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VALORIZATION OF CLAY-BASED MINING WASTES FROM OUED EL ANEB MARBLE QUARRY FOR TERRACOTTA PRODUCTION

Purpose. Valorization of marble quarry waste for the production of terracotta meeting industrial requirements. In this context, our work has focused on the use of clayey waste from the Oued El Aneb marble quarry for the manufacturing of terracotta bricks. The principle is based on using a mixture of different clays (yellow, red, black, and gray clays) derived from marble extraction to produce terracotta under various firing temperatures.

Methodology. After sampling and mechanical preparation of the samples in the laboratory, the clayey waste was characterized using several analytical techniques, including X-ray diffraction (XRD), infrared spectroscopy (IR), atomic absorption spectroscopy (AAS), scanning electron microscopy coupled with EDS analysis (SEM-EDS), and X-ray fluorescence (XRF). For the manufacturing of terracotta bricks, tests were conducted under various firing temperatures.

Findings. The use of waste materials from marble quarrying represents an innovative approach to the recovery and valorization of mining waste. The tests conducted for the production of terracotta bricks from these materials have yielded promising results. The fired clay materials obtained from these formulations exhibit high performance characteristics, particularly in terms of firing shrinkage (ranging from 0.47 to 1.44 %), water absorption (between 10.87 and 15.47 %), and flexural strength (ranging from 3.82 to 8.72 MPa).

Originality. The results of the research into the use of mining waste from the marble quarry of Oued El Aneb under different firing temperatures has made it possible to substantiate the effectiveness of producing high-quality terracotta bricks that meet industrial requirements.

Practical velue. The technical properties (physical and mechanical) of the terracotta materials produced from waste raw materials, particularly firing shrinkage, water absorption, and flexural strength, demonstrate the effective valorization of clay waste from marble extraction at Oued El Aneb.

Keywords: *mining exploitation, clayey wastes, SARL SOPRAM, Annaba, North-East Algeria*

Introduction. The extractive industry, particularly the mining industry, is one of the world's vital resources [1]. In Africa, revenues derived primarily from oil and gas extraction and the exploitation of various minerals play a key role in countries' economic and social development [2, 3]. This highlights the importance of improving and optimizing the use of natural resources. Among the most in-demand industrial minerals worldwide is marble. It is extracted from a granular metamorphic rock formed from limestone or dolomite, consisting of a mass of calcite or interlocking dolomite grains. The exceptional beauty, durability, and versatility of marble make it a highly sought-after material for a wide range of applications, depending on the characteristics of the available raw material [4]. Beyond its technical properties, its uses are limited only by the creativity and vision of designers, architects, and artisans [5, 6].

However, despite these advantages, mining residues resulting from ore processing, including those derived from marble, pose a major threat to the environment and biodiversity. Historical failures, along with poor management of tailings storage facilities, have led to some of the most severe environmental disasters [7]. This situation highlights the urgent need to develop new valorization strategies, supported by rigorous and effective environmental management of mining waste.

In the field of mining waste valorization, various techniques have been used, each with its own advantages and disadvantages. One of the most widely employed methods involves integrating these wastes into the production of construction materials, which significantly reduces the volume of waste sent to storage sites and helps conserve natural resources [8, 9]. Regarding the waste from marble extraction, several solutions have been proposed, including recycling marble powder into fine aggregates for concrete, cement, or ceramics [10]. The main challenge lies in choosing the most effective method, both in terms of technical performance and cost-effectiveness.

Algeria is a country rich in various useful substances, the majority of which are still unexploited. Its subsoil contains important mineral resources, often in large quantities and of strategic importance to the country [11]. The exploitation of these deposits can also generate

large volumes of waste [12]. In fact, this research project exemplifies the characterization of mining waste from the Aggregate and Marble Production Company – (SARL SOPRAM Oued El Aneb – Annaba) aiming for its valorization through terracotta manufacturing.

