

## Article

# Manufacture and Characterization of Fired Bricks from Gold Mine Waste Rocks

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**Abstract:** The purpose of this research was to evaluate the possibility of using gold mine waste rocks (GMWRs) as alternative raw material for the manufacturing of fired bricks. The feasibility study was assessed through (i) physical, chemical, mineralogical and environmental characteristics of GMWRs; (ii) determination of the natural clay (NC) substitution effect when using GMWRs; (iii) the effect of the firing temperature on the mechanical and physical properties of the fired bricks. Five mixtures of NC and GMWRs were studied. The percentages of substitution of NC with GMWRs varied from 0 to 100%. The brick specimens were fired at 900 °C, 1000 °C and 1050 °C. The results show that increasing the firing temperature improved the flexural strength and density of the bricks, while the substitution of NC with GMWRs caused a reduction in the mechanical resistance of the bricks and an increase in their porosity and, consequently, their water absorption rate. However, the properties of bricks that contained up to 80 wt% of GMWRs and fired at 1000 °C and 1050 °C satisfied the requirements set by the applicable civil engineering and environmental standards. This was found to be an efficient and sustainable solution to mitigate environmental hazards and better manage mining wastes, concurrently producing marketable products from them, which is in accordance with the circular economy concept.

**Keywords:** valorization; mining waste rocks; fired bricks; environment; clays; mechanical properties



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## 1. Introduction

The generation of mining waste rocks is an inevitable consequence of the mining industry. It constitutes an inseparable part of the ore extract and treating process [1]. The management of mining wastes is becoming a world concern due to: (i) their high amount, which is estimated to be between 20 and 25 billion tons of solid waste each year [2]; (ii) their environmental impact [3]; (iii) their expensive restoration costs [4]; (iv) and potential human health risks [5–7]. The implementation of both efficient and sustainable solutions to better manage this waste is therefore becoming a more serious priority. Recently, a lot of research has been conducted to find and develop alternative solutions for the reuse of these mining wastes [8–15], especially as alternative secondary raw materials in the construction sector [16–22]. The aim is to reduce the huge quantity of waste, its environmental impact, and to contribute to the conservation of non-renewable material resources for future generations.

On the other hand, the new environmental regulations insist on efficient and sustainable management of all types of wastes [23,24]. Consequently, all the mining companies

are trying to manage their wastes, either by reducing their production upstream, or by recovering/recycling/reusing these wastes if possible, or both.

In the literature, many economic and environmentally friendly solutions were shown to be effective and safe for reusing mining waste as alternative raw materials. The aim is to manufacture building and construction materials from waste, such as fired bricks, ceramics, lightweight aggregates, concrete, mortar, geopolymers and road construction. For example in Morocco, Taha et al. [25–27] showed that fired bricks with properties similar to conventional ones can be produced by substituting 30% of natural clay with calamine waste and 100% of coal waste. Loutou et al. [28] and Bayoussef et al. [29] produced high-performance ceramics and lightweight aggregates using phosphate sludge and clay by-product from phosphate mines. Argane et al. [30–32] and Elmachi et al. [33] used mining wastes as alternative fine sands for mortar and aggregates for concrete. Cementitious materials, including geopolymers and ecological cement, were developed using phosphate mining by-products [34,35]. Through an experimental study, Amrani et al. [36] investigated the possibility of phosphate wastes disposal in the road construction field. The results of this study confirmed that the obtained physical, geotechnical, chemical, mineralogical and environmental properties of wastes satisfy the conventional characteristics to be used as materials for embankments. Therefore, phosphate waste can be used successfully in the construction of roads by replacing natural aggregates.

