

Composition and Ceramic Properties of Carbonate-Bearing: Illitic Clays from North-Eastern Tunisia

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Abstract The Aptian clays of Jebel Ressas (north-east of Tunisia) have been studied for their use in ceramic industry. At first, mineralogical, chemical, physical, and thermal analyses of these clays are given. Indeed, illite is the main mineral (60–65 wt%) but other minerals; quartz, kaolinite, interstratified illite/smectite, calcite and feldspar, are present in small quantities. Next, this study reveals that the average amounts of silica and potassium are 51.57 and 3.35 wt%, respectively. The percentage of potassium is also quite high, suggesting the presence of illite. The amount of alumina is in average of 19.01 wt%. The contents of lime and iron vary between 5 and 8 wt%. The grain size data indicate a silt-dominated assemblage. The plasticity test shows a medium value (PI = 16–20 wt%). The firing shrinkage and the expansion are limited. The absence of expansible minerals in these clays explains why the plasticity and the linear shrinkage are limited. Finally, two mixtures M1 and M2 prepared from these clays show that ceramic properties respect the norm and the industrial tests confirm that these clays could be used in manufacturing of bricks and earthenware tiles.

Keywords Illitic clays · Ceramic properties · Ceramic production

الخلاصة

تمت دراسة عيّنات طينية أخذت من جبل الرصاص بالشمال الشرقي لتونس وذلك لاستخدامها في صناعة السيراميك . وتم - في البداية - القيام بالتحليلات المعدنية والكيميائية والفيزيائية والحرارية للعينات. وبيّنت الدراسة أن هذه العيّنات غنيّة بالإليت " illite " بوصفه معدناً (60 - 65%) . وقد وجدت بعض المعادن الأخرى ولكن بكميات صغيرة كالكوارتز ، والكاؤولينيت kaolinite ، والكالسيت calcite وخليط من الإليت/سمكتيت illite/smectite mixed layer والفلدسبار feldspar .

وبيّنت التحاليل الكيميائية أن هذا الطين يحتوي على نسبة عالية من و السيليكا و البوتاسيوم (51.57% و 3.35%) . والمعدل الوسطي لكمية الألمنيوم هو 19.01% . و يتميز هذا الطين بلدانة منخفضة حيث تتراوح بين (16 - 20%) . وتعتبر درجة التمثط لهذا الطين منحصرة أيضا الأمر الذي يمهّد الطريق إلى استخدامه في صناعة السيراميك المطابق للمواصفات العالمية.

1 Introduction

Tunisia is a sedimentary country in which clays occur widely. Besides their geological interest, clays are of importance for local industry. For the most case, they have been used to produce bricks and earthenware tiles. Today, there is an increase in construction activity in Tunisia. The industries cannot meet the ever-increasing market demand for the construction materials needed. Few studies have been carried out on the clays or of raw materials used for traditional ceramic in Tunisia. For example, Ben M' Berek et al. [1] studied nature and distribution of the north-east Tunisian clays and their possibility in ceramic industry. El Hechi [2] studied the physico-chemical properties of clays associated to

