Research Article

A preliminary mineralogical and physicochemical characterization of the Neogene clays from the Timgad Basin (Massif of Aurès, NE Algeria): potential use in the manufacturing of bricks and ceramic industry

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Received: 17 December 2021 / Accepted: 25 March 2022
Published online: 13 April 2022

Abstract

Abstract This study aims at the mineralogical and physicochemical characterization of the clay formations of the Miocene age from the Timgad Basin (Massif of Aurès, NE Algeria), in order to evaluate their possible valorization, notably for their potential use in the manufacturing of bricks and ceramic industry. For this purpose, four samples were taken from a clay-dominated formation that outcrops 5 km east of the Timgad city. Each sample was collected, prepared, and analyzed by the appropriate analytical methods of characterization such as X-ray diffraction, X-ray fluorescence, and scanning electron microscopy. Moreover, other complementary analyses are performed in this study such as laser granulometry and geotechnical tests. The results of the different tests revealed that the clay formations of the Timgad Basin are non-refractory clays and characterized by medium plasticity. These clays are constituted of more than 50% of fine fraction, mainly represented by kaolinite in association with non-negligible proportions of illite, chlorite, mixed-layer clay minerals, and traces of smectite. Besides, this clayey assemblage is accompanied by some proportion of quartz and calcite, as well as traces of hematite, feldspar, and gypsum. In the light of these results and in combination with the particle size distribution, as well as the results of geotechnical tests, it is concluded that the Neogene clays of the Timgad Basin present high limits of Atterberg. Consequently, their use in the field of manufacturing of bricks and terra cotta 'Terre Cuite' products is subordinated to a preliminary treatment with addition of a degreasing agent, in the form of coarse sand, in order to improve their plasticity.

Article Highlights

- Mineralogical and physicochemical properties of the Neogene clays from the Timgad Basin have been investigated.
- The studied clays have ideal mineralogical composition and physicochemical properties thus making them applicable in the manufacturing of bricks and ceramic industry.
- Addition of coarse sand to the Neogene clays will greatly improve the plasticity of these raw materials.

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Keywords Clay · Mineralogical · Physicochemical · Characterization · Terra cotta · Timgad

1 Introduction

According to their properties and various applications through centuries, clays and clay minerals are at the center of interest of many people in several fields, principally because of their numerous fundamental or applied uses [1].

Clays are classified as industrial minerals whose use is subject to several factors and technological requirements mainly related to mineralogical composition, physicochemical and behavioral properties. Among these factors and requirements, nature and grain size, overall geochemical and mineralogical composition [2], specific surface area, cationic and anionic exchange capacity can be cited [1]. Other specific properties related to each particular area of application are also crucial for investigation such as viscosity, plasticity, abrasiveness, heat hardening, ability to form colloids, sorption [3], reactivity in the presence of fluids and impurities of harmful elements [4]. It is the solicitation of one or more of these properties of clay minerals that justifies their use in various industrial fields such as the environmental field, the pharmaceutical industry, the building materials, the food industry and the oil industry [5, 6].

Prior to the use of any type of natural raw materials, this requires detailed characterization and investigation [7]. Furthermore, knowing that the Neogene basins of the Aurès Massif are well-known for their abundance of clay formations, which are left generally unstudied, both of these situations motivated us to start this research that perfectly falls within the framework of the valorization of the local mineral resources. Indeed, the present work focuses on the clay formations of the Aurès in SE Algeria firstly with the aims at characterizing them by delimiting the potential sites, and as well as highlighting its potential application in the manufacturing of bricks and ceramic industry. To investigate the mineralogical and physicochemical properties of these clay formations, the realized studies were carried out on four different samples T1 to T4. These most representative samples were taken from a clay-dominated formation that outcrops 5 km east of the Timgad city.

2 Material and method

2.1 Material

The study area is part of the Neogene Timgad Basin located at the northern limit of the Aurès Massif (Fig. 1). Outcrops along this area, near east of the Timgad city, offer exceptional exposure of Upper Tortonian to Messenian (Upper Miocene) sections [8, 9], and more specifically, the formation of brown clays with gypsums content. This sampled clay-dominated formation, which is in concordance with the basal Tortonian sandstones, constitutes the main filling of the central part of the Timgad Basin (Fig. 2). In contrast, the overlying Plio-Quaternary formations of the basin are dominant with whitish colour, essentially due to white clays and in association with gravels, conglomerates and a coarse material (Fig. 3).

