Available online at www.CivileJournal.org

Civil Engineering Journal

(E-ISSN: 2476-3055; ISSN: 2676-6957)

Vol. 12, No. 04, April, 2026

Mineralogical, Chemical, and Geotechnical Characterization of Natural Clay for Ceramic Applications

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Received 06 November 2025; Revised 24 March 2026; Accepted 29 March 2026; Published 01 April 2026

Abstract

In the framework, the sustainable local development of the Adrar region is one of the largest in the Algerian Sahara. The Algerian government has launched a search for useful local substances to cover the need for building materials in the construction sector. However, the Algerian Sahara has a variety of mineral resources, including clays. This work aims to characterize and identify a natural Algerian clay from the Reggane basin (Paleozoic sedimentary basin) in southwestern Algeria. This is for use in the manufacture of ceramic products. For this, numerous analyses were carried out using techniques such as X-ray Diffraction (XRD) to determine the different crystalline mineral phases, X-ray Fluorescence (XRF) to identify the elemental composition, and Infrared Spectroscopy (FTIR) to study the molecular structure along with the geotechnical identification in order to better understand the main properties of this clay. The findings indicated that Reggane clay is silty and highly plastic (21.94-31.7). It contains a mixture of illite, kaolinite, and quartz, in very significant proportions, as well as hematite, orthoclase, and palygorskite. Furthermore, elemental chemical analyses were conducted, and the results showed that the main constituents of this clay are SiO_2 (58.19%-61.71%), Al_2O_3 (13.32%-13.50%), and Fe_2O_3 (6.13%-6.40%). These findings could eventually be used to target applications of this clay in the production of local fired materials.

Keywords: Silty Clay; Reggane; Identification; Raw Material; Ceramics.

1. Introduction

Clay, like stone or wood, was one of the first materials to be used by humans, and several civilizations employed it in their various constructions. It is readily available throughout the world. Clay, through the centuries, has been exploited through different techniques, depending on its characteristics and on the region where it is used. According to Bergaya and Lagaly [1], clays are sedimentary rocks that are mainly composed of clay minerals that contain hydrated phyllosilicates formed by a stack of sheets, i.e., alternation of octahedral layers of $Al_2(OH)_6$ or $Mg(OH)_6$ and tetrahedral layers of SiO_4, with interlayer spaces [1-3]. Further, in general, the structure of the sheet allows distinguishing different types of clay minerals (Type 1/1, Type 2/1) [4, 5–17].

Several works, carried out throughout the world, have shown that clay minerals are now widely used in various sectors, such as the production of ceramic materials, like earthenware, tiles, and bricks [18-27]. They are also utilized in the environmental sector (wastewater treatment and pollutant adsorbents; they are also employed as adsorbents and

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https://doi.org/10.28991/CEJ-2026-012-04-012

catalysts) [5, 26, 28-32] in various medical applications and in the cosmetics industry [33-36]. These clays are particularly important for the construction of sustainable structures as well, which have proven their usefulness through remarkable heritage sites around the world. They are also used in other domains due to their physical and chemical properties and their mechanical characteristics as well [37-39].

It is also interesting to emphasize that the town of Reggane is located in the Reggane Basin (Southwest Algeria), which is one of the largest sedimentary basins on the Saharan platform. This region has clay reserves that are widely sufficient to fully or partially supply several industrial sectors, such as the manufacture of clay-based terracotta building materials, which constitutes a significant ecological and economic advantage. Unfortunately, this clay is little exploited, except for the few applications in the manufacture of raw earth bricks and the production of traditional pottery.

The present work was carried out with a view to achieving sustainable local development for the purpose of enhancing the economic, social, and environmental well-being of our society. The goal was to present the potential of locally available clays in the region of Reggane, which have not yet been fully identified. To this end, this study seeks, first, to carry out an extensive geotechnical, chemical, and mineralogical identification of this clay in order to classify it and, hence, confirm its potential use in the ceramic products and building materials industry in general. The second objective is to generalize the use of local materials, which are abundantly available and inexpensive.

2. Study Area and Material

2.1. Geographic Context of Reggane Basin

The Reggane Basin is largely covered by the impressive dune belts of Erg Chech. It extends along the southwest edge of the Ougarta Mountains to the Réguibat Shield (Figure 1). Towards the South-Southeast, this basin extends through the Bled El Mass-Azzel Matti shoal; it is separated to the west by the Bou Bernous Sill. The northeastern flank of the basin has a steep inclination in contact with the Ougarta folded system. This basin, with a sedimentary column 6500 meters thick, shares remarkable similarities with that of Tindouf, with a Mesozoic sedimentary layer not exceeding 300 meters thick [6, 8, 40].