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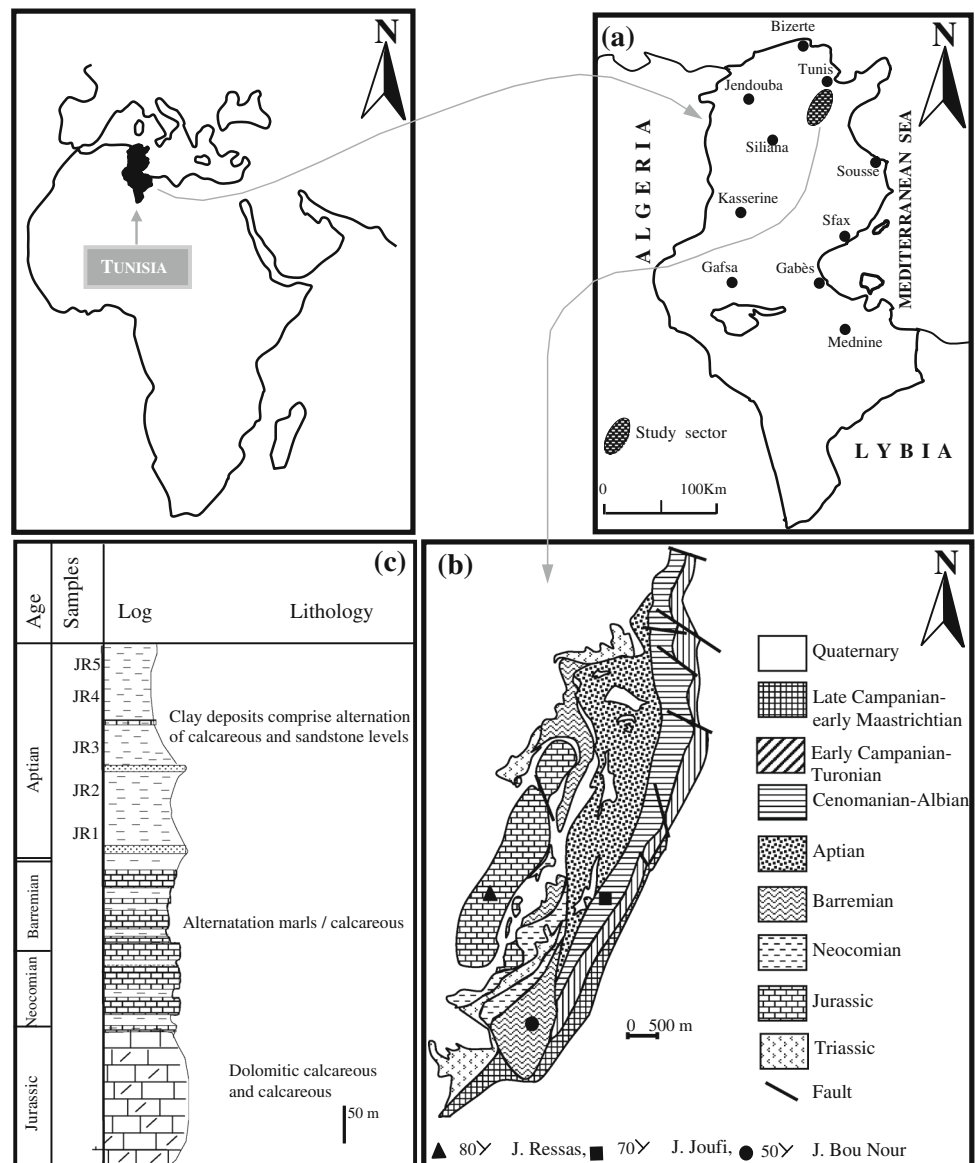
great fault of Zaghouan, located in the south east of Tunis. Mahmoudi et al. [3,4] studied the characteristics of the clay deposit at Jebel Oust (see the geological map of Bir M'Cherga No. 28 at 50,000 scale) and their application in traditional ceramic.

The studied sector is the Jebel Ressas (see the geological map of Grombalia No. 29 at 50,000 scale), located in the south of Tunis, as a neighbouring area to the ceramic factories (<40 km). Jebel Ressas is composed of Jurassic limestones and dolomitic limestones. These facies are characterized by subvertical layers limited by two vertical faults having a direction north–east/south–west. The Jurassic ground is surrounded by cretaceous clays (Fig. 1). The clay deposits contain an important reserve which comprises alternation of

calcareous and sandstone levels [5,6]. The average thickness of useful clays is 400 m. Sedimentologically speaking, the variation of lithology and thickness which affected the Jurassic series indicates sedimentary movements. The discontinuous sedimentary passage from Jurassic to cretaceous was provoked by tectonic events [6]. Our interest centred around the Aptian clays alternate with occasional sandstone levels.

There is an ample demand for quality of ceramic products, and, thus, the present study evaluates the mineralogical, chemical, physical characteristics, and technological behaviour of the raw materials for use in traditional ceramic. This will help to give a better understanding of the clay materials to industrialists.

Fig. 1 a The geographic, b geological situation of the study sector [6], and c synthetic lithostratigraphic log of J. Ressas



2 Materials and Methods

2.1 Sample Preparation

For the characterization of the clay deposits, five bulk samples representative of the prevailing facies were collected from Jebel Ressay area. They were initially dried at 105 °C until a constant weight was achieved, and they were powdered for 30 min. To prepare the oriented aggregates, the clay fraction was purified by classical methods. In fact, the raw material was completely decarbonated by adding a small amount of 1 N HCl, and treated with H₂O₂ to eliminate organic matter. The material was washed five times with 1 N NaCl, excess salt was removed by dialysis. The fraction <2 μm was separated in a centrifuge, and suspension was dried at 60 °C [7,8].

For technological tests, the clays humidified at 6 wt% water content, mixed and sieved (Ø 1 mm) until homogeneous paste was obtained. Specimen tiles were prepared using 60 g of green material per test piece by pressing at 20–25 N/mm² using 100 × 50 × 10 mm molds and then dried in air at room temperature during 1 week and then at 105 °C for 24 h.

