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Reuse of Lead Mine Processing Tailings for Ceramic Tiles Production: Zeïda Mine Case Study, Morocco

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Abstract

Unrehabilitated abandoned mining sites pose significant risks to the surrounding ecosystems through toxic releases. This study aims to assess the feasibility of converting mine tailings from the processing of lead ore from the abandoned Zeïda mine (Upper Moulouya, Morocco) as an alternative raw material for the production of ceramic tiles by dry pressing. The raw materials were characterized by assessing the chemical composition (lead mine processing tailings, clays, and industrial sodium feldspar), as well as the mineralogical properties and particle size distribution of the lead mine processing tailings. Various formulations were developed by incorporating proportions of mine tailings ranging from 0 to 50%, combined with clays and sodium feldspar. Dry-pressed ceramic materials were characterized for their mechanical properties (modulus of rupture), physical properties (water absorption, density, shrinkage, weight loss on ignition and Surface Morphology). Principal Component Analysis (PCA) was applied to identify relationships among the variables related to the properties of the fired manufactured materials. The main properties were evaluated against the industrial standards (ISO 10545 and ISO 13006). The fired products formulated with 10–20% lead mine processing tailings achieved compliance with group BIIa of ISO 13006, demonstrating a modulus of rupture of 22–25 N/mm^2 and water absorption of 3–4%, along with favourable other physical properties. This study confirms the feasibility of reusing Zeïda lead mine processing tailings for ceramic tile production, thereby providing a sustainable solution that offers both environmental remediation and economic advantages.

Keywords: Abandoned Mines, Ceramic Tiles, Mine Tailings, Mine Waste Management, Zeïda Mine.

Introduction

Morocco's mining industry substantially impacts the nation's economy through exports and gross domestic product (GDP). As a leading global supplier of phosphate and a major barite and silver miner across Africa, minerals represent the economic backbone (1). The country is one of the largest phosphate exporters and a leading producer of barite and silver in Africa (2). Morocco has many large mining areas rich in deposits that have been exploited for many decades (3). The number of abandoned mines in Morocco has been reported to exceed 200 (4). Abandoned mines in Morocco are of great concern because of their potential impacts on ecological biodiversity and human health (5). Studies have shown that abandoned mines in Morocco, notably the Touissit and Sidi-Boubker mines and the Zeïda mine, contain high levels of heavy metals in their tailings and the surrounding soils (6). The Zeïda mine, in the semi-arid region of Upper Moulouya of morocco, has a special geological and environmental context. Its semi-arid climate and alkaline geological conditions prevent the formation of acid mine drainage, an issue that has been a problem at many abandoned mining sites (7). Leaching tests on mine tailings have indicated neutral pH values and low levels of dissolved metals, a phenomenon known as neutral mine drainage (NMD) (8). The Zeïda mine is an abandoned lead (Pb) mine with ore reserves estimated at 10 million tonnes to be mined by open pit, with a processing capacity of 1.4 Mt/year through a washing plant (9). Production of 40% to 70% lead concentrate reached 630,172 tons after 13 years of operation (1972, 1985) (10). This operation has generated lead mine processing tailings (LMPT), which are defined as a set of residual substances generated during the extraction of minerals, metals and other valuable resources from ores using ore processing methods (11).These tailings contain trace metal elements (TMEs) in the Zeïda mine which are

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lead (Pb), zinc (Zn), copper (Cu), cadmium (Cd), arsenic (As) and barium (Ba), with high average contents compare with normal average contents in the earth's crust (10). At Zeïda the deposits consist of stratiform formations, and the paragenetic association is composed of well-crystallized cerussite [PbCO3] and galena [PbS], with accessory minerals such as chalcopyrite [CuFeS2] and, pyrite [FeS2], as well as large quantities of pink barite [BaSO4] and rare small yellow cubes of fluorite [CaF_2] (7). Substantial amounts of silicate minerals are present in the Zeïda mine, whereas the presence of carbonate minerals (such as cerussite) and clay minerals is minimal. Significant quantities of Ba were found, whereas sulfides were absent (12). The disposal of mine tailings at the Zeïda abandoned mine has been addressed using methods aimed at mitigating environmental and health risks. One approach involves the reuse of low-sulfide base-metal tailings as construction materials, such as surface finishing mortars, which have shown potential for sustainable tailing management (8). Additionally, phytostabilization has been explored as a method to limit the transfer of heavy metals from tailings to the environment. This technique utilizes native plants, such as Retama sphaerocarpa, which can stabilize metals and reduce their mobility, offering a promising solution for tailings management in the Zeïda mining area (13). This study presents a simple, cost-effective, and environmentally friendly strategy to assess the feasibility of valorizing mine tailings from lead ore processing at the Zeïda mine for the manufacture of ceramic tiles. Our objective was to replace the raw materials traditionally used in ceramic tile production with an optimised composition that incorporates LMPT. This new composition should enable the production of tiles that meet the industrial standards (ISO 10545 and ISO 13006) by exhibiting optimal physical and mechanical properties. Consequently, this production process will contribute to the conservation of non-renewable natural resources. Several steps were taken to achieve this objective. The first involved sampling and preparing the raw materials and tailings for experimentation. This was followed by the characterizing of the physical, chemical, and mineralogical properties of the raw materials. The next step was the formulation and fabrication of ceramic tiles with different compositions, incorporating the LMPT. The manufactured ceramic materials were thoroughly characterized to evaluate their physical, mechanical, and microstructural properties, focusing on essential parameters such as the modulus of rupture, water absorption, apparent relative density, post-firing bulk density, shrinkage, and weight loss on ignition. These properties were studied to ensure compliance with the international ceramic tile standards. Finally, the economic benefits of reusing mine tailings in ceramic tile manufacturing are assessed, demonstrating the potential for cost savings and resource optimization.

Materials and Method

Presentation of the Study Area

From 1972 to 1985, the 300 km^2 Zeïda mining centre on the banks and course of the Oued Moulouya was a site of intense lead ore mining (Figure 1). In Zeïda, mining is conducted in open-pit quarries. A processing plant with a capacity of 1, 400,000 tons per year is responsible for enriching the ore by crushing, grinding, flotation, and filtration. After 13 years of operation, the balance sheet is as follows: surface facilities (workshop, laundry, abandoned equipment), production of 630,172 tons of lead concentrate with a grade of 40-70%, approximately 12 million tons of processing rejects, and approximately 70 million tons of overburden waste rock, both stored on the banks of the Oued Moulouya (7). The LMPT being studied for valorization in this study consists of residues from tailings deposits generated during the processing and enrichment of lead ore near the Zeïda mining center, located in the northeastern region of Morocco, within the upper Moulouya watershed (Figure 2).