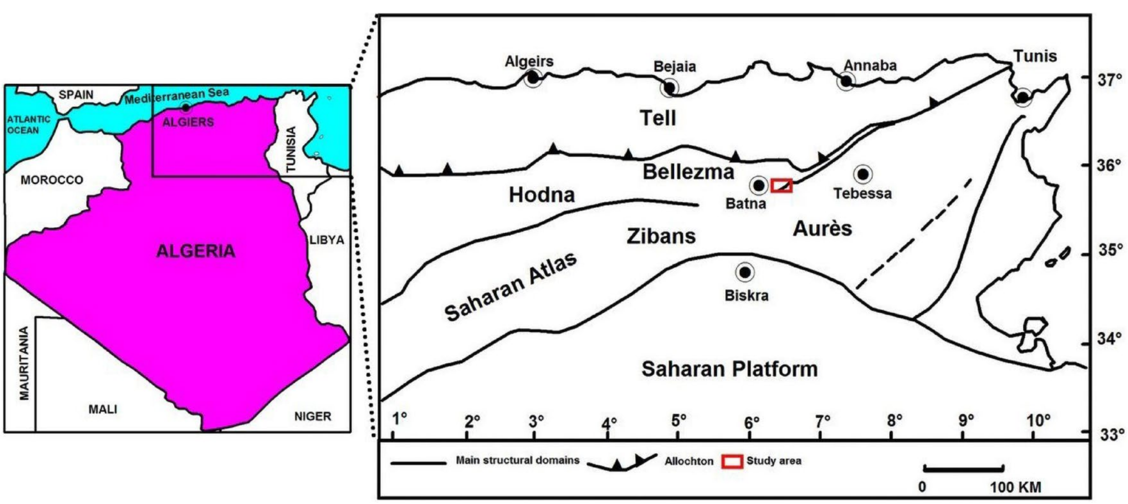

Fig. 1 Situation of the study area [10] (Missoum 2007)

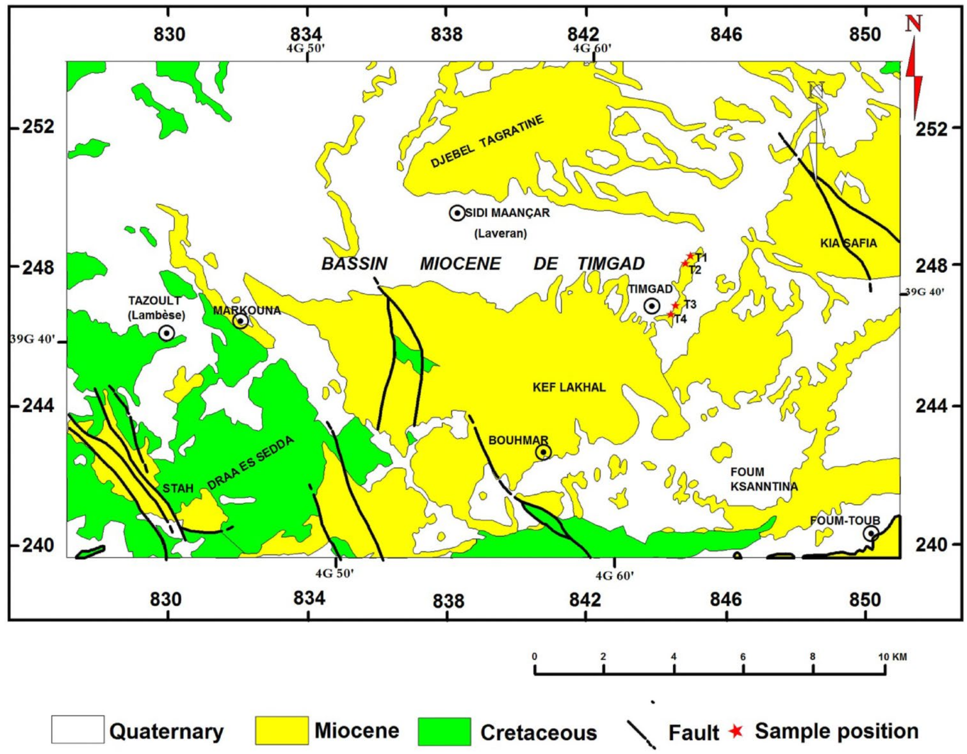

Fig. 2 Geological map of the Neogene Timgad Basin [11] (Sonatrach 1977)

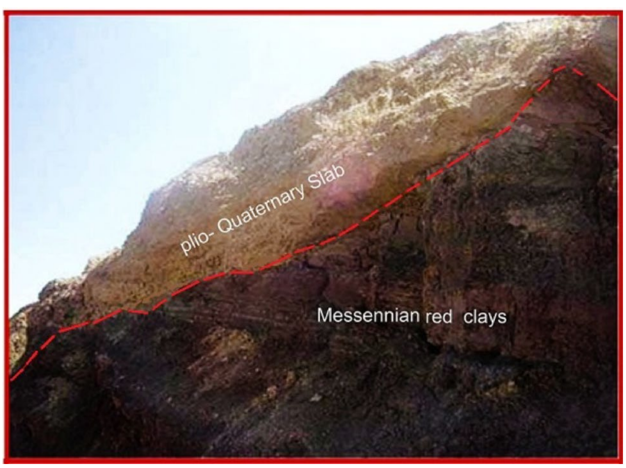

Fig. 3 Messenian red clays enclosed by a Plio-Quaternary slab

2.2 Analytical methods

In spite of the diversification of the methods of analysis of the clayey materials, which are closely related to their fields of use, the characterization of these materials starts essentially by the study of their mineralogical and geochemical composition [7]. Indeed, both form the basis of the clay identification but also allow indications of its physicochemical properties.

For this purpose, the four collected clay samples T1 to T4 from the basin center have undergone the appropriate treatments for their characterization firstly by classical analytical methods, i.e., X-ray diffraction (XRD), X-ray fluorescence (XRF) and advanced scanning electron microscopy (SEM). Moreover, other complementary analyses such as laser granulometry and geotechnical tests are also performed in this present study.