Location of the deposit. Located approximately 300 m from the exit of Oued El Aneb towards Draa Errich, the quarry can be found on the right side of the road after a 1km track used exclusively by quarry operations and local residents (Fig. 1). The AbdRabbouh Quarries feature limestone formations characterized by a single bench of white limestone, which is covered with a layer of topsoil in the southern area, extending beyond the limits of the concession. This topsoil is underlain by clay deposits that can vary in thickness from 1m to over 7m at the higher elevations of the terrain. Beneath these limestone formations lies a less durable shale substrate [11].

Moreover, Annaba, the city where this quarry is located, is situated in the northeast of Algeria along the coast; it boasts a Mediterranean climate, featuring hot and rainy winters alongside hot summers. The city's humidity levels are notably high due to its geographical position, nestled between the mountains and the sea [12, 13].

Geological setting of the region. Annaba is generally characterized by a varied relief consisting of mountains, hills, and plains. The mountains cover 52.16 % of the territory, which corresponds to an area of 736 km^2. Particularly, the Edough massif features the highest point, Mount Bouzizi, which rises to an altitude of 1,100 m. The hills and foothills occupy 25.82 % of the total area, equal to 365 km^2. The plains make up 18.08 % of the territory, totaling 255 km^2, and are primarily represented by the Kherraza plain. The remaining relief consists of plateaus and other formations, accounting for 3.94 % of the area, or 56 km^2. The tectonics of the Annaba region is characterized by intense metamorphism, giving rise to the schists of Edough, the marbled limestones of Oued El Aneb, and depressions such as that of Fezzara [11].

Materials and Methods. ***Overview of mining waste from SOPRAM – Annaba.*** The mining wastes generated by the SOPRAM – Annaba Marble Aggregates Company predominantly consist of various types of clays which are the uncovered layer that surrounds the marble. These clays can be classified based on their coloration and properties into several categories: yellow clays, red clays, black clays, and gray clays (Fig. 2). In addition to these clay types, the waste also contains small marble grains, which result from the extraction of marble. They represent considerable reserves which really require valuation.

Fig. 1. Location maps of the study area

The diverse composition of mining waste presents both challenges and opportunities for management and recovery efforts, with each type of clay having distinct properties and potential applications in various industries. The identification of the characteristics and potential uses of these clays is essential to identify ways to reduce the environmental impacts of mining activities.

Sampling. In the process of assessing mining waste for valorization, samples were collected from the land. The coordinates of the sampling locations for the mining waste are displayed in Table 1.

Mineralogical and chemical analysis. A comprehensive characterization study was conducted to determine the mineralogical and chemical composition of our samples. Various qualitative and quantitative analytical methods were employed, including X-ray diffraction (XRD), scanning electron microscopy (SEM), X-ray fluorescence (XRF), atomic absorption spectroscopy (AAS), and infrared (IR) analysis.

Valorization tests. As part of this study, tests were carried out to produce fired bricks using waste materials derived from marble extraction. The briquettes were prepared by mixing clays, followed by molding, drying, and finally firing at various temperatures.

Physico-mechanical tests. The fired clay briquettes were subjected to various physical and mechanical characterization tests, including strength testing, shrinkage, water absorption, and loss on ignition.

Results and discussions. ***XRD analysis.*** The DRX analysis of mining waste from SOPRAM-Annaba is presented in Fig. 3.

