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Immobilization of Trace Metal Elements from Lead Ore Processing Tailings in Ceramic Tiles : Case of the Zeïda Mine (High Moulouya, Morocco)

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Abstract

Mine tailings, which result from mining activities, generate hazardous wastes containing metal trace elements (MTEs). These metals contribute to environmental pollution and pose serious risks to both environmental and human health. In light of this issue, this study investigates the feasibility of immobilizing metal trace elements (MTEs) within lead ore processing tailings (LOPT) from the abandoned Zeïda Mine (High Moulouya, Morocco) as secondary raw materials for ceramic tile production. The chemical composition of the LOPT and clay were analyzed using X-ray fluorescence (XRF), and the mineralogical phases were identified using X-ray diffraction (XRD). The particle size distribution of LOPT was characterized, and the physical and mechanical properties of the produced ceramic tiles were assessed. Scanning electron microscopy (SEM) was used to examine the microstructure of the ceramic matrix. Additionally, the leaching behavior of MTEs was evaluated according to the EN 12457-2 standard to assess the environmental safety of sintered products. The results revealed that the incorporation of LOPT into the ceramic mixture produced tiles with excellent physical and mechanical properties. XRD analysis confirmed the formation of new crystalline phases, such as $AlAsO_4$, $CuAl_2O_4$, $ZnAl_2O_4$, $ZnFe_2O_4$ and $BaFe_2O_4$, which effectively immobilized MTEs, including As, Pb, Cu, Zn, and Ba. These findings suggest that LOPT can be successfully valorized in ceramic tiles, reducing environmental pollution from mining waste while promoting sustainable industrial practices. This strategy provides a practical solution for MTEs immobilization and supports the circular economic principles. This study demonstrates a viable pathway for waste valorization, environmental protection, and sustainable ceramic tile manufacturing.

Keywords: Ceramic Tiles, Lead Ore Processing Tailings, Metal Trace Elements Immobilization, Mine Waste Management, Zeïda Mine.

Introduction

Mining tailings, a significant by-product of ore processing, requires extensive land use, involves high construction and maintenance costs, and poses considerable environmental and ecological risks (1). However, they also contain valuable raw materials that can be recovered and utilized in various industrial applications, thereby reducing the demand for natural resources and supporting sustainable resource management (2). In the transition toward a circular economy, mine tailings are regarded as a critical resource for reuse and recycling because of their large volumes, environmental impact, and growing need for sustainable utilization of natural resources (3, 4). Mine tailings, leftover waste from the extraction and processing of valuable minerals, pose serious and complex environmental risks owing to the release of hazardous contaminants and their long-term effects on ecosystems and human health (5, 6). Improper handling of this waste can exacerbate environmental deterioration and lead to widespread contamination (7). High concentrations of metals in tailings can lead to their mobilization into groundwater through leaching and infiltration (8, 9). These pollutants compromise the soil integrity and, reduce the shear strength and load-bearing capacity, which may result in foundation settlement and structural damage. Additionally, soil fertility declines, leading to lower crop yields and indirect risks to human health (10). Mine tailings contribute to the dispersion of toxic elements in nearby soil and water, severely impacting the surrounding environment (11). Addressing this issue not only reduces the land occupied by tailings piles and minimizes the loss of mineral resources, but also

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mitigates the associated pollution risks. This global challenge has prompted extensive research on tailings management and solid-waste treatment (12–14). The major environmental concern in mine tailings disposal stems from the potential release of hazardous substances into the environment. The most common management strategy involves the construction of tailings pond that store waste materials in water-filled containment areas (15). These pond are widely used because of their low cost and regulatory acceptance; however, they pose risks of dam failure and groundwater contamination (16). In response, alternative disposal methods such as dry stacking and valorization of tailings for construction materials are being increasingly explored (17, 18).

The recycling and reuse of mine tailings refer to the process of utilizing the entire waste material in its original state for a specific purpose, without requiring any reprocessing (19). Among these remediation methods, solidification/stabilization technology is distinguished by its cost-effectiveness and operational simplicity, establishing it as a reliable solution for treating toxic metal-contaminated soils (10). This process combines waste materials with binding agents to create products with enhanced physical properties (20).

Building on this approach, sintering stabilization offers an effective technique to reduce the leachability of toxic metals in waste materials while preserving the original volume of stabilized products (20). In addition to offering a cheap source of readily available building materials, thermal processes for materials that contain tailings as a raw material can stabilize toxic metals in the newly created materials.

The abandoned Zeïda mine, which is the focus of this study, is located in northeastern Morocco and has been operational from 1972 to 1985. During the extraction of minerals, metals, and other valuable resources through ore processing methods, mines generate mine tailings (LOPT) containing metal trace elements (MTEs) such as lead (Pb), zinc (Zn), copper (Cu), arsenic (As), and barium (Ba). These metals are present in concentrations significantly higher than the average levels found in the Earth's crust (21). The Zeïda mine is situated in the semi-arid region of Upper Moulouya and is, characterized by a unique geological and environmental context. A semi-arid climate, combined with alkaline geological conditions, inhibits the formation of acid mine drainage, which is a common issue at many abandoned mining sites. However, despite the absence of acid mine drainage, this region experiences a phenomenon known as neutral mine drainage (NMD) (22).

This study investigates the integration of lead ore processing tailings (LOPT) of abandoned Zeïda mine into ceramic tiles to immobilize metal trace elements within the ceramic matrix. The hazardous waste after treatment not only helps to immobilize the MTEs, but also might transfer the wastes into eco-friendly products, such as ceramsite. Combining the control of contamination and tailings utilization is a recent trend in the disposal of tailings containing toxic metals (23). Their addition to construction materials is considered the best fit among the options for the utilization of solid waste in large quantites, and many successful studies have been reported recently regarding the use of tailings to prepare sintered bricks and ceramic tiles (24–27).