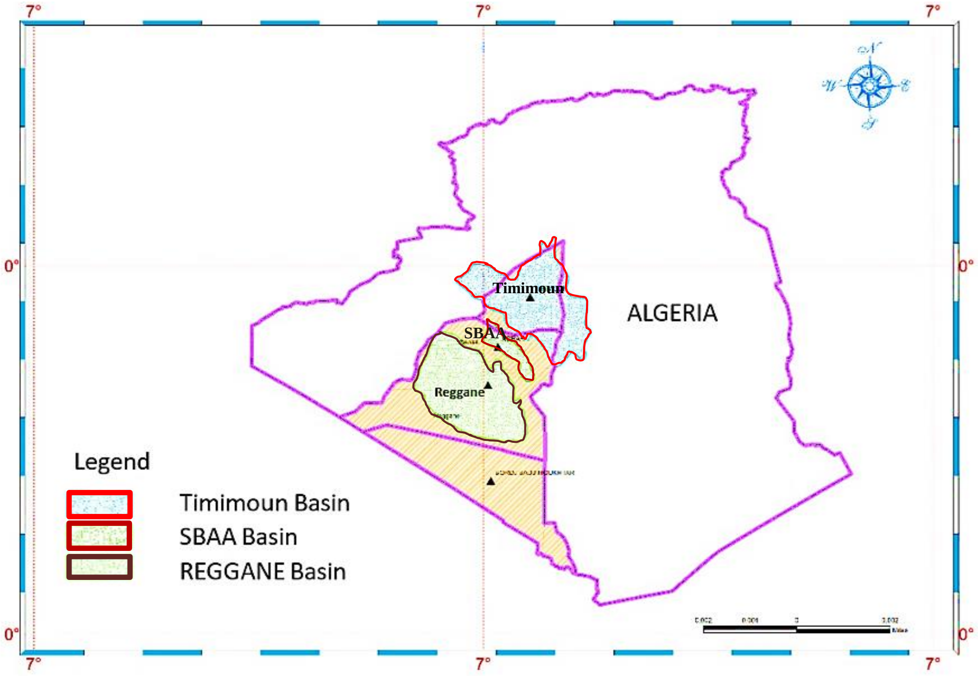

Figure 1. Location of the sedimentary basin of Reggane

2.2. Geological Context of Reggane Basin

The Reggane pericratonic basin is a vast Paleozoic depression filled primarily with detrital sediments, hence providing a complete petroleum system. The sedimentation transition from the Silurian to the Devonian is characterized by the predominance of clayey-sandstone deposits. In the Lower Devonian, deposits on the southern part of the platform are predominantly clayey-sandstone. They contain numerous oblique strata, reflecting a dynamic fluvial environment, as illustrated in Figure 2.

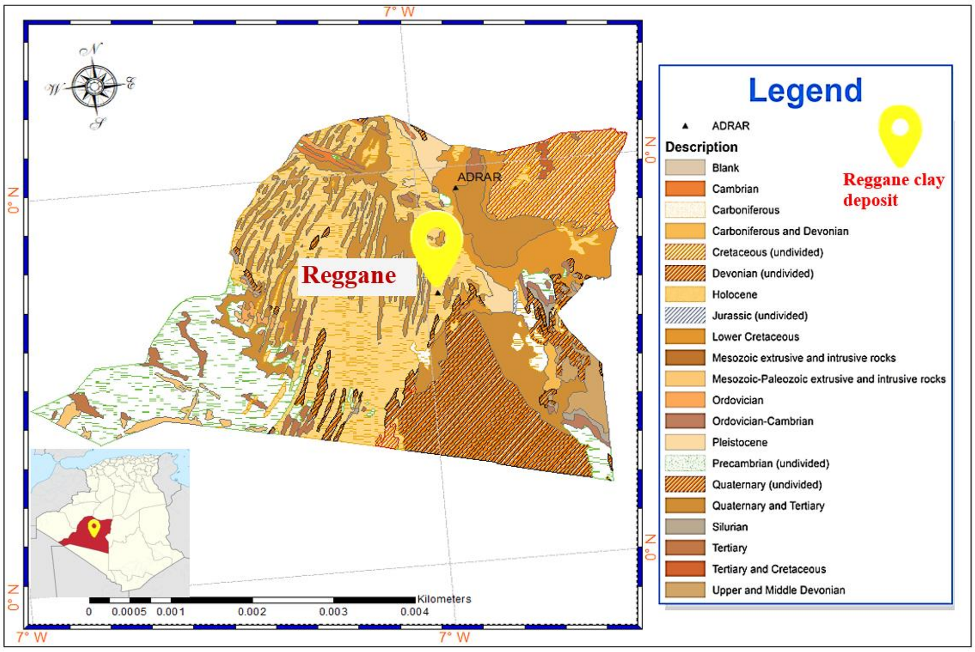

Figure 2. Geological map of the study area, extracted from Touat geological map NG_30_XXIV

2.3. Location and Lithology of the Deposit

The deposit is located 400 meters from the road leading to Kasr Timadanine, on the left side. It is bordered to the north by the road leading to Timadanine, on the left side, at a distance of 400 m; to the south and west by a vacant lot; and to the east by National Road (NR) number 06, at a distance of approximately 450 m, with geographical coordinates of latitude 26°43'47.23''N and longitude 0°8'56.69''E. Description of the lithological section of the deposit, as shown in Figure 3:

- 0.00 - 0.30 m: Fine, gravelly reddish sand;
- 0.30 - 3.00 m: Consolidated reddish clay-sandstone with greenish marly inclusions in places.

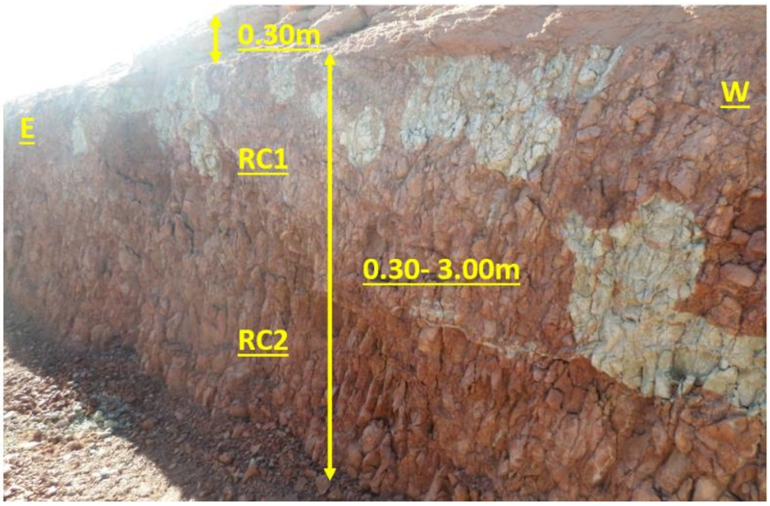

Figure 3. Panoramic view of the Reggane deposit

3. Techniques and Methods Used

Identification tests were carried out on two samples, RC1 and RC2, that were taken from the Reggane clay deposit, as depicted in Figure 3. These tests consisted of performing the following unit operations (Figure 4):