The mineralogical analysis by X-ray diffraction (XRD) was carried out at the Laboratory of Clays, Geochemistry and Sedimentary Environments "AGEs" of the University of Liege-Belgium using a Bruker D8-Advance diffractometer with CuKα radiations. The analysis focused on the identification of the bulk and clay minerals following the protocol of analysis of the clays as described respectively by Nathalie FAGEL [12] and Moore and Reynolds [13].

The identification of the mineral phases was carried out on the basis of the position of the main reflections characteristic of each mineral in the case of random powders. And on the combination of the position and intensity of the three diffraction patterns acquired (air-dried (N), ethylene–glycol solvated (EG), and heated (500)) for clay minerals [14]. As for the semi-quantitative estimates (± 5–10%) [15] of the main mineral species were based on the height of their specific reflections [16] multiplied by a corrective factor [17, 18].

The quantitative analysis in major elements on pellets was carried out by X-ray fluorescence spectrometry, on an XRF instrument (ARL Perform-X 4200, equipped with a Rhodium anode) at the Petrology, Geochemistry and Petrophysics Laboratory, ULiège. The morpho-structural analysis and the acquisition of the SEM photomicrographs (backscatter images) were carried out, after the Silver coating of the powder samples, with a TESCAN VEGA3 apparatus of the Laboratory of Physics of the thin layers and applications "LPCMA" at the University Mohamed Kheider Biskra—Algeria. Concerning the particle size distribution, i.e., granulometric analysis, it was analyzed using a Mastersizer 2000 laser granulometer (Malvern Instruments) of the Laboratory of Structural Inorganic Chemistry "LCIS" at ULiège. Finally, the Atterberg limits were determined at the Geotechnology Laboratory, ULiège, where the liquid limit (LL) and the plastic limit (PL) were estimated by the Casagrande cup apparatus and the roller test, respectively.

Table 1 Mineralogical composition of the four different samples T1 to T4 (bulk mineralogy and the clay fraction)

Min (%)	Sample	T1	T2	T3	T4
Clay Minerals (Oriented clay powders)	Kaolinite	54	48	43	44
	Illite	21	21	14	21
	Chlorite	15	16	22	18
	Mixed-layer clay minerals (10–14)	9	12	18	14
	Smectite	02	04	04	04
Total Clay (Random powders)	Total clay	45	56	43	49
	Quartz	27	26	22	28
	Calcite	17	14	13	19
	Gypsum	Traces	Traces	14	Trace
	Dolomite	Traces	Traces	Traces	Traces
	Hematite	Traces	Traces	Traces	Traces
	Feldspar	Traces	Traces	Traces	Traces

3 Analytical results

3.1 Mineralogy

Formations of the Timgad Basin can reach a total clay fraction from 43% to up to 56% (Table 1), with kaolinite as a dominant clay mineral, reaching a percentage of 43–54%, accompanied by 15–22% of chlorite, 14–21% of illite, 9–18% of mixed-layer clay minerals, and traces of smectite < 4% (Figs. 4, 5, 6 and 7).

For the non-clay accessory minerals, they consist mainly of 22% to 28% quartz, 13% to 19% calcite, and some traces of dolomite, gypsum, hematite, and feldspar (Fig. 8).

3.2 Geochemical analysis

As the XRD bulk mineralogy composition and the XRD clay fraction analysis revealed no significant variations, only two samples were selected for geochemical analysis by XRF, i.e., T1 and T2, most representative samples. Their composition (cf. Table 2) shows that the main oxides (SiO_2+Al_2O_3) represent more than 50% with a rate of alumina much more lower than 45%. These confirm the non-refractory clayey aspect of these materials of the Timgad Basin [4]. The presence of ferric oxide Fe_2O_3 > 6% testify the reddish-brown of the investigated Messenian red clays, while the potassium oxide K_2O point and confirm the presence of illite-type minerals. Nevertheless, in both of the analyzed sample, the high percentage of loss on ignition