Methodology

Presentation of the Study Area and Raw Materials Used

The Zeïda mining center is an abandoned Pb mining district located in northeastern Morocco, within the upper Moulouya watershed. It lies in the province of Midelt, within the Middle Atlas Mountains, at an altitude of 1603 m (32°53'41.8'' N, 5°00'08.010'' W). The site covers approximately 300 km² along the Oued Moulouya and was actively mined for Pb between 1972 and 1985. Mining operations were conducted in open-pit quarries, and a processing plant with a capacity of 1.4 million tons per year was used to enrich the ore through crushing, grinding, flotation, and filtration. After 13 years of exploitation, the mining activities have resulted in the accumulation of substantial waste materials, including 630,172 tons of lead concentrate (40–70% grade), approximately 12 million tons of processing tailings, and approximately 70 million tons of overburden waste rock, all stored along the Oued Moulouya banks.

In this study, lead ore processing tailings (LOPT) from the Zeïda mining center were investigated for their reuse in sustainable application for the immobilization and stabilization of metal trace elements (MTEs) in ceramic tiles.

The sampling process involved preparing a composite LOPT sample for this study by collecting material from the tailings deposits at the abandoned Zeïda Mine. A composite sample in Figure 1 was assembled from five subsamples manually collected at a depth of 40 cm from various locations using a plastic scoop.

Substantial quantities of natural clay, specifically CL1 and CL2, are utilized in the production of ceramic materials Figure 1. These clays, sourced from the Berrechid quarry in Morocco, represent two distinct types : CL1 is a high-grade clay commonly used in the production of floor and wall tiles, serving as the primary skeletal component in pressed tile formulations.

Figure 1 : Raw Materials for Ceramic Production : (A) LOPT (Lead Ore Processing Tailings), (B) Clay 1 (CL1), (C) Clay 2 (CL2)

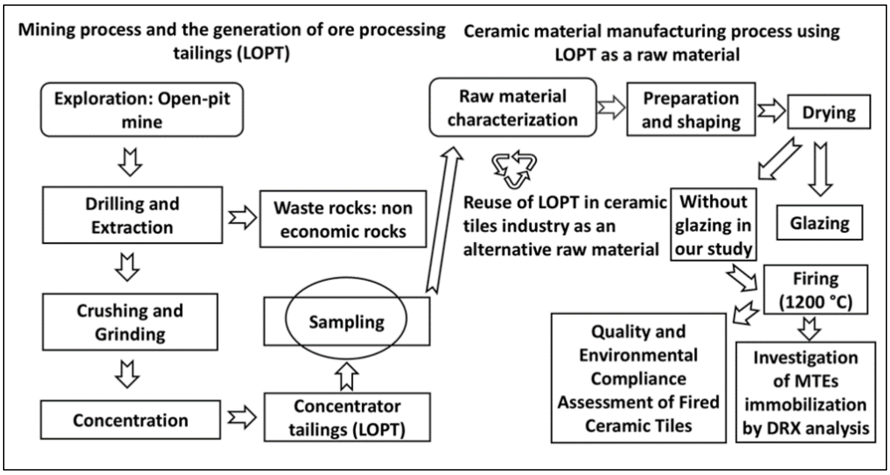

Figure 2 : Mining and Ceramic Material Manufacturing Processes

After sampling and characterization, the mine tailings (LOPT) were reused as alternative raw materials for ceramic material production, as illustrated in Figure 2.

Techniques for Characterizing Raw Materials

The raw materials were analyzed using a range of advanced techniques. The chemical composition of LOPT, CL1 and CL2 were determined through X-ray fluorescence (XRF) spectroscopy, using a wavelength dispersive spectrometer (Axios model) for precise quantitative elemental analysis. The mineralogical composition of LOPT, CL1 and CL2, were investigated using X-ray diffraction (XRD) on an EMPYREAN device equipped with a reflection-transmission spinner setup (MALVERN PANALYTICAL), Diffraction patterns were recorded with CuKα radiation and phase identification was performed using the ICDD database, with crystalline phases further identified using the X'pert HighScore Plus software (Version 3.0, Malvern Panalytical).

The particle size distribution of the lead ore processing tailings (LOPT) was examined using a combination of methods. Dry sieving was applied to analyze the sand fraction, whereas sedimentation techniques were employed for the silt and clay fractions, following the ISO 17892-4 (28) standard. LOPT is categorized into three main fractions based on their size : clay (< 2 µm), silt (2 – 50 µm), and sand (50 – 2 mm), according to the United States Department of Agriculture (USDA) classification. The plasticity of the clay samples was evaluated according to the standard procedures described in ISO 17892-12. The plastic limit (Wp) was determined by rolling a semi-soft clay paste into threads approximately 10–15 cm in length and 3 mm in diameter, continuing until the thread cracked before reaching the specified diameter. The wet mass (Mh) and dry mass (Ms), obtained after oven drying at 140°C for 2 h, were used to calculate Wp as the water content relative to the dry mass. The liquid limit (WL) was measured using the Casagrande apparatus, where a clay paste was spread in the apparatus cup, grooved with a V-shaped tool, and subjected to controlled drops at two drops per second until the groove closed along a length of 1 cm. The water content measurements were valid only for tests with 15-35 drops. The plasticity index (IP) was calculated as IP = WL – Wp.

Manufacturing Process of Ceramic Materials

Five different formulations were developed and investigated for ceramic tile production shows in Table 1. LOPT was introduced as a partial replacement for CL2 clay, with substitution levels ranging from 5% to 50% in formulations CM1 to CM4. However, its incorporation was limited to a maximum of 50% owing to its non-plastic nature, which affects the workability and shaping of the ceramic mixture. Clay, which plays a crucial role in providing plasticity and binding properties, remains the primary component of the formulations. To maintain adequate workability and structural integrity, CL1 clay, known for its superior quality and widespread availability, was included at a fixed proportion of 40% in CM1 to CM4. This ensured sufficient plasticity in the mixture, facilitating molding and preventing excessive brittleness during the drying and firing processes. The CM5 formulation, on the other hand, was composed entirely of LOPT without the addition of other raw materials, serving as a reference for evaluating the impact of LOPT alone on ceramic properties. This composition allowed for a comparative assessment of the influence of LOPT content on the final ceramic product in terms of its physical, mechanical, and microstructural properties.