2.2 Methods

X-ray diffraction (XRD) patterns were recorded on a Philips X'Pert diffractometer at the Centre of Researches and Technology of Energy (CRTE) using CuKα radiation (1.5418 Å), both on bulk or powder samples and oriented aggregates [natural (N) and treated with ethylene glycol (EG) and heated to 550 °C for 2 h (H)]. A scanning rate of 1° 2θ/min from 5° to 60° is considered. For the quantitative analysis of the samples, the relative abundance of minerals was estimated from the chemical data and taking into TG curve.

The major element compositions (SiO₂, Al₂O₃, Fe₂O₃, K₂O, Na₂O, MgO, CaO, MnO and TiO₂) were determined by atomic absorption spectroscopy.

Loss on ignition (LOI) was measured from total weight after ignition at 1,000 °C for 2 h.

The parameters of plasticity [liquid limit (LL), plastic limit (PL) and plastic index (PI), PI = LL – PL] were defined by Proust et al. [9] and Modesto et al. [10]. These parameters were determined by the Casagrande method, in accordance with the French Standard NF P 94-051.

The grain size analysis distribution of as-received samples was obtained by wet sieving, using an AFNOR series device adopted by the French standardization system.

The differential thermal analysis and thermal gravimetric DTA/TG were obtained with a SETRAM type 124. The temperature was increased from room temperature to 1,000 °C at a rate of 10 °C/min, maintained at this temperature for 10 min then reduced again. The expansion and firing shrink-

age were measured using the dilatometer Adamel Lhomargy DM 15. The samples were heated from ambient temperature to 900 °C with 10 °C/min.

The linear firing shrinkage, the water absorption, and the bending strength were measured according to the norms ISO 10545-2, ISO-10545-3, and ISO 10545-4, respectively. These technological tests were performed on ten specimens of each sample.

3 Results and Discussion

3.1 Mineralogy

The mineralogical compositions of the bulk and <2 μm fraction are reported in Table 1. The following peaks were used for the identification of different minerals (Fig. 2): quartz (4.26–3.33 Å), calcite (3.03 Å), albite (sodic plagioclase) (3.18 Å), illite (10–5 Å), interstratified illite/smectite (11.5–12 Å), and kaolinite (7.1–3.57 Å). Indeed, XRD shows that the non-clay minerals in the samples are quartz (23–34 wt%), calcite (9–15 wt%) and minor amounts of feldspars (5–9 wt%). The content of quartz is tolerable since it opposes to shrinkage and partly dissolved in the vitreous phase at high firing temperatures during firing [3].

The <2 μm fraction indicates the predominance of illite (50–60 wt%). The illite is characterized by favourable properties: it's the main clay for ceramic use [11–14], whereas interstratified illite/smectite and kaolinite exist in similar proportions (average 17.5 wt%). The absence of smectitic minerals will ensure a ceramic body against possible difficulties during drying.

With reference to the ternary diagram of Fiori et al. [15], we deduced that these clays are rich in clay minerals (Fig. 3). These clays were classified into one basic type according to their clay mineralogy (Table 1): those containing more illite and moderate amounts of illite/smectite and kaolinite. On the basis of these results, and the criteria established by Fiori et al. [15], clays with the highest clay fractions are most suitable for manufacturing ceramic bodies.

3.2 Chemical Composition

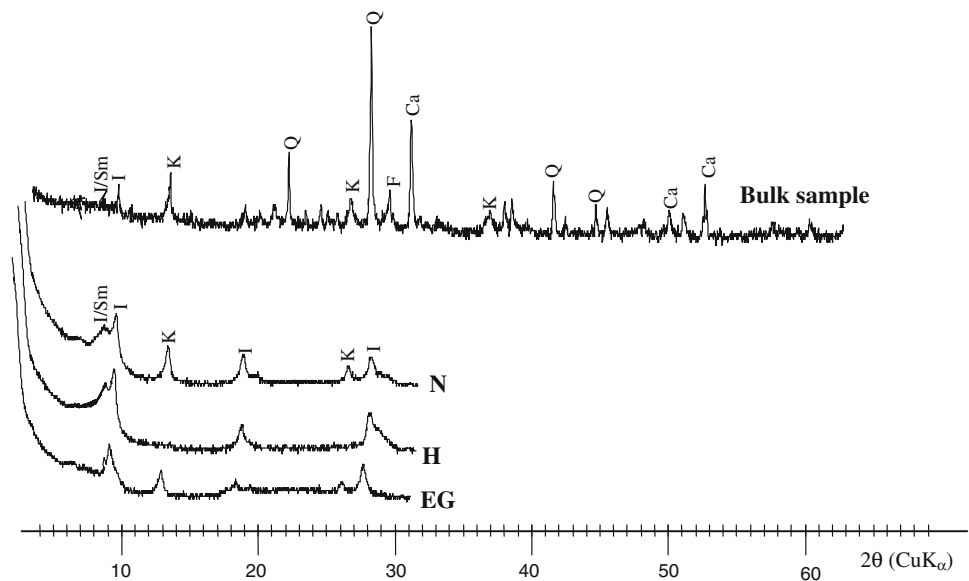
The chemical compositions of the raw materials are given in Table 1. The main oxides are SiO₂, Al₂O₃, Fe₂O₃, K₂O, Na₂O, MgO, and CaO, whereas MnO and TiO₂ are present only in small amounts. The SiO₂ (average 51.57 wt%) content varies inversely with Al₂O₃ content (average 19.02 wt%). The samples contain a relatively high quantity of potash (average 3.35 wt%), feldspar is always low or very low. The content of alkalis (K₂O + Na₂O) is high (average 4.11 wt%). The main effect of alkalis in clays is to reduce their refractory characteristics and, therefore, they are fluxes [16]. More-