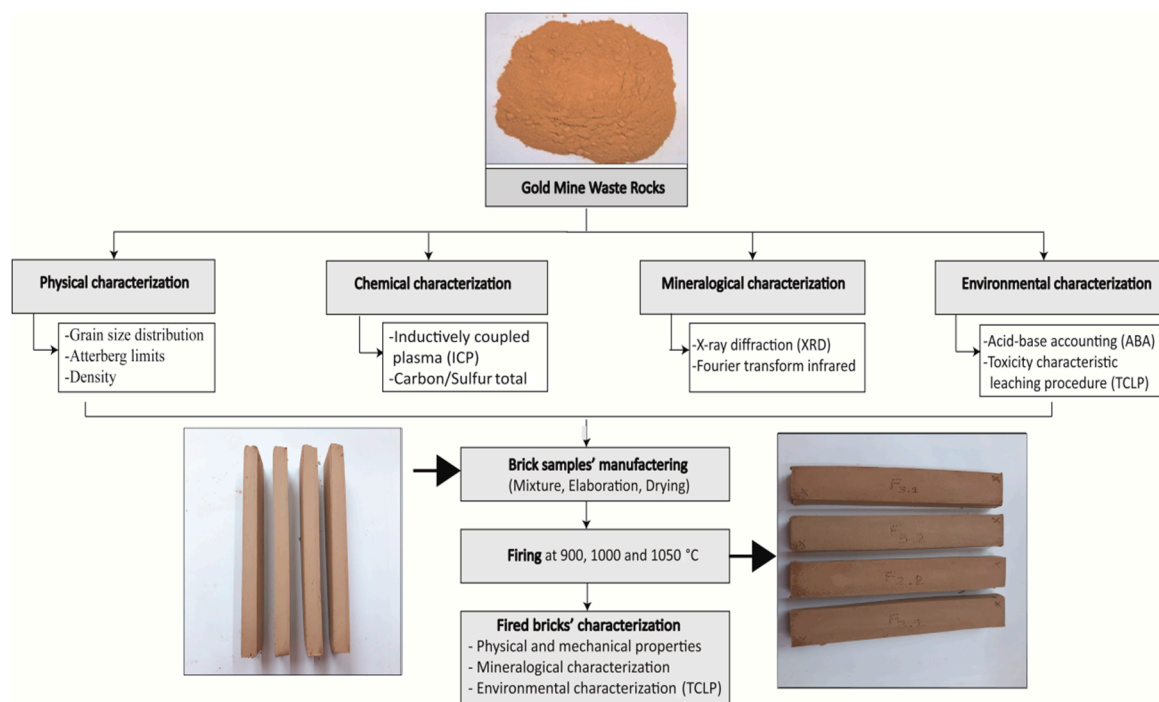
Internationally, Contreras et al. [37] demonstrated the feasibility of integrating “ilmenite mud” waste in the fabrication of ceramic tiles. The results of their study showed that the use of mud as an additive (3–10%) can improve the sintering properties, increase the mechanical strength (about 15%), and reduce the apparent porosity and water absorption (about 50%). Onuaguluchi et al. [38,39] evaluated the incorporation of copper residues in mortar formulation. The obtained samples provided high resistance to compression, abrasion, acid attack and penetration chlorides compared to a reference mortar. Thomas et al. [40] studied the feasibility of using copper tailings as a substitute for natural sand for concrete production. In their study, concrete samples were formulated by using copper tailings as fine aggregates (0 to 60%). The results showed that concrete mixtures made with a 60% substitution rate present high mechanical strength and satisfactory durability. Chen et al. [41] investigated the potential of producing bricks by using hematite residues. It was observed that the proportion of 84% hematite residues can provide high-quality bricks that satisfy the required characteristics in China according to the conventional standards. Ali Umara Shettima Shettima et al. [42] evaluated iron ore tailing (IOT) as a replacement for river sands in concrete. Five concrete mixtures were formulated with different mass ratios of 0, 25%, 50%, 75% and 100% of IOT as sand and using a 0.5 water/cement ratio (W/C). The results revealed that the compressive strength of the fabricated samples was improved in comparison with the reference concrete sample no matter what the considered age was. Therefore, using IOT is recommended for concrete as a sand replacement. In the same approach, Benarchid et al. [43] assessed the reuse of low-sulfide mining waste rocks as aggregates for concrete according to recycling guidelines of non-hazardous inorganic waste in Quebec, Canada. The obtained results of the compressive strength test revealed the feasibility of manufacturing concrete using low-sulfide mining waste.

In the same context, this study was conducted to evaluate the feasibility of producing fired bricks using GMWRs from the Agadir region, Morocco. The choice of this valorization process was based on the characteristics of the wastes and especially the biggest challenge of reducing the transport costs for waste producers and potential consumers. The aim of this research was to partially substitute the natural clays (NC) used in the fabrication of fired bricks with the gold mine waste rocks (GMWRs) from Agadir region, Morocco. The obtained specimens were sintered at different temperatures (900 °C, 1000 °C and 1050 °C). Further to this, the mechanical, physical, mineralogical and environmental characteristics of the specimens (flexural strength, water absorption, density, porosity and toxicity characteristic leaching procedure (TCLP) test) were evaluated.

## 2. Materials and Methods

### 2.1. Materials and Research Methodology

Representative samples of gold mine waste rocks were sampled from a gold mining site located in the Agadir region, Morocco. The GMWR sample was crushed and ground according to the desired granulometry for brick preparation. The natural clay that is commonly used in the production of high-quality fired bricks was also sampled in the same region. The raw materials were prepared for further characterizations and processing into fired bricks through the methodology described in Figure 1.



**Figure 1.** Summary of the followed methodology.

### 2.2. Characterization Methods

The physical properties, chemical and mineralogical composition and thermal and environmental behavior of the NC and GMWRs were determined through laboratory tests, in accordance with the French Association for Standardization (AFNOR) and the American Society for Testing and Materials (ASTM) standards. The toxicity characteristic leaching procedure (TCLP) was used to determine the mobility of heavy metals in the fired bricks made from GMWRs. The thermogravimetric analysis was performed in an air atmosphere with 10 °C/min as the heating rate to analyze the thermal behavior of the raw materials.

For the X-ray diffraction (X'pert Philips, PANalytical X'Pert-Pro, 2009 vintage, Nottingham, UK), Cu K $\alpha$  radiation was used to identify the crystalline phases in the NC, GMWRs and fired samples. The chemical composition of the raw materials was analyzed using inductively coupled plasma with atomic emission spectroscopy (ICP–AES) (Perkin Elmer Optima 3100 RL, PerkinElmer, Waltham, MA, USA) following a total HNO<sub>3</sub>/Br<sub>2</sub>/HF/HCl digestion, with a detection limit that varied depending on the analyzed element.

