Physicochemical and mineralogical characterization of clays from the Tcheriba zone in the Boucle of Mouhoun region (Burkina Faso)

Dieudonné Muvunyi [1,2], Mahamane Sani Ousmane [3*], Moussa Bougouma [1]**

[1]1Laboratory of Materials and Environmental Chemistry (LCME), UFR/Sciences et Technologies (ST), Norbert ZONGO University, Avenue Maurice Yaméogo, BP 376 Koudougou, Burkina Faso
[2]Laboratory of the Office of Mines and Geology of Burkina, 01 BP 601 Ouaga 01 – Burkina Faso
[3]Departement of Chemistry, University of Agadez, BP 199, Agadez, Niger
*Corresponding author, Email address: Mahamane Sani Ousmane, msaniousmane@gmail.com
**Corresponding author, Email address: Moussa Bougouma bmoussaraphael@gmail.com

Abstract

This work presents the results of the physicochemical and mineralogical characterization of clays from the Tcheriba zone in the Boucle of Mouhoun region (Burkina Faso). Various analysis methods such as thermo gravimetric analysis (TGA) and differential thermic analysis (DTA), infrared spectroscopy (FTIR), X-ray diffraction (XRD) and ICP-MS elemental analysis have been used. The results show a significant amount of Quartz, Kaolinite and low amounts of Muscovite as well as Illite and Microcline in traces. By XRF chemical analysis, the predominant constituents are silica and alumina. The low content of heavy elements such as Mo, W, Ag, As, Cd is a guarantee of the quality of these clays from an environmental point of view with regard to their use in the manufacture of ceramic membranes for water filtration.

1. Introduction

The physico-chemical and mineralogical properties of clays arouse particular interest in several fields of application including water treatment, use as a catalyst, adsorbents, barriers for pollutants, paint, etc. This is why, several researches are interested in the development of new mineral membranes based on low-cost raw materials such as clays [1-8]. Previous work has shown that several varieties of local clay materials have the ability to fix significant amounts of metallic trace elements from solutions [9-14]. Recently, studies carried out on two clay materials from the Niger River valley have shown their effectiveness in removing copper from drinking water [7]. Other studies on clay materials from Bikougou (Gabon) have shown their effectiveness in eliminating manganese (II) in aqueous solutions [14]. Another example is the recovery of Zinc from sulphate medium by purified bentonite of Maghnia (Algeria) impregnated by an organophosphorus acid [15].

Clay rocks are made up of mixtures of clay minerals, with which endogenous minerals (quartz, feldspars, micas, heavy minerals) or sedimentary (anatase, sulphates, etc.) are associated [5]. Because of the progress of modern analysis techniques, in particular differential thermic and thermo gravimetric analyses, infrared spectroscopy and especially X-ray diffraction, clay minerals are beginning to be well known. In this context, this study aims at the use of different characterization methods for the

physicochemical and mineralogical study of local clay materials of the Tcheriba area in the Boucle of Mouhoun region (Burkina Faso), a pottery area very rich in clay martial. These materials used locally in pottery or ceramics are likely to be used in the manufacture of ceramic membranes for water filtration. Thus, the main long-term objective which is the implementation in the laboratory of new ceramic membranes based on local materials with improved performance which are inexpensive, more accessible, and more efficient. This requires a preliminary study of the physicochemical and mineralogical of these materials

2. Materials and methods

2.1 Source

The samples of the clay materials analyzed were taken from the Tcheriba area in the Boucle of Mouhoun region. The sample was taken between 15 and 40 cm deep. Four samples were collected at two sites (Sao and Tikan) and the GPS coordinates of these sites are shown in **Table 1**.

Table 1. Coordinates of sampling sites

Samples	GPS Coordinates	Sites
S1	N12°15.77' W003°00.74'	Sao
S2	N12°15.61' W003°00.30'	Sao
T1	N12°18.27' W003°09.12'	Tikan
T2	N12°18.33' W003°09.12'	Tikan

2.2 Drying and grinding

After drying at 40°C, the samples are ground and then sieved to obtain a powder of less than 250 μm and then less than 63 μm. The sieve obtained is subjected to a series of physico-chemical analyses such as density, loss on ignition, pH.

2.3 Shrinkage and plasticity

A mass of 750 g of each raw sample is introduced into a plastic bucket containing hot water at approximately 100°C. The sample is allowed to stand for at least 24 hours to allow good dispersion of the particles. Next, sieving is carried out using a sieve 63 μm in diameter and the thin part is poured onto a plaster which absorbs the water in order to obtain a fairly consistent paste. The pastes obtained are then used to make briquettes in order to determine the rate of shrinkage after drying at 110°C. The rate of shrinkage or shrinkage is determined by measuring a marked dimension of the briquette before and after drying at 110°C for 24 hours. The residues that do not pass through a 63 μm sieve are dried and weighed to determine the residue rate in relation to the initial weight (750 g) in order to arrive at the recovery rate characterizing the clay material.