X-ray diffraction (XRD) analysis results of the clay samples (grey, yellow, black and red) reveal a relatively homogeneous mineralogical composition, dominated by quartz, observed by characteristic peaks between 20–25 and 35° 2θ, present in all samples. Clay minerals, notably kaolinite and illite, are also identified, with a more marked abundance of kaolinite in the grey clays, and peaks specific to both minerals in the 2θ range close to 12°. Calcite, detected by a distinctive peak at 29° 2θ, is present in the majority of samples and

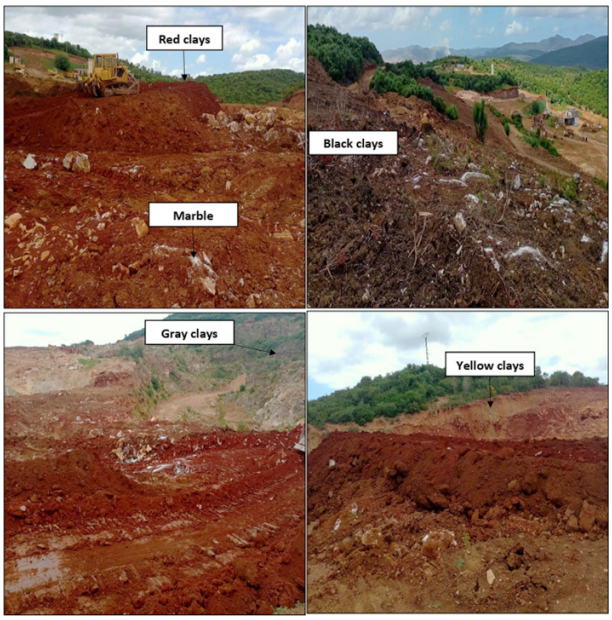

Fig. 2. Mining wastes from Oued El Aneb

Table 1

Sampling points of mining wastes

Sampling point	Latitude	Longitude	Altitude (m)
1	36.8685356	7.4891665	89.4
2	36.8671409	7.4896899	93.5
3	36.8681153	7.488986	97.7
4	36.8679558	7.4890187	98.2
5	36.867318	7.4895237	98.4
6	36.8677917	7.4890435	102.7
7	36.8652588	7.4882075	135
8	36.8652465	7.4882331	135.4
9	36.8652737	7.4881946	135.7

could reflect more alkaline depositional conditions, potentially influenced by local metamorphism. Furthermore, the presence of secondary minerals such as corundum (Al_2O_3) and hematite (Fe_2O_3), particularly in clays containing iron oxides, suggests complex geochemical processes related to high temperature and pressure conditions. These results indicate that the clay samples likely originate from sedimentary deposits undergoing diagenetic processes, with local variations in mineral composition.

The mineralogy of the gray clay shows that the large main peak at approximately 8–10° 2θ likely corresponds to a clay mineral, such as kaolinite or illite, which is commonly observed in clay samples. Kaolinite, for example, has a main peak of around 12–15° 2θ, but variations may occur depending on the degree of hydration and other factors. Quartz is also present, with spectra of around 20–25° 2θ, which is common in clay formations containing sand grains or unaltered silica particles. Peaks at 35, 50, and around 60° 2θ correspond to minerals such as calcite (a prominent peak around 29° 2θ), or corundum (a mineral in the oxide family), indicating mineral contamination or secondary materials present in the rock.

Yellow clays are composed of kaolinite, quartz as the dominant mineral, and calcite and corundum.

This black clay sample has a high concentration of quartz, while calcite and corundum are present in lesser amounts.

This red clay sample is primarily quartz, with lesser amounts of corundum and calcite.

SEM analysis. The subsequent figure (Fig. 4) illustrates the SEM analysis of representative samples taken from the site.

The SEM observation and EDS diffractogram of gray, yellow, black, and red clays revealed the microscopic composition of various elements such as Ca, O, Si, Ti, Fe, Mg, and Al, along with other minor elements.

The significant presence of oxygen is attributed to its involvement in the composition of CaO, SiO_2, Al_2O_3, Fe_3O_4, MgO, and other oxides.

XRF analysis. The results of the quantitative characterization of SOPRAM mining waste by XRF are mentioned in Table 3.