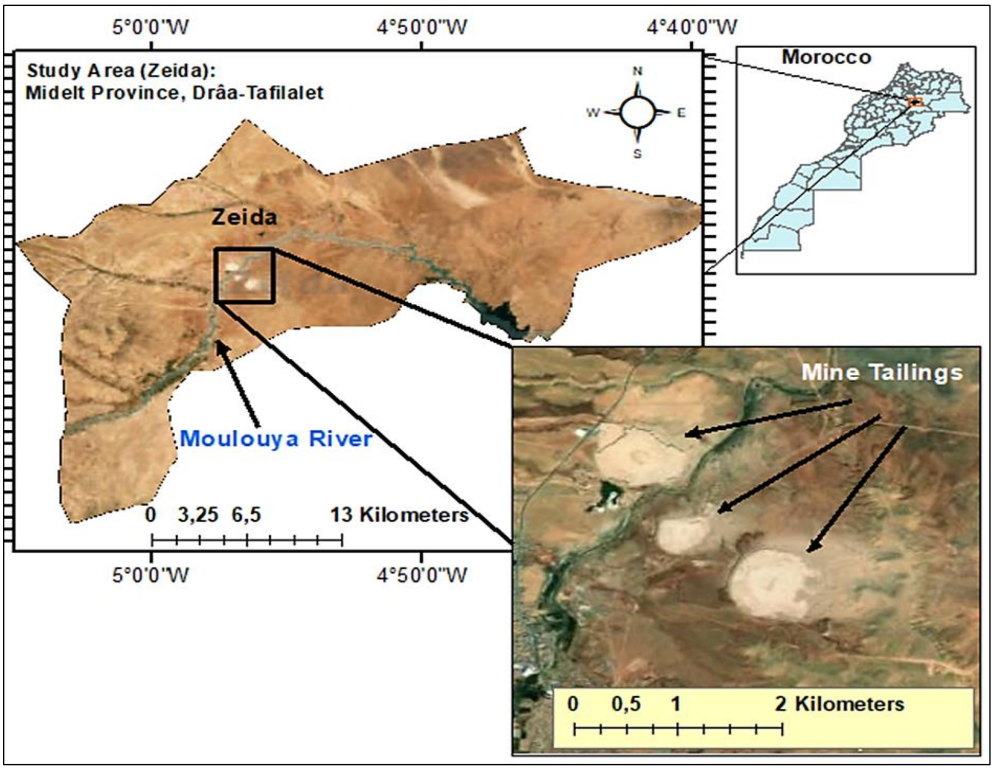

Figure 1: Geographical Location of the Lead Mine Processing Tailings (LMPT) Study Area

Figure 2: Lead Mine Processing Tailings (LMPT) from Zeïda Mining Centre, Upper Moulouya, Morocco

Sampling and Mixture Preparation

The composite LMPT sample used in this study was collected from tailings deposits of the Zeïda mine. Five subsamples were used to create the composite sample. A manual plastic scoop was used to collect the samples from various locations at a depth of 40 cm. Large quantities of natural clay (C1 and C2) and sodium feldspar (SF) were used to produce ceramic materials (Figure 3). More specifically, clays C1 and C2, sourced from the Berrechid quarry in Morocco, represent two distinct clay varieties. C1 is a high-quality clay used in the formulation of floor and wall tiles, serving as the primary component of the skeleton of pressed tiles, whereas SF is a sodium feldspar that is also utilised as a raw material in the ceramic tile industry.

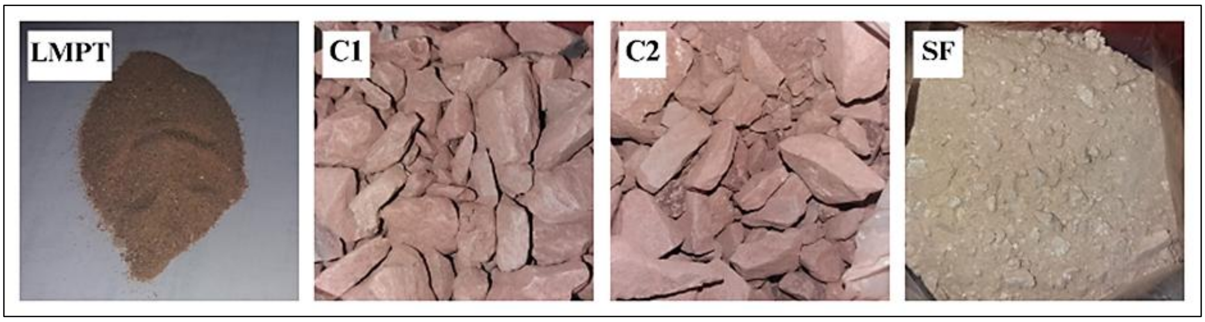

Figure 3: The Raw Material Used to Produce Ceramic Materials. LMPT : Zeïda Lead Mine Processing Tailings, C1 and C2: Clays from the Berrechid Quarry, SF: Sodium Feldspar

Seven distinct formulations were prepared and examined (Table 1). LMPT was introduced into formulations F1 to F6 to substitute C2 clay, with percentages ranging from 5% to 50% for formulations F1 to F4. In formulations F5 and F6, the LMPT replaced C2 clay by no more than 15%, with the addition of SF as a raw material, 8% in F5, and 3% in F6. Owing to its relative abundance and good quality, C1 clay was introduced into formulations F1 to F6 in proportions ranging from 40% to 42%. Ultimately, F0 formulation consisted entirely of LMPT with no other raw materials.