2.4 pH

A 10% m/v clay solution is prepared with distilled water. After stirring and homogenization, the mixture is left to stand for 4 hours at 25°C, to allow the ions to pass into solution, then the reading is taken directly on a HANNA type pH meter [5].

2.5 Density

The density was measured using a helium pycnometer which is used to measure, at a given temperature, the density of solids or liquids. This works with a helium bottle and makes it possible to precisely determine the volume of a solid sample (massive, divided or porous) of known mass. The density corresponding to the pycnometric density is given by the following relation:

$D = m/Vp$ **(Eqn 1)**

m = mass of the sample (about 5 g) and Vp = Volume of the powder (cm³)

2.6 Thermo gravimetric analyse-Differential thermic analyse (TGA-DTA)

The differential thermic analysis measurements are carried out on the clay fraction of each sample under atmospheric pressure and using the SETARAM device equipped with a device for attaching the DTA sensor to the weighing module to perform the simultaneous TGA measurement. and DTA on the same sample. Thermo Gravimetric (TGA) and Differential Thermic (TDA) curves of the clay were produced using Sigma Plot 10 software.

2.7 XRF and ICP-MS chemical analysis

The analyses for the major elements were carried out by X-ray fluorescence spectrometry (XRF) using an Axios-Advenced type spectrometer provided by PANAlytical. The samples were prepared using the molten pearl technique: the sample, previously heated to 1050°C to free it from water and organic substances, is mixed with a flux consisting of lithium metaborate and tetraborate. This mixture is placed in a platinum crucible and heated in an oven (Katanax X-300 fluxer) at 1050°C. A transparent bead, ready to be analysed, is thus obtained after cooling. The analysis of trace elements was carried out by digestion with three acids (hydrochloric, nitric and hydrofluoric) in the microwave oven and by determination with the ICP-MS (Inductively Coupled Plasma Mass Spectroscopy) brand Perkin Elmer of the Elan 9000 type equipped of a plasma. Mineralogical analyzes by X-Ray Diffraction (XRD)

2.8 Mineralogical analysis by X-Ray Diffraction (XRD)

The diffractogram used for the X-ray diffraction is of the X'pert Pro MRD type from the PANalytical company of the PHILIPS brand, equipped with a copper anticathode (monochromatic radiation CuKα (λ=1.54056 A°) operating at a voltage of 45 kV and an emission current of 30 mA. The scanning speed is 0.039 °/s. For qualitative peak identification, the data is processed using MDI-JADE 2010 software.

2.9 Infrared spectroscopy FTIR

FOURIER transform infrared spectroscopy (FTIR) was performed using a Perkin Elmer Precisely Spetrum 100 spectrometer over the spectral range of 400-4000 cm⁻¹. The pressed disc technique was employed by mixing a few milligrams (2 mg) of the sample powder and (200 mg) of potassium bromide (KBr).

3. Results and Discussion

3.1 Shrinkage and plasticity

The shrinkage rate is given in **Table 2** below. The results show that the shrinkage rate being in the vicinity of 7%, these clays have a fairly good plasticity. To improve plasticity, a degreaser can be added to lower the shrinkage rate to less than 7%. Knowledge of the shrinkage rate allows control of the dimensions of the molds according to the desired dimensions of the finished products. Regarding the

recovery rate, the results obtained show good recovery for all the samples (greater than 70%) as indicated in Table 2.

Table 2. Shrinkage rate and recovery rate of the clays studied

Samples	Shrinkage rate (%)	Recovery rate (%)
S1	6.90	78.39
S2	6.94	77.01
T1	7.56	79.23
T2	7.59	73.52

3.2 Density and pH

The determination of the pH is necessary to quantify the contribution of the acidity when the solid is in contact with the solution. We note that the pH value around 6 (**Table 3**), reveals the acidity of the clay samples which would be due to the low contents or the absence of soluble salts with a basic character such as alkaline carbonates and bicarbonates or silicates, and which are generally included in the composition of clay. In the literature, pH is the most important parameter controlling the adsorption of heavy metals reported [16]. An acid pH would slow down adsorption by competition between the H_3O^+ ions in solution and the ions in solution. Whereas at basic pH, adsorption is improved by increasing the number of negative sites [17-18]. Thus, for use of the clays studied in this work, for the adsorption of metals, it would be important to adjust the pH to values greater than 7. The density value of the four samples is around 2.63. This value is high and could reflect a low permeability of these clays. The higher the density, the less porous the rock and the lower the permeability.

Table 3. Density and pH of the clays studied

Samples	Ph	Density
S1	6.2	2.65
S2	6.4	2.63
T1	5.0	2.63
T2	5.0	2.61

3.3 Thermal analysis

For clays S1 and S2, two main endothermic peaks are observed on the DTA curve (**Figures 1 and 2**). The first peak between 22°C and 200°C corresponds to the departure of hygroscopic water from the illite sheets [19]. This departure of water leads to a loss of about 2.30% in mass on the TGA curve, corresponding to 1.40 mg for 62.282 mg of S1. The second peak between 320°C and 920°C is characteristic of the elimination of constitutional water resulting from the release of hydroxides belonging to the kaolinite and illite or quartz networks. It corresponds to the dehydroxylation of the clay structure [19,20].