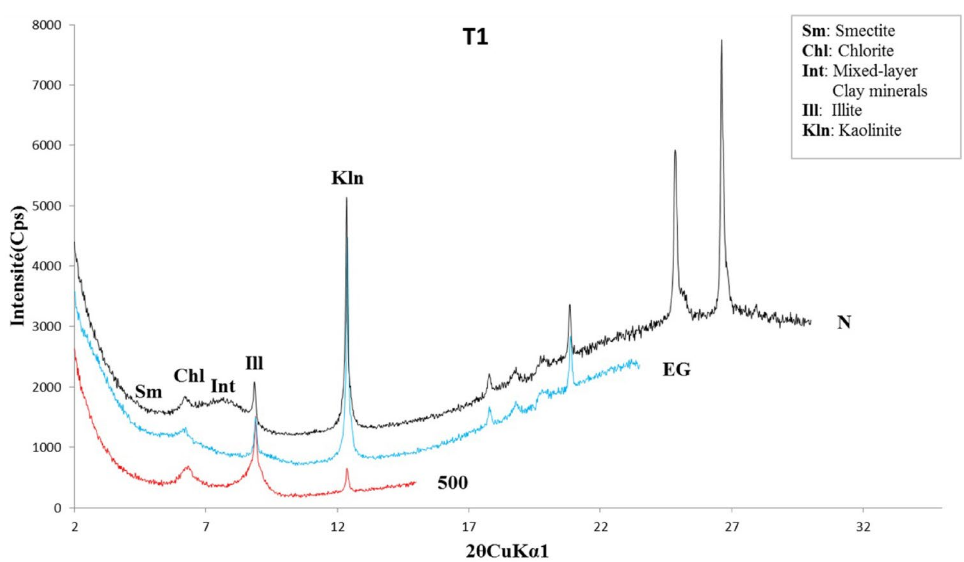

Fig. 4 X-ray diffraction patterns (N, EG, 500) for oriented preparations in < 2 µm clay fractions (**Sample: T1**)

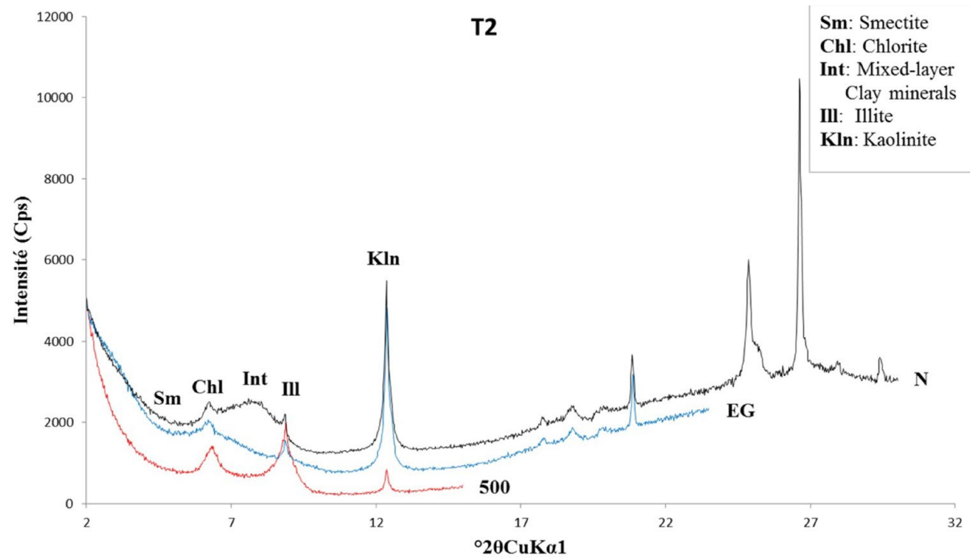

Fig. 5 X-ray diffraction patterns (N, EG, 500) for oriented preparations in < 2 µm clay fractions (**Sample: T2**)

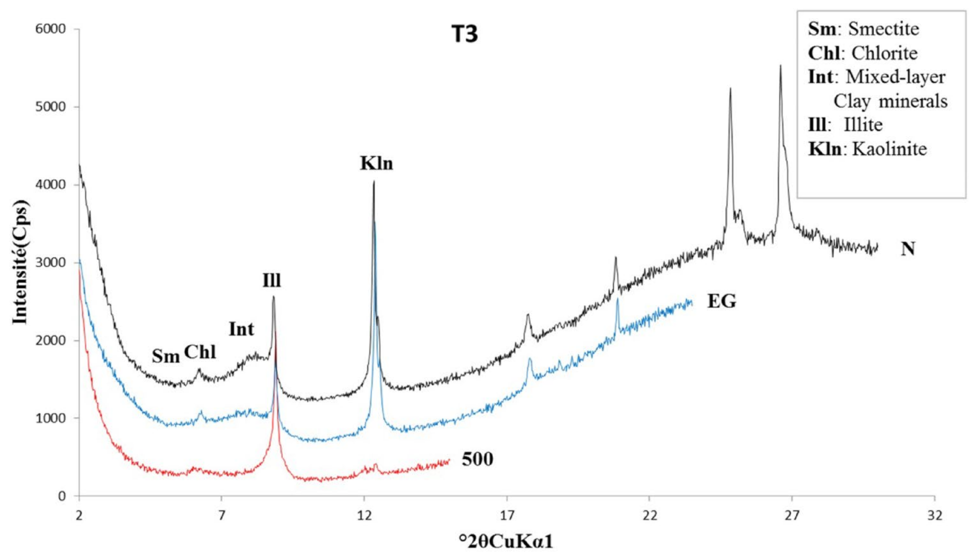

Fig. 6 X-ray diffraction patterns (N, EG, 500) for oriented preparations in < 2 µm clay fractions (**Sample: T3**)

(LOI ~13%) is strongly related to carbon dioxide released from the presence of calcite.

3.3 Morpho-structural characterization

The textural and spatial relationships of the samples examined from rock-chips under SEM (cf. Figs. 9 and 10), led to the confirmation of the abundance of the clayey fraction composed mainly of kaolinite crystals [19]. Their crystal shapes are reasonably close to hexagon platelets. However, the observed illite and chlorite display elongated fine-grained shapes and rosettes, respectively [20]. The photomicrographs also highlighted crystals of quartz and calcite, which are both presented in the form of small grains.