Table 1 The physico-chemical analyses of studied clays

Parameters		Sample JR1	Sample JR2	Sample JR3	Sample JR4	Sample JR5	
Chemical composition (wt%)	SiO ₂	52.75	51.03	50.51	52.01	51.55	
	Al ₂ O ₃	19.34	19.38	17.87	20.52	18.03	
	Fe ₂ O ₃	6.51	6.21	5.39	7.69	6.88	
	CaO	5.27	6.88	8.26	5.07	7.51	
	MgO	1.95	1.52	1.34	1.21	1.03	
	Na ₂ O	1.02	0.77	0.88	0.47	0.69	
	K ₂ O	3.02	3.48	3.77	2.92	3.58	
	TiO ₂	0.41	0.34	0.17	0.37	0.21	
	MnO	0.32	0.18	0.23	0.51	0.27	
	LOI	8.54	9.31	10.57	9.69	9.61	
Mineralogy (wt%)	Clay minerals	55	60	47	50	63	
	Whole sample	Calcite	11	10	12	15	9
		Quartz	27	25	34	26	23
		Feldspar	7	5	7	9	5
< 2 μm fraction	Illite	53	60	61	58	54	
	Illite/Smectite	30	25	16	19	23	
	Kaolinite	17	15	23	23	23	
	Plasticity (wt%)	Liquid limit	44	40	40	42	44
Plastic limit		26	21	24	26	24	
Plastic index		18	19	16	16	20	
Microgranulometry (wt%)	Fraction >63 μm	15.5	13.7	10.1	4.4	5.5	
	Fraction 50–63 μm	7.8	13.4	10.3	10.2	11.5	
	Fraction 20–50 μm	15.5	13.1	12.1	17.3	15.8	
	Fraction 10–20 μm	21.7	23.4	19.5	19.3	19.7	
	Fraction 2–10 μm	15.3	16.1	18.3	20.1	18.1	
	Fraction <2 μm	24.2	20.3	29.7	28.7	29.4	

Fig. 2 XRD patterns of bulk sample and fine fraction of studied sample; *N* normal, *EG* saturated with ethylene glycol, *H* heated at 550 °C, *Q* quartz, *Ca* calcite, *F* feldspars, *I* illite, *K* kaolinite and *I/Sm* interstratified illite/smectite



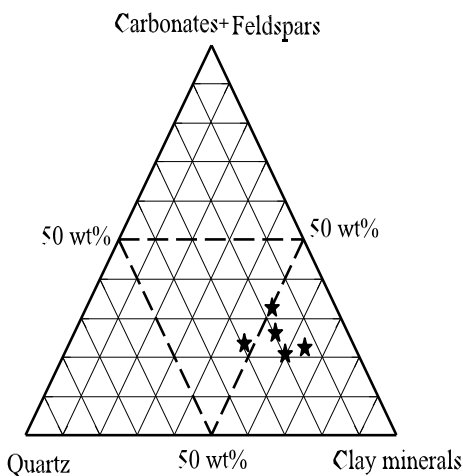


Fig. 3 Ternary diagram: quartz/carbonates + Fe-oxides + feldspars/clay minerals for the studied clays from J. Ressay, after Fiori et al. [15]

over, the $\text{SiO}_2:\text{Al}_2\text{O}_3$ ratio (average 2.73 wt%) is consistent mainly with the amounts of quartz and clay minerals. Concerning the distribution of iron, the mineralogical analysis reveals the absence of iron minerals in the clay samples. We can suggest that this element is probably combined with the illite structure, as the positive correlation between illite and iron is found. This content of iron (average 6.53 wt%) is homogeneous; it will reflect a uniformity and stability of the tonality of color after firing [17].