Table 1: Prepared Formulations of Ceramic Materials

Formulations	C1 (%)	C2 (%)	LMPT (%)	SF (%)
F0	0	0	100	-
F1	40	55	5	-
F2	40	50	10	-
F3	40	40	20	-
F4	40	10	50	-
F5	42	40	10	8
F6	42	40	15	3

Characterization Techniques Used For Raw Materials

The raw materials were subjected to a grinding process using an electric grinder commonly used in the ceramic tile industry. The grinding involved two stages: an initial wet grinding for 10 min to produce a slurry, followed by drying the slurry in a furnace at 110°C for 4 h under controlled conditions. Once dried, the material was subjected to dry grinding to obtain a fine powder suitable for chemical and mineralogical characterization. The raw materials were characterized using various analytical techniques. The fine powders obtained after grinding were subjected to chemical and mineralogical analyses. The chemical compositions of the LMPT, C1, C2, and SF were determined via X-ray fluorescence (XRF) spectroscopy using a wavelength dispersive spectrometer (Axios model) for quantitative elemental analysis. Mineralogical analysis of the LMPT was performed using X-ray diffraction (XRD) with an EMPYREAN device with a reflection-transmission spinner setup (MALVERN PANALYTICAL). Diffraction patterns were created using CuKα radiation, and phase identification was performed using the ICDD database. The scans covered a range from 2θ = 5.0328° to 89.9748°, with a step size of 0.0660° and a total scan time of 113.2 seconds in the continuous mode for high-resolution data acquisition. The particle size distribution of the LMPT was assessed using a combination of methods. Dry sieving was employed for the sand fraction, whereas sedimentation techniques were used for the silt and clay fractions, adhering to the ISO 17892-4 (2018) standards. This method allowed for a thorough analysis of the particle size distribution of LMPT.

Ceramic Materials Manufacturing Process

A mixture of 300 g of the total mass from each formulation was ground in a 500 gram capacity cylindrical ceramic ball mill for 10 min, using 80 ml of water and 1% deflocculant [Fluicer: a mineral dispersant with a density of 1.51-1.54 g/cm^3 at 20 °C, effective in reducing the viscosity of the slurry during discontinuous grinding]. The resulting slurry was placed in an oven for 4 h at 110°C until completely dried. The obtained product was ground into a fine powder, sieved, and moistened via spraying. The wet powder obtained was pressed using a manual hydraulic pellet press in a rigid mould [165 bars] to obtain test specimens [pellets]. They were oven-dried at 110°C for 40 min, resulting in a residual moisture content of less than 1% (the optimum condition to avoid product cracking during firing). The final firing phase was conducted in an industrial rotary kiln for 44 min, during which the temperature gradually reached 1,200°C. The ceramic materials produced through this process were unglazed, indicating that no glass-like coating was applied to their surfaces after the firing phase. As a result, these materials retain a natural matte finish, which emphasizes their raw texture and earthy appearance.

Characterization of Manufactured Ceramic Materials

Various essential tests have been conducted to analyse the mechanical and physical properties of ceramic materials. Initially, the three-point flexural strength (mechanical Bending Strength or modulus of rupture) was evaluated using a Gabbrielli Crometro CR/650 R160 machine. Rectangular plate samples were manufactured with dimensions of 151.7 mm x 50.30 mm x 7.21 mm (as indicated in Figure 4). Seven tests were conducted and averaged for each formulation, in compliance with the ISO 10545-4 standard (14).

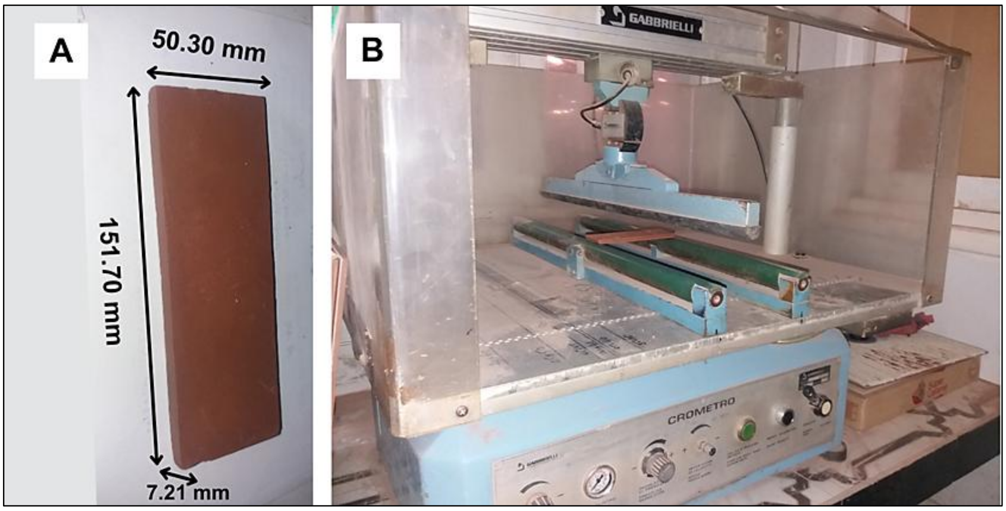

Figure 4: Measuring Mechanical Bending Strength (Modulus of Rupture): (A) Rectangular Plate and (B) Machine for Measuring Modulus of Rupture

The fire shrinkage was determined by measuring the diameters of the dry cylindrical samples (Figure 5) before and after firing. Five tests were performed and averaged for each formulation using an industrial rotary kiln, in compliance with the ISO 10545-3 standard (15). Water absorption was assessed by measuring the weight difference in dry cylindrical samples before and after immersion in boiling water at 100°C for 4 h. Five tests were performed and averaged for each formulation following the ISO 10545-3 standard (15). The apparent relative density was determined by calculating the ratio between the dry fired mass and the saturated suspended mass of the dry fired cylindrical samples. Five tests were conducted for each formulation in accordance with ISO 10545-3 standard (15). The bulk density was measured as the ratio of the dry fired mass to the external volume of the dry fired cylindrical samples, including their pores. Five tests were conducted for each formulation according to the ISO 10545-3 standard (15). Surface analysis was performed on the dry fired samples using scanning electron microscopy (SEM) to determine the compactness and cohesiveness of the grains. Microscopic observations were conducted using a QUATTRO S-FEG-Thermofisher Scientific Instrument. Finally, statistical analysis using Principal Component Analysis (PCA) was conducted with OriginPro software version 9.9.0.225 to identify correlations among the different properties of ceramic materials.