- Crushing the sample lumps into pieces using a mortar with a rubber head to preserve the grains,
- Drying in an oven for 24 hours (T = 60°C),

- Grinding the clay sample lumps. The particle size distribution was determined by two complementary methods, namely Wet Sieving and Sedimentation Analysis, according to the AFNOR [41, 42] standards, respectively. Furthermore, the plastic properties of the fine fraction, containing particles smaller than 400 µm, were measured in accordance with the recommendations of the AFNOR [43] standard. In addition, the specific gravity of solid particles (γ_S) was measured using a pycnometer, according to Standard AFNOR [44]. Likewise, the dry density was also determined using the Normal Proctor test [45].

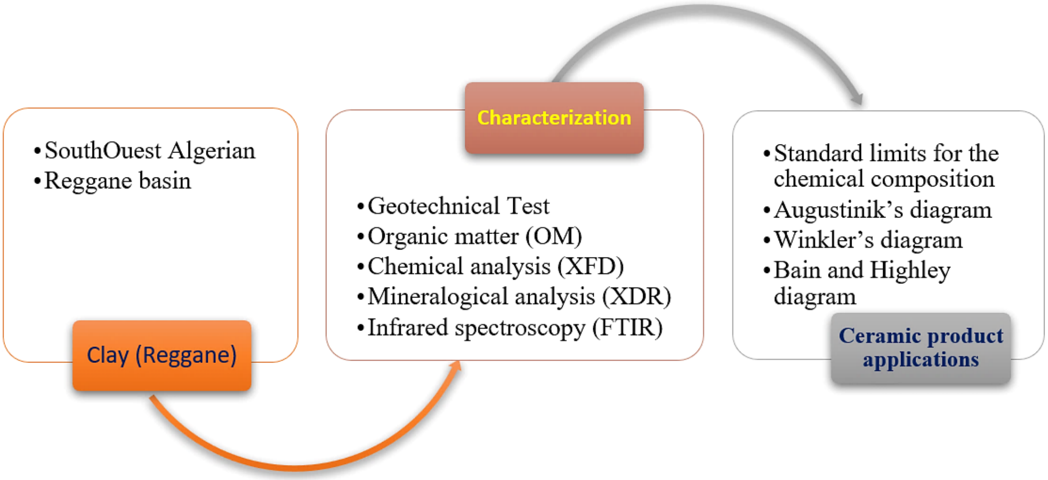

Figure 4. Study methodology

Furthermore, it was necessary to determine the pH for the purpose of quantifying the contribution of acidity when the solid is in contact with the solution. To do this, a 10% w/v clay solution was prepared with distilled water. This mixture was then allowed to stand for 4 hours, at 25°C, to enable the ions to dissolve. The resulting clay solution was then homogenized using a magnetic stirrer. The readings were taken directly from a pH meter [46].

Afterwards, the organic matter (OM) content was determined using the calcination method (loss on ignition) [47]. For this, the sample under study was dried in an oven for 24 hours at 105°C to remove all moisture from the sample. The mass of the dry sample (A) was thus determined. Then, the sample obtained was placed in a ceramic crucible and calcined at 550 °C for 4h in a furnace [48-52]. The mass of the calcined sample (B) was then determined. Consequently, the loss on ignition (LOI) was determined using the following expression:

$$MO = (\text{Masse A} - \text{Masse B})\ /100 \tag{1}$$

Furthermore, the crystalline phases were identified using a Proto AXRD Benchtop Powder X-ray Diffractometer (instrument at the laboratory of the University of Adrar) with CuKα radiation (λ = 1.5406 Å) at 30 kV and 20 mA. The scan range was set from 5° to 70° 2θ with a step size of 0.02° and a scan rate of 1.2°/min. Then, the diffractograms were obtained from disoriented powdered samples that were placed directly in a conventional sample holder.

Similarly, Fourier Transform Infrared Spectroscopy (FTIR) was conducted with the Agilent Cary 660 FTIR spectrometer (at the laboratory of the University of Adrar) using the KBr disc method. Scans were recorded in the 400–4000 cm^{-1} range at a resolution of 4 cm^{-1}.

Likewise, the elemental chemical analysis of the clay sample was performed using X-ray fluorescence spectroscopy (Rigaku ZSX Primus IV X-ray fluorescence (WDXRF) spectrometer, 4kW). This type of chemical analysis was carried out at the Laboratory of the Center for the Study and Technological Services of the Building Materials Industry (CETIM) in Boumerdès (Algeria).