The Gray clays exhibit a typical silicate clay composition, dominated by SiO_2 (38.09 %) and Al_2O_3

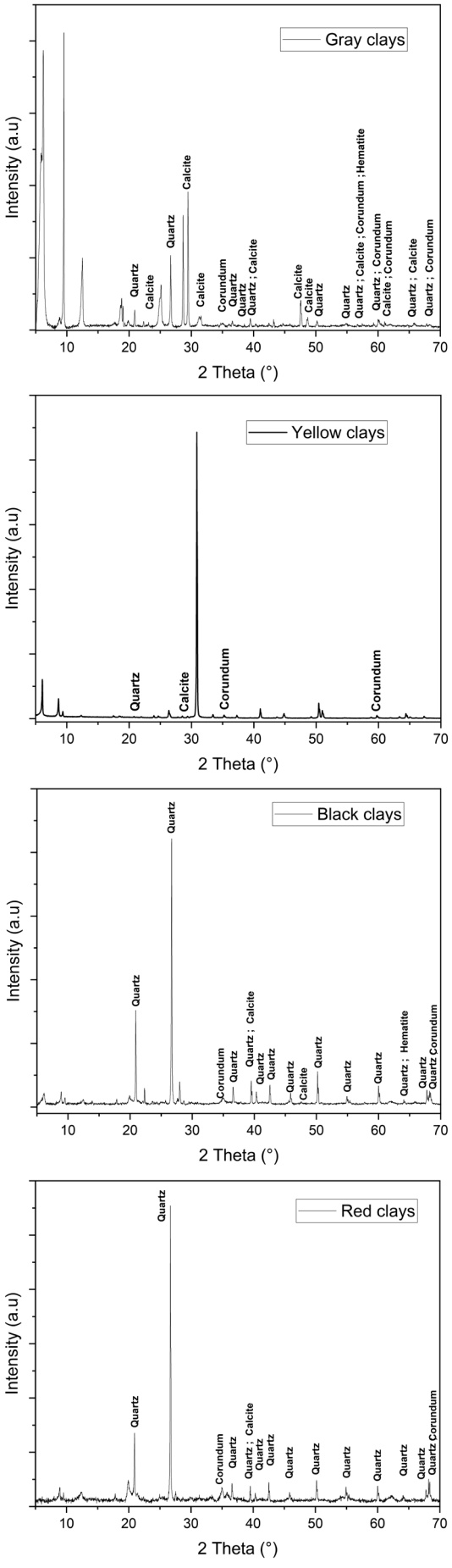

Fig. 3. XRD analysis of Gray clays; Yellow clays; Black clays; Red clays

(14.22 %), along with carbonates (CaO, MgO) and iron oxides (Fe_2O_3), which explains their gray color. Their high loss on ignition (LOI) of 16.94 % reflects the presence of water bound to clay minerals or organic matter, making them suitable candidates for fired clay products (bricks and/or ceramics, etc.), provided that carbonate content is carefully controlled.

Chemical analysis by AAS. The analysis by Atomic Absorption Spectroscopy (AAS) of the mining waste from SOPRAM – Annaba gave these results (Table 2).

Infrared analysis. The IR analysis of the SOPRAM-Annaba mine tailings are given in Fig. 5.