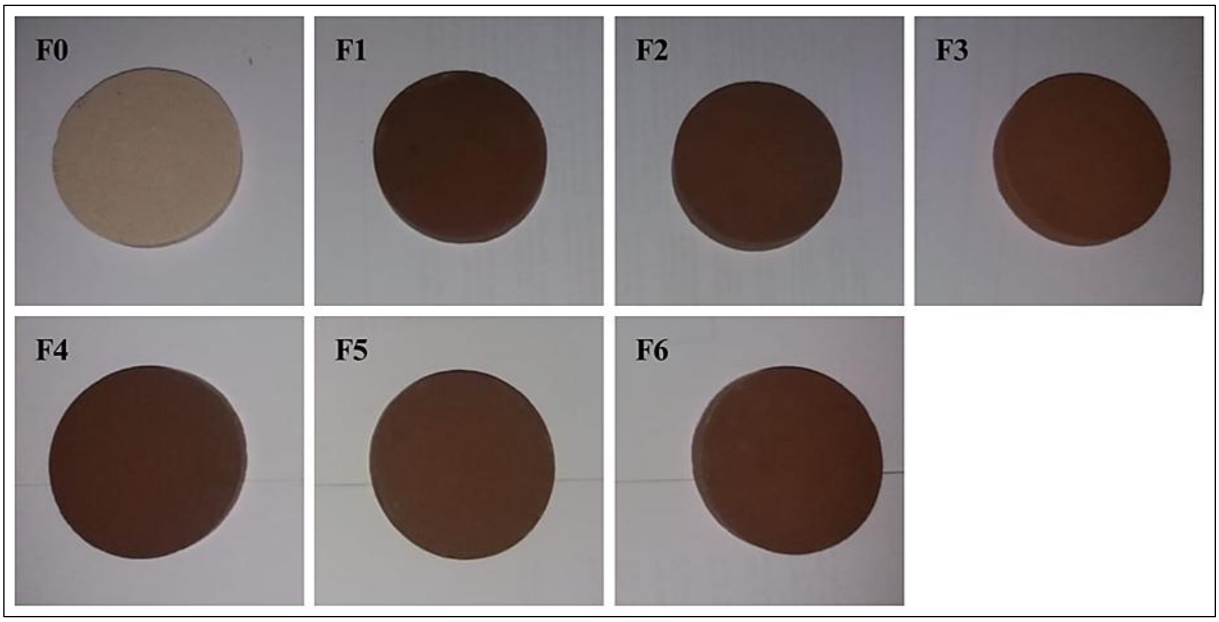

Figure 5: Ceramic Materials Produced from Formulations F0 to F6

Results and Discussion

Chemical Analysis Results

The results of the chemical characterization of the LMPT, C1, C2, and SF, as analyzed by XRF spectrometry, are presented in Table 2. This chemical analysis of LMPT showed a high silica (SiO_2) content of 68.92%, while the alumina (Al_2O_3) content was relatively low at 12.03%. The use of alumina in ceramics reduces shrinkage during drying and firing and contributes to the creation of refractory materials at high temperatures. For optimal ceramic production, it is essential to combine these tailings with alumina-rich clays to achieve a well-balanced formulation that enhances the properties of the final ceramic product (16,17). The presence of potassium oxide K_2O (3.88%) indicated the presence of feldspar in the mining waste. K_2O and Na_2O are common oxides found in feldspars and are known for their ability to induce the melting process (18). They play a significant role in ceramic tiles as fluxes, lowering the sintering temperature, and promoting the formation of a glassy phase (19). LMPT contain barium oxide (BaO) (4.59%), which acts as an effective flux owing to the formation of a eutectic in both SiO_2-BaO-Na_2O and SiO_2–BaO-K_2O systems (20). These eutectic processes promote the melting and fusion of ceramic materials at low temperatures, thereby optimising the sintering process. The presence of calcium oxide (CaO) (1.52%) in the LMPT supports the presence of plagioclase minerals (12,21). Additionally, the presence of a small amount of iron(III) oxide (Fe_2O_3) (0.53%), which acts as a fluxing agent, is responsible for the brown or red colour that occurs in the products after firing when present in sufficient quantities (21,22).

Table 2: Chemical Composition (wt %) by X-ray Fluorescence of Clays C1, C2, Lead Mine Processing Tailings (LMPT), and Sodium Feldspar (SF)

	SiO_2	Al_2O_3	K_2O	Na_2O	Fe_2O_3	CaO	BaO	TiO_2	SO_3	F	PbO	P. F*
LMPT	68.92	12.03	3.88	0.88	0.58	1.52	4.59	0.11	2.19	1.74	0.46	2.21
C1	54.42	27.27	2.07	1.27	4.38	0.31	0.05	0.72	0.03	-	-	7.20
C2	57.50	23.78	2.76	1.26	4.32	1.08	0.03	0.59	0.02	-	-	6.16
SF	67.36	18.22	0.09	10.17	0.53	0.20	-	0.10	0.02	-	-	2.12

* Loss on ignition at 1140 °C

Mineralogical Analysis Results

The mineralogical analysis by XRD, as shown in the graph in in Figure 6, demonstrates that the mineralogy of the LMPT comprises quartz SiO_2 (reference code 01-079-1910 based on the ICDD database), anorthoclase $(Na,K)(Si_3Al)O_8$ (00-009-0478), barite $BaSO_4$ (00-024-0020), fluorite CaF_2 (01-089-4794) and muscovite $(KAl_3Si_3O_{10}(OH)_2$ (01-084-1306). This mineralogical analysis illustrates a significant mineral diversity in these mine tailings, presenting interesting views for their possible valorization and environmental management. Quartz (SiO_2) is the dominant mineral in the sample. Its substantial presence is beneficial for ceramic tile production, because it enhances the structure and mechanical strength of the final product (23–25). Anorthoclase ($(Na,K)(Si_3Al)O_8$), an alkali feldspar, was also identified. This mineral can act as a natural flux, promoting vitrification during firing, thus aiding in the densification of the ceramic material. The presence of barite ($BaSO_4$) is particularly noteworthy. As mentioned above, barium oxide (BaO) can act as an effective flux in ceramic systems, potentially lowering the melting temperature and improving the densification of the material.