Table 1 : Ceramic Materials Prepared with Varying Percentages of CL1 (Clay 1), CL2 (Clay 2), and LOPT (Lead Ore Processing Tailings)

Ceramic Materials	CL1 (%)	CL2 (%)	LOPT (%)
CM1	40	55	5
CM2	40	50	10
CM3	40	40	20
CM4	40	10	50
CM5	0	0	100

A total of 300 g of each formulation was mixed and milled in a cylindrical ceramic ball mill with a 500-gram capacity for 10 minutes, using 80 ml of water and 1% deflocculant. The moistened powder was compacted into test specimens using a manual hydraulic press at 175 bars in two types of rigid molds: rectangular and cylindrical. The rectangular specimens had dimensions of 151.7 mm × 50.30 mm × 7.21 mm, while the cylindrical specimens had a diameter of 5 mm. After compaction, all specimens were oven-dried at 110°C for 40 min, ensure a residual moisture content below 1% to prevent cracking during firing. The final firing process was conducted in an industrial rotary kiln over 47 min, during which the temperature was progressively increased to 1200°C. The shapes of the produced ceramic materials are shown in Figure 3.

Cylindrical specimens were designed to measure the shrinkage, weight loss on ignition, and water absorption, whereas rectangular specimens were used to measure the modulus of rupture.

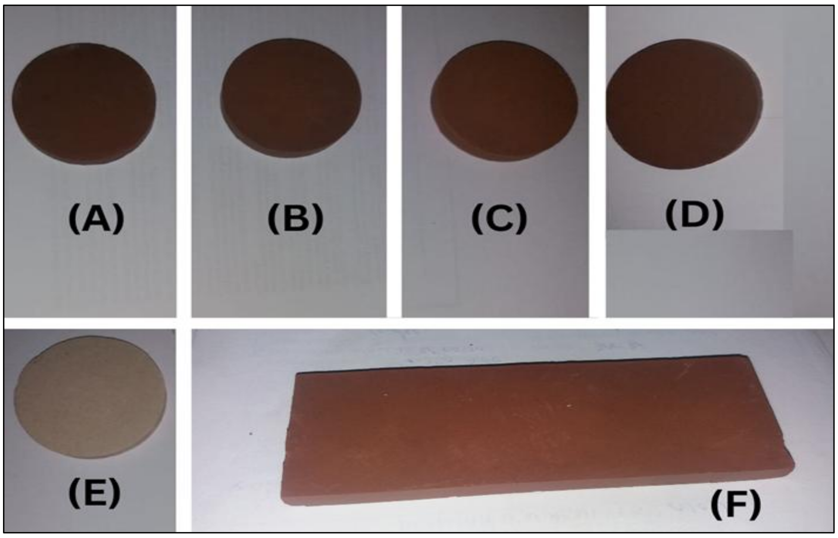

Figure 3 : Produced Ceramic Materials : (A) CM1 Material, (B) CM2 Material, (C) CM3 Material, (D) CM4 Material, (E) CM5 Material, (F) Shows a Rectangular Sample Used for Modulus of Rupture Testing of Each Formula

Characterization of the Produced Ceramic Materials

Several important tests were performed to evaluate the mechanical and physical properties of the ceramic materials. The three-point flexural strength, also referred to as the bending strength or modulus of rupture, was measured by using a Gabbrielli Crometro CR/650 R160 machine. Rectangular ceramic specimens were used for these tests in Figure 3. Seven tests were conducted for each formulation and the results were averaged in accordance with the ISO 10545-4 starndard (29).

Firing shrinkage was evaluated by measuring the diameters of the dry cylindrical samples shown in Figure 3 both before and after the firing process. Five tests were conducted for each formulation and the results were averaged according to the ISO 10545-3 standard (30).

Water absorption was determined by calculating the weight difference between the dry cylindrical samples before and after immersion in boiling water at 100°C for 4 h. Five tests were performed for each formulation and the results were averaged in accordance with the ISO 10545-3 standard (30).

Surface analysis of the dry-fired samples was conducted using scanning electron microscopy (SEM) to assess the compactness and cohesiveness of the grains. Microscopic observations were performed using a QUATTRO S-FEG scanning electron microscope, provided (Thermo Fisher Scientific).

Leaching Test for Ceramic Materials

The leaching behavior of the ceramic materials was assessed following the standard procedure provided by EN 12457-2 (31), which characterizes the release of inorganic substances from the solid materials. The Crushed ceramic samples, with particles smaller than 4 mm, were dried at 105°C to a constant mass, and a representative sample of approximately 100 g was taken. The prepared sample was transferred to an appropriate container and demineralized water was added at a liquid-to-solid ratio of 10 L/kg. The mixture was agitated at 10 rpm for 24 h to achieve a full contact between the solid and liquid phases. The leachate was filtered using a 0.45 µm filter for eluate collection. The concentrations of MTEs in the leachate were determined by ICP-AES and thus provided a quantitative assessment of the possible mobility of pollutants from the ceramic matrix.

To evaluate the environmental safety of the produced materials, the leaching results were compared with the WHO drinking water standards and the Moroccan irrigation water regulations. WHO standards serve as a global benchmark to ensure that any potential leaching remains within safe limits for human health, particularly in cases where ceramic products are exposed to water. Moroccan irrigation guidelines are relevant for assessing the suitability of these materials for agricultural applications, where water quality can affect soil and crop health. By considering both standards, this study provides a comprehensive assessment of MTE immobilization and the potential environmental impact of ceramic tiles.

Results and Discussion

Results of Chemical Analysis of Raw Materials

Chemical composition analysis of Lead Ore Processing Tailings (LOPT) and the two clay types (CL1 and CL2) showed significant potential for ceramic manufacturing in Table 2. These raw materials exhibit distinct compositional profiles, with LOPT characterized by high silica content (68.92%), moderate aluminum oxide levels (12.03%), and a distinctively high barium oxide concentration (4.59%). In contrast, the clay samples (CL1 and CL2) exhibited higher aluminum oxide percentages at 23% and 27%, respectively, along with elevated iron oxide levels (4.38% for CL1 and 4.32% for CL2), providing a complementary chemical foundation for ceramic manufacturing. For optimal ceramic production, these tailings should be combined with alumina-rich clays to create a well-balanced formulation that improves the properties of the finished ceramic product. Alumina helps to create refractory materials at high temperatures and reduces shrinkage during drying and firing (32).