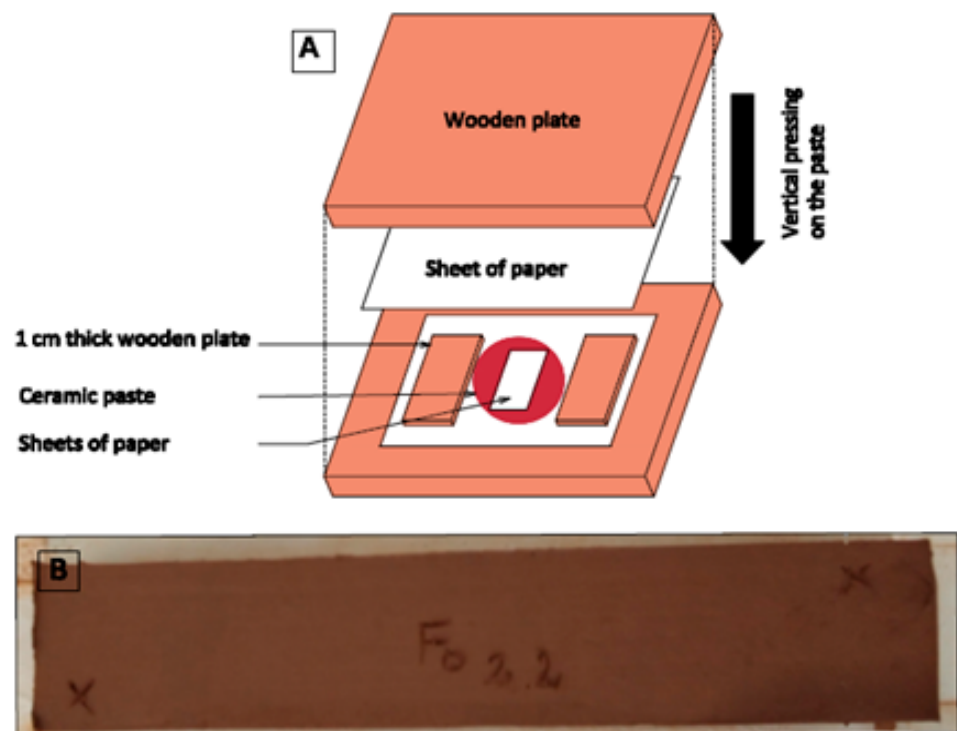
The carbon (C) and sulfur (S) contents were determined using LECO equipment with a detection limit of  $\pm 0.05$  wt%.

A laser analyzer (Malvern Mastersizer 2000 Ver. 5.12F, Malvern Instruments Ltd., Malvern, UK) was used to determine the particle size curve of each sample. The specific gravity (GS) and bulk density were measured following the NF EN 1097-6 (pycnometer method) and NF EN 1097-3 standards, respectively. The plasticities of the NC and GMWRs were assessed using Atterberg limits according to NF P 9405 1. During the analysis, three

replicates of each analysis were performed. The results presented in the figures represent the average of the values.

### 2.3. Manufacture of Laboratory Brick Samples

In this study, the approach adopted is described in Figure 1. First, the raw materials (NC and GMWRs) were prepared and homogenized to formulate mixes with different portions of GMWRs (0, 20, 40, 60, 80 and 100 wt%). The formulations were mixed with an adequate amount of water and rectangular bricks with dimensions of 10 mm × 20 mm × 100 mm were formed, employing a conventional method used by potters and adopted by Geoenvironment and Civil Engineering Laboratory (L3G) of the Faculty of Sciences and Techniques of Marrakech [44]. Figure 2 presents the device used to produce the brick specimens. Two small wooden plates with a thickness of 1 cm served as the bilge during pressing so that all the bricks were of the same thickness. After pressing, the wooden plate was superimposed on paper of the same size. The base area of the wood plate corresponded to that of the brick to be prepared. The brick was then cut with a sharp blade using the wooden plate as a model.



**Figure 2.** (A) Device for producing test pieces using the plate method. (B) Example of a specimen with two reference crosses for measuring the shrinkage.

The brick samples were air-dried at ambient temperature for 48 h, and then fired at the three temperatures—900, 1000 and 1050 °C—using a muffle furnace, with 30 °C/h as the heating rate and held for 3 h at the required temperatures.

### 2.4. Fired Bricks' Characterization

To evaluate the influence of the incorporation of GMWRs in fired clay bricks, the technological properties were determined. Four brick samples per mixture were tested. Flexural strength was measured using a SYNTAX testing machine (SYNTAX 100 kN and 300 kN, 3R company, Montauban, French) following the ASTM C 674 (1999) standard [45]. The density, porosity and water absorption of the bricks were determined according to ASTM-C373 (1999) [46].

Finally, the leaching of heavy metals in the fired bricks was assessed using the TCLP test, following the Environmental Protection Agency (EPA) method 1311 [47] and the MA-100-Lix.com.1.1 standard (2010) [48].

### 3. Results and Discussion

#### 3.1. Raw Materials Characterization

##### 3.1.1. Physical, Chemical and Mineralogical Properties

Table 1 summarizes the different physical and chemical properties of the raw materials used in this research. Figure 3 illustrates the grain size distribution of the raw materials. The NC and GMWR samples show a relatively similar grain size distribution, with the proportion of particles between 20 μm and 70 μm greater in the GMWRs. Furthermore, they presented other similar physical properties, such as bulk density (1.5 g·cm<sup>-3</sup>) and specific gravity (2.6 g·cm<sup>-3</sup>). The plasticity index of the GMWRs was lower than the one of the NC. This was due to the amount of clayey materials in the NC compared to those in the GMWRs.