An endothermic quartz transition hook is observed around 573°C. It may be the transformation of α quartz into β quartz ($\alpha SiO_2 \rightarrow \beta SiO_2$) and this small hook is therefore linked to the presence of quartz [19,21]. Between 900 and 1000°C, the exothermic peak is due to the structural reorganization of clay minerals or crystallization [19, 22-23]. This exothermic phenomenon is not associated with a considerable loss of mass. The exothermic peak observed could be due to the formation of mullite. At this temperature, the metakaolinite undergoes a structural reorganization associated with an exothermic

peak on the ATD curve and can form a spinel (mullite over-stoichiometric in Al_2O_3) or a γ-alumina [19,24]. At high temperature, kaolinite undergoes dehydroxylation, associated with endothermic mass loss, which destructures the crystal lattice. Kaolinite can be transformed into metakaolinite which is however supposed to undergo mineralogical transformations during the thermal analysis. Then there should be the formation of mullite. Heat treatment above 1000°C then leads to decomposition into recrystallized phases such as mullite and a liquid phase (viscous flow) assimilated after cooling to a vitreous phase [25].
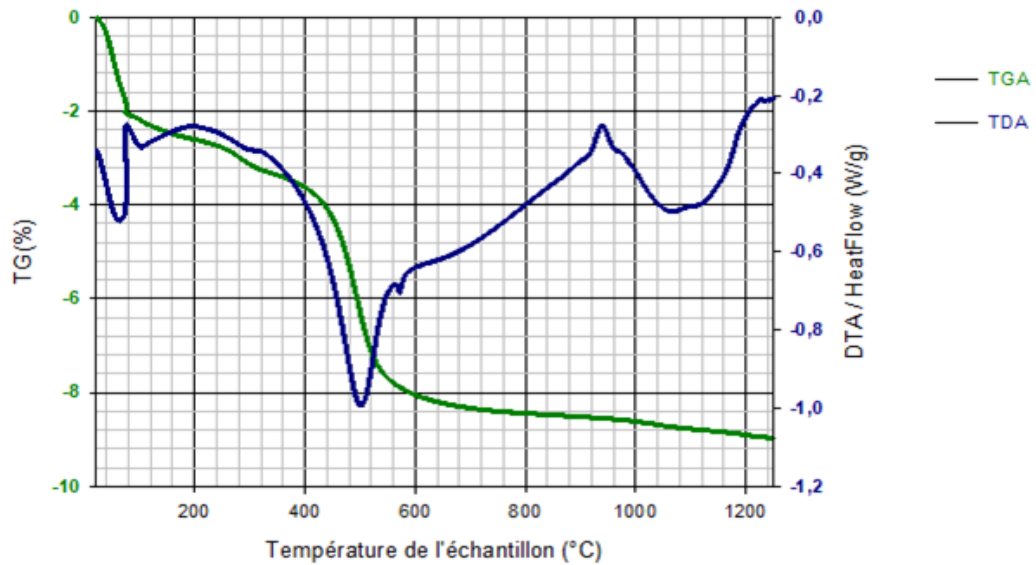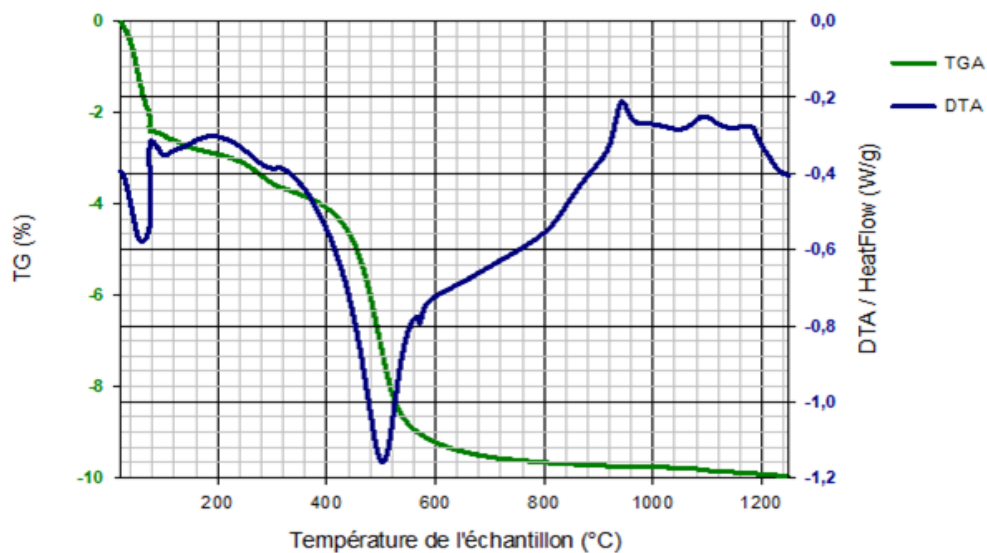

Figure 1. TGA/DTA curves of sample S1.