The studied samples showed mineralogical and chemical compositions similar to those exhibited by materials currently used in Tunisia to produce ceramic bricks and earthenware tiles [18–20].

3.3 Microgranulometry

The grain size analysis results are reported in Table 1. The analyzed samples show a large variation in grain size. For example, the sand content ranges from 4.9 to 15.5 wt%, the silt ratio ranges from 60.2 to 69.9 wt%, and the clay proportion ranges from 20.3 to 29.4 wt%. Grain size data indicate a silt-dominated assemblage. Indeed, the studied samples fall in the clayey–silt field of the Shepard’s diagram [21], except for samples JR1 and JR2, which fall on the boundary between clayey–silt and sandy silt (Fig. 4).

The Mc Manus diagram indicates that the grain size cumulative curve gives well-sorted sediments [22]. The mineralogical and grain size features of the clays suggest that a significant amount of clay minerals occurs in the silt fraction.

Referring to the points location representing the clay grain size fractions in the Winkler’s diagram (Fig. 5) [23], the studied raw material is neither suitable for manufacturing common bricks or vertically perforated bricks.

For ceramic products, the industrialists search for the clays rich in finer fraction (lower than $2\ \mu\text{m}$). This property allows

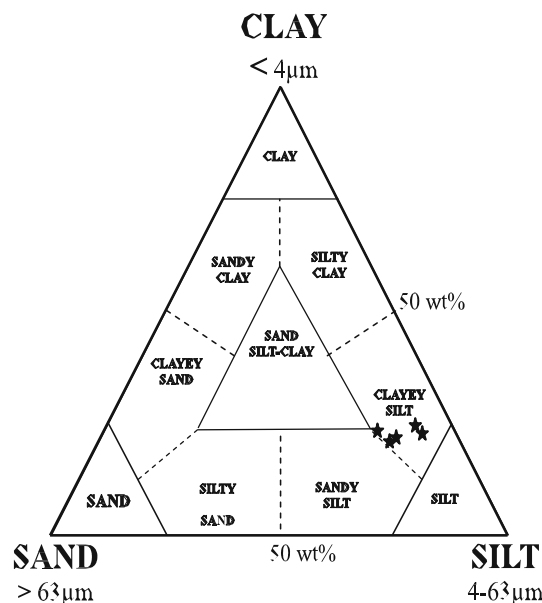


Fig. 4 Classification of studied clay raw materials from J. Ressay based on the sand–silt–clay ratios [22]

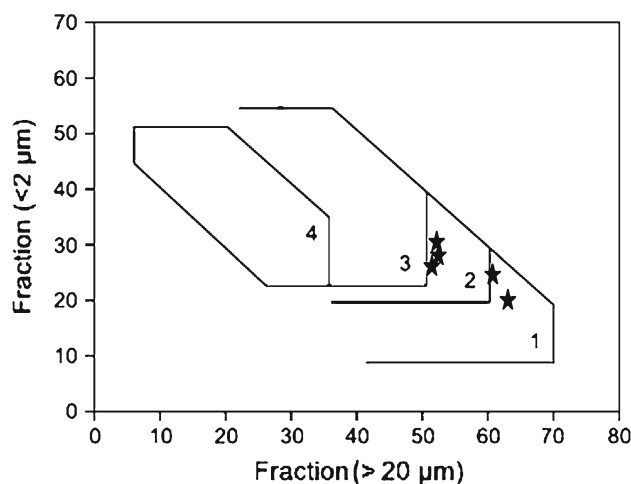


Fig. 5 Position of the study clays on Winkler diagram [23] for manufacturing common bricks (1), vertically perforated bricks (2), roofing tiles and light blocks (3) and thin-walled hollow bricks and blocks (4)

cohesion of ceramic paste. The problem arising the presence of particles $>63\ \mu\text{m}$ can be solved by simply crushing and sifting.

3.4 Plasticity

The plasticity parameters are illustrated in Table 1 and showed that the plasticity index and liquid limit are 18 ± 2 and 42 ± 2 wt%, respectively. These parameters, plotted in the Holtz and Kovacs diagram [24] indicate that these samples are illitic clays, having medium or moderate plasticity (Fig. 6). These values of plasticity are suitable and encouraging for