3.4 Physical characterization

3.4.1 Particle size distribution

The grain-size distribution curves of the four studied samples T1 to T4 revealed the existence of three distinct granulometric fractions (Fig. 11). The most dominant granulometric population is related to the fraction between 2 and 63 µm that represents between 73.5 and 80.8% of the mass of the samples. This is followed by the second class of particle size < 2 µm with a percentage ranging from 13.2 to 18.6%, and finally the fraction > 63 µm with a percentage not exceeding 1% for all the samples. Indeed, these grain-size results are strongly in agreement with the whole mineralogical composition (cf. Table 1), which is indicated by the presence of high predominance of fine crystals of

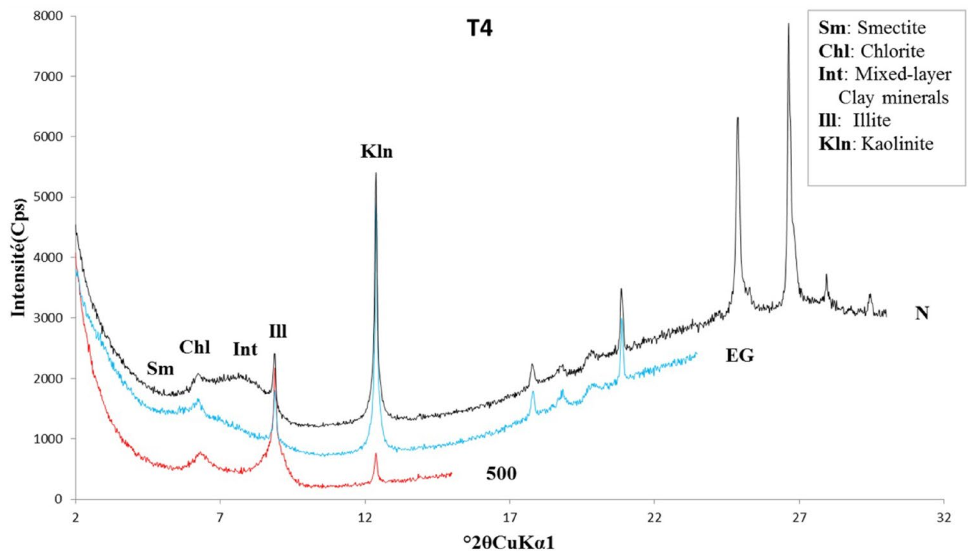

Fig.7 X-ray diffraction patterns (N, EG, 500) for oriented preparations in < 2 µm clay fractions (**Sample: T4**)

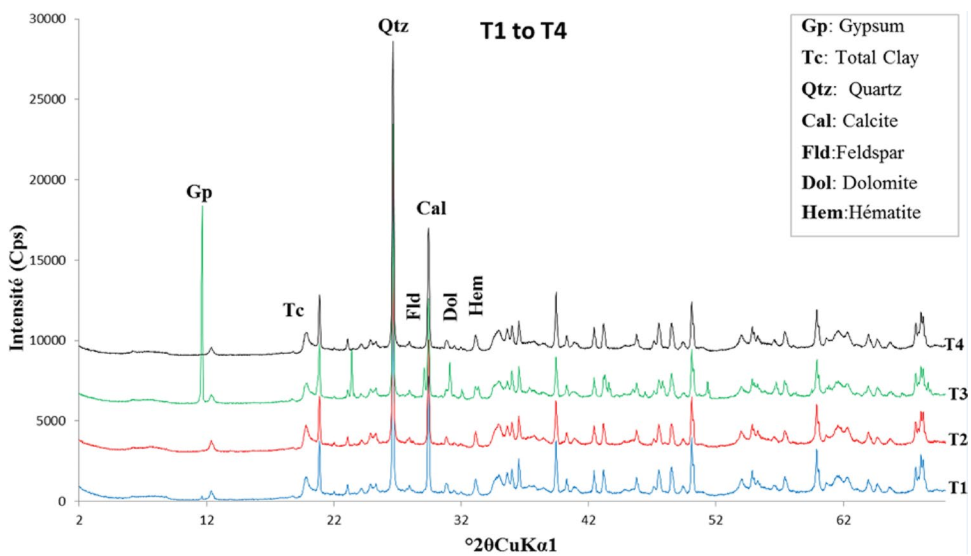

Fig. 8 X-ray diffraction pattern for bulk mineralogy "random powders" (**Sample: T4**)

Table 2 Results of the geochemical analysis of the two analyzed samples T1 and T2 by X-ray fluorescence spectrometry

Sample	SiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5	TiO_2	SO_3	LOI	Σ
T1	48.49	14.38	6.33	0.08	2.63	10.83	0.26	2.01	0.29	0.75	0.36	13.47	99.88
T2	49.28	15.17	6.24	0.06	2.39	9.45	0.63	2.11	0.24	0.81	0.49	13.06	99.93

Whole-rock analysis of major elements (oxide wt.%) with the integration of LOI

quartz and calcite, but also the variation in proportion of the clayey fraction, as well as the nature of the clay mineral assemblages, mostly dominated by large crystals of kaolinite in comparison with the other clayey minerals [7].