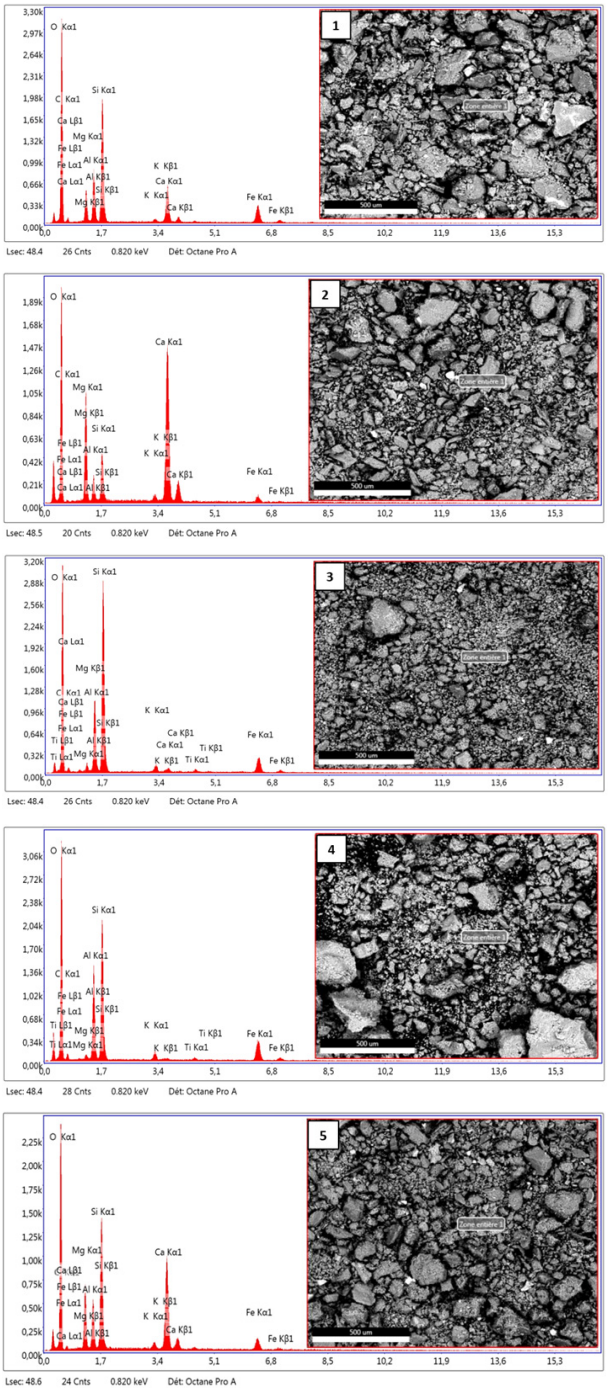

Fig. 4. SEM observation of:

1 – gray clays; 2 – yellow clays; 3 – black clays; 4 – red clay; 5 – mining wastes

The infrared absorption bands are directly related to the chemical elements Zn, Ni, Pb, Cu, Cd, Mn present in clay, although infrared spectroscopy is generally less specific for directly identifying elements than for characterizing functional groups or bonds in mineral structures.

Here is what can be deduced for each band with these elements:

3,691 and 3,621 cm^{-1}: These bands generally correspond to O—H vibrations and are typical of hydroxyl groups. Metal ions such as Fe, Mn or Zn can bond with hydroxyl groups in clays, which is often the case in hydrated minerals containing transition metals.

1,117 cm^{-1}: This band is close to those attributed to asymmetric vibrations of M—O bonds (M being a transition metal such as Zn, Fe, Ni, or Mn), particularly in oxide or silicate structures.

997 cm^{-1}: This band could be associated with M—O—M vibrations where M is a metal such as Zn, Ni, or Mn in mixed oxide structures.

905 cm^{-1}: This band may be due to O—H vibrations associated with metals in a hydrated environment, for example Fe(OH), $Ni(OH)_2$ or $Mn(OH)_2$.

749 and 678 cm^{-1}: These bands are located in the region of M—O deformation vibrations. Clays containing elements such as Fe, Ni, and Zn in oxide or silicate environments often show bands in this range.

520 cm^{-1}: This band could be attributed to Fe—O deformation vibrations, which can be found in iron oxides (such as Fe_2O_3 or Fe_3O_4) or in structures containing iron as an impurity in the clay.

In summary, although infrared spectroscopy is not specifically quantitative for each metal, the observed bands can be associated with M—O or M—OH bond vibrations in oxide or hydroxide structures that incorporate the metals Zn, Ni, Pb, Cu, Cd, Mn, and Fe in the clay. A complementary technique, such as X-ray absorption spectroscopy, would be more suitable for the specific analysis of each metal.