Figure 2. TGA/DTA curves of sample S2.

The thermograms of the T1 and T2 clays (**Figures 3** and **4**) show similarities as in the case of S1 and S2 with some observed differences which would result from the chemical composition of the minerals and their relative contents. A first endothermic peak is observed between 22°C and 400°C corresponding to the departure of hygroscopic water from the illite sheets [19]. This leads to a loss of approximately 2.00% in mass on the TGA curve, corresponding to 1.23 mg for 62.282 mg of T1 and T2. The second peak between 400°C and 920°C is characteristic of the elimination of the water of constitution resulting from the release of hydroxides belonging to the networks of kaolinite and illite

or quartz. It corresponds similarly to the previous result (S1 and S2) to the dehydroxylation of the clay structure [19,20]. Heating between 450°C and 600°C leads to the endothermic dehydration of kaolinite to metakaolin ($Al_2Si_2O_5$), which releases around 5% of the initial mass. And at a temperature of about 950°C, metakaolin is sintered by exothermic reaction into more stable minerals such as mullite and quartz [22]. It appears from these spectra that samples S1, S2, T1 and T2 contain illite, kaolinite and quartz.
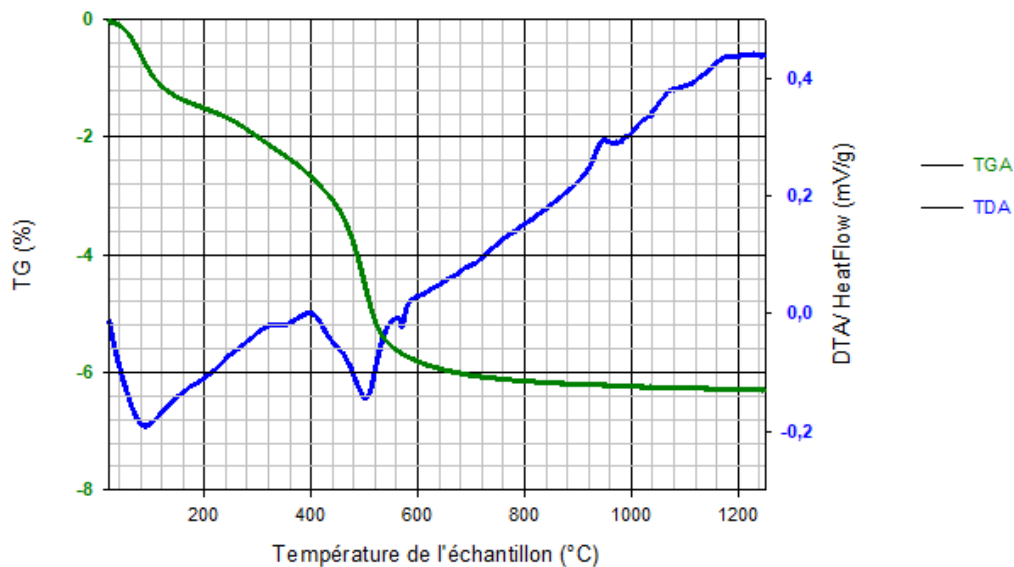

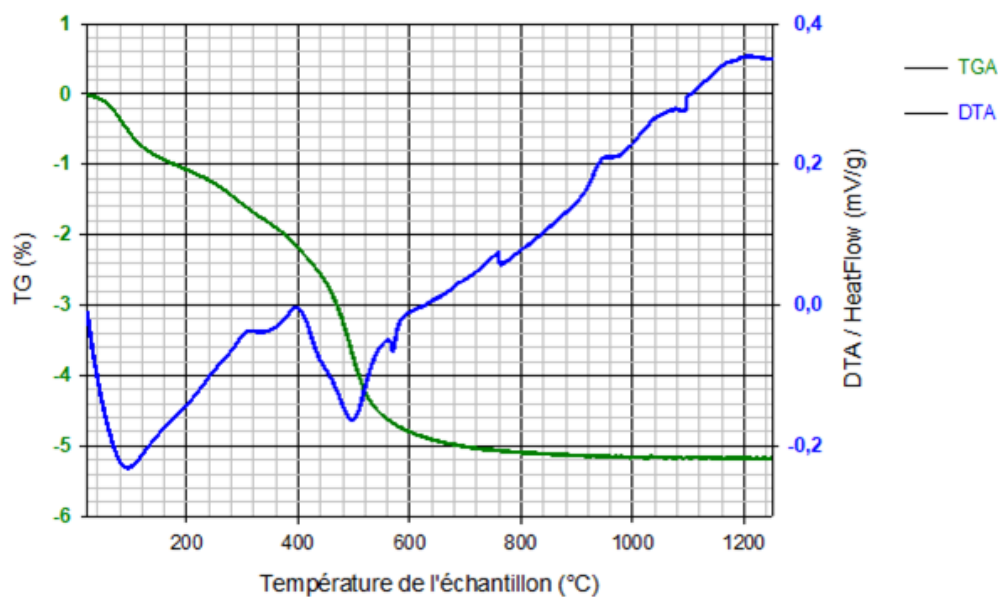**Figure 3.** TGA/DTA curves of sample T1

Figure 4. TGA/DTA curves of sample T2.