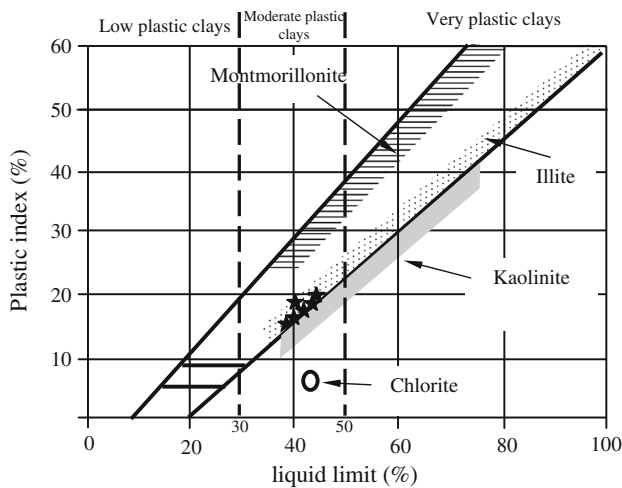


Fig. 6 Position of the study clays on the Holtz and Kovacs diagram [24]

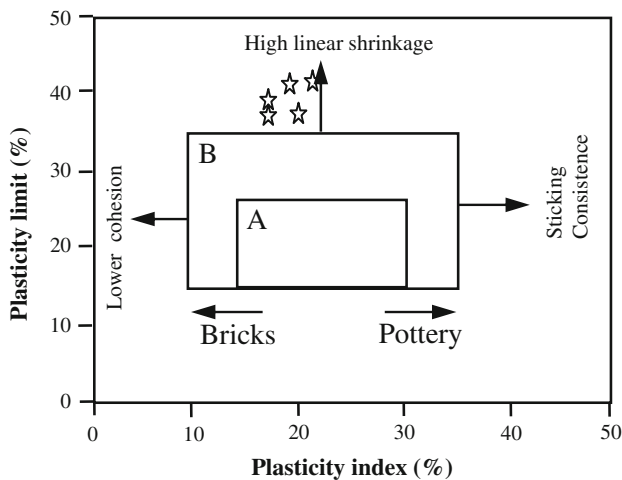


Fig. 7 Diagram of Bain and Highley [26] showing the optimum (A) and acceptable (B) domains for clay shaping by extrusion

optimal behaviour in pressing (cohesion) and drying (negligible contraction) which are in agreement with the range defined in the literature for ceramic production [25].

Because of its high amount of the fraction $>20 \mu\text{m}$ ($>52 \text{ wt}\%$), these clays are rather convenient for bricks and the high linear shrinkage can be expected. This could be supported by the position of the clay Atterberg limits in the diagram of Fig. 7 [26]. In view of these results, the manufacturing of these products required for instance additions of coarse grains of tectosilicates and/or carbonates.

3.5 Thermal Analyses

Figure 8 shows the results of differential thermal analysis (DTA), thermogravimetry analysis (TG), and dilatometry analysis (DA) of the clay sample. It can be observed that