Feldspar was detected in the LOPT in the presence of potassium oxide K_2O (3.88%). Feldspars frequently contain K_2O and Na_2O oxides, which are well-known for their capacity to cause melting (33). These are important fluxes that lower the sintering temperature and encourage the development of a glassy phase in ceramic tiles (34).

Table 2 : Chemical Composition (wt%) of Clays (CL1, CL2) and Lead Ore Processing Tailings (LOPT) Determined by X-ray Fluorescence

	SiO_2	Al_2O_3	K_2O	Na_2O	Fe_2O_3	CaO	BaO	SO_3	PbO	ZnO	Other oxides	LOI*
LOPT	68.92	12.03	3.88	0.88	0.58	1.52	4.59	2.19	0.46	0.01	1.84	2.21
CL1	54.42	27.27	2.07	1.27	4.38	0.31	0.05	0.03	-	-	0.72	7.20
CL2	57.50	23.78	2.76	1.26	4.32	1.08	0.03	0.02	-	-	0.59	6.16

* Loss on ignition at 1140°C

Results of Mineralogical Analysis of Raw Materials

The primary phases identified through mineralogical analysis using XRD, presented in Figure 4, reveal that the LOPT is composed of quartz (SiO_2), anorthoclase ($(Na,K)(Si_3Al)O_8$), barite ($BaSO_4$), fluorite (CaF_2), and muscovite ($KAl_3Si_3O_{10}(OH)_2$). In addition to these phases, LOPT also contains minerals associated with trace metal elements (MTEs), such as scorodite ($FeAsO_4{\cdot}2H_2O$), cerussite ($PbCO_3$), lead sulfide (PbS), lead oxide (PbO), copper oxide (Cu_2O), and zinc oxide (ZnO).

The mineralogical composition of CL1 and CL2, as determined by XRD analysis in Figure 4, reveals that CL1 is primarily composed of quartz (SiO_2), illite [$(K, H_3O)Al_2Si_3AlO_{10}(OH)_2$], and clinochlore [$(Mg, Fe, Al)_6(Si, Al)_4O_{10}(OH)_8$], while CL2 consists of quartz (SiO_2), hematite (Fe_2O_3), and vanadian muscovite [$K(Al, V)_2(Si, Al)_4O_{10}(OH)_2$]. These mineral compositions provide valuable insights into the characteristics and potential use of these clays in ceramic production.

The clay and its possible applications in the manufacturing of ceramics were revealed by its mineral composition (35, 36). The optimal amount of illitic clay contributes to a dense microstructure with improved physical characteristics, such as less water absorption and greater bending strength, improving the quality of ceramic materials (37). Therefore, the CL1 content was constant in all formulations.

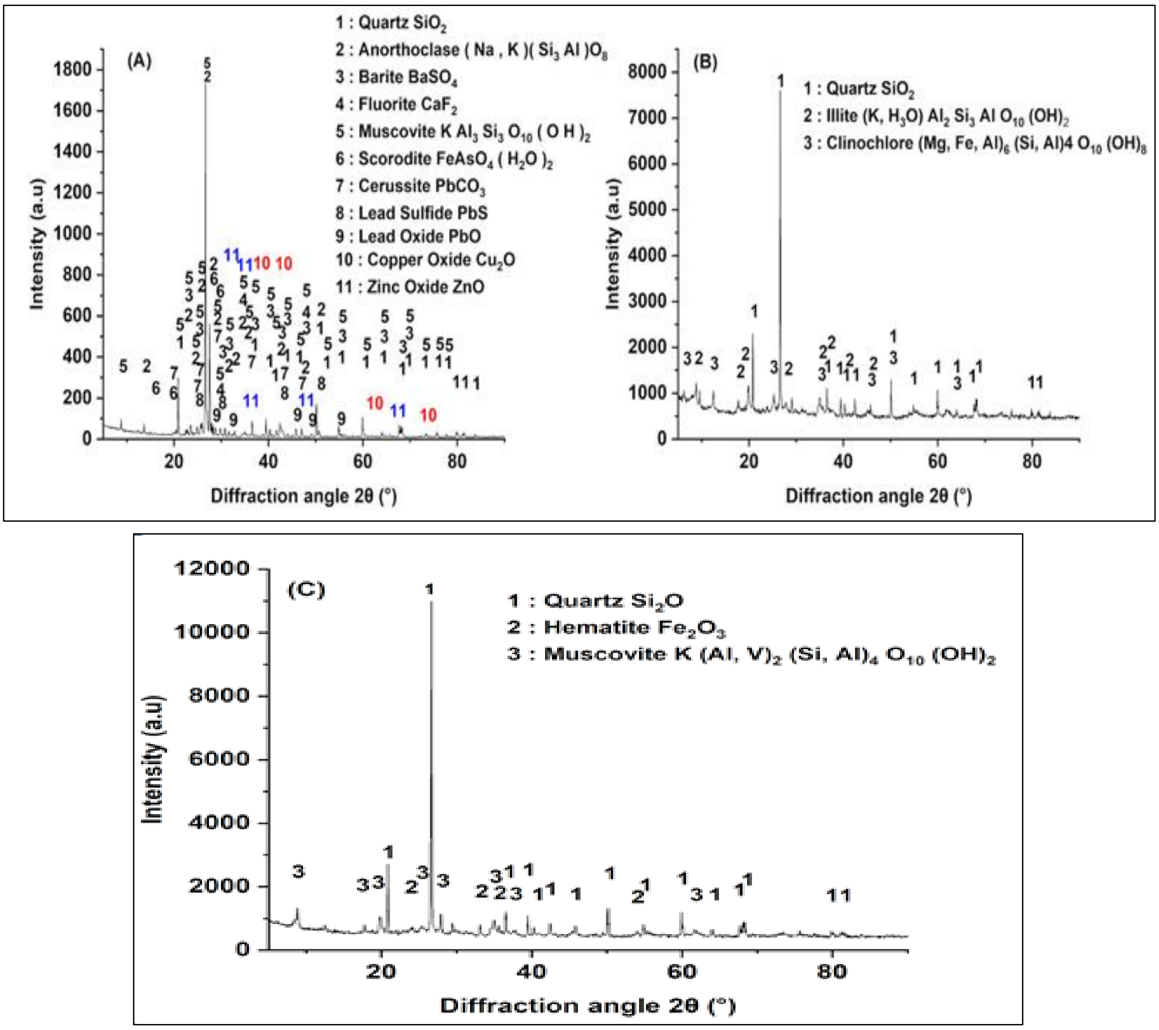

Figure 4 : Crystalline Phases of Raw Materials : (A) Lead Ore Processing Tailings LOPT, (B) Clay CL1, (C) Clay CL2