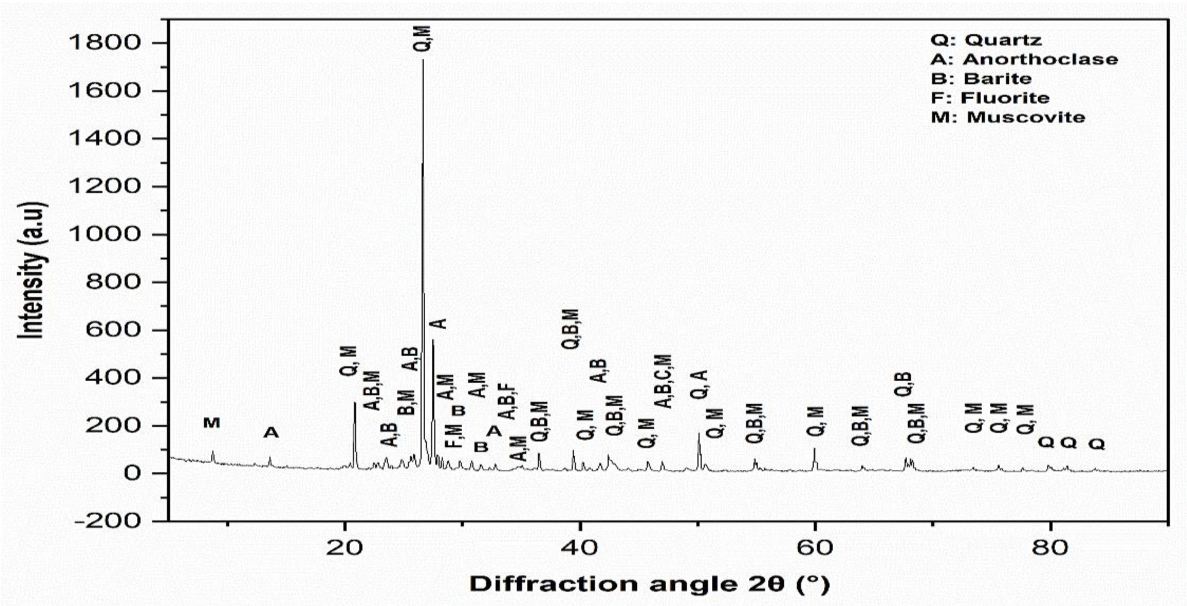

Figure 6: X-ray Diffraction Phases Measured from Lead Mine Processing Tailings (LMPT)

Finally, muscovite ($KAl_3Si_3O_{10}(OH)_2$), a clay mineral, was detected. Although likely to be present in smaller quantities, they can contribute to the plasticity of the mixture and aid in the formation of crystalline phases during firing (26,27).

Particle Size Distribution Results

Particle size analysis allows the determination of various material classes without considering their chemical compositions. Table 3 presents the particle size distributions of the LMPT.

Table 3: Particle Size Distribution (%) of Lead Mine Processing Tailings (LMPT)

Clay (< 2 µm)	**Silt** (2 µm - 50 µm)	**Very fine sands** (50 µm - 100 µm)	**Fine sands** (100 µm - 250 µm)	**Coarse sand** (250 µm - 2000 µm)
27.276	57.586	12.436	2.702	0

The particle size distribution of the LMPT showed a large amount of silt and clay. Most of the LMPT consisted of fine particles, with 57.586% silt and 27.276% clay for 84.862% of the sample. This particle size distribution, which is dominated by fine particles, supports cohesion and contributes to the strength and moldability of ceramic formulation mixtures (28,29). The 15.138% sand fraction is mainly due to the presence of quartz in the LMPT (30). The sand fraction acts as a natural degreasing agent, reducing shrinkage during drying and increasing the physical strength of the final product. Additionally, the absence of coarse sand (0%) helps achieve the fine texture required for ceramic tiles (31,32).

Physical and Mechanical Properties Results

The ceramic tiles sector is highly complex, with a well-established correlation between the raw material composition and quality of the final ceramic products (16,33,34). Owing to this complexity. A series of formulations (F0 to F6) was developed by progressively incorporating LMPT as raw materials to find formulations that meet international industrial standards. The physical and mechanical properties of the final products were evaluated as a part of the quality control procedures (Table 4 and Figure 7). The materials produced with high concentrations of LMPT (F0 and F4) exhibited low shrinkage. Conversely, formulations F2, F3, and F5 displayed markedly higher shrinkage rates (7.72 %, 7.43 %, and 7.57 %, respectively). This increased shrinkage was likely due to the elevated plastic clay content (35). Clays contain phases, such as illite and kaolinite, which are responsible for the plasticity required during shaping (alteration of form depending on the amount of water added to the formulation) and mechanical strength in the green state (36). Interestingly, despite these fluctuations, all formulations remained within the industrially acceptable shrinkage range of 2.3-7.9% for firing temperatures between 1100°C and 1250°C (19,37). The weight loss on ignition values varied considerably across the formulations owing to compositional differences. Formulations F0 and F4, characterised by a high LMPT content, showed minimal weight loss on ignition of 2.53% and 3.90%, respectively. This low weight loss on ignition can be attributed to the reduced organic matter and clay content in the LMPT-rich mixtures. Indeed the process of mass loss include the release of carbon dioxide (CO_2) during carbonate decomposition, dehydroxylation of clay minerals, and degradation of organic matter (38). Conversely, formulations F1, F2, F3, F5, and F6, which incorporated high amounts of clay, exhibited substantially higher weight loss on ignition (ranging from 4.81% to 6.00%), as confirmed in the literature (39). The apparent relative density measurements revealed notable variations across formulations, providing insights into their structural characteristics. Formulations F2, F3, F5, and F6 demonstrated higher densities, ranging from 2.33 to 2.42 g/cm^3, in comparison to F0, F1, and F4. This disparity suggests that the latter group represents suboptimal formulations, causing the ceramic body to become under-compacted. The bulk density measurements showed a correlation with the apparent relative density. This correlation is consistent with the established relationship between the two density parameters (40). Formulations F2, F3, F5, and F6 exhibited elevated bulk densities, ranging from 2.22 to 2.28 g/cm^3, compared to formulations F0, F1, and F4. It is important to note that the acceptable bulk density range is 1.98–2.28 g/cm^3 (41). These results highlight the importance of balanced raw material proportions in achieving the desired density during ceramic tile production, which affects final product performance (42).

Table 4: Physical Analysis of Ceramic Materials Developed

Finished Product	Shrinkage (%)	Weight loss on Ignition (%)	Apparent Relative Density (g/cm³)	Post Firing Bulk Density (g/cm³)
F0	4,66±0,57	2,53±0,80	2.33±0.09	1.82±0.06
F1	6,80±0,50	5,96±0,43	2.40±0.03	2.13±0.05
F2	7,72±0,45	4,81±0,31	2.41±0.02	2.22±0.07
F3	7,43±0,43	4,83±0,22	2.41±0.03	2.19±0.09
F4	5,33±0,26	3,90±0,21	2.34±0.04	2.01±0.07
F5	7,57±0,32	5,69±0,54	2.42±0.07	2.28±0.09
F6	6,95±0,29	6,00±0,47	2.41±0.05	2.25±0.06

The results of the modulus of rupture (N/mm^2) and water absorption (%) of the ceramic materials produced are presented in Figure 7. Analysis of the modulus of rupture revealed large variations among the formulations tested. The F0 composition, consisting solely of LMPT, exhibited the lowest strength at 4.01 N/mm^2. Similarly, F4, which contained 50% LMPT, demonstrated inadequate strength of 15.01 N/mm^2. In contrast, formulations F2, F3, F5, and F6 displayed superior flexural strengths ranging from 22.75 to 25.31 N/mm^2. The balanced composition of these formulations, consisting of a mixture of clays (C1 and C2), LMPT (10%-20%), and SF (for F5 and F6), contributed to their enhanced performance. Water absorption analysis revealed marked differences among the tested formulations. F0 exhibits the highest water absorption (11.98 %). F4 demonstrated an elevated water absorption of 7.11%. In contrast, formulations F2, F3, F5, and F6 displayed water absorption values ranging from 3.21% to 4.12%. Their balanced compositions contribute to their superior performance by reducing water absorption. The classification of ceramic materials based on the modulus of rupture and water absorption, in accordance with the ISO 13006 standard, is addressed later in this paper.