Table 2

Analysis by AAS of the mining waste

Sample (ppm)	Zn	Ni	Pb	Cu	Cd
Yellow clays	<0.01	25.94	<0.01	11.32	<0.01
Black clays	<0.01	25.00	<0.01	26.33	10.01
Red clays	<0.01	48.13	<0.01	47.30	4.05
Gray clays	42.12	47.58	<0.01	24.45	<0.01

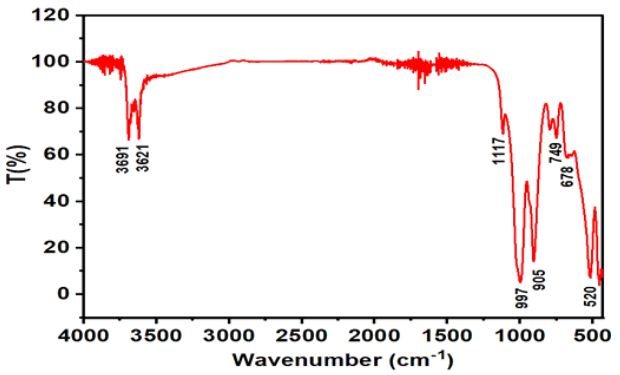

Fig. 5. IR analysis of the mining wastes

For the elements Na, Mg, and K in clays, here is how the infrared bands could correspond:

3,691 and 3,621 cm^{-1}: These bands are related to the O–H valence vibrations, often observed in hydrated minerals. Na^+, K^+, and Mg^{2+} ions can be present in clays as exchangeable ions or associated with hydroxyl groups in the structure of clay minerals such as smectites or illites.

1,117 cm^{-1}: This band could be due to the vibrations of the Si—O or M—O bonds (M for a metal) modified by the presence of Na, K, or Mg ions. These ions do not participate directly in the covalent bonds with oxygen in clays but can influence the structure and the Si—O vibrations.

905 cm^{-1}: This band is in the region of O—H vibrations and may indicate hydroxyl groups associated with Mg^{2+}, which is often present as hydroxides or in clays containing brucite ($Mg(OH)_2$) layers.

997 cm^{-1}: As with 1,117 cm^{-1}, this band may correspond to Si—O bond vibrations in which Na^+ or K^+ are present as compensating ions in the structure of silicate clays.

749 and 678 cm^{-1}: These bands are in the region of Si—O or M—O—Si bond deformation vibrations. The K^+ and Na^+ ions may modify the structure vibrations by acting as compensating ions for the charge in the clay layers.

520 cm^{-1}: This band could be associated with Mg—O deformation vibrations, especially in clay structures where magnesium is present as silicate or oxide.

Then, Na and K elements are generally present as exchangeable ions in clays and only slightly affect M—O band by electrostatic interaction. Mg is often integrated directly into the structure of clay minerals, further influencing the observed bands, especially those of O—H and M—O vibrations in hydrated minerals.

Results of Characterization of manufactured terracotta briquettes. The making of briquettes from SOPRAM-Annaba mining waste under different temperatures are tested, the physical and technological properties of the samples are illustrated in Table 4.

The absorption values [14] were calculated from the formula

$$Absorption = \frac{Wet\ weight - Dry\ weight}{Dry\ weight} \cdot 100.$$

Note that the resistance values [15] were calculated using formula

$$Rupture\ Modulus = \frac{3}{2}\frac{Force \cdot Distance\ between\ two\ Supports}{Width \cdot (Thickness\ of\ piece)^2}.$$

According to the Standard [16], the firing temperatures significantly affect the mechanical resistance of construction materials such as special concretes and bricks (Fig. 6). Here is a summary of the findings based on different firing temperatures:

At 900 °C: the measured compressive strength is 66.42 kg/cm^2 (equivalent to 6.51 MPa, classified as RC 60). This level of strength indicates suitability for construction materials used in applications requiring high durability and resistance, such as: high-rise structures (skyscrapers), bridges, viaducts, and tunnels, foundations and slabs subjected to heavy loads, environments where concrete must withstand severe climatic conditions.