4. Results and Discussion

4.1. Geotechnical Parameters

The findings indicate that both samples, RC1 and RC2, under investigation contain an average clay particle proportion, varying between 42% and 45%, in accordance with the Winkler diagram. According to Figure 5, this soil is classified as clayey-loamy. Also, the values obtained for the plasticity index, i.e., between 21.94 and 31.7, confirm that this is a high-plasticity soil. In this regard, Holtz & Kovacs [45] found out that the analyzed soil contained kaolinite and illite, as shown in Figure 6. In addition, the result of the methylene blue test gave a value ranging from 7.25 to 7.5, which reveals that the samples tested contained a high proportion of clay particles, as clearly indicated in Table 1.

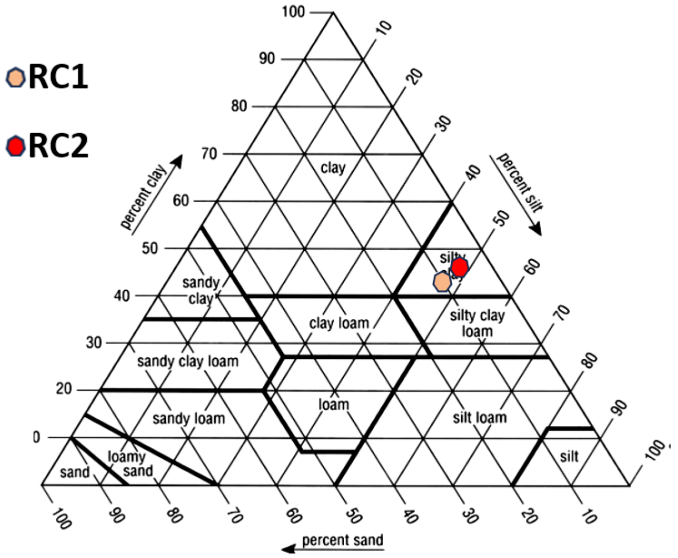

Figure 5. Granulometric classification of the studied samples according to Winkler's diagram

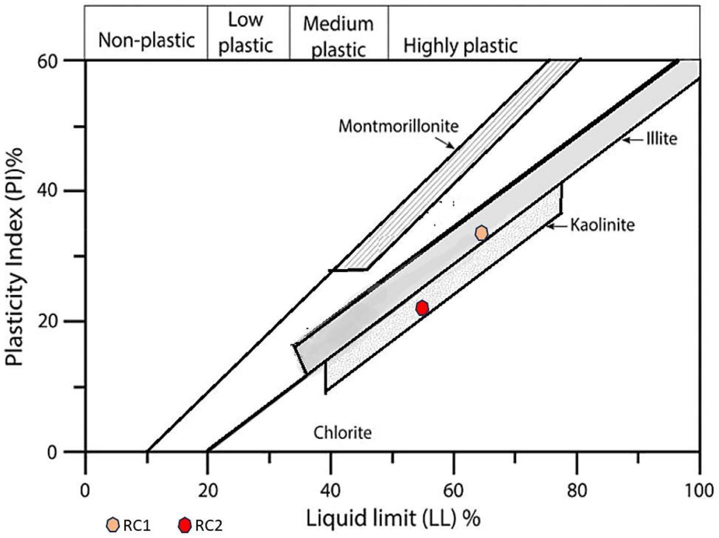

Figure 6. Position of samples RC1 and RC2 in the Holtz & Kovacs diagram [45]

Table 1. Geotechnical properties of clay in this study

Sample	RC1	RC2
γ_S (g/cm^3)	2.66	2.60
Sand (%) (> 0.02mm)	10	6
Silt (%) (0.02 - 0.002mm)	48	49
Clay (%) (< 0.002mm)	42	45
LL (%)	64.7	55.1
PL (%)	33	33.16
PI	31.7	21.94
A_c	0.75	0.48
$\gamma_{d\,max}$ (g/cm^3)	1.55	-
ω_{opt} (%)	23	-
VBS	7.25	7.5
SST (m^2/g)	151.74	156.97
pH	8.42	-
OM	0.17	0.172
$CaCO_3$	4.5	-

According to the USCS (Unified Soil Classification System), this is a fine-grained soil with high plasticity and cohesion, classified as CH. The analyzed clay is not very active. Furthermore, the low values of the specific surface area, i.e., 151.74 and 156.97 m^2/g, suggest that this soil contains clay minerals, such as illite, kaolinite, or chlorite, but does not contain expandable clay minerals, such as vermiculite or montmorillonite, which encourages its use in the building materials industry.

Furthermore, the analyzed samples had an organic matter (OM) content of approximately 0.17%, indicating that the soil is very poor in organic matter. It can therefore be classified as an inorganic soil, as higher organic content may lead to excessive porosity and cracking during firing [53]. Moreover, the two samples, RC1 and RC2, exhibited almost identical geotechnical parameters, confirming that the geological formation of the studied deposit is relatively homogeneous. In addition, the pH value indicates that the soil is slightly basic, which is favorable for its use as a building material (ceramic products). Finally, the $CaCO_3$ content suggests that the soil has a predominantly clayey nature.

4.2. X-ray Diffraction

Figure 7 depicts the X-ray diagrams of the samples under study. Spectral analysis indicates that these samples are composed of quartz (SiO_2), illite [$(KH_3O)Al_2Si_3AlO_{10}(OH)_2$] [18, 54], kaolinite ($Al_2Si_2O_5(OH)_4$) [18, 54], palygorskite (attapulgites), and hematite (Fe_2O_3), in addition to orthoclase ($KAlSi_3O_8$) and gypsum.