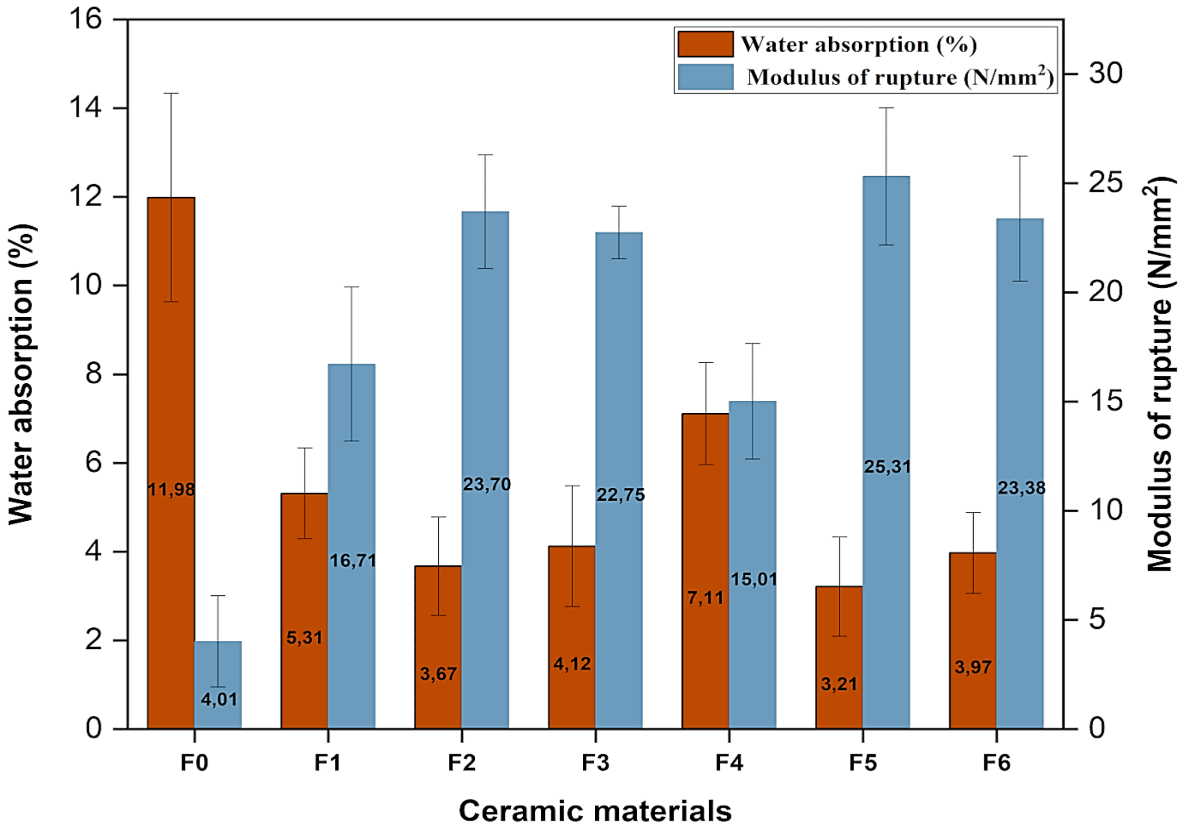

Figure 7: Modulus of Rupture and Water Absorption of the Produced Ceramic Materials

Principal Component Analysis of the Properties of Manufactured Ceramic Materials

The results of the Principal Component Analysis (PCA) of the physical, mechanical, and technological properties of ceramic materials are shown in Figure 8 .PCA is a dimensionality reduction technique that generates new principal components through linear combinations of the original variables. These derived components are orthogonal and uncorrelated (43,44).

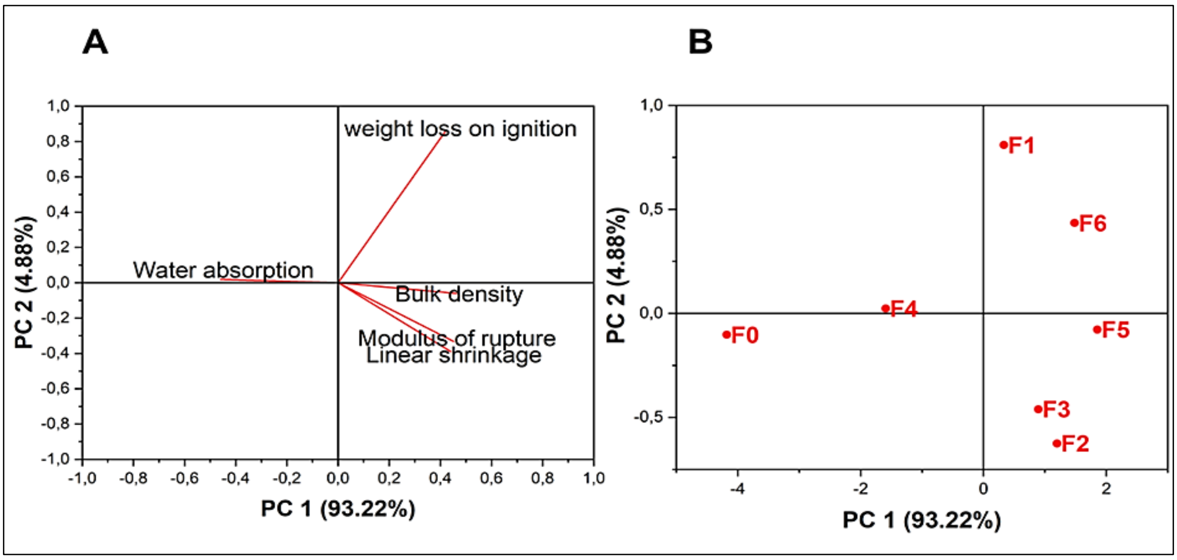

Figure 8: Principal Component Analysis (PCA) of the Physical, Mechanical and Technological Properties of Ceramic Materials : (A) Vectors of Variables and (B) Distribution of Formulations

This graphical representation can be interpreted along two distinct lines of analysis:

Variable vectors

The correlations between various characteristics were examined in this analysis. There was a strong positive correlation between the bulk density and modulus of rupture. In contrast, the water absorption showed an inverse relationship with these two factors, indicating a negative correlation. A positive correlation was also observed between shrinkage and weight loss on ignition ; however, the strength of this correlation was less pronounced than that of the other variables.