3.4.2 Geotechnical tests

The geotechnical tests aim at determining the physical and mechanical properties of the soil and its behavior towards the water and consequently, its potential of

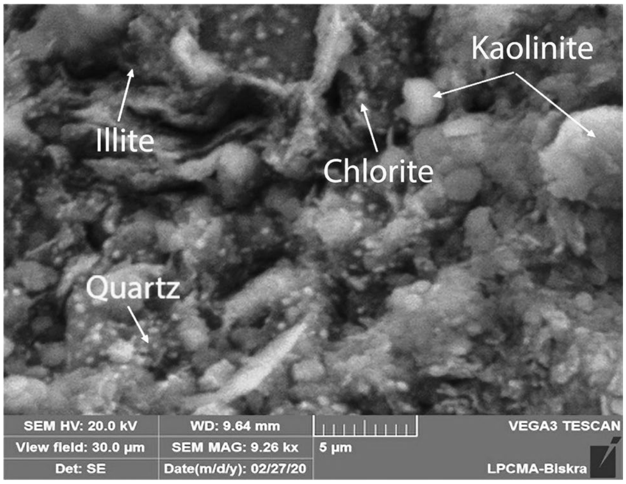

Fig. 9 SEM photomicrographs (backscatter images) of sample T1

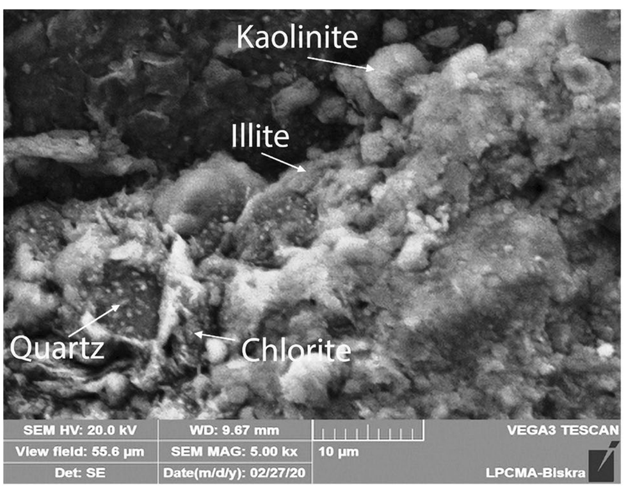

Fig.10 SEM photomicrographs (backscatter images) of sample T2

swelling. The results of these tests, displayed in Table 3, show that the clay-dominated formation from the Timgad Basin present liquid limit (LL) that vary between 38.61 and 40.94%, plastic limit (PL) 23.33–25.67% and plastic index (PI) 13.73–16.61%. According to the Casagrande plasticity chart [21], projection of the acquired dataset allowed to classify the Neogene clays as inorganic clays of medium plasticity, which corroborate the abundance of kaolinite, illite, and the high percentage of quartz (Fig. 12).

4 Discussion

Some clays and clay minerals on the Earth's surface are classified as industrial minerals whose use is subject to several factors and technological requirements [2, 22]. Considering our results from this study on the characterization of the Neogene clays of Timgad Basin (Massif of Aurès, NE Algeria), this clay-dominated formation does not present qualities that allow to qualify them as noble clays, nor as absorbing clays. However, they present qualities that rank them in the category of common clays [23], i.e., relatively impure, composed of a clay minerals mixture of at least 50%, and accompanied by accessory minerals such as quartz, calcite, feldspars, and iron oxides.

Therefore, the use and application of Timgad Basin clays in some fields should be excluded, e.g., fine manufacturing industries (paper, pharmaceuticals, paint, fine ceramics, and among others), environmental field (geomembrane for technical landfills [24], purification or sorption of heavy metals [25, 26]), because all requires very pure clayey materials with high absorbent properties.

Besides the aforementioned potential areas of application, the properties of the investigated clays seem to be favorable for possible use in the field of coarse ceramics and the manufacturing of terra cotta 'Terre Cuite' products. Because, such applications requires raw material that meets some technical requirements, which are indeed present within the Timgad Basin clays such as the grain size, the mineralogical and geochemical composition [27, 28].

Therefore, the clay raw materials used in the field of coarse ceramics [27], and in particular for the terra cotta products manufacturing (common bricks and tiles) must be made of balanced materials containing at least 40% of clay fraction, which is essentially characterized not only by appreciable plasticity, low shrinkage, absence of swelling,

Fig. 11 Particle size distribution for each sample (T1 to T4) from the Timgad Basin

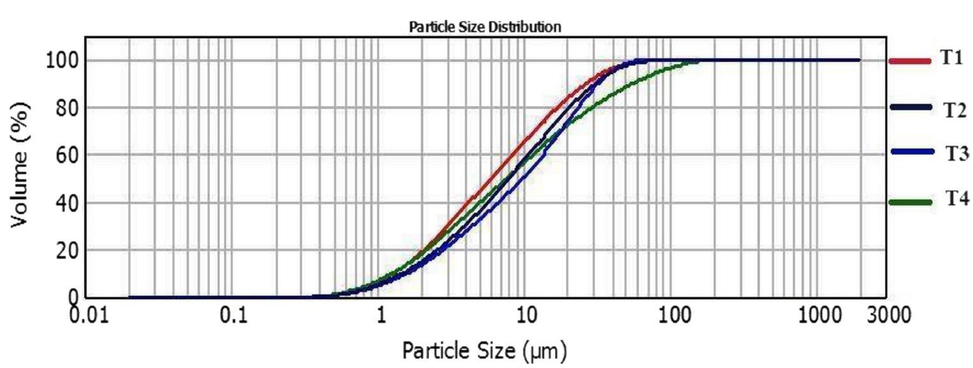

Table 3 Atterberg limits data of the different four samples

Sample	Liquid limit (WL)%	Plastic limit (WP)%	Plastic index (PI)
T1	38.61	23.33	15.28
T2	40.94	25.33	16.61
T3	39.30	25.67	13.73
T4	40.08	24.33	15.75