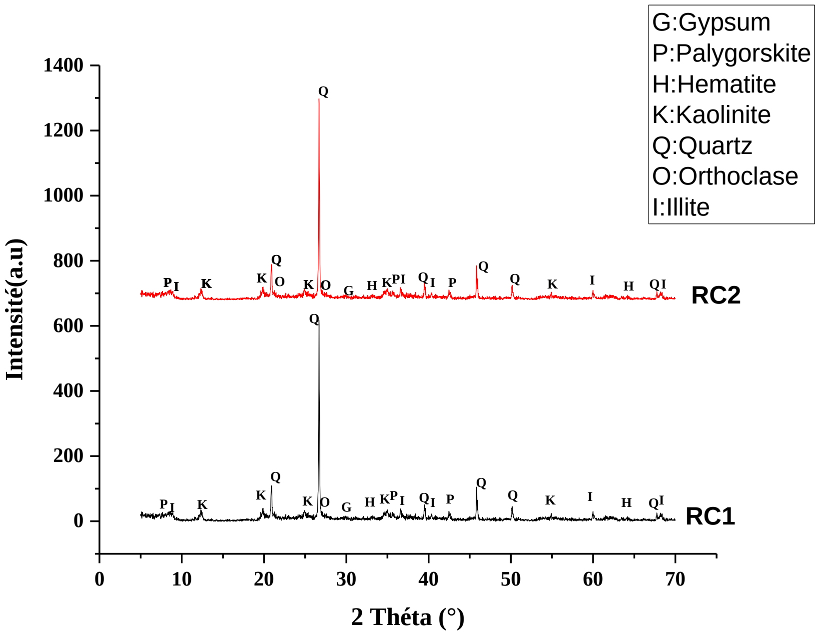

Figure 7. X-ray diffractograms of samples RC1 and RC2

Moreover, these diagrams reveal the presence of a prominent peak at approximately 26.6° (2θ, CuKα), corresponding to quartz. Illite is identified by a reflection around 8.8° (2θ) (10 Å), indicating a significant presence of K-clay minerals and suggesting moderate plasticity. Kaolinite is also detected, with a characteristic peak near 12.3° (7 Å), indicating a mixed clay assemblage dominated by both 1:1 and 2:1 layer silicates. In addition, weaker intensity peaks corresponding to orthoclase confirm the presence of feldspar. Minor peaks associated with palygorskite and gypsum indicate limited amounts of sulfate and Mg-bearing fibrous clay minerals [50, 55]. Hematite is also identified by weak peaks, suggesting the presence of iron [27, 56].

4.3. Infrared Spectroscopy

Infrared (IR) spectroscopy was used to complete the analysis of the clay from Reggane. The spectrum obtained for sample RC1 is illustrated in Figure 8, which explicitly shows two adsorption bands located between 1600-1700 cm^{-1} and 3200-3800 cm^{-1}. Hence:

- The band within the 1600-1700 cm^{-1} region can be attributed to the valence vibrations of the OH group of the constituent water and to band corresponding to the bond vibrations of the adsorbed water, located at 1644 cm^{-1} [56, 57].
- The band within the 3200-3800 cm^{-1} range, located exactly at 3616.0 cm^{-1}, corresponds to the stretching vibrations of the internal hydroxyl (OH) groups in the octahedral layer as well as to the band of the OH-Fe^{3+} valence vibrations, located at 3410 cm^{-1} [55-58].

- The Si-O bond is characterized by an intense band between 900-1200 cm^{-1}, centered around 1057 cm^{-1}. It corresponds to the valence vibrations of the Si-O bond [55, 59].
- The bands detected between 795 and 748 cm^{-1}, corresponding to the Si-O-Al bond, also give way to a band around 787 cm^{-1} [59]. However, the absorption bands seen at 797 and 779 cm^{-1} may correspond to quartz [60, 61]. The Si-O-Al bond forms a band around 541 cm^{-1} [62, 63]. The band detected at 450 cm^{-1} is caused by the Si-O-Si bond [61]. These findings are consistent with those obtained with the XRD (X-ray Diffraction) technique. They also confirm the presence of quartz, kaolinite, and illite in the clay from the region of Reggane (Table 2).