Distribution of Formulations

This analysis investigated the distribution of formulations based on the properties obtained from post-firing ceramic material characterisation. Materials F0 and F4 were isolated on the left side of the graph, showing high water absorption and low values for the other properties, indicating poor mecanical and physical performance. F1 also appeared isolated, although it was less extreme than F0 and F4. The lower-right quadrant contains a cluster comprising F2, F3, and F5, which is characterised by a high modulus of rupture, high bulk density, and low water absorption. This positioning supports superior physical and, mechanical performance. Notably, F6 was situated in the upper-right section of the graph near the vector, indicating weight loss on ignition. This positioning suggests that F6 exhibits a higher weight loss on ignition than the other high-performance formulations. The increased weight loss on ignition may be attributed to F6's unique composition. Specifically, it contained 15% LMPT and 3% SF, which could result in a greater release of volatile substances during firing. Despite this higher weight loss on ignition, F6 still exhibited a strong performance in terms of the modulus of rupture, bulk density, and water absorption, as indicated by its position in the right quadrant of the graph.

Surface Analysis Results

The cohesion and texture characteristics of the produced materials were examined using scanning electron microscopy (SEM), as shown in Figure 9. The SEM micrograph of sample F0, produced entirely from LMPT (100%), displayed significant porosity and apparent heterogeneity. This observation supports the data from the water absorption assessment, which provided a value of 11.98%. The micrograph of sample F4, containing 50% LMPT, showed also surface heterogeneity, suggesting mediocre characteristics for the ceramic tiles generated from this composition.

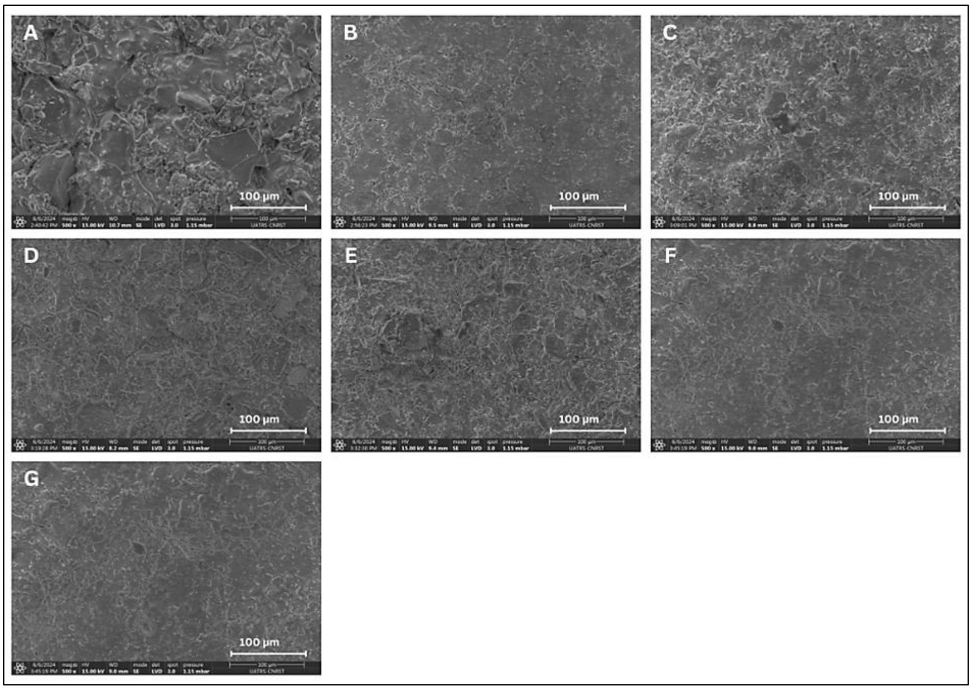

Figure 9: Scanning Electron Microscopy (SEM) Observations at 500x Magnification Showing the Cohesion and Texture of the Developed Materials: (A) F0 Material, (B) F1 Material, (C) F2 Material, (D) F3 Material, (E) F4 Material, (F) F5 Material, (G) F6 Material

In contrast, the SEM micrographs of F2, F3, F5, and F6 showe a more compact microstructure characterised by smaller grain sizes. This observation can be directly correlated with the higher proportion of clay present in these formulations, which facilitates increased compaction during the manufacturing process (45). The incorporation of LMPT within the range of 10%-20% served an important role as a degreasing agent, generating a skeletal structure inserted within the solid matrix. A comparative analysis of these micrographs revealed that the most coherent and compact surface microstructures were achieved in samples F5 (10% LMPT and 8% SF) and F6 (15% LMPT and 3% SF). This finding confirms the assumption that the presence of feldspar in adequate quantities can improve the quality of ceramic tiles (18).

Classification and Justification of the Choice of Ceramic Materials

The classification of ceramic tiles in industrial manufacturing processes is governed by the standards established by the International Organization for Standardization (ISO) under ISO 13006 (46). These standards specify two primary criteria for commercial classification : the production method and water absorption rate. The manufacturing process is classified into Method A, which involves extrusion, and Method B, which involves dry pressing. It is noteworthy that in the tile group nomenclature, capitalised "B" denotes production via dry pressing. Furthermore, the ceramic tiles were subjected to additional commercial classifications, as listed in Table 5. Hereafter, F0, F1, F2, F3, F4, F5, and F6 refer to the ceramic materials fabricated according to their respective compositions. Based on the categorisation established by ISO 13006, F0, which contains 100 % LMPT, exhibits poor mechanical properties (modulus of rupture of 4.01 N/mm^2) and high water absorption of 11.98%. This is because they lack sufficient clay content, which is a essential component for ceramic tile production (47–49). The complete substitution of raw materials with LMPT results in products that do not satisfy the minimum standards required for ceramic tile manufacturing. Material F1, with 5% LMPT, showed a modulus of rupture of 16.71 N/mm^2 and a water absorption of 5.31%. These characteristics prevent its categorisation into existing groups owing to a significant reduction in degreasing agents (50), which are essential for the structural integrity of ceramic compositions. Conversely, F2, which incorporated 10% LMPT, met the criteria for classification within the BIIa group, demonstrating a modulus of 23.70 N/mm^2 and water absorption rate of 3.67%. The significance of water absorption lies in its correlation with open porosity, which influences the consolidation and compaction of ceramic bodies before firing. This relationship directly affects the physical and chemical properties of the final product (51). The additional physical parameters of F2, as detailed in Table 5, align with the industrial standards documented in the literature (see Table 5).