At 1,000 °C: the resistance drops to 39.38 kg/cm^2 (approximately 3.82 MPa, classified as RC 28). This strength range is typical for bricks used in general construction, prefabricated construction elements, applications requiring resistance to humidity and adverse weather conditions, sound insulation properties.

At 1,100 °C: the strength rises to 89.38 kg/cm^2 (equivalent to 8.72 MPa, classified as RC 80). This indicates a material suitable for industrial structures, high-rise buildings.

At 1,200 °C: the resistance increases to 187.03 kg/cm^2 (equivalent to 18.33 MPa, classified as RC 180). This indicates a category of bricks suitable for use in advanced and resilient structures, particularly under extreme conditions.

At 1,250 °C: the measured resistance reaches 145 kg/cm^2 (equivalent to 14.21 MPa); however, it is important to note that at this temperature, the sample is destroyed by the high heat, indicating the limitations of material stability at extreme temperatures.

Conclusion. Based on the principles of sustainable development and the recognition that the environment is a vital asset that must be preserved, a characterization

Table 3

Chemical composition of SOPRAM mining wastes

Sample	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	MNO	TIO_2	K_2O	Na_2O	SO_3	P_2O_5	LOI
Gray clays	38.09	14.22	4.36	9.1	15.06	<0.05	0.74	1.34	<0.05	<0.05	0.16	16.94
Yellow clays	9.55	2.57	0.73	28.68	17.16	<0.05	0.1	0.69	<0.05	<0.05	0.05	40.56

Table 4

Physical and technological properties of briquettes

Temperature, °C	Cooking shrinkage, %	Total shrinkage, %	Loss on ignition, %	Porosity, %	Density, g/cm^3	Absorption, %	Color
900	0.47	11.18	3.22	18.62	1.80	15.47	Brick Red
1,000	1.18	12.60	4.15	18.16	1.86	14.25	Brick Red
1,100	1.44	13.12	4.19	14.41	1.95	10.88	Brick Red
1,200	2.63	15.50	4.59	2.33	2.20	1.44	Dark grayish red
1,250	Fusion	Fusion	Fusion	18.62	1.80	15.47	Merger

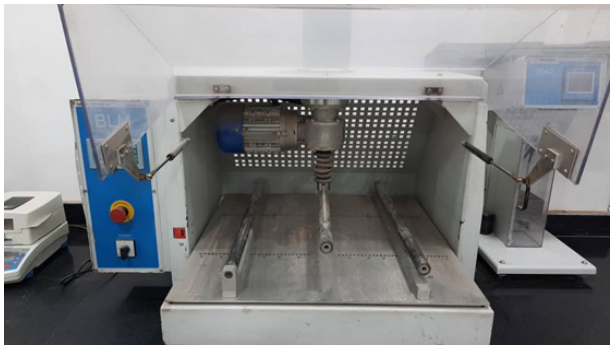

Fig. 6. Bending tests on briquettes

Table 5

Mechanical resistance properties of the briquettes produced

Class, C°	Force	Strength distance between supports, cm	Resistance, kg/cm^2
900	23.25	6	66.42
1,000	12.23	6	39.38
1,100	28.9	6	89.38
1,200	50.5	6	187.03
1,250	45.32	6	145

study was conducted on the mining waste from the Marble Grain Production Company in Oued El Aneb (SOPRAM) – Annaba.

Through various analytical techniques, including Scanning Electron Microscopy (SEM), X-ray Diffraction (XRD), Infrared Spectroscopy (IR), X-ray Fluorescence (XRF), and Atomic Absorption Spectroscopy (AAS), we determined the geological and mineralogical composition of the mining waste.