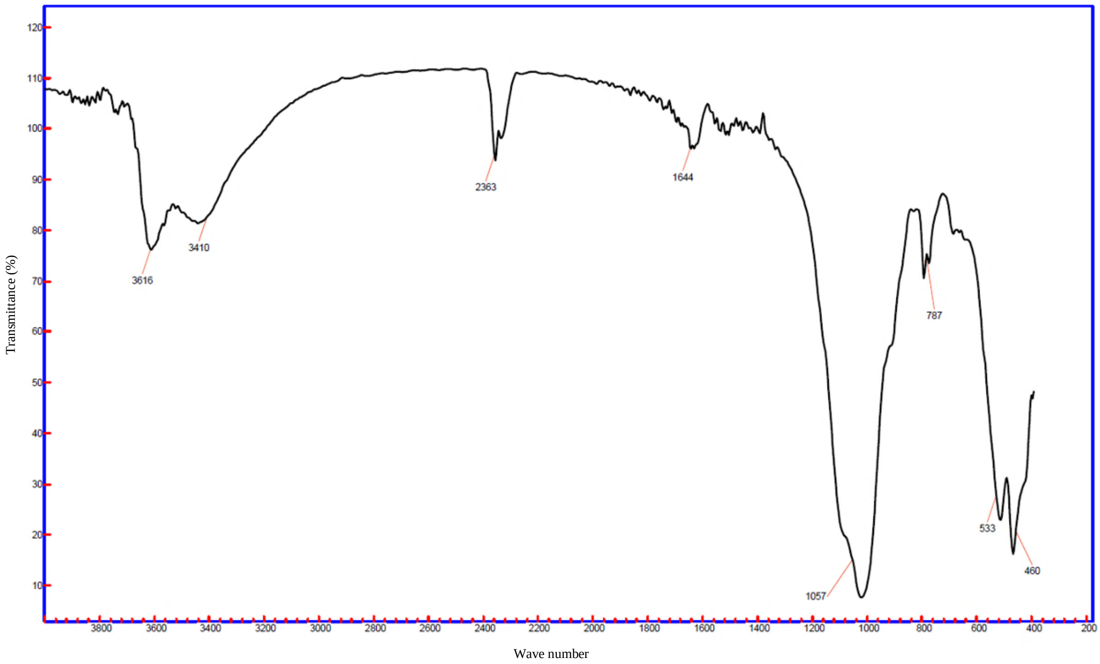

Figure 8. Infrared spectrum of sample RC1

Table 2. Principal FTIR absorption bands and their mineralogical assignments

Wave number (cm^{-1})	Vibrational mode	Relative intensity	Assigned phase
3616	O-H stretching structuralhydroxyl	Medium	Kaolinite/Illite
3410	O-H hydrogen-bonded OH-Fe^{3+}	Medium	Adsorbed water/minor Fe^{3+} substitution in octahedral layer
1644	H-O-H bending vibration	Medium	Interlayer/adsorbed water
1057	Si-O stretching vibration	Very Strong	Aluminosilicate framework
797	Si-O symmetric stretching	Medium	Quartz
787	Si-O-Al bending Si-O symmetric	Medium	Clay mineral lattice / quartz contribution
541	Si-O-Al bending vibration	Weak-Medium	Aluminosilicate framework
450	Si-O bending vibration	Medium	Silicate tetrahedral network

4.4. X-ray Fluorescence

Table 3 summarizes the chemical composition of the analyzed clay. The main constituents are three oxides, namely SiO_2, Al_2O_3, and Fe_2O_3. It should be noted that silica accounts for more than 50% of the total mass. The clay samples exhibit fairly high SiO_2 and Al_2O_3 contents, indicating that they are mainly composed of aluminosilicates [49], which makes this clay exhibit some characteristic phyllosilicate properties [57]. It also includes a high K_2O content, indicating that this clay is rich in illite [9], hence the presence of the illite peak.

Table 3. Elemental chemical composition of the clay samples

Oxide (%)	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	P_2O_5	TiO_2	Cl^-	LOI	SiO_2/Al_2O_3
RC1	58.19	13.32	6.13	3.12	3.67	0.35	3.96	0.20	0.11	0.82	0.024	10.14	**4.36**
RC2	61.71	13.50	6.40	1.14	2.35	0.57	4.06	0.27	0.08	0.83	0.036	7.08	**4.57**

The other elements that were detected exist in minor proportions. This clay has also a high content of colored oxides (a composite of iron (III) oxide (Fe_2O_3) and titanium dioxide (TiO_2) with more than 5% iron oxide), which explains its bright color [64]. The iron oxide content (6.13–6.40%) explains the existence of hematite [53], which is characteristic of clays commonly used for red ceramic products.

The alkali oxides (K_2O + Na_2= 4.1–4.3 %) indicate the presence of illitic phases or feldspathic components. It has also been revealed that, in general, alkalis in clays can reduce their refractory properties [65]. Furthermore, the LOI value (10.14% for RC1, 7.08% for RC2) of the studied clay indicating variations in structural water, organic matter (OM=0.17), and/or carbonate contents. The CaO value (RC1=1.14, RC2=3.12), then reported ranges of 0.5–18% [14, 15], Indicating minimal carbonates and a non-calcareous composition favorable for ceramics. Low CaO reduces the risk of lime bursting and efflorescence and reflects high chemical stability [62].

Moreover, the SiO_2/Al_2O_3 mass ratio mass ratios are 4.36, 4.57 in RC1, and RC2, respectively, indicating the presence of free silica (quartz) in the clay fraction [54] and (2/1) clay minerals, such as illite, in the materials, in addition to the chemical maturity of the studied clay material [65, 66].

The XRF, XRD, and FTIR results obtained for the clay studied are consistent, confirming its potential use in ceramic applications.

5. Ceramic Product Applications

The nature of clays used in the Algerian ceramic industry is extremely varied and diversified due to the many different geological formations found across the country. The chemical compositions of clay mixtures must be determined before using these clays. These compositions have a direct impact on the characteristics of the clay mass and on the products made of this clay [15]. Table 4 lists the common limits for the chemical composition of mixtures used in the manufacture of terracotta. The data in Table 4 may serve as a basis for the preparation of a clay mixture.

Table 4. Standard limits for the chemical composition of clay mixtures for the manufacture of terracotta products [14, 15]

	Standard limits (%)		Clay under study	
	Minimum	**Maximum**	**RC1**	**RC2**
LOI	3	18	10.14	7.08
SiO_2	35	80	58.19	63.71
Al_2O_3	8	30	13.32	13.50
TiO_2	0.3	2	0.82	0.83
Fe_2O_3	2	10	6.13	6.40
CaO	0.5	18	3.12	1.14
MgO	0	5	3.67	2.35
Na_2O	0.1	1.5	0.20	0.27
K_2O	0.1	4.5	3.96	4.06

LOI: Loss on Ignition

The results of chemical analyses conducted on clays from the region of Reggane suggest that this clay can be used as a raw material for the manufacture of fired products like bricks. Figure 9 depicts the Augustinik diagram, which shows the evolution of the ratio (Al_2O_3/SiO_2) as a function of the sum of the contents (R_2O + RO + Fe_2O_3). Here, R_2O corresponds to the sum (K_2O + Na_2O) while RO corresponds to (CaO + MgO + MnO) [15, 67]. Figure 9 clearly shows the clay from Reggane can serve for the production of bricks and tiles.