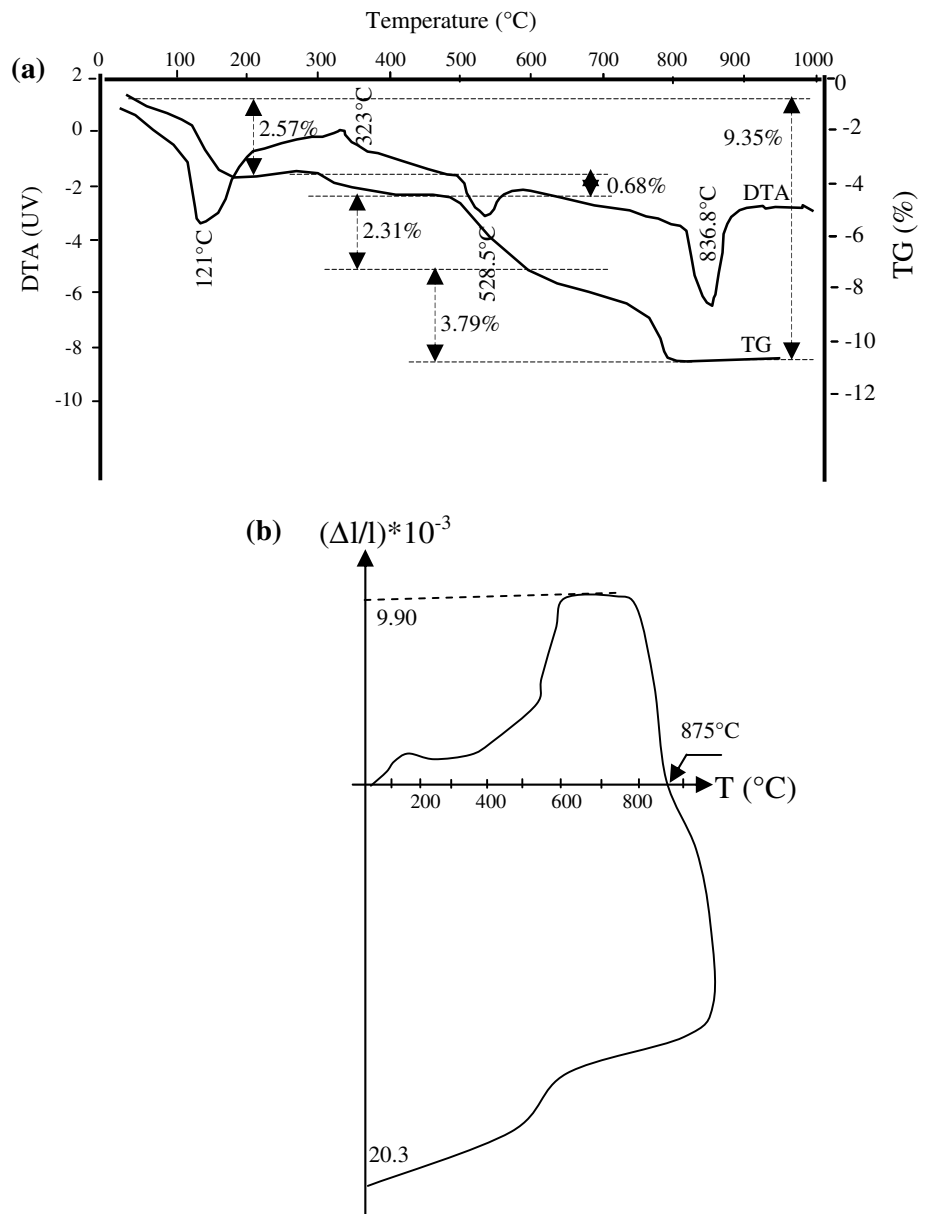
in DTA curve, there are three endothermic peaks and one exothermic peak. Their temperatures are similar which represent the similar reaction that occurred at those temperatures. For this reason, we interpreted just one curve for the sample JR1. Indeed, at a low temperature ($121 \text{ }^\circ\text{C}$ for DTA and $135 \text{ }^\circ\text{C}$ for DA), an endothermic reaction occurs due to the removal physically bound water (Fig. 8a). At this temperature, TG curve shows a weight loss $2.57 \text{ wt}\%$. The exothermic effect is owing to decomposition of organic matter, detected at $323 \text{ }^\circ\text{C}$, which corresponds to a minor weight loss $0.68 \text{ wt}\%$. As the temperature increases, the dehydroxylation around $528.5 \text{ }^\circ\text{C}$ is typical of kaolinite, observed in DTA curve and to the polymorphic α to β transition of quartz at $573 \text{ }^\circ\text{C}$ (a small endothermic peak on the DTA curve), observed in DA curve, of the clay minerals structure and formation of quasi-amorphous materials [27–31]. The endo-reaction for decarbonation is detected at $836.8 \text{ }^\circ\text{C}$ [32]. The weight loss attributed to carbonate decomposition is estimated at $3.79 \text{ wt}\%$.

The typical clay behaviour is characterized by an expansion till to about $600 \text{ }^\circ\text{C}$ (Fig. 8b), which indicates a richness of illite, with reference to Cizeron [33], which revealed that illitic clays present a stage or landing in the temperature range $600\text{--}800 \text{ }^\circ\text{C}$. This expansion is followed by a structural collapse of some clay minerals due to the loss of structural water, and a moderate shrinkage due to the carbonate decomposition [34]. Finally, during clay cooling, we can observe a small effect on the curve attributed to the β to α inversion of quartz. In short, the firing shrinkage is limited (about $2 \text{ wt}\%$); the clays could be used in the manufacturing of ceramic products. A lower firing temperature can be translated into significant energy savings.

3.6 Industrial Tests

These clays are characterized by similar mineralogical, geochemical, and physical composition. Consequently, they show similar firing behaviour that is of decisive importance for the quality of the end products [35,36]. Indeed, the study proved that these clays are illitic with a discrete content of carbonates and have moderate plasticity. The firing shrinkage and expansion are limited; thus these clays could be used in the manufacturing of traditional ceramic products. But according to Ferrari et al. [15], who used illitic clays for traditional ceramic and showed that the high amount of illite is necessary in ceramic mixtures but it provokes a larger percentage of glass phase, lower water absorption and a higher linear shrinkage, thus the addition of a degreaser (sands, crushed glass, grogs, feldspars, pegmatite, and chamotte; the latter is made up of brick debris and biscuit waste) is necessary to reduce the plasticity and the linear shrinkage,