Table 5: Requirements for the Validation of Ceramic According to ISO 13006 Standard

Ceramic Tiles Properties	Group I $E_v \leq 3\%$		Group II $3\% < E_v \leq 10\%$		Group III $E_v > 10\%$
	BIa	BIb	BIIa	BIIb	BIII
Water absorption E_v (%)	$E_v \leq 0.5\%$	$0.5\% < E_v \leq 3\%$	$3\% < E_v \leq 6\%$	$6\% < E_v \leq 10\%$	$E_v > 10\%$
Modulus of rupture (N/mm^2)	Minimum 35	Minimum 30	Minimum 22	Minimum 18	Minimum 12

The same analysis was applied to F3, which contained 20% of LMPT. aligned with the BIIa group classification. This formulation exhibited a modulus of rupture of 22.75 N/mm^2 and a water absorption of 4.12% By contrast, F4, which contained 50% LPMT, displayed characteristics that differed from those of the established ceramic tile categories. This formulation was marked by a notably low modulus of rupture (15.01 N/mm^2), coupled with high water absorption (7.11%). The increased proportion of LMPT, specifically the abundance of silica (SiO_2) in the form of quartz particles, contributes to a reduced modulus of rupture (24). This high silica content adversely affects the mechanical strength. This is due to the correlation between the compression induced by quartz crystals in the vitreous phase and microstructural imperfections, such as cracks surrounding or within the quartz particles. The analyses of F2 and F3, incorporating 10% and 20% LMPT, respectively, displayed promising technological characteristics. These findings prompted the development of two additional compositions, F5 and F6, which maintained similar mine tailings proportions of 10% and 15% (between 10% and 20%) while introducing SF to the formulation. This modification aims to improve the physical and mechanical properties of the resulting material. Owing to their lack of plasticity, degreasing agents form a rigid interconnected skeleton within the ceramic body. Typically present as coarse particles (>10 µm), these agents play an essential role by contributing significantly to the consolidation of the ceramic body while also reducing shrinkage during the drying and firing stages of production (50). Material F5, which was composed of 10% LMPT and 8% SF, exhibited notable physical properties. Its modulus of 25.31 N/mm^2, and water absorption of 3.21%, placing it within the BIIa classification group. Similarly, F6, which contained 15% LMPT and 3% SF, was assigned to the BIIa group. This formulation achieved a modulus of rupture of 23.38 N/mm^2 and a water absorption of 3.97%. These results demonstrate the importance of appropriate raw material proportioning to achieve desirable mechanical properties in ceramic tile production (42).

Economic Benefits of Using LMPT in Ceramic Tiles

The reuse of LMPT in ceramic tile production optimizes resource use by reducing reliance on non-renewable raw materials. Traditional raw materials are often costly and less sustainable, making the substitution of these materials with mine tailings a practical and economical alternative. By incorporating mine tailings, the ceramic tiles industry can make more efficient use of available resources, which is particularly beneficial in regions where access to conventional materials may be limited or expensive (52). In addition to resource optimization, the substitution of mine tailings for traditional raw materials leads to significant cost savings. With the rising prices of conventional materials, using mine tailings can substantially reduce procurement expenses. Studies have shown that this substitution can result in notable savings for manufacturers (30). These cost reductions directly impact the overall cost structure of ceramic tile production, offering a more affordable solution for manufacturers.

Conclusion

Characterization of the mechanical and physical properties of fired ceramic materials revealed that the incorporation of lead mine processing tailings (LMPT) of the Zeïda mine, Morocco, in proportions between 10% and 20%, combined with abundant local clays and sodium feldspar, permits the production of ceramic tiles that meet applicable standards (ISO 10545 and ISO 13006). The use of 100% LMPT in ceramic tile production is not feasible, owing to the inferior characteristics achieved. Indeed, the quality control evaluation of the ceramic materials demonstrated that formulations incorporating 10-20% LMPT (F2, F3, F5, and F6) are effective for producing ceramic floor tiles that meet the ISO 13006 standards, which belong to the BIIa group (good balance between mechanical strength and water absorption). By contrast, the exclusive use of mine tailings or high concentrations (50%) did not meet the necessary requirements.The reuse of LMPT as an alternative raw material in the ceramic industry has several advantages. Incorporating LMPT into ceramic tile production not only addresses waste management but also coincides with both ecological and economic aims. Environmentally, this method mitigates the disposal of potentially harmful products and is dependent on non-renewable resources. It provides a cost-efficient alternative by partially replacing imported raw materials such as sand and feldspar with locally produced mine tailings, thereby lowering production expenses. In summary, this study indicates the significant potential of utilising Zeïda LMPT as an alternative raw material in the ceramic industry, delivering both environmental and economic benefits while matching the industrial standards for floor tiles. Large-scale investigations and additional industrial experiments are required to confirm the applicability of the most appropriate formulations developed in this study for commercial ceramic tile manufacturing. In addition, a more extensive study of the environmental impact should be performed, particularly by assessing the leaching of heavy metals from ceramic tiles, to verify their long-term environmental safety.

Abbreviations

C1: Variety 1 of clay sourced from the Berrechid quarry, Morocco, C2: Variety 2 of clay sourced from the Berrechid quarry, Morocco, ISO : International Organization for Standardization, LMPT: Lead mine processing tailings, PCA: Principal component analysis, SEM: Scanning electron microscopy, SF: Sodium feldspar, XRD: X-ray diffraction, XRF: X-ray fluorescence.

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Author Contributions

Authors contributed to data analysis and manuscript writing for this study.

Conflict of Interest

The authors declared that there is no conflict of interest.

Ethics Approval

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