Ceramics can be manufactured from one or more types of clay mixed with non-plastic mineral modifiers, such as quartz powder and feldspar, which are known for their degreasing effect [68]. It must be emphasized that the particle size distribution of clay mixtures has a major influence on their properties and, therefore, it is necessary to use specific clay and silt contents in the mixtures that are intended for the production of ceramic products.

In addition, the Winkler diagram (Figure 10) provides some industrial data that help classify clays based on their particle size distribution in order to determine their suitability for ceramic applications. These data may also aid in the extrusion shaping of the products to be manufactured. Figure 10 shows that the clays studied, given their particle size distribution, appear quite suitable for use as single components for the manufacture of thin-walled hollow bricks. These same observations are also true for many other traditional quarried materials that can be employed in the preparation of mixtures intended for the manufacturing of various clay products with optimal characteristics.

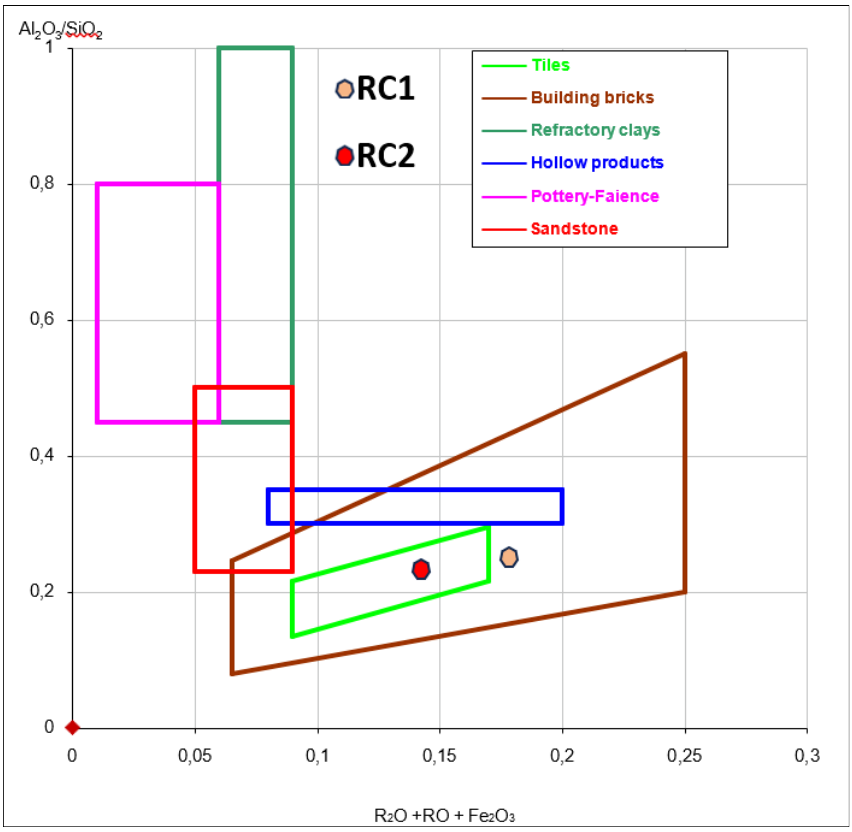

Figure 9. Augustinik's diagram for the clay samples under study [15]

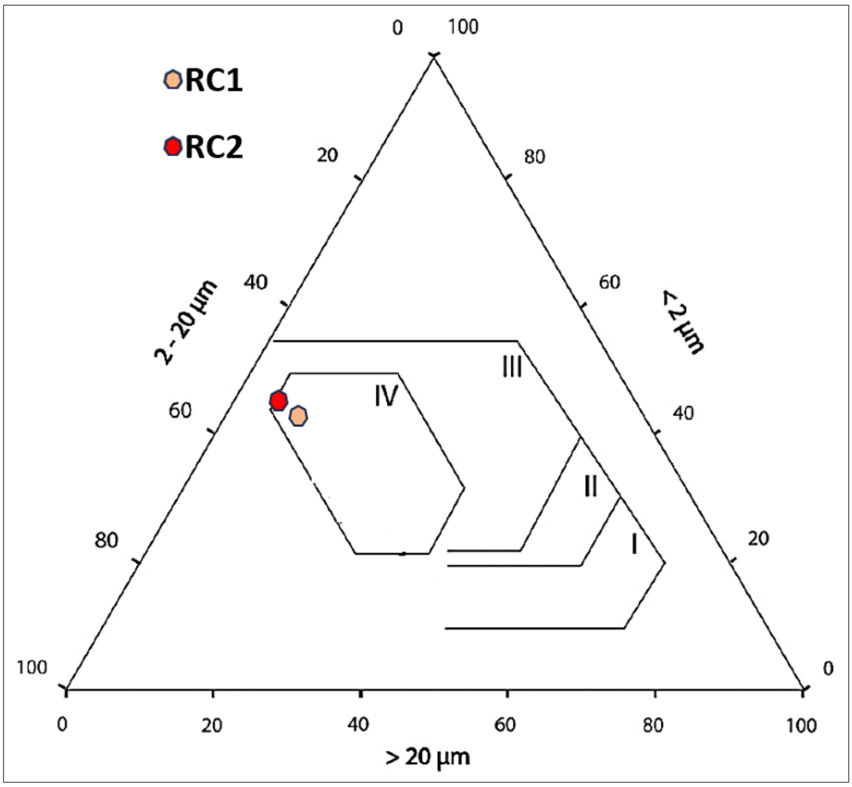

Figure 10. Winkler's diagram for the clay samples studied. I: Solid bricks, II: Perforated bricks, III: Tiles and lightweight blocks, IV: Thin-walled hollow bricks

With regard to the production of terracotta materials, the adopted extrusion shaping process requires the use of plastic pastes. Indeed, the clay under study can only be used with appropriate water contents [69]. The shaping of clay material products, such as ceramics like pottery and porcelain, construction materials such as bricks and tiles, is generally carried out by extrusion. In this regard, Holtz & Kovacs [45] defined the ranges of plasticity limits and plasticity indices in order to determine the characteristics of the mixtures and achieve optimal shaping of the products [19, 54, 64, 66, 68, 70-78]. Samples RC1 and RC2 can be located in the plasticity chart. Both samples (RC1, RC2) are located in the acceptable extrusion zone, which suggests that these samples are suitable for extrusion (Figure 11).

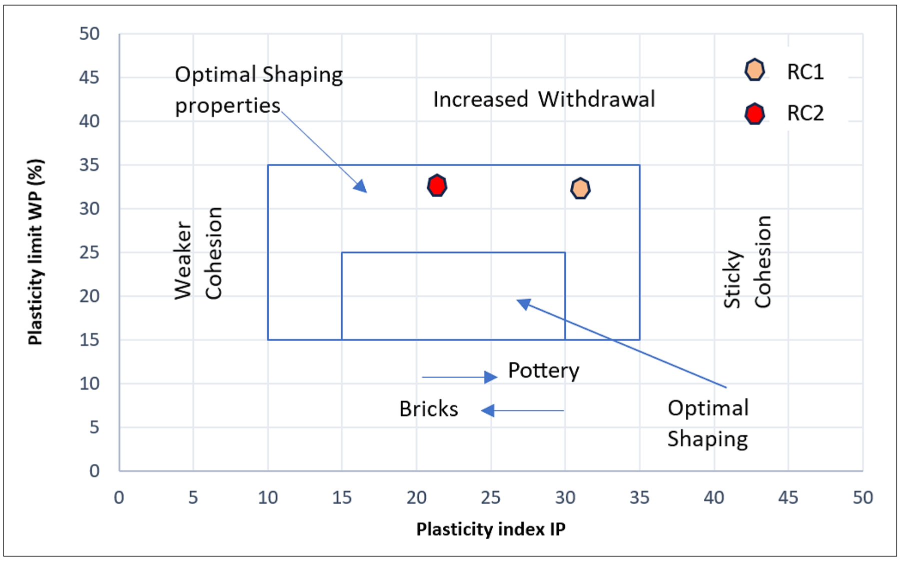

Figure 11. Bain and Highley diagram showing the molding capacity of clay samples from Reggane

6. Conclusion

In the geological context of the Reggane region, the surface formations are generally derived from ancient lacustrine or alluvial deposits, known for their lateral uniformity over large desert areas. This homogeneity can be explained by a stable sedimentary environment, clay deposits formed under uniform conditions, low recent tectonization, and the absence of significant lithological variability in the exploited horizons.

As the clay deposit studied is homogeneous in terms of facies, mineralogy, and geotechnical properties, it is scientifically acceptable to consider that two well-taken samples RC1 and RC2 can be representative of the deposit. This study helped identify the geotechnical characteristics, as well as the chemical and mineralogical compositions of the samples (RC1, RC2) from the Reggane clay deposit. It was found that the clayey-silt samples (RC1, RC2) under study have an average clay content of approximately 44%. A high plasticity index makes it necessary to implement a set of technical measures to control shrinkage, limit cracking, and improve the dimensional stability of ceramic products. These measures include adjustments to the clay formulation, such as the addition of corrective materials like sand (as a degreaser).

As well as with high concentrations of SiO_2 (58.19% - 63.71%), Al_2O_3 (13.32% - 13.50%), and Fe_2O_3 (6.13% - 6.40%). It is also to be noted that the significant quantities of these oxides reflect the presence of aluminosilicates and hematite in this clay. However, the contents of alkali (K_2O and Na_2O) and alkaline earth (MgO) oxides are quite low, implying that this clay is primarily composed of illite, kaolinite, and quartz.

In conclusion, based on the geotechnical, mineralogical, and chemical analysis results, it can be asserted that the clay under study is well-suited to the manufacture of clay products, such as bricks. This type of clay may equally be utilized in numerous traditional applications like pottery and earth bricks.

7. Declarations

7.1. Author Contributions

Conceptualization, A.M. and S.A.; methodology, A.M.; formal analysis, A.M.; investigation, A.M. and B.Z.; data curation, A.M. and S.A.; writing—original draft preparation, A.M.; writing—review and editing, A.M. and B.Z.; supervision, S.A. All authors have read and agreed to the published version of the manuscript.

7.2. Data Availability Statement

The data presented in this study are available on request from the corresponding author.

7.3. Funding

The authors received no financial support for the research, authorship, and/or publication of this article.

7.4. Acknowledgments

The authors would also like to thank the Director of the Center for the Study and Technological Services of the Building Materials Industry (CETIM) in Boumerdès, Algeria, for their assistance in carrying out this work. In addition, thanks are extended to the Manager of the Chemistry Laboratory at the University of Adrar, Algeria, for their support in completing this work.

7.5. Conflicts of Interest

The authors declare no conflict of interest.

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