Fig. 8 a DTA, TG, and b dilatometry curves of sample JR 1

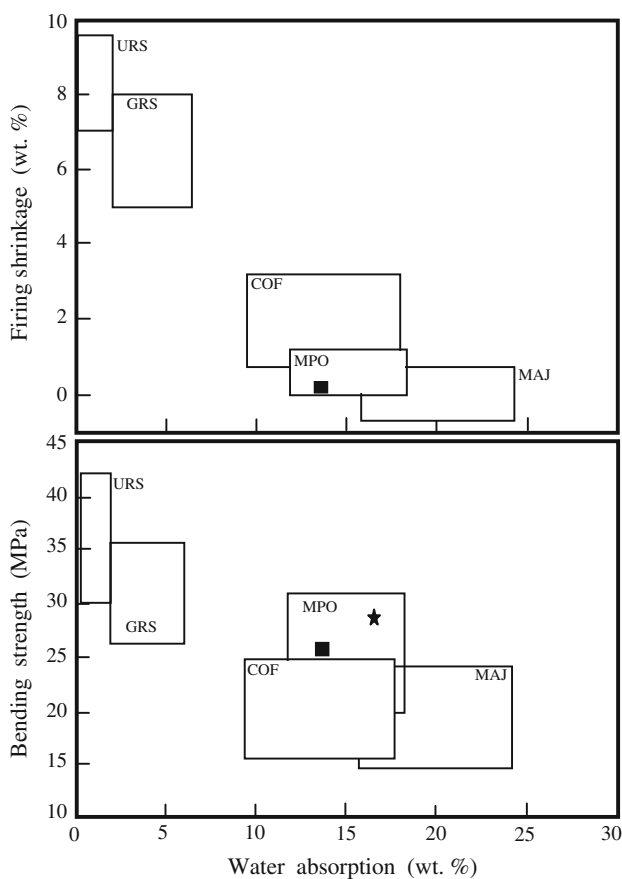


to improve water absorption and makes the clays easy to dry. To judge the quality of ceramic products, two mixtures of clays were prepared from Jebel Ressay M1 = JR1 and M2 = JR1+15 wt% sand, to determine the water absorption, linear firing shrinkage, and the flexural strength. The sand added to mixture M2, comes from Sidi Aïch formation, which was well-studied by Gallala et al. [37]. Table 2 shows the variation of the firing shrinkage, the flexural strength and the water absorption with temperature for the mixtures M1 and M2 of studied clays. It's clear and well-known that the increase of temperature provokes the increase of firing shrinkage and flexural strength and the decrease of water absorption. All ceramic bodies produce red colour

after firing at 1,100 °C and therefore will be more suitable for the production of bricks and earthenware tiles, which confirm chemical analysis, with high content (6.53 wt%) of iron. The high water absorption due to the evolution of more CO₂ outside the structure, derived from carbonate minerals decomposition [38,39]. Indeed, a value of temperature >1,100 °C shows good linear firing shrinkage, water absorption, and flexural strength according to ISO 10545-2, ISO 10545-3 and 10545-4, respectively for commercial use of ceramic bodies. No significant differences can be recognised in these parameters between 1,100 and 1,150 °C. These behaviours perfectly agree with the thermal treatment of illitic clays dealt within literature [36,40]. These firing behav-

Table 2 Results of the physical parameters according to the temperature of the mixtures M1 and M2 of ceramic faïences

Mixtures	Temperature (°C)	Drying shrinkage (wt%)	Firing shrinkage (wt%)	Water absorption (wt%)	Bending strength (MPa)
M1	1,000	4.9	0.15	22.70	8.33
	1,050		0.35	18.10	9.74
	1,100		0.44	16.10	26.88
	1,150		0.45	16.1	28.13
M2	1,000	4.1	0.05	20.52	6.04
	1,050		0.20	16.34	6.88
	1,100		0.22	13.44	20.72
	1,150		0.24	13.10	25.42

**Fig. 9** Relationship between water absorption and bending strength and firing shrinkage of the mixtures M1 (filled star) and M2 (filled square) fired at 1,150 °C and comparison with some commercial products. MAJ majolica, COF cottoforte, MPO monoporosa, GRS glazed red stoneware, URS unglazed red stoneware

iors can be compared with the technological requirements for ceramic tiles with coloured body (Fig. 9) and showed that the mixtures M1 and M2 correspond well with those of monoporosa.

4 Conclusion

The mineralogical analysis of studied clays, collected from Jebel Ressa indicates the presence of illite as the main mineral. The chemical analysis reveals that the amounts of SiO₂ (average 51.57 wt%), Al₂O₃ (average 19.02 wt%), and K₂O (average 3.35 wt%) are suitable for ceramic products. These clays are characterized by a richness of finer fraction, medium, and a low firing shrinkage.

The ceramic properties and industrial tests show appropriate industrial characteristics of these clays, which make able to find application in the production of bricks and earthenware tiles.

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