Table 3 : Particle Size Distribution (%) of Lead Ore Processing Tailings (LOPT)

Clay fraction (< 2 µm)	**Silt fraction** (2 µm - 50 µm)	**Sand fraction** (> 50 µm)
27.276	57.586	15.138

Results of Particle Size Distribution of LOPT

Particle size analysis helps to classify materials by size, regardless of their chemical composition. The particle size distributions of the LOPT are listed in Table 3.

The particle size distribution of the LOPT revealed a significant presence of silt and clay, with 57.586% silt and 27.276% clay. The dominance of fine particles enhances cohesion, which contributes to the strength and moldability of ceramic formulations (38). The 15.138% sand fraction, primarily due to quartz, functions as a natural degreasing agent, helping to reduce shrinkage during drying and improve the physical strength of the final product (39).

Results of Plasticity Measurements of Clay Samples

The plasticity of the clay samples was significantly influenced by their mineralogical compositions, as shown in Table 4. CL1, with a liquid limit of 41.3% and a plasticity index of 21.41, exhibited high plasticity, primarily owing to its dominant illite content. Illite is known to enhance the plasticity of clay, making it more suitable for various applications (40). In contrast, CL2, with a liquid limit of 26.38% and plasticity index of 15, is less plastic. This reduced plasticity is attributed to the higher content of nonplastic minerals, such as quartz and hematite. Quartz and hematite do not contribute to the plasticity of clay and can decrease its workability (41).

Table 4 : Atterberg Limits (%) with Corresponding Plasticity Indices

Clay	Liquid Limit (%)	Plastic Limit (%)	Plasticity Index
CL1	41.3	19.89	21.41
CL2	26.38	11.38	15

Results of Physical and Mechanical Properties of Ceramic Materials

The evaluation of ceramic tile performance in Table 5 provides significant insights into the mechanical and physical characteristics of materials incorporating lead ore processing tailings (LOPT). According to the ISO 13006 (42) standard in Table 6, the modulus of rupture and water absorption are important parameters for assessing material quality. The modulus of rupture demonstrated a remarkable transformation with LOPT incorporation, with CM2 and CM3 exhibiting exceptional mechanical performance. Specifically, CM2 achieved a peak modulus of rupture of 27.25 N/mm^2, while CM3 reached 26.16 N/mm^2, representing substantial improvements over CM1 (19.22 N/mm^2) and CM4 (17.26 N/mm^2).

Table 5 : Physical and Mechanical Properties of Ceramic Materials Developed

Finished Product	Shrinkage (%)	Weight loss on Ignition (%)	Water Absorption (%)	Modulus of Rupture (N/mm^2)	Ceramic tile Classification (ISO 13006 Standard)
CM1	7.14±0,40	5.80±0,39	4.52±1,15	19.22±2,24	-
CM2	7.99±0,41	4.70±0,33	3.11±1,11	27.25±1,17	BIIa
CM3	7.80±0,36	4.80±0,24	3.50±1,46	26.16±1,13	BIIa
CM4	5.59±0,28	3.81±0,20	6.10±1,13	17.26±2,28	-
CM5	4.90±0,62	2.49±0,78	10.18±2,37	4.61±2,02	-

Complementing the modulus of rupture analysis, the water absorption characteristics provided additional evidence of material enhancement. The materialCM1 demonstrated a water absorption rate of 4.52%, whereas CM2 and CM3 dramatically reduced water absorption by 3.11% and 3.50%, respectively. This reduction suggests an improved material density and cohesion, which are critical attributes for ceramic tile durability and performance.

Regarding the shrinkage, values between 4.90% and 7.99% indicated structural refinement, with CM2 achieving a shrinkage of 7.99% and CM3 7.80%, both indicating optimal shrinkage performance (34, 43). Similarly, weight loss upon ignition ranged from 2.49% to 5.80%, reflecting the removal of organic matter and volatiles during firing. Collectively, these properties, along with the classification of CM2 and CM3 as BIIa group materials, validate the integration of LOPT into ceramic production as a sustainable and performance-enhancing approach for waste valorization.

Table 6 : ISO 13006 Standard Requirements for Ceramic Tile Classification

Group	Water Absorption (Ev) (%)	Modulus of Rupture (N/mm^2)
BIa	Ev ≤ 0.5%	Minimum 35
BIb	0.5% < Ev ≤ 3%	Minimum 30
BIIa	3% < Ev ≤ 6%	Minimum 22
BIIb	6% < Ev ≤ 10%	Minimum 18
BIII	Ev > 10%	Minimum 12

The morphologies of CM2 and CM3 were examined using scanning electron microscopy (SEM) shown in Figure 5. SEM micrographs revealed that the microstructures were more compact. In contrast, CM1, CM4, and CM5 exhibited apparent heterogeneity, which directly correlated with their higher water absorption values in Table 5. These results indicate that LOPT content between 10% and 20% provides optimal mechanical and physical properties.