Table 1. Physico-chemical properties of the raw materials.

Chemical Compositions (%)			Physical Properties			Mixture Designs (%)		
DL *	NC	GMWRs		NC	GMWRs		NC	GMWRs
SiO <sub>2</sub> 0.1	50.8 ± 1.5	43.26 ± 1	Moisture (%)	10.4	11.5	F0	100	0
Al <sub>2</sub> O <sub>3</sub> 0.1	16.4 ± 1	10.13 ± 0.5	Plasticity limits (%)	25	19	F20	80	20
TiO <sub>2</sub> 0.1	1.1 ± 0.7	1.1 ± 0.2	Plasticity index (%)	27	12	F40	60	40
Fe <sub>2</sub> O <sub>3</sub> 0.1	5.9 ± 0.9	8.59 ± 1				F60	40	60
MnO 0.01	0.1 ± 0.05	0.156 ± 0.02				F80	20	80
MgO 0.1	3.5 ± 0.5	2.65 ± 0.2	Specific gravity (g·cm <sup>-3</sup> )	2.6	2.61	F100	0	100
CaO 0.1	8.5 ± 1	11.23 ± 1.2	Bulk density (g·cm <sup>-3</sup> )	1.5	1.5			
Na <sub>2</sub> O 0.03	0.3 ± 0.01	–						
K <sub>2</sub> O 0.01	3.0 ± 0.1	1.19 ± 0.3	LOI (%)	11.5	21.53			
P <sub>2</sub> O <sub>5</sub> 0.1	–	0.16 ± 0.01						

\*: detection limits.

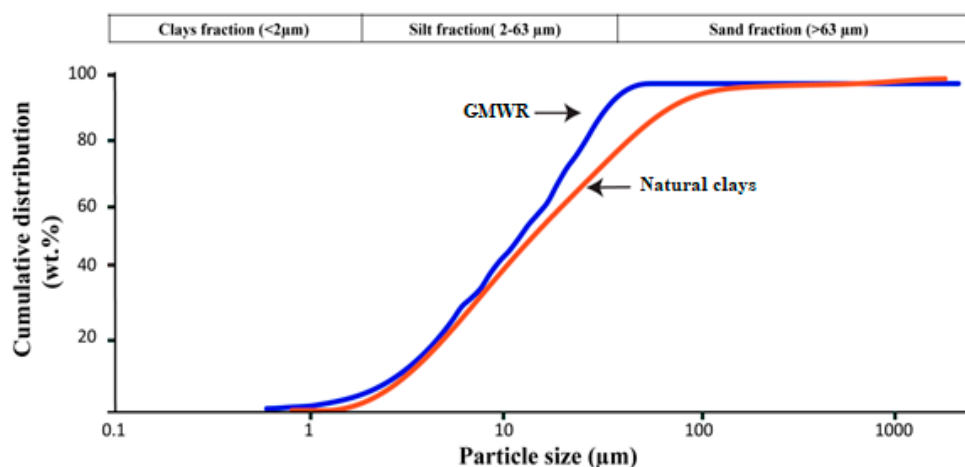
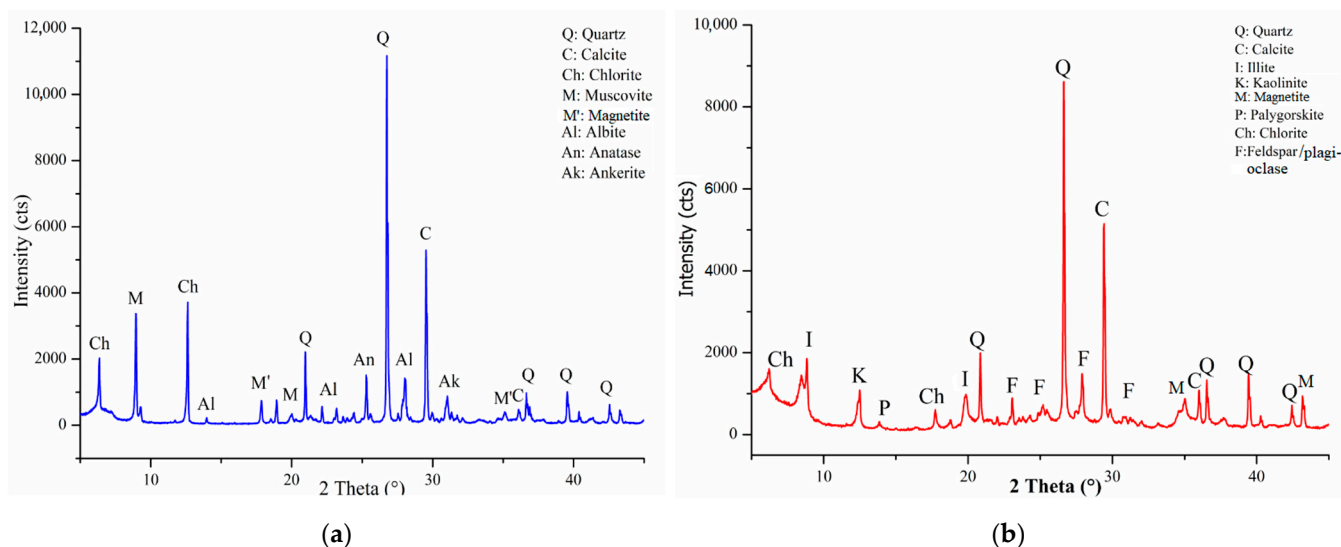


Figure 3. Particle size distribution of the GMWRs and NC.

The chemical compositions of the NC and GMWRs identified using ICP–AES, and summarized in Table 1 show that silica SiO<sub>2</sub> was the main component of both materials

with 51 wt% and 43 wt%), alumina (16 wt%, 10 wt%), CaO (8.5 wt%, 11 wt%) and iron oxide (5.9 wt%, 8.59 wt%). The CaO richness for solid GMWRs was confirmed by a considerable loss of ignition (21.5 wt%). Moreover, the existence of carbonates in the raw materials contributes to stabilize the acid generation phenomena.

The mineralogical composition of the GMWRs and the NC are presented in Figure 4. From the obtained result, the GMWRs were mainly dominated by quartz, calcite, albite and muscovite, while magnetite and chlorite were present as minor phases. The NC (Figure 4b) was composed of quartz, calcite, clays minerals (illite and kaolinite), magnetite and feldspar as minor phases. In comparison to the NC, the GMWRs were poor in clay minerals, which explains the lower value of the plasticity index (11.5%).



**Figure 4.** The X-ray patterns of gold mine waste rocks (GMWRs) (a) and natural clay (NC) (b).

Depending on the chemical composition of the GMWRs, its ability to be used in the manufacture of fired bricks was evaluated using the ternary diagram in Figure 5. This diagram was developed based on a literature synthesis of the chemical compositions of natural clays used for the fabrication of fired bricks. The diagram in Figure 5 shows the difference in chemical composition between GMWRs and the NC. The composition of the GMWRs was close to that of natural clays. Based on the results obtained, the GMWRs showed a high probability to be successfully used in the fired bricks industry. Thus, this study was conducted to evaluate the feasibility of using GMWRs as a substitute for natural clays according to the applicable civil engineering and environmental standards.

### 3.1.2. Thermal Behavior of Raw Materials

Figure 6 presents the thermogravimetric analysis (TGA) curves for the NC and GMWRs up to 1050 °C. The total weight loss of the GMWRs was 16.57%. Three main stages of weight loss steps are illustrated. The first loss in the 40–210 °C range could have been related to the loss of moisture and the dehydration of clay minerals (loss of hygroscopic and crystal water). The second loss in the 230–334 °C range was related to the dehydroxylation of iron hydroxides and the evolution of anhydrite III to anhydrite II. The last and greatest weight loss (13.94%) occurred in the 550–800 °C range and was associated with the degradation of the calcium and magnesium carbonates found in the GMWR.

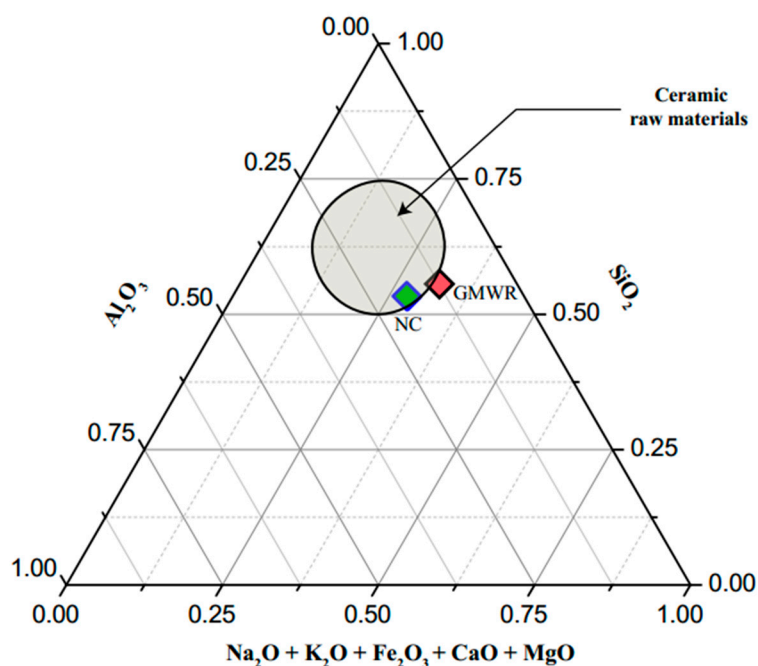


Figure 5. Representation of the studied samples in the ternary diagram of SiO<sub>2</sub>–Al<sub>2</sub>O<sub>3</sub>–fluxing oxides showing the zone of suitable materials for fired bricks based on [49].

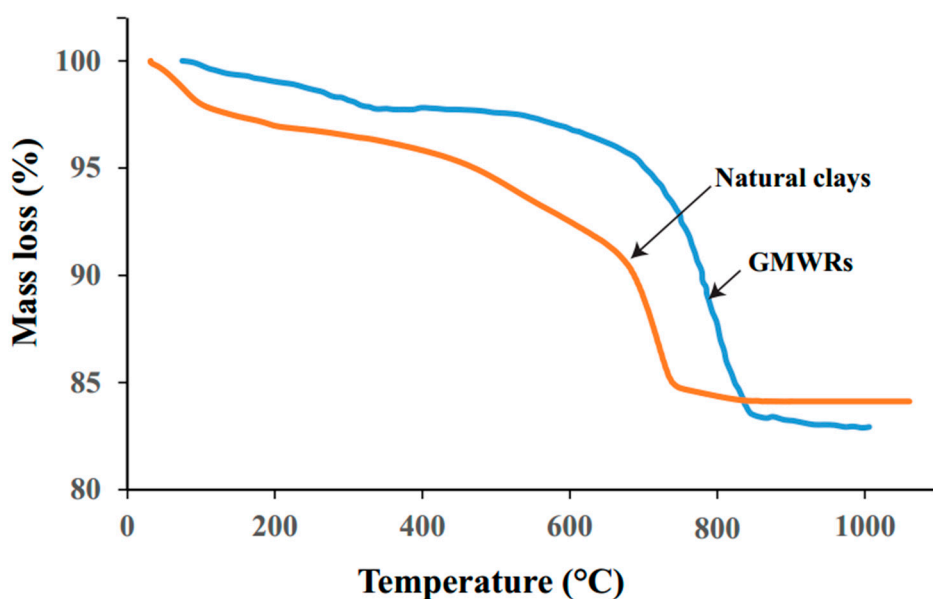


Figure 6. Thermal analysis (TGA) of the NC and GMWRs.

The TGA curve of the NC shows four main weight loss steps. The first one was centered at 200 °C and represents the dehydration of moisture and intercalated water, the second loss between 400–600 °C corresponded to the vaporization of the adsorbed water and the combustion of organic substances, followed by the dihydroxylation of the clay minerals between 600 °C and 750 °C. The last weight loss between 750 °C and 850 °C was attributed to the destruction of carbonates.

### 3.1.3. Evaluation of Acid Generation Potential

The prediction of the environmental behavior of the GMWR samples was performed through the static test called acid–base accounting (ABA) [50,51], which provides important

information to decide on the nature of the acidity of the mining wastes and therefore on their potential risk to the environment.

The overall principle of this test is to take a balance sheet of the acid potential of generation (AP) and the neutralization potential (NP) of the selected materials. The AP and NP are determined from the total sulfur and carbon contents existing in the solid wastes. It should be mentioned that these contents are specified in kg equivalent of calcite per ton of material (kg CaCO<sub>3</sub>/t).

Table 2 summarizes the values of the total carbon and sulfur in the GMWR samples. The total sulfur and inorganic carbon contents were analyzed using an induction furnace (ELTRA CS-2000, ELTRA ELEMENTAL ANALYZERS, Stevensville, MI, USA) with a detection limit = 0.05 wt%.

**Table 2.** Test acid–base accounting (ABA) of GMWR samples.

Sample	C%	S%	NP	AP	NNP	NP/AP
GMWR	2.28	0.14	189.9	4.37	185.53	42.45

The authors did not evaluate the presence of sulfides/sulfates in the GMWRs in detail as the S content was very low. Therefore, the AP was calculated assuming that all the sulfur occurred as pyrite that will oxidize and generate acidity.

The AP value was determined from the sulfide content according to the following formula:

$$AP = 31.25 \times S_{\text{total}} (\%) \quad (1)$$

where:

AP: acidity potential (kg CaCO<sub>3</sub>/t of material);

S<sub>total</sub> (%): total sulfur weight content (%);

31.25: conversion factor 1000 kg/t × M(CaCO<sub>3</sub>)/100% × Ms.

However, there are various variants to NP determination. Each of these variants is carried out under different conditions leading to different interpretations. This is especially true for materials whose generating nature is less obvious, i.e., they are neither highly acid generating nor highly acid consuming [52].

In this study, the NP was determined with the standard carbonate NP method, which consists of measuring the total inorganic carbon with an induction furnace and converting the value into kg CaCO<sub>3</sub>/t equivalents. This method is simple and widely used in the industry [53].

The CNP was evaluated assuming that the C<sub>total</sub> (wt%) measured is totally present in carbonates (C<sub>inorg</sub>). The results were then converted to calcite equivalents (kg CaCO<sub>3</sub>/t) by multiplying by a factor of 83.33:

$$CNP = \%C_{\text{inorg}} \times 83.33 \quad (2)$$

CNP: carbonate neutralization potential (kg CaCO<sub>3</sub>/t of material);

%C: carbon weight content (%);

83.33: conversion factor.

Finally, to know the nature of the material (generator or consumer of acid), we compared the values of AP and NP found with the static ABA test. The difference between these two values gives the net neutralizing potential (NNP). When the NNP is less than −20 kg CaCO<sub>3</sub>/t, it indicates an acid-producing material, whereas materials with an NNP greater than 20 kg CaCO<sub>3</sub>/t are considered to be acid consumers. However, there is an area of uncertainty for this technique between 20 > NNP > −20 kg CaCO<sub>3</sub>/t, as indicated by Skousen et al. [54]. Another useful tool for assessing the potential for acidity production is from NP/AP ratio results. In general, Materials are considered not to be acid generators if the NP/AP ratio is superior to 2.5. However, when the NP/AP ratio is less



than 1, they are considered acid generators. Materials providing an NP/AP ratio ranging between 1 and 2.5 are in an area of uncertainty.

The results presented in Table 2 show that the GMWR sample contained very low sulfur content, the net neutralization potential (NNP) was positive and higher than 20 kg CaCO<sub>3</sub>/t and the NP/AP ratio was above 42.45. From these results and Figure 7, this shows the variation of NNP as a function of AP and the no-acid-, uncertain and acid-generation zones, it appears clearly that the GMWR samples were located in the no acid generation zone.

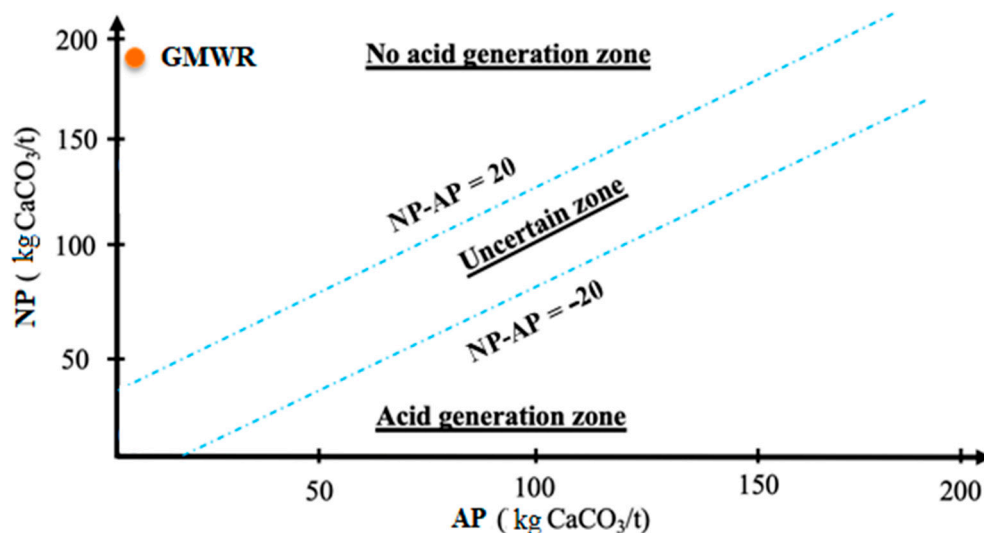


Figure 7. Illustration of the location of GMWR samples.

### 3.2. Detailed Fired Bricks Properties

#### 3.2.1. Physical and Mechanical Properties

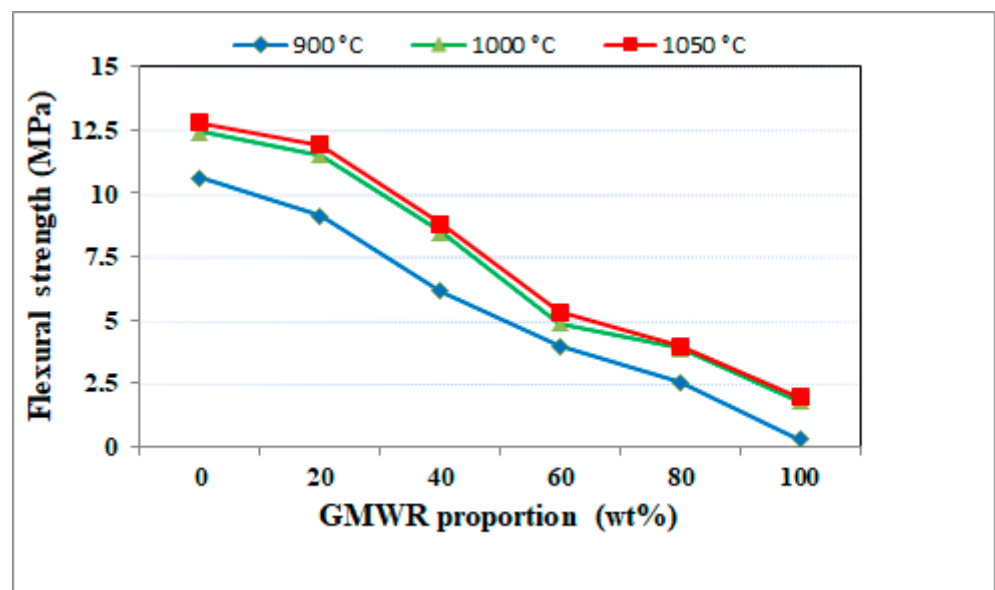
In order to investigate the ability to use GMWRs in the manufacture of fired bricks, they were qualified in terms of their mechanical, physical and mineralogical properties. The obtained results were compared to the control specimen and the required engineering standards. Figure 8 illustrates the behavior of the technological properties of the fired bricks as a function of the proportion of waste and the firing temperature. Generally, no significant difference was observed for the values of various characteristics of the fired bricks sintered at 1000 and 1050 °C. The results derived from each experimental analysis were taken as the average of four measurements for each brick mixture.

According to the results, it is noteworthy that the bricks containing 100% GMWRs and fired at 900 °C developed significant cracks. This caused the deterioration of the bricks in water. Consequently, their density, water absorption and porosity could not be determined.

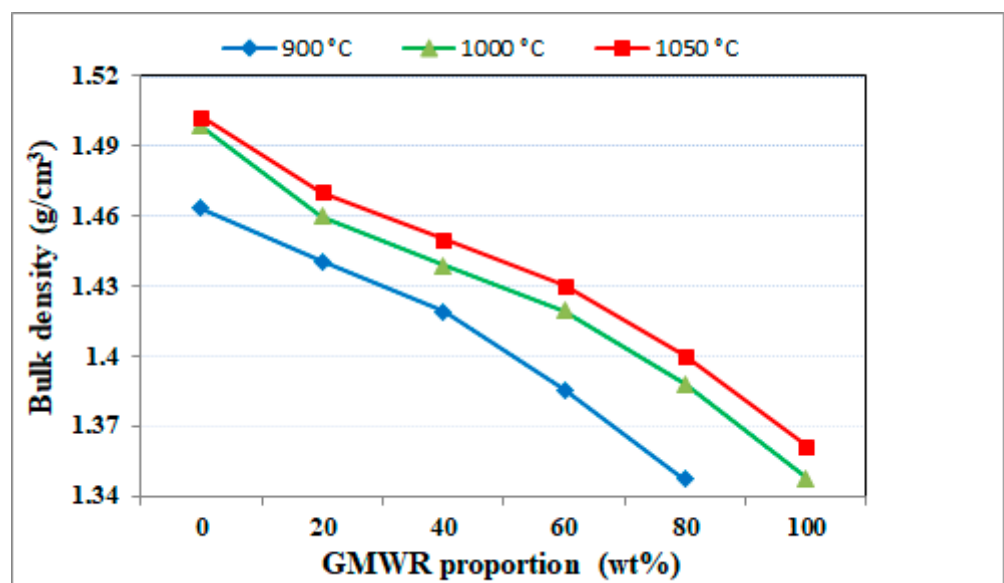
As shown in the results, the incorporation of GMWRs and the firing temperature significantly affected the mechanical and physical properties of the specimens (strength, density, water absorption and porosity). The incorporation of GMWRs caused a decrease in the flexural strength and density. On the other hand, porosity and water absorption were affected differently and tended to be enhanced.

The flexural strength of the produced bricks is presented in Figure 8a. This parameter is considered as the main quality indicators required by building standards, which allows for the assessment of the suitability of the material for use in the construction industry. Figure 8a shows the influence of the amount of GMWRs and the firing temperature on the flexural strength. As shown, the flexural strength of bricks decreased with the increasing proportion of GMWRs. The results show that up to an 80% GMWR mix, the strength of the bricks was greater than 2.5 MPa, which represents the minimum strength specified by the standards. Furthermore, at 1050 °C, the flexural strength values varied between 12.77 and 3.96 MPa, when GMWR addition increased from 0 to 80%, respectively. The

decrease observed in the flexural strength with the addition of GMWR was due to the main source of reductions in mechanical properties, namely, the open porosity, which increased with GMWR addition. On the other hand, the flexural strength of specimens slightly increased as the firing temperature increased from 900 to 1050 °C. It went from 10.62 to 12.77 MPa for the 0% reference brick and from 2.53 to 3.96 MPa for F80. This improvement in flexural strength was attributed to the improvement in the consolidation or vitrification processes when the firing temperature was increased. Bricks produced using GMWRs alone (100%) presented poor mechanical properties, where the flexural strength was less than 2.5 MPa. Therefore, we can conclude that GMWRs cannot be used alone to manufacture high-quality fired bricks. The acquired results are an average of the measurements performed on four specimens.

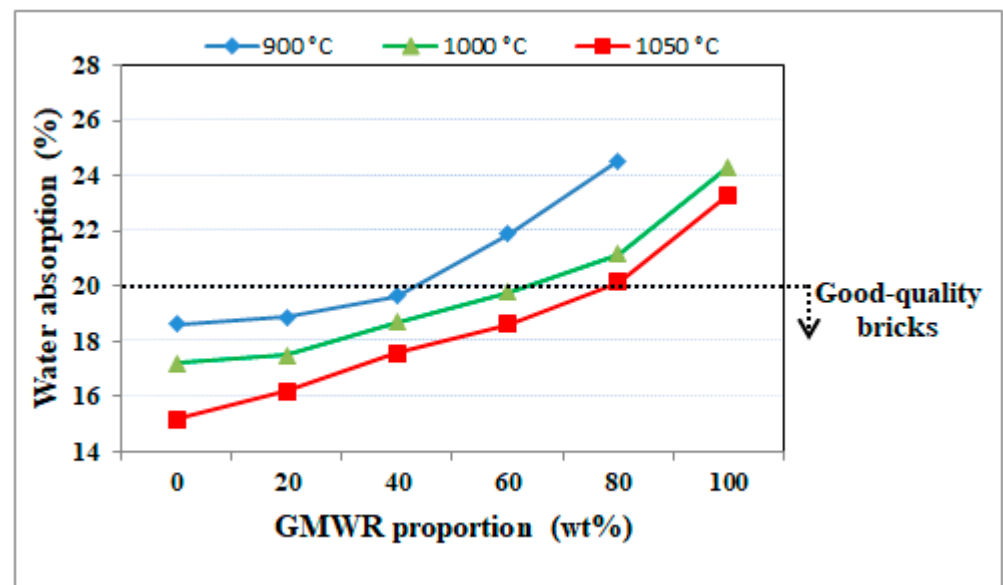