The various analyses carried out on our samples indicate that these are mainly composed of quartz, carbonates (calcite, dolomite) and potentially other minerals such as gyps. This composition suggests a sedimentary origin and demonstrates that waste from marble extraction at the SOPRAM-Annaba quarry can be used in the manufacture of construction materials, such as terracotta, including bricks and ceramics.

Furthermore, five samples were studied at different firing temperatures (900; 1,000; 1,100; 1,200 and 1,250 °C) to produce fired clay briquettes. These briquettes were obtained by mixing clayey waste materials derived from the marble extraction process at SOPRAM-Annaba quarry.

The manufacturing tests and characterization analyses of the obtained products revealed that the incorporation of mining waste into fired clay production should be carried out at a temperature below 1,250 °C. Beyond this temperature, the product undergoes degradation due to excessive fusion of the clay.

The fired clay materials produced from these formulations exhibit good performance, particularly in terms of firing shrinkage (ranging from 0.47 to 1.44 %), water absorption (between 10.87 and 15.47 %), and flexural strength (ranging from 3.82 to 8.72 MPa).

The technical properties of the terracotta produced demonstrate promising characteristics and good overall performance. The implementation of this approach requires a minimal investment to use the important reserves of clay found in mining waste, positioning the company as a responsible entity that not only produces the product, but also manages its mining waste effectively. This initiative strengthens and diversifies the company's financial resources while contributing positively to the preservation of the environment.

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Валоризація глиновмісних відходів видобутку з мармурового кар'єру Уед Ель Анеб для виробництва теракоти

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Мета. Валоризація відходів видобутку в мармуровому кар'єрі для виробництві теракоти, що відповідає промисловим вимогам. У цьому контексті наша робота була зосереджена на використанні глиновмісних відходів з мармурового кар'єру Уед Ель Анеб із виготовлення теракотової цегли. Принцип полягає у використанні суміші різних типів глин (жовтої, червоної, чорної й сірої), отриманих під час видобутку мармуру, для виробництва теракоти за різних температур випалювання.

Методика. Після відбору проб і механічної підготовки зразків у лабораторних умовах, глиновмісні відходи були охарактеризовані за допомогою кількох аналітичних методів, зокрема: рентгенівської дифракції (XRD), інфрачервоної спектроскопії (IR), атомно-абсорбційної спектроскопії (AAS), сканувальної електронної мікроскопії в поєднанні з енергодисперсійним аналізом (SEM-EDS), рентгенівської флуоресценції (XRF). Для виготовлення теракотової цегли були проведені випробування за різних температур випалювання.

Результати. Використання відходів мармурового видобутку є інноваційним підходом до відновлення й валоризації відходів видобутку корисних копалин. Проведені випробування із виготовлення теракотової цегли із цих матеріалів дали обнадійливі результати. Випалені глинисті матеріали, отримані за запропонованими рецептурами, демонструють високі експлуатаційні характеристики, зокрема з точки зору усадки при випалюванні (у межах від 0,47 до 1,44 %), водопоглинання (від 10,87 до 15,47 %) й міцності на вигин (від 3,82 до 8,72 МПа).

Наукова новизна. Результати дослідження використання відходів гірничодобувної промисловості з мармурового кар'єру Уед Ель Анеб за різних температур випалювання дозволили обгрунтувати ефективність виробництва високоякісної теракотової цегли, що відповідає промисловим вимогам.

Практична значимість. Технічні властивості (фізичні й механічні) теракотових матеріалів, вироблених із сировини відходів, зокрема усадка при випалюванні, водопоглинання й міцність на вигин, демонструють ефективну валоризацію глиновмісних відходів видобутку мармуру в Уед Ель Анеб.

Ключові слова: *розробка родовищ, глиновмісні відходи, SARL SOPRAM, Аннаба, Північно-Східний Алжир*

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