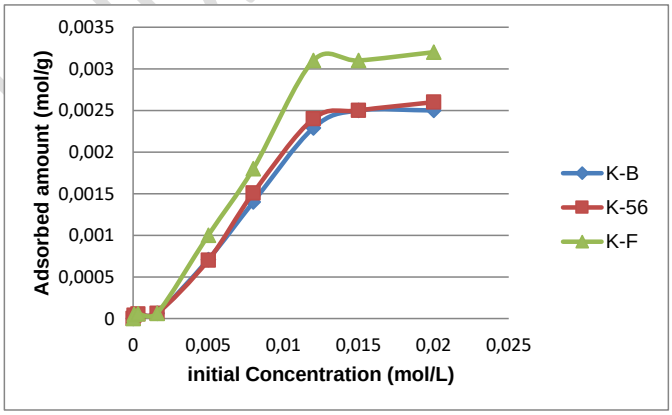

Figure 7: Adsorption isotherm for Nitrate ions on samples of K-B, K-56 and K-F

Analysis of the curves in Figures 6 and 7 reveals that the quantities of chromate and nitrate adsorbed on the unsieved crude sample (K-B) are lower than those adsorbed on the 56 µm sieved sample (K-56). These quantities adsorbed on the unsieved crude sample and on the 56 µm sieved sample are themselves lower than those adsorbed on the fine fraction (K-F).

The small difference in adsorption quantities between the 56 µm sample and the crude sample is due to the fact that the particle size distribution between these two fractions is not very different. However, the considerable difference observed between these two fractions (crude and 56 µm) and the fine fraction is directly related to the enrichment of fine particles in the latter fraction. Fine particles offer a larger specific surface area, which acts as an ultra-efficient sponge, capable of capturing many more chromate and nitrate ions. In other words, the finer the particles, the more efficient our adsorbent is [13], because in this case the adsorbent is enriched with clay particles which are responsible for adsorption; which consequently improves the adsorbed quantities of chromate and nitrate ions. This was explained in a study on the permeability of clay layers to polluting effluents [22], which showed that permeability increases when the clay content decreases. Another study on the migration of heavy metals in subsoils showed that they were completely retained at a depth of a few centimeters (36 cm) below the clay-waste limit for sites with 40 to 50% clay, and that it reaches a few decimeters (+ 50 cm) in sites with 20% clay [23]. At low concentrations of chromate and nitrate solutions, the difference between the adsorbed quantities on the three samples (K-B, K-56 and K-F) is small. Whereas at high concentrations, the difference is large between the fine fraction (K-F) and the other two fractions (K-B, K-56).

Referring to Giles' classification [24], the curves obtained in Figure 6 are S-type isotherms. This type of isotherm signifies that there is competition between the solvent and the solute for the occupation of the adsorbent sites [25]. In our case it would be competition between chromate or nitrate ions and water molecules, since both would seek to bind to clay sites with positive charges.

4. CONCLUSION

The objective of this work was to characterize the physicochemical and mineralogical properties of Kombé clay materials in order to understand their properties and evaluate their potential practical applications.

The results of the grain size analysis reveal that the Kombé material has a clayey texture with a composition of 52% clay, 18% silt, and 30% sand. Atterberg limits indicate that this material is plastic, with a plasticity index of 27%. These two results combined demonstrate that this material can be used in traditional and structural ceramics.

X-ray diffraction (XRD) analysis of this sample shows that the Kombé material is primarily composed of kaolinite, with significant amounts of quartz. Chemical analysis confirmed the presence of kaolinite due to its high silica (SiO_2) and alumina (Al_2O_3) contents, which are typical of kaolinitic clays. The silica-alumina ratio indicated that this material could be used as a refractory material.

A chemical property study revealed moderate values for cation exchange capacity (CEC) and specific surface area, but nevertheless suggested the possibility of interaction with various substances, including pollutants.

Adsorption tests for chromate and nitrate ions demonstrated that Kombé clay can capture and remove these pollutants from aqueous solutions. However, the adsorption efficiency was low and must be optimized to confirm the material's use in environmental remediation applications as a low-cost adsorbent.

In summary, the results of this study, particularly the mineralogical, physicochemical, and adsorption properties, suggest that Kombé clay material is a local resource that can be used for applications in

the production of structural ceramics (such as bricks and tiles), due to its kaolinitic nature and refractory properties, and also as an adsorbent for water treatment.

Future research should focus on optimizing the properties of this clay through various treatments (such as thermal activation, acid activation) to improve its adsorption capacity and better exploit it as a local resource.

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Code de champ modifié

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