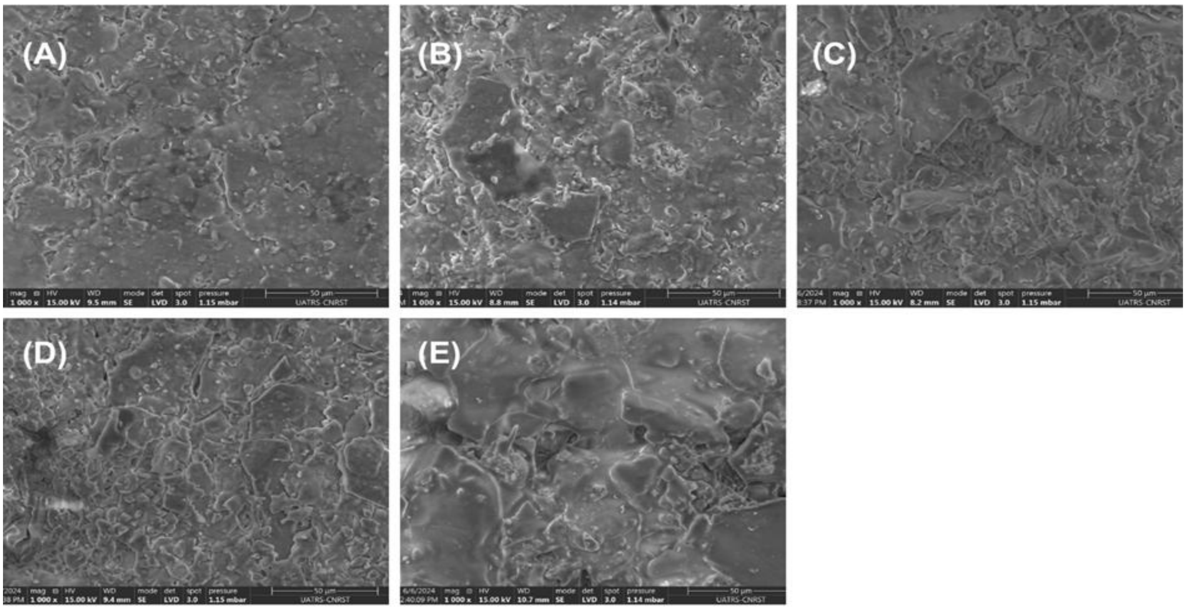

Figure 5 : Scanning Electron Microscopy (SEM) Images at 1000x Magnification Highlighting Cohesion and Texture of Developed Materials : (A) CM1, (B) CM2, (C) CM3, (D) CM4, (E) CM5

Impact of Sintering on MTE Immobilization

Different crystalline phases were formed during the sintering process, as shown in Figure 6 when LOPT was incorporated into ceramic materials. The phases that contained MTEs were focused upon in accordance with the objectives of this study. MTEs including arsenic (As), lead (Pb), copper (Cu), zinc (Zn), and barium (Ba) are largely immobilized within these phases. Immobilization lowers the leaching potential of these hazardous elements and ensures environmental safety by converting them into stable water-insoluble crystalline structures (44). By interacting with particular elements of the ceramic raw materials (clay and LOPT), Each MTE was immobilized within distinct crystalline phases in the ceramic matrix. The following section discusses the main crystalline phases that immobilize each MTE.

Aluminum arsenate ($AlAsO_4$) is mostly formed, which immobilizes arsenic (As). This phase is the result of the reaction between the aluminum (Al) and arsenic (As) during high-temperature sintering (45, 46). The high thermal stability and water insolubility of this phase ensure the long-term immobilization of arsenic within the ceramic matrix by incorporating it into the structure of $AlAsO_4$, significantly reducing its potential release (47). Arsenic immobilization is indirectly facilitated by the formation of a glassy phase during sintering, which is promoted by a flux agent (48). This glassy phase reduces porosity and forms a compact structure that further prevents arsenic leaching while enhancing the densification of the ceramic matrix (33). The synergistic interaction between AlAsO4 and the glassy matrix created a highly effective stabilizing effect on As.

Lead (Pb) is immobilized in the ceramic matrix mainly by the formation of lead sulfate ($PbSO_4$). In the sintering process, sulfate ions (SO_4^{2-}), mostly coming from the decomposition of sulfates present in the raw materials, react with lead to form $PbSO_4$ (49). This phase remains stable at elevated sintering temperatures, exhibits minimal solubility in water (showing greater stability at pH levels above 3), and effectively lowers the potential for Pb leaching from the ceramic matrix (50). $PbSO_4$ forms a crystalline structure that acts as a stable host to lead ions ; their mobility under environmental conditions is limited. Moreover, the denser and more compact ceramic structure produced during sintering may encapsulate $PbSO_4$ more effectively, which should significantly improve the overall environmental safety of the material (51).

Copper (Cu) and alumina (Al_2O_3) interact at high temperatures, forming a spinel-like phase, CuAl2O4 (52). This spinel structure is well known for its thermal stability and chemical resistance, which are crucial for immobilizing copper and reducing its leaching potential. During sintering, CuO serves as an intermediary phase, and its conversion into $CuAl2O_4$ over time further enhances the stability of Cu in the ceramic material.

The immobilization of zinc (Zn) in ceramic materials is accomplished by two important spinel-type phases: zinc aluminum oxide ($ZnAl2O_4$) and zinc iron oxide ($ZnFe_2O_3$) (53). When zinc (Zn) and alumina (Al_2O_3) react, a phase with high chemical stability and low solubility is created, effectively trapping zinc within the ceramic matrix and forming $ZnAl2O_4$. Similarly, when zinc (Zn) and iron oxide (Fe_2O_3) react, $ZnFe_2O_3$ is formed (54). Zinc is effectively hosted by $ZnFe_2O_3$, a spinel structure that provides strong resistance to chemical weathering (55).

The ceramic matrix immobilizes barium (Ba) through the formation of barium iron oxide ($Ba\ Fe_2O_4$) and barium aluminum oxide ($BaAlO_4$) (56, 57). Iron oxide (Fe_2O_3) and alumina (Al_2O_3) react with barium (Ba) during sintering to produce these phases. Their inability to dissolve in water prevents Ba from escaping from the ceramic matrix. Ba should form stable, insoluble crystalline structures that can be safely immobilized (58).