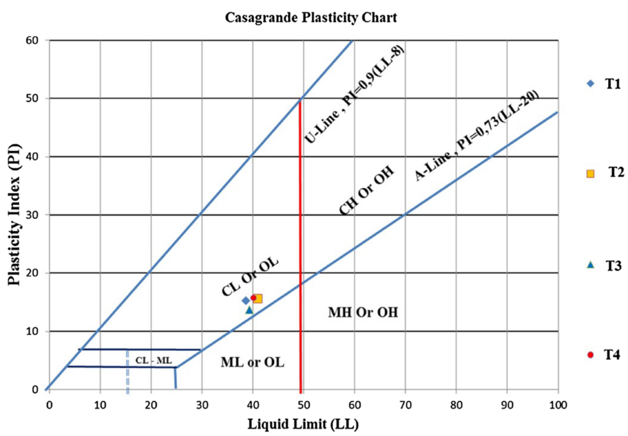

Fig. 12 Projection of the different four samples in the Casagrande plasticity chart

and rich in elements with a melting and grease-remover effect, but also free of harmful elements such as high proportions of carbonates, sulfates, sulfides, and chlorides. This is justified by:

- An adequate geochemical composition represented by acceptable proportions of different oxides of the major elements [28], essentially SiO_2 (35–85%), Al_2O_3 (9–45%), CaO (0–25%), Fe_2O_3 (0–10%), and $Na_2O + K_2O$ (1–5%).
- A mineralogical composition represented by a clay mineral assemblage mainly composed of kaolinite and illite [2]. Indeed, these minerals significantly improve the plasticity and do not cause any shrinkage problems during the drying and firing processes, accompanied by other non-clay minerals having grease-remover and/or melting effects such as quartz, feldspars, and calcite.
- A particle size distribution favoring acceptable geotechnical properties [29], characterized by a liquid limit (LL) between 30–35%, plastic limit (PL) 12–22%, and finally plastic index (PI) that vary between 7 and 18%.

As the characterized clay formations of the Timgad Basin are very rich in fine fractions estimated between 43 and 56% and represented mainly by kaolinite (43–54%), illite, and chlorite, these non-swelling minerals guarantee perfectible and optimal plasticity of the dough during manufacturing. While the absence of harmful elements is also a positive point, except for some traces of gypsum, which are present on the ground in the form of small strings, but easy to remove. Moreover, the good proportion of quartz (22–28%), and calcite (13–19%) both act as a grease-remover and melting agents, respectively (cf. Table 1). This mineralogical composition is confirmed by the geochemical composition with SiO_2 (48.5–49.3%), Al_2O_3 (14.4–15.2%), CaO (9.5–11%), Fe_2O_3 (6.2–6.3%), and $Na_2O + K_2O$ (2.3–2.7%) (cf. Table 2).

Furthermore, based on these cited criteria and in comparison with the requirements in terms of mineralogical and geochemical composition, which define the field of valorization of the clayey material and in particular those related to the field of ceramics and the production of terra cotta, it is noted that the Neogene clays from the Timgad Basin not only meet the requirements in this field of manufacturing and production [27], but also they are strongly comparable with the earthen materials used for the common bricks manufactured in the majority of the European countries [30] (cf. Tables 4 and 5), and this considering the concordance of all their mineralogical and chemical data with the tolerable thresholds in the matter. With the exception of the contents of quartz which appear below the required limit, which requires a pre-treatment with the addition of a degreaser.

Nevertheless, the comparison of particle size and the geotechnical properties with the acceptable standards in this field of ceramics and the production of terra cotta

Table 4 Ranges of geochemical compositions of clay raw materials used in the brick and tile industry in 11 European countries (Austria, France, Netherlands, Hungary, Italy, Greece, Denmark, Belgium, UK, Switzerland, Germany) (European Commission 2007) [30]

Chemistry (wt-%)	SiO_2		Al_2O_3		Fe_2O_3		MgO		CaO		Na_2O		K_2O		TiO_2	
	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max
Countries																
European range in 11 countries	33.05	**80.60**	5.47	30.0	1.0	11.5	0.0	7.21	0.0	26.0	0.1	14.13	0.1	5.9	0.3	2.0
Timgad Basin Algeria (average of the 04 samples T1 to T4)	48.49	**49.28**	14.38	15.17	6.24	6.33	2.39	2.63	9.45	10.83	0.26	0.63	2.01	2.11	0.75	0.81