3.4 XRF and ICP-MS chemical analysis

Table 4 gives the results of the XRF analysis and the loss on ignition (PAF) at 1050°C. The main oxides such as SiO_2, Al_2O_3, Fe_2O_3, TiO_2, K_2O contained in these samples are typical of clay minerals. Silica appears as the main component of these samples with more than 50% in mass proportion. Alumina is the second component with more than 10% for all samples. The SiO_2 and Al_2O_3 content shows that these clays are aluminosilicates. The K_2O contents in a minor proportion (around

1%) probably indicate that these clays are poor in illite [19]. The SiO_2/Al_2O_3 ratio of 4.15 and 3.45 respectively for S1 and S2 is higher than the classic value for bentonites which is 2.7. That of samples T1 and T2 respectively 7.17 and 7.82 is much higher than the latter. This difference indicates the presence of free quartz in the clay fraction [26].

Table 4. Chemical composition of major oxides of S1, S2, T1 and T2

Samples	S1	S2	T1	T2
SiO_2	68.26	63.87	76.90	78.86
Al_2O_3	16.46	18.52	11.61	10.09
Fe_2O_3	4.30	5.54	2.13	2.36
TiO_2	1.35	1.30	1.61	1.74
Mn_3O_4	0.05	0.04	0.02	0.05
MgO	0.29	0.28	0.25	0.25
CaO	0.13	0.17	0.06	0.08
Na_2O	0.00	0.00	0.03	0.02
K_2O	0.96	0.86	0.92	0.95
P_2O_5	0.04	0.04	0.04	0.04
V_2O_5	0.01	0.02	0.01	0.01
Cr_2O_3	0.01	0.01	0.01	0.01
SrO	0.01	0.01	0.00	0.01
ZrO_2	0.12	0.12	0.13	0.12
BaO	0.10	0.10	0.10	0.12
PbO	0.01	0.01	0.00	0.00
HfO_2	0.01	0.00	0.00	0.01
LI	8.14	9.33	6.41	5.57
Total	99.98	99.98	99.98	99.99

LI : Loss of Ignition

On the other hand, the Alumina/Silica ratio provides information on the permeability of the material towards to humidity. The greater this ratio, the greater the permeability [27]. In our case, this ratio (Al_2O_3/SiO_2) respectively around 0.24; 0.28; 0.12 and 0.13 for S1, S2, T1 and T2 is small. These low values could reflect a low permeability and a low percentage of humidity of these clays. These results are consistent with the density values found in **Table 3** which indicated low permeability. The loss on ignition (LI) recorded at 1050°C corresponds to a mass loss of 8.14% to 9.33% for samples S1 and S2; from 5.57% to 6.41% for samples T1 and T2. This could justify the mass losses observed on the TGA curves (approximately 9% for S clays and 5% for T clays). Moreover, This Loss on ignition reflects that the amount of the organic matter in this sample is low compared to that of mineral fraction. The analysis of trace elements by ICP-MS gave the results recorded in **Table 5**.

Table 5. Chemical composition in trace elements (in ppm) of S1, S2, T1 and T2

ID	Ag	As	B	Be	Bi	Cd	Ce	Co	Ga	Ge	Mo	W	Li
S1	1.07	6.00	25.42	1.12	0.26	0.07	0.48	9.93	17.24	1.11	0.77	1.50	12.79
S2	1.12	5.33	26.08	1.11	0.94	0.09	0.04	8.83	17.42	1.12	0.81	1.38	11.64
T1	0.36	4.50	40.99	0.97	0.43	0.11	4.04	5.64	18.54	1.05	0.54	1.82	14.96
T2	0.50	4.50	39.16	0.98	0.33	0.08	18.38	9.66	19.34	1.03	0.61	2.02	13.51

The low contents of heavy elements such as Mo, W, Ag, As, Li, Cd is a guarantee of the quality of these clays on the environmental plan regarding their use in the manufacture of ceramic membranes for water filtration.

3.5 X-ray diffraction analysis (XRD)

The diffractograms of raw samples S1, S2, T1 and T2 are shown in **Figure 5**. The data were processed using the MDI-JADE 2010 software for qualitative peak identification. This qualitative identification indicates that the samples studied contain quartz, kaolinite, illite and microcline. The quantitative composition of the different phases determined using a rapid detector (PiXel) according to the Rietveld method more or less confirmed the results obtained qualitatively. Quantitative mineralogical compositions are recorded in **Table 6**. The dominant presence of free quartz in proportion greater than 50% followed by kaolinite in proportion greater than 30% in all samples. Illite is in a minor proportion (1% to 3%). These results are in agreement with those of Fluorescence X which shows high proportions of SiO_2 (Quartz) and AL_2O_3 as well as the low proportion of K_2O.
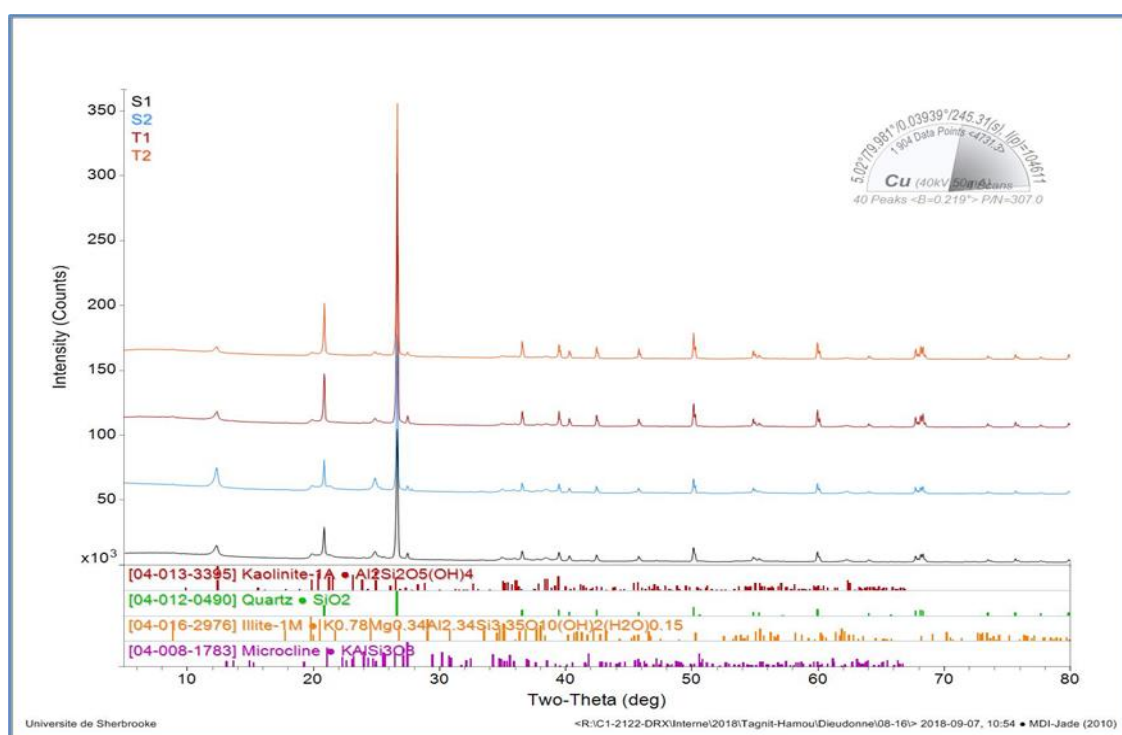

Figure 5. XRD spectra of raw clays of S1, S2, T1 and T2

Table 6. Quantitative Mineralogical Compositions of Samples S1, S2, T1 and T2.

Minerals (%)	S1	S2	T1	T2
Quartz	54.6	51.2	69.2	71.4
Kaolinite	30.3	34.5	17.3	15.1
Illite	1.6	0.0	2.8	2.8
Microcline	4.5	2.3	0.3	2.0
Muscovite	7.5	10.0	8.2	6.9
Rutile	1.5	2.0	2.2	1.7

3.6 Infared spectroscopy (FITR)

Infrared spectroscopy was used to complete the analysis of clay samples. **Figures 6, 7, 8** and **9** show the infrared spectra of the four clays studied. Generally, the adsorption bands that appear in the

3620 cm^{-1} to 3700 cm^{-1} regions correspond to the vibrations of the characteristic structural hydroxyl groups of kaolinite [6]. the exact position of these bands and their intensities vary according to the nature of the bonds of molecules. They occur in clays S1, S2, T1 and T2 between 3621 and 3700cm^{-1}.
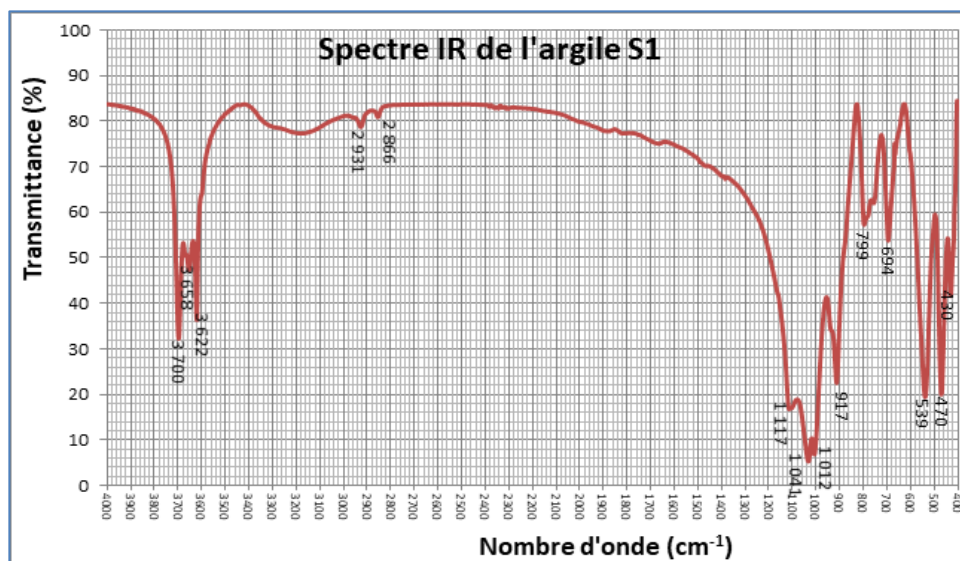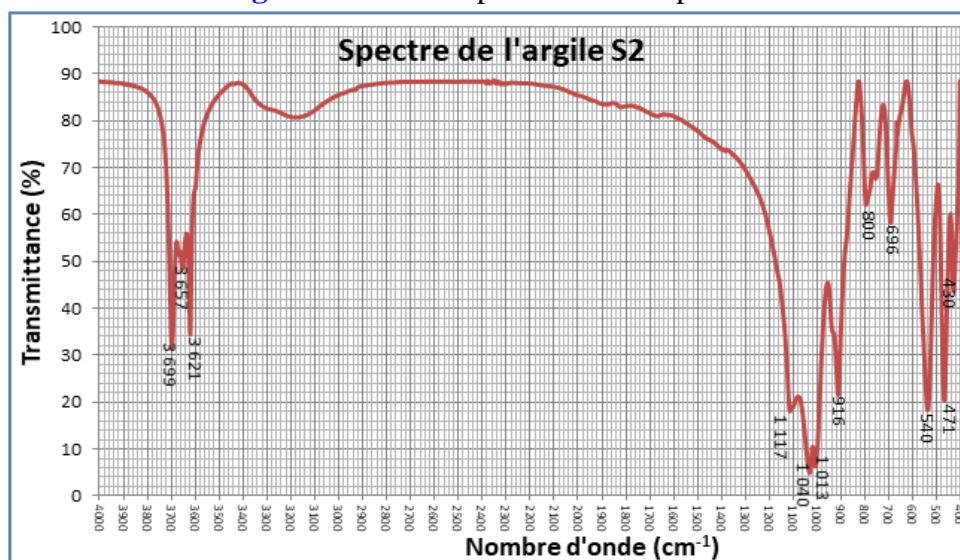

Figure 6. Infrared spectrum of sample S1

Figure7. Infrared spectrum of sample S2

The band observed around 3620 cm^{-1} is attributed to the inner hydroxides [28] and the bands observed around the other three characteristic bands are generally attributed to the vibrations of the outer hydroxides. The position of the absorption peak close to 3620 cm^{-1} of the OH groups could indicate the presence of smectites [29].

The infrared spectra of these solids also show signals corresponding to the Si-O-Si group elongation vibration of kaolinite or quartz at about 1040 cm^{-1} for clays S and about 1035 cm^{-1} for clays T. The characteristic bands of the deformation vibrations of the Si-O bond of the quartz occur for clays S1, S2, T1 and T2 around 430 cm^{-1}, 470 cm^{-1}, 540 cm^{-1}, 696 cm^{-1} and 800 cm^{-1} [5, 29-30]. These bands can also be attributed to the vibrations of the Si-O-M angular deformations of the smectites where M may be : Al, Mg, Fe, Ti. A wide band from 3400 to 3070 Cm^{-1} represents a vibration band of OH bound to octahedral aluminum as obtained by other authors [20]. These results confirm the important proportions of quartz and kaolinite found in X-ray diffraction.
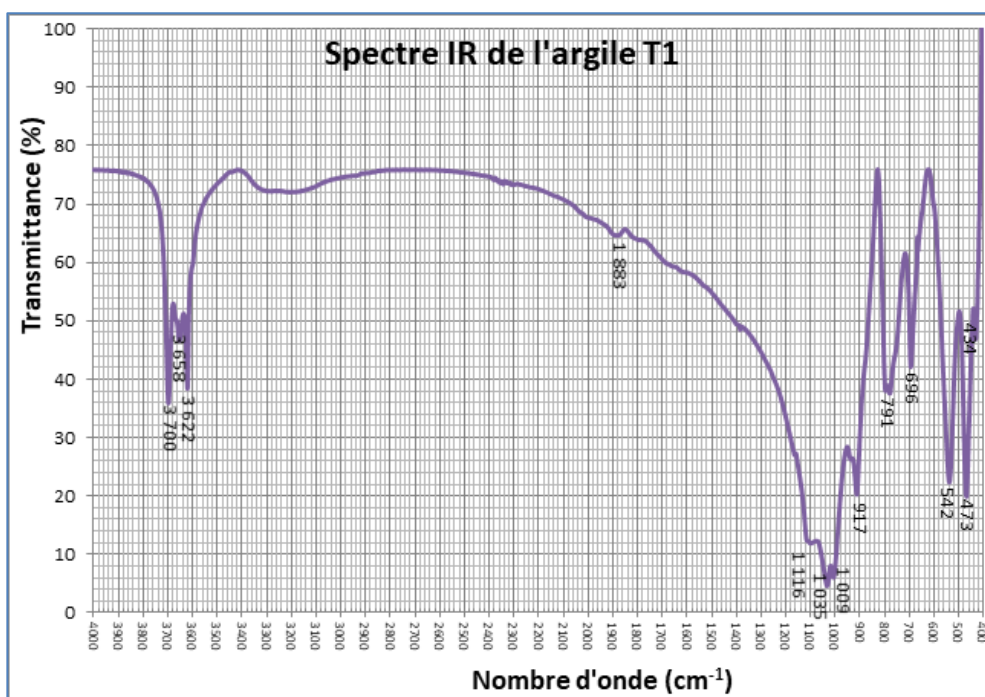
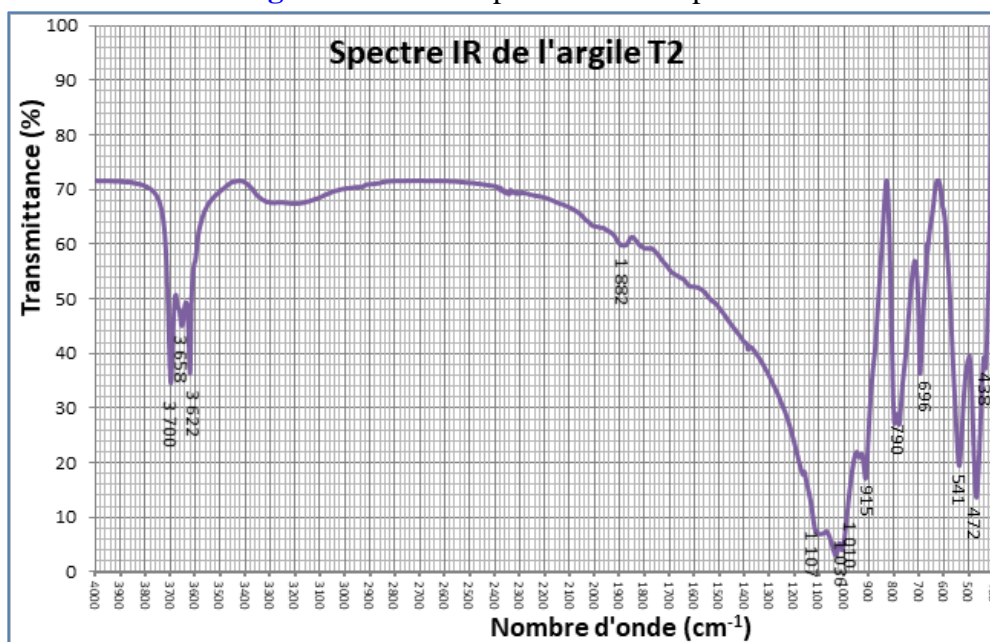**Figure 8.** Infrared spectrum of sample T1

Figure 9: Infrared spectrum of sample T2

The use of several analytical methods allowed investigations to establish a complete characterization. The application of a single method defines only one aspect and it must be confirmed using the other methods. In most cases, the methods of analysis complement each other. The results obtained by DRX more or less confirm those of other methods.

Conclusion

The physicochemical and mineralogical characterization of clay materials S1, S2, T1 and T2 was carried out using thermal analyses, infrared spectroscopy as well as X-rays. The results showed the mineral phases and the chemical composition of each clay analyzed. Qualitative and quantitative

analysis by X-ray diffraction showed that the samples consist mainly of Quartz and Kaolinite, as well as trace illite and microcline. These results are confirmed by the FTIR infrared analysis which revealed the presence of kaolinite or quartz at approximately 1040 cm^{-1} for clays S and at approximately 1035 cm^{-1} for clays T. The thermal analysis mainly indicates that clays S and T consist of illite, kaolinite or quartz. The pH value around 6 reveals the acidity of the clay samples.

This study demonstrated that the development of clayey membrane is a reality. S clays which have a slightly higher SiO_2/Al_2O_3 ratio than bentonite could be better adapted than T clays. Other experiments are possible, such as electrochemical characterization tests of the slurries and the study of their stability, in order to study its influence on the performance of water filtration membrane. This study can be used to make easy the accessibility of membranes of good performance and elaborated in local raw materials in Burkina Faso.

Acknowledgement

Disclosure statement: *Conflict of Interest:* The authors declare that there are no conflicts of interest. *Compliance with Ethical Standards:* This article does not contain any studies involving human or animal subjects.

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