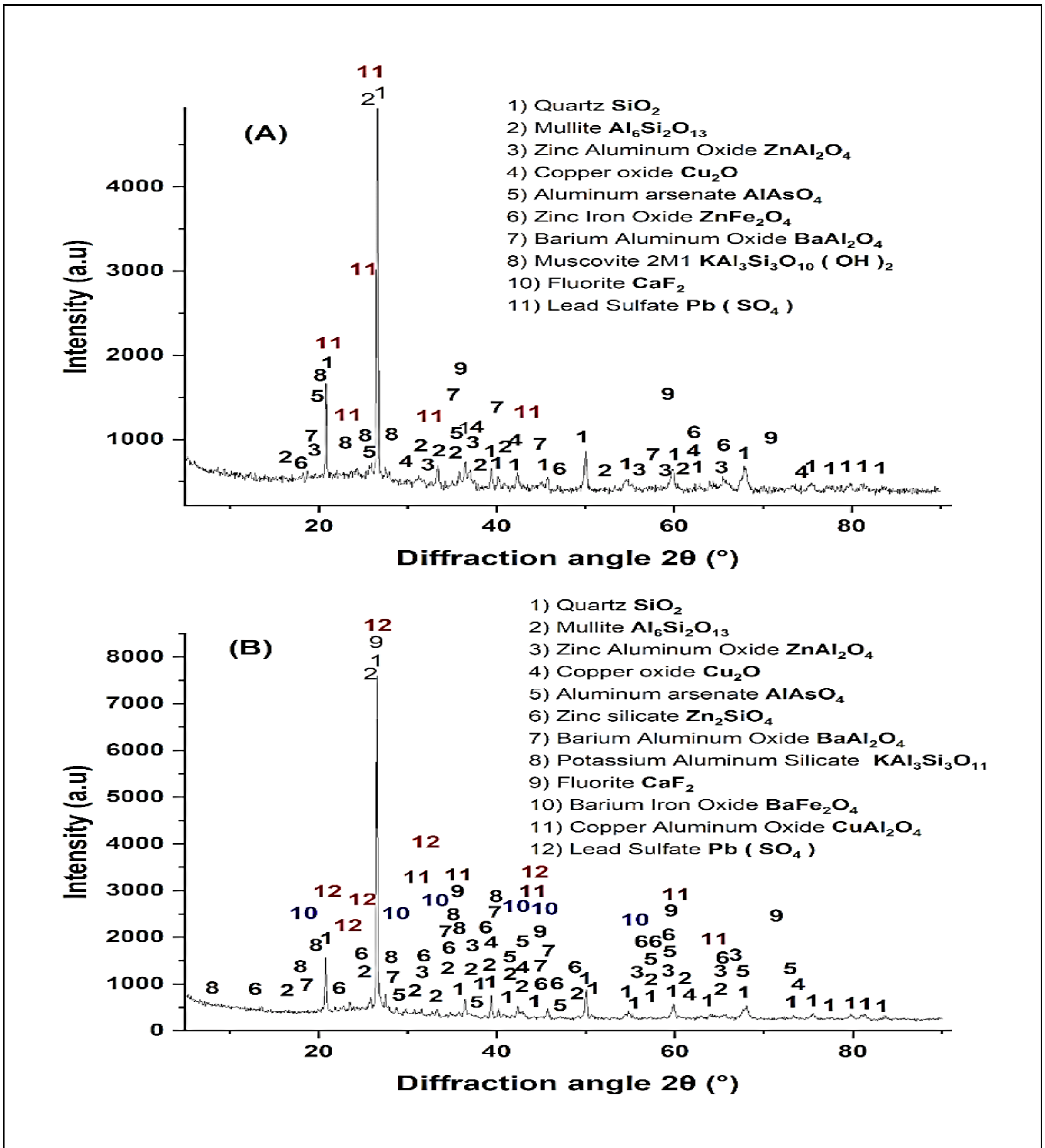

Figure 6 : Crystalline Phases of Produced Materials with High Physical and Mechanical Performance : (A) CM2, (B) CM3

MTE Leaching Test

Leaching is a process that determines whether contaminants migrate from a stabilized matrix to a liquid medium, and is essential for evaluating the environmental impact of a particular disposal method. The leaching of MTEs (As, Pb, Cu, Zn, and Ba) from ceramic samples CM2 and CM3, which exhibited good physical and mechanical properties, was analyzed in accordance with the EN 12457-2 standard shown in Table 7.

Table 7 : Concentrations of Metal Trace Elements (MTEs) Measured in the Leachate of Ceramic Materials CM2 and CM3

Materials	As (mg/L)	Pb(mg/L)	Cu(mg/L)	Zn(mg/L)	Ba (mg/L)
CM2	Not detected	0.011	0.1125	0.0813	0.3544
CM3	Not detected	0.013	0.1421	0.0458	0.3804

The results showed that arsenic (As) was undetected in both samples, thereby complying with the stringent standard of 0.01 mg/L established by the World Health Organization (WHO), as presented in Table 8. The concentrations of lead (Pb) detected (0.011 mg/L in CM2 and 0.013 mg/L in CM3) were near the WHO limit of 0.01 mg/L. Copper (Cu) was found at 0.1125 mg/L in CM2 and 0.1421 mg/L in CM3, which is well below the WHO drinking water limit of 2 mg/L. Zinc (Zn) concentrations, detected at 0.0813 mg/L in CM2 and 0.0458 mg/L in CM3, remained far below the WHO limit of 3 mg/L. The barium (Ba) concentrations (0.3544 mg/L for CM2 and 0.3804 mg/L for CM3) adhered to the WHO limit of 1.3 mg/L.

These leaching levels can be directly attributed to the formation of stable crystalline phases during sintering, which effectively immobilizes MTEs within the ceramic matrix. The incorporation of aluminum arsenate ($AlAsO_4$) plays an important role in binding arsenic (As) and preventing its release into the environment. The immobilization of lead (Pb) is mainly achieved through the formation of lead sulfate ($PbSO_4$). Although $PbSO_4$ formation contributes significantly to Pb stabilization, minor fractions may still be present in less stable phases or residual glassy structures, which could explain the slight presence of lead in the leachates.

During sintering, copper (Cu) interacts with alumina (Al_2O_3) to form a $CuAl_2O_3$ spinel phase.

Table 8 : WHO Drinking Water and Moroccan Irrigation Water Limits for (As, Pb, Cu, Zn, and Ba) Metals

Metal	WHO Drinking Water Limits (mg/L)	Moroccan Irrigation Water Limits (mg/L)	Health Impact (WHO Guidelines)
Arsenic (As)	0.01	0.1	Established due to cancer and other serious health risks.
Lead (Pb)	0.01	5	Established to address significant health risks, such as neurological impairment and developmental delays in children.
Copper (Cu)	2	0.2	Concentrations above 1 mg/L can cause staining, levels exceeding 2.5 mg/L affect taste, and high levels may lead to gastrointestinal effects
Zinc (Zn)	3	2	Not a health concern typically, but levels >3 mg/L may alter taste.
Barium (Ba)	1.3	Not specified	High levels linked to cardiovascular issues; guideline set for health protection.

This phase was thermally stable and chemically resistant, effectively reducing the leaching potential of Cu. CuO serves as an intermediary phase during the reaction, and its conversion to $CuAl_2O_3$ further enhances copper retention within the ceramic matrix.

The immobilization of zinc (Zn) in ceramic materials occurs through the formation of two key spinel-type phases : zinc aluminum oxide ($ZnAl_2O_3$) and zinc iron oxide ($ZnFe_2O_3$). When Zn reacts with alumina (Al_2O_3), it forms $ZnAl_2O_3$, a highly stable and low-solubility phase that effectively traps Zn within the matrix. Similarly, the reaction of zinc with iron oxide (Fe_2O_3) results in $ZnFe_2O_3$, which provides strong resistance to chemical weathering and prevents Zn leaching.

The barium (Ba) detected in the leachates was primarily stabilized within the $BaFe_2O_4$ and $BaAlO_4$ phases, which are known for their low solubility and chemical stability, preventing Ba from escaping the ceramic matrix. With regard to MTE leaching in relation to irrigation water, the results demonstrate that the ceramic samples effectively limit contaminant release while meeting Moroccan water quality standards. According to the Moroccan guidelines in Table 7, arsenic (As) was not detected in either CM2 or CM3, ensuring compliance with the threshold of 0.1 mg/L. Lead (Pb) concentrations were 0.011 mg/L in CM2 and 0.013 mg/L in CM3, well below the permissible limit of 5 mg/L, indicating no significant risk. Copper (Cu) concentrations remained within the specified limit of 0.2 mg/L, with values of 0.1125 mg/L in CM2 and 0.1421 mg/L in CM3. Similarly, zinc (Zn) concentrations were 0.0813 mg/L in CM2 and 0.0458 mg/L in CM3, which remained far below the Moroccan limit of 2 mg/L. Although no specific limit was set for barium (Ba), the detected concentrations were 0.3544 mg/L in CM2 and 0.3804 mg/L in CM3, which are minimal, thereby reducing the risk of soil contamination.

The same reasoning applied to WHO standards regarding phase formation and MTEs immobilization also holds for irrigation water, as the stabilized crystalline structures effectively limit metal leaching, ensuring compliance with the Moroccan regulations. The sintering process plays an important role in this immobilization by incorporating MTEs into stable crystalline and glassy phases within the ceramic matrix, significantly reducing their leaching (51, 59). This strategy offers a sustainable solution for mine tailing management, transforming hazardous waste into safe ceramic products while minimizing the environmental impact (60, 61).

Conclusion

This study illustrates the effective valorization of lead-ore-processing tailings (LOPT) into ceramic tile formulations, providing both environmental and industrial advantages. The reuse of 10–20% LOPT in a ceramic mixture produced materials with superior physical and mechanical qualities. The CM2 and CM3 materials, categorized as Group BIIa according to the ISO 13006 (42) standard, demonstrated superior performance, with flexural strengths of 27.25 N/mm^2 and 26.16 N/mm^2, respectively, and minimal water absorption rates of 3.11% and 3.50%, respectively. The findings demonstrated a dense and cohesive microstructure, which was validated using SEM. The immobilization of metal trace elements (MTEs) was accomplished by the development of solid crystalline phases, including $AlAsO_4$, $PbSO_4$, $CuAl_2O_4$, $ZnAl_2O_4$, and $BaFe_2O_4$ during the sintering process. These phases efficiently immobilize metals, thereby diminishing their mobility and reducing their environmental hazards. The environmental safety of the produced ceramic materials was confirmed through leaching tests performed in accordance with the ISO EN 12457-2 (31) standard. The MTE concentrations in the leachates were substantially lower than the regulatory levels. Arsenic (As) was not detected in either CM2 or CM3, ensuring compliance with the WHO (0.01 mg/L) and Moroccan irrigation water standards (0.1 mg/L). Lead (Pb) was measured at 0.011 mg/L in CM2 and 0.013 mg/L in CM3, slightly exceeding the WHO limit of 0.01 mg/L but remaining well below the Moroccan irrigation threshold of 5 mg/L. Copper (Cu) was measured at 0.1125 mg/L in CM2 and 0.1421 mg/L in CM3, far below the WHO limit of 2 mg/L, and the Moroccan irrigation standard of 0.2 mg/L. Zinc (Zn) concentrations were 0.0813 mg/L in CM2 and 0.0458 mg/L in CM3, which were significantly lower than the WHO (3 mg/L) and Moroccan (2 mg/L) limits. Barium (Ba) levels were 0.3544 mg/L in CM2 and 0.3804 mg/L in CM3, well within the WHO guideline of 1.3 mg/L. These levels are far below the WHO limits for drinking water and correspond to Moroccan requirements for irrigation water. This strategy highlights the potential of LOPT as a secondary raw material for the production of high-performance, environmentally friendly ceramic tiles. To further strengthen these results, future research could focus on improving the formulations of ceramic tiles using LOPT as a secondary raw material, including studying the effects of firing temperature and cycle duration on the physical and mechanical qualities. Such investigations could provide greater insights into improving the performance and scalability of this strategy.

Abbreviations

CL1: Type 1 clay obtained from the Berrechid quarry in Morocco, CL2: Type 2 clay obtained from the Berrechid quarry in Morocco, ISO: International Organization for Standardization, LOPT: Lead Ore Processing Tailings, MTE: Metal Trace Element, SEM: Scanning Electron Microscopy, XRD: X-ray Diffraction, XRF: X-ray Fluorescence.

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Author Contributions

The authors contributed to data analysis and writing of the manuscript for this study.

Conflict of Interest

The authors declare that they have no conflict of interest.

Ethics Approval

Not applicable.

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