Table 5 Ranges of mineralogical compositions of clay raw materials used in the brick and tile industry in in 11 European countries (Austria, France, Netherlands, Hungary, Italy, Greece, Denmark, Belgium, UK, Switzerland, Germany) (European Commission 2007) [30]

Mineralogy (%)	Quartz		Feldspar		Calcite		Pyrite		Kaolinite		Illite		Montmo-rillonite		Verm-iculite	
	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max
Countries																
European range in 11 countries	0	**71**	0	33	0	41	0	7	0	40	0	60	0	50	0	20
Timgad Basin Algeria (average of the 04 samples T1 to T4)	22	**28**	Small amount	13	19	–	–	18	27	6	12	1	2	none		

Table 6 Ranges of Atterberg limits required for clay bricks by the manufacturing of products from earthen materials [29]

Sample	Liquid limit (WL)%	Plastic limit (WP)%	Plastic Index (PI)
Standards	**30–35**	**12–22**	**7–18**
Timgad clay (average of the 04 samples T1 to T4)	38.61–40.94	23.33–25.67	13.73–16.61

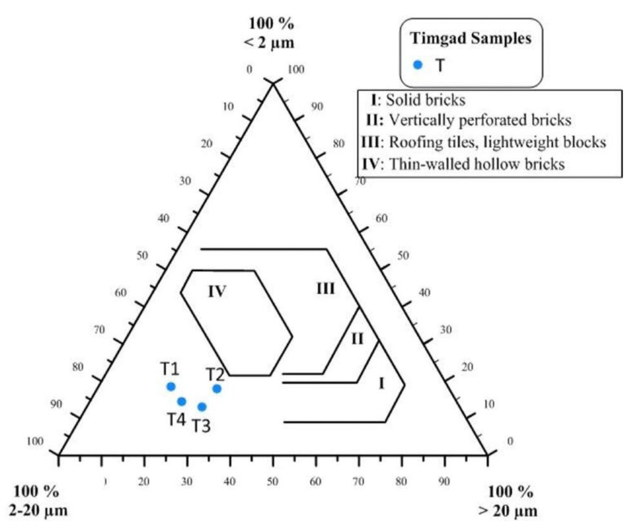

Fig. 13 Particle size data projection in the Winkler diagram [31]

reveals that the studied clays are non-refractory material [30]. Mostly with medium plasticity (Fig. 12) and liquid limit higher than the recommended standard values by the manufacturing of products from earthen materials (see Table 6) [29]. Indeed, this was confirmed by the projection of the results in the Winkler diagram (Fig. 13), which takes into account the particle size distribution [31], and highlight that the Timgad Basin samples do not coincide with any type of raw material intended for the manufacturing of various brick types, unless these clays are subject both to the grain-size improvement and to enhance their geotechnical properties for an optimal plasticity, notably by the addition of a grease-remover in the form of coarse sand.

5 Conclusion

Through centuries, clays and clay minerals have been at the center of interest of many people in several fields, principally because of their numerous uses and applications. Each valorization and delimiting of the potential sites of the local mineral resources starts with detailed characterization and investigation, which constitutes a pertinent starting point and a great asset for the present study.

Based on the preliminary mineralogical and physico-chemical characterization of the Neogene clays from the Timgad basin (Massif of Aurès, NE Algeria), it was concluded for the first time that these clay formations are non-refractory materials, and have a great potential area of application, accordingly to their numerous properties, namely their average plasticity and ideal mineralogical composition. Indeed, each of the four characterized samples T1 to T4, the most representative, taken from a clay-dominated formation that outcrops 5 km east of the Timgad city are constituted of more than 50% of fine fraction. This fraction indicates the presence of kaolinite as a dominant clay mineral, reaching a percentage of 43–54%, accompanied by non-negligible 15–22% percentage of chlorite, 14–21% of illite, 9–18% of mixed-layer clay minerals, and traces of smectite < 4%. Besides, this clayey assemblage is accompanied by some proportion of quartz 22% to 28%, and calcite 13% to 19%, as well as some traces of dolomite, gypsum, hematite, and feldspar.

Finally, in light of these results and both in combination with the grain-size distribution, as well as the geotechnical test results, the studied Neogene clays from the Timgad Basin not only can find application in the field of the brick production industry and terra cotta 'Terre Cuite' products, but also they are strongly comparable with the earthen materials used for the bricks manufactured in the majority of the European countries. Nevertheless, to better improve the plasticity of these raw materials, a preliminary treatment with an addition of a grease-remover, e.g., in the form of coarse sand, is highly recommended.

Funding The present study was fully funded personally by the first author (Bachir LAMOURI).

Data availability The data used in this study are available upon request from the corresponding author.

Declarations

Conflict of interest The authors declare that there is no conflict of interest. The author Bachir LAMOURI declares that he has no conflict of interest. The author Sedik Abdallah LABADI declares that he has no conflict of interest. The author Mechati BOUKOFFA declares that he has no conflict of interest. The author Ibtissem CHOUAF declares that she has no conflict of interest. The author Hocine DJOUDER declares that he has no conflict of interest. The author Fouad DJAIZ declares that he has no conflict of interest. The author Lakhdar BOUABSA declares that he has no conflict of interest.

Human or animal rights This article does not contain any studies with human participants or animals performed by any of the authors.

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