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Physico-Chemical and Mineralogical Characterizations of Tchiky Clays (Thies, Senegal) for Pharmaceutical Uses

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Abstract

Our study focused on the valuation of Tchiky clays. This work aims to evaluate its properties to explore possible uses in pharmacy. Physico-chemical and mineralogical characterizations were carried out, as well as pharmacopoeial tests and an evaluation of the antioxidant activity. Thus, chemical analysis by X-ray fluorescence spectrometry gave silicon (55.65%), iron (15.73%), aluminum (13.53%), potassium (6.05%), titanium (3.98%), magnesium (2.10%), and calcium (0.82%). X-ray diffraction showed the presence of kaolinite, quartz and illite. This study also revealed that the sample studied was essentially a plastic clay of hard consistency, with average flowability. The evaluation of the antioxidant activity gave a percentage inhibition of 62.97% for a concentration of 7.5 g/l with an IC50 of 5.5 g/l. These results should allow use as an excipient in pharmacy, particularly in liquid, semi-liquid and pasty formulations.

Keywords

Clays, Tchiky, Minerals, Pharmacopoeia, Antioxidant Activity

1. Introduction

Clay is one of the raw materials used since antiquity. Indeed, thanks to its plasticity, it makes it possible to manufacture both containers necessary for daily life as well as statuettes. Clays are thus of considerable technical interest, both for geologists and agronomists, but also miners and public works engineers as well as doctors and pharmacists [1]-[5].

Clayey rocks are used as raw materials in ceramics and several industries thanks to their properties: absorbance and adsorbance, thixotropic, coverage, ion exchange, photovoltaic, colloidal, catalyst, etc. [6] [7].

Moreover, better knowledge of its structure, chemical composition, reactivity, etc., made more applications [8]-[13].

Thus, they are used as geological tracers. They can be used as a vehicle, fertilizer, bedding, and animal feed. Clays find their applications as catalysts for chemical or biochemical reactions, water and oil purifying agents, bleaching agents, and gas adsorbents. Clays are also used in the containment of radioactive waste, as drilling muds, as fillers in plastics, rubbers, and paints, in the paper industry and even in cooling and air conditioning systems [14] [15] [16].

In addition, the pharmaceutical and cosmetologically applications of clays are numerous. They can be used as active ingredients for their antacid and antidiarrheal effects by protecting the gastric mucosa and their gas and microorganism adsorbing effects [17] [18] [19]. As an excipient, they serve as a lubricant, anti-caking agent, and hardener in the manufacture of tablets, for example [20] [21] [22] [23].

In cosmetology, they are used as an active ingredient through the role as ion exchangers and as an excipient; through the role as an absorbent, opacifying, expansion, suspension, and viscosity control agent [22] [23] [24].

While the consumption of these products tends to become widespread, their production remains very little exploited in certain developing countries. In Senegal, clays are most often exported or used by cement factories. However, local workers have been interested in their valorization [25]-[32].

The village of Tchiky is in the rural community of Diass to Mbour department in Senegal. The Tchiky clay, a very abundant deposit, is exploited by private cement factories [25].

Thus, the objective of this work was to determine the physico-chemical and mineralogical characteristics of a clay sample from Tchiky as well as pharmacopoeia tests and finally, evaluate the antioxidant activity.

2. Experimental Part

2.1. Material

The Tchiky clay samples were collected in 2014 and stored at room temperature, away from humidity (**Figure 1**).

Figure 1. Raw Tchiky clay.

2.2. Methods

2.2.1. Morphological Analysis: Laser Granulometry

The purpose of the particle size analysis was to determine the percentages of different fractions of mineral particles constituting this material. This analysis was performed on crushed clay. The laser granulometry device was of the Beckman Coulter LS 230 type, the optical model of the Fraunhofer type, the dispersion liquid, and ethanol.

The samples were dispersed in ethanol until an optical density of approximately 9% was obtained, then the analysis lasted 60 seconds.

2.2.2. Physico-Chemical and Mineralogical Characterization

1) Loss of drying

The loss on drying was determined by heating 20 g of clay in an oven at 105˚C until a constant weight was obtained after 48 hours.

2) Elemental chemical composition: X-ray fluorescence spectrometry

The elemental composition of our samples was determined with an S2 Ranger Bruker AXS type device comprising a vacuum chamber, a Pd tube and an X Flash 145 Ev detector at 100,000 counts with Mn Kα lines.

3) Mineralogical composition

This test was carried by BRUKER D8 type diffractometer with monochromatic radiation such as the Kα line of copper.

4) Measurement of the cationic exchange capacity by the cobaltihexamine ion

Phyllosilicates are characterized by their cation exchange capacity, which is defined as the number of cations that can be substituted for the charge compensating cations to balance the electrical charge of 100 g of dry clay.

The cobaltihexamine ion $[(Co[NH_3]_6)^{3+}]$ has a very high displacement power on all exchangeable cations.

To a test sample of 2 g of Tchiky clay were added 40ml of cobaltihexamine solution (Sigma Aldrich, purity for analyses, Switzerland) 0.05N, (which corresponds to 50 mEq/l) of orange color, then stirring for 2 hours at room temperature. The cobaltihexamine ion was then measured by colorimetry using a JENWAY 6300 UV-Visible spectrophotometer at 472 nm.

A series of dilutions were carried out to verify the linearity of the absorbance of the cobaltihexamine solution as a function of the concentration.

5) Liquidity and plasticity limits

These limits are used to characterize the consistency of fine soils, which vary greatly depending on the water content. Their state ranges from solid when dried out to liquid when soggy. Between these two states, there is an intermediate state called plastic; the plasticity defines the property of the clay to undergo deformations without notorious cracking. The consistency of a clay is the rheological characteristic reflecting the plastic or non-plastic behavior and which changes with the variation of the water content. This index makes it possible to situate the behavior of clay in its natural state in relation to its plasticity defined by the Atterberg limits.

The liquid limit WL is the water content above which the clay becomes semi-liquid and flows under its own weight. It corresponds to the quantity of water necessary to dissociate the grains, so that the cohesive forces become negligible.

The plastic limit WP is the water content below which the clay loses its plasticity and becomes friable. It corresponds to the quantity of water which allows a relative movement of the grains without causing the rupture of bonds.

A sample of sieved clay (sieve Ø 0.5 mm), washed then dried, was wetted with water and homogenized. It was then placed in a dish and placed on the Casagrande device. The material in the cup was split into two parts by a groove and then subjected to several blows at the rate of one blow per second until the two parts came together on a length greater than or equal to 1cm. This number of strokes depends on the water content.

The same mixed material, used to determine the liquid limit, was desiccated a little and small sausages of 3 mm in diameter were made. These sausages before and after steaming gave a water content corresponding to the plastic limit WP.

6) Pharmacopoeia characterization methods

Clays are widely used by the pharmaceutical industry. Talc and kaolin officinalis are used in the composition of medicinal products and thus appear in the European Pharmacopoeia [33].

a) Density after settlement

The bulk density (MV) was obtained first by weighing a 100 ml flask (M1), then a volume V of 100 ml of the product was added slowly, leveling and without any possible compaction, with an accuracy of 1 ml. The bottle was then weighed with the product and the annotated result (M2).

Thus, the bulk density MV (g/ml) is given by the formula:

$$\text{MV} = \left(\text{M2} - \text{M1}\right) - \text{V}$$

The density after settlement (MT) is the value of the increased density, when the settlement of the powder sample contained in a container (graduated cylinder or weighing vessel) is mechanically caused. Mechanical shocks are caused by raising the container to a specified height and then dropping it, under the effect of its own weight.

The tapped density (MT) was obtained by putting a mass in a 100 ml flask then raising it to a certain height (arbitrarily) and dropping it until having a constant MT weight. The test was repeated three times.

b) Swelling power

2 g of substance to be analyzed, mixed with 2 ml of ultra-pure water, the mixture obtained should not flow.

c) Shear test

The shear test allows for a more complete and accurate evaluation of the flow properties of powders. This method is widely used for the study of pharmaceuticals. It allows the determination of multiple parameters, in particular the plasticity criteria representing the shear stress-strain, the angle of internal friction, the elastic limit in a confined environment, the tensile strength, as well as a series of derived parameters such as the flow coefficient and other indicators of flowability. Allowing more precise control of experimental parameters, it allows the determination of flow properties as a function of consolidation load, time, and other environmental conditions. It also makes it possible to determine the critical design parameters of hoppers and silos [33].

This study was carried out at the local Experimental of Research and Studies for Equipment Center (C.E.R.E.E.Q.) according to standard NF 94-071-1.

Three clay samples were prepared under the same conditions, sheared at the same speed but subjected to different vertical forces: 1.8 kg, 7.4 kg and 13.5 kg corresponding respectively to the 50 kPa, 150 kPa and 250 kPa levels. After having placed the box in the sealed frame fixed to the frame, the piston equipped with its draining plate was placed there. The original value of the force sensor was noted, as well as the value of the position of the sensor for measuring the relative horizontal displacement between the two half-boxes. The force was then applied to the upper part of the piston and the vertical displacement of the piston was measured as a function of time (every 10 seconds for 6 minutes).

d) pH

The use of universal colored indicator paper allowed the measurement of the acidity or the basicity of the sample in solution.

2 g of the sample are put in 100 ml of pure water and the hydrogen potential (pH) is measured after shaking.

2.2.3. Antioxidant Activity Evaluation

The evaluation of the antioxidant activity was carried out by visible spectrophotometry with the 1,1-diphenyl-2 picryl hydrazyl DPPH• reagent, following the protocols described by Popovici *et al.* [34] and Scherer *et al.* [35]. Thus, an antioxidant will have the ability to donate a singlet electron to the DPPH• radical. Ascorbic acid was used as a reference. 100 g of the sample are extracted with 100mL of a methanol solution under magnetic stirring for 24 hours, then filtration and vacuum evaporation to dryness. The dry residue is taken up with 4 mL of methanol for a stock solution. 3.9 ml of DPPH• (25 mg per 100 ml methanol or 63.4 μM) were added to 0.1 ml of the stock solution of methanolic extract of Tchiky clay. This mixture is placed away from light. After 30 min, the spectrophotometer reading at 517 nm was made. Each measurement was performed three times.

3. Results

3.1. Morphological Analysis: Laser Granulometry

According to these results (**Figure 2**), the Tchiky clay sample was essentially clayey, meaning grain size less than 2 μm but contains a low rate of very fine silt.

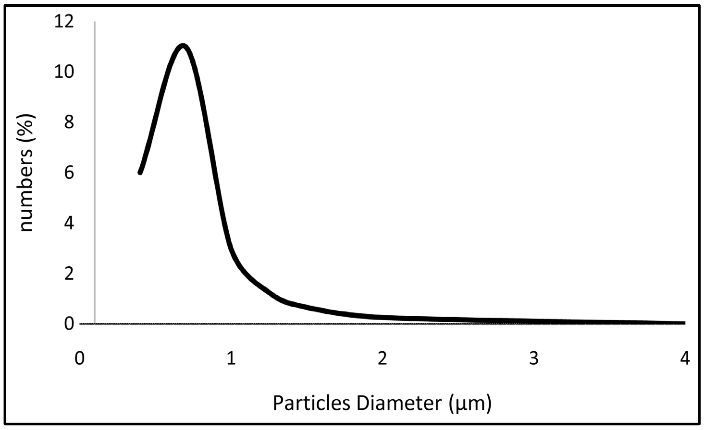

Figure 2. Laser grain size of the Tchiky clay sample.

3.2. Physico-Chemical and Mineralogical Characterization

3.2.1. Loss on Drying

A stay in the oven of 20 g of Tchiky clay at 105˚C for 48 hours showed a loss rate of 3.09%.

3.2.2. Elemental Chemical Composition

The analysis showed significant contents of Si (55.65%), Fe (15.53%) and Al (13.53%) (Table 1).

Table 1. Elemental chemical composition.

Elements	Si	Mg	Ca	Fe	Al	K	Ti	Cl	S	Zr	Zn
%	55.65	2.10	0.82	15.73	13.53	6.05	3.98	0.79	0.47	0.80	0.03

3.2.3. Mineralogical Composition

The diffractogram (Figure 3) showed that this Tchiky clay sample composed of kaolinite (11˚, 37˚, 50˚, etc.), quartz (21˚, 27˚, 40˚, 60˚ etc.) and illite (9˚ and 19˚).

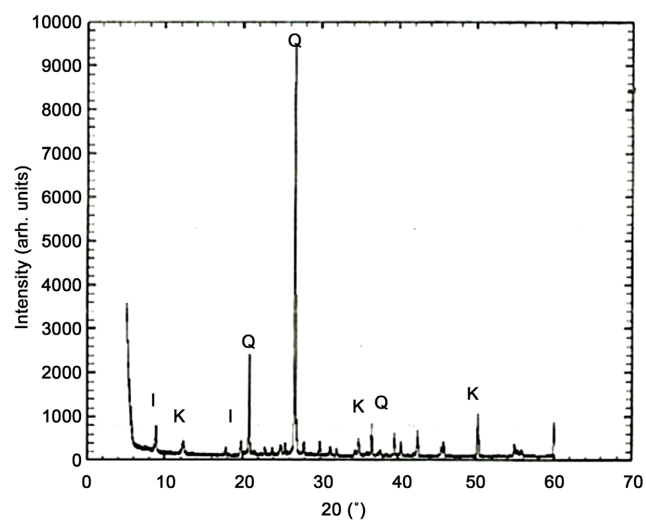

Figure 3. Diffractogram of the Tchiky clay sample.

3.2.4. Cation Exchange Capacity by the Cobaltihexamine Ion

The results obtained according to the established protocols were 43% for liquidity limits and 19% for plasticity limits.

From Atterberg limits found, the results of the associated indices were:

- plasticity index: Ip = 19;
- relative consistency index Ic = 2.1.

3.2.5. Bulk and Tapped Density

The results obtained with the sample of pulverized raw clay and according to the protocol described above were bulk density MV = 0.97 g/ml and density after settling MT = 1.36 g/ml.

This made it possible to find the compressibility and Hausner indices CI and HI respectively 25% and 1.33%.

Referring to the classification of Carr [33], the pulverized sample of Tchiky clays gave fair flowability.

3.2.6. Swelling Power

The mixture of 2 g of Tchicky clay sample with 2 ml of water sank. The Tchiky raw clay sample did not have good swelling power.

3.2.7. Shear Test

The calculations yielded the following results: 22˚ internal friction angle (or friction); 0.14 kg/cm^2 of Cohesion C.

3.2.8. pH

The pH paper showed a range between 6 and 7.

4. Antioxidant Activity Evaluation

From the stock solution of 2,2-diphenyl-1 picryl hydrazyl (DPPH•), various dilutions were carried out and the reading of their absorbances with the spectrophotometer were carried out after 30 minutes of contact and protected from light.

The antioxidant activities of the Tchiky clays were evaluated by calculating their percentage inhibition of the oxidative activity of the 0.6 mM DPPH• solution according to the method described by Scherer R. *et al.* [35]. Percentage inhibition of 62.97% was obtained for a concentration of 7.5 g/l of the stock solution. As the percentages of inhibition vary according to the concentrations, the IC50 of 5.5 mg/ml was obtained. The IC50 of the ascorbic acid used was 0.16 mg/ml.

5. Discussion

Laser granulometry showed that the studied Tchiky clay sample was 99% clayey and subsequent acid treatment can dissolve fine silts and make them purely clayey.

The diffractogram of the raw clay showed the majority presence of kaolinite (11˚, 37˚, 50˚, …) and quartz (21˚, 27˚, 40˚, 60˚, …). These results were confirmed by studies on the Tchiky clay [21] where the diffractogram revealed the majority presence of kaolinite and quartz. Illite (9˚ and 19˚) was also found in the sample. Studies on Cameroon clays have shown kaolinite, micas, quartz, hematite, maghemite, goethite, sanidine, microcline, and anatase mainly [36]. A study on Portuguese geological materials has reported mostly the presence of kaolinite and quartz [37].

Kaolinite, illite and micas are used in therapy and cosmetology. Kaolinite can be used as a mineral excipient because of its physico-chemical characteristics close to those of medicinal kaolin. Kaolin is a naturally hydrated aluminum silicate stripped of its impurities. It is a water-insoluble, hydrophilic, very slightly hygroscopic product. It is used as an excipient is (topical and oral adsorbent). It is well tolerated by the skin and mucous membranes and can be administered orally [19] [20] [38]. Kaolin is used as a factor XII activator in the test for exploring the endogenous and common pathways of plasma coagulation named Test of Cephalin Kaolin (TCK). Elsewhere, it is studied as a hemostatic in post-tonsillectomy, the induced hemorrhage being the main complication. Kaolinite easily and quickly adsorbs many viruses. Double-blind protocols with randomization on adults and children have shown the shortening of the duration of diarrhea, and the decrease in stool frequency following treatment with kaolin.

Illite/kaolinite type clay is used in France. Illite is used as a thick poultice to be applied to various bruises (sprains, strains, etc.), or to absorb impurities (organic waste, micro-organisms, etc.) [2] [24].

The chemical composition of the Tchiky clay sample showed a level of silica equal to 55.65%, alumina at 13.53%, iron oxide at 15.73%, 3.98% titanium and 2.10% magnesium. A study [27] obtained the following rates: silica 62.1%, alumina 13.6%, iron oxide 8.38%, titanium oxide 13.05%, and magnesium oxide 0.73%, thus showing practically identical contents except for those of iron oxide and titanium oxide. Other studies have shown silica levels of 53.67%, alumina of 22.33%, iron oxide of 8.55%, titanium oxide of 1.25%, and oxide magnesium of 0.89% [39].

Silica, being very electronegative, makes it possible to neutralize electropositive charges.

It is used in therapy and cosmetics as a thickening agent to stabilize dispersed systems, texturizing agent for pasty systems such as toothpaste, powder settling retarder for the manufacture of suspensions, coatings, suppositories, and sticks, "anti-caking" agent to facilitate resuspension, adsorbent support for liquid and pasty active ingredients, anti-caking agent for wet and cohesive powders (grinding aid), flow lubricating agent widely used in the manufacture of tablets and the filling of capsules, protectors of dry forms against humidity [21].

Silicon is very important because plays a role in many physiological activities. Its presence allows the proper functioning of certain metabolisms and the establishment of molecular structure. Thus, various properties are attributed to it, contributing to the architecture and elasticity of connective tissue. Therefore, it is essential to the formation and regeneration of the skin, joints, nails, and hair, bone calcification, healing, vessel flexibility, and protects the arteries from cardiovascular risks. It contributes to the improvement of endocrine functioning, to the protection of inflammation. Silicon opposes the proliferation of fatty tissue, protects against the harmful effects of aluminum (at bone and brain level), against tissue aging [21].

Aluminum is present in the mineral kingdom in the form of aluminum silicates. Aluminum hydroxide is used as an activator of vaccine immunity by creating, after subcutaneous injection, a slight inflammatory reaction. Aluminum is proposed in antacid medication, in gastrointestinal protection in the form of various salts such as carbonate, hydroxide, silicate or phosphate [21].

Iron is known for its physiological role as an oxygen carrier in the blood. It is present in the hemoglobin of red blood cells which carry oxygen to all cells. It is also present in myoglobin, a substance like hemoglobin, which helps the muscles to store oxygen. An iron deficiency will then cause anemia in the patient which is compensated for by medicinal intake of different iron salts (chloride, sulphate, fumarate, oxalate, gluconate, and ascorbate). Iron oxide is used in pharmacy and cosmetology as an excipient, as well as water-insoluble coloring pigments, and is therefore completely harmless [19].

The cation exchange capacity (C.E.C.) found (14.85 meq/100g) confirmed the presence of kaolinite whose C.E.C. is between 10 - 20 meq/100g [38].

The water content obtained (3.09%) is low compared to other clays, for example, Mbodiène attapulgite (8.4%) [31] [32].

The determination of the liquid limit (WL = 43%) and of plasticity (WP = 19%) made it possible to calculate the indices of plasticity (IP = 19), of relative consistency (IC = 2.1). Thus, according to these results, our sample is a plastic clay with a hard consistency. These limits of liquidity and plasticity were determined in this study because they provide valuable information in the context of a liquid, semi-liquid, or pasty formulation.

The results of the bulk density and the density after settling, as well as the compressibility and Hausner indices which were deduced, made it possible to conclude that the sample of pulverized Tchiky clay has an average flowability. This is linked to the fact that the sample studied is a raw material that has not undergone any treatment. Adjuvants could be used to improve flowability. This is a solution that was advocated in a study of various clayey rocks, some of which had poor flowability. The determination of this property is important in the context of the formulation of dry forms [35].

The pH measurement gave a value between 6 and 7. This result remains within the pH limits of medicinal kaolin which are between 4 and 7.5 and indicates that, like kaolin, the raw sample of clay de Tchiky may contain soluble salts. Indeed, it was reported in a study on kaolin that the latter was more likely to contain soluble salts if its pH values were above 6.5 [40].

The cation exchange capacity of the raw sample of Tchiky clay with a 0.05N cobaltihexamine solution was 14.85 mEq/100g of material.

The limits of liquidity and plasticity found were respectively 43% and 19%, they made it possible to determine a plasticity index equal to 19 and a relative consistency of 2.1. These results led to the conclusion that the raw sample of Tchiky clay is a plastic clay with a hard consistency.

A water content of 3.09% was found.

The compressibility indices of the order of 25% and of Hausner of the order of 1.33 which were deduced from the bulk density and after settling, made it possible to conclude that the raw sample of clay of pulverized Tchiky has an average flowability.

The antioxidant activity of Tchiky clay was evaluated by the spectrophotometric method using 2,2-diphenyl-1-picrylhydrazyl (DPPH•).

The DPPH• test is used according to the protocol described by Scherer et al. The DPPH• radical is one of the most used substrates for the rapid and direct assessment of antioxidant activity due to its stability and simplicity of analysis [34] [35].

The DPPH• test showed that the sample studied has an interesting antioxidant power with an IC50 of 5.5 mg/ml, against an IC50 of 0.16 mg/ml obtained with ascorbic acid used as reference. This weak antioxidant activity was been observed by others authors [39].

6. Conclusions

This study was conducted with the aim of enhancing our local resources, and has given encouraging results regarding the use of Tchiky clay in the field of pharmacy and cosmetology, particularly in liquid, semi-liquid, and pasty formulations.

However, further studies will have to be carried out, to be able to use it as an excipient in tablet forms as a thick poultice to be applied to various bruises, etc.

Finally, the use of clays in cosmetology gives good prospects and Tchiky clays because of their abundance and their properties could also be used in this field.

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Conflicts of Interest

The authors declare no conflicts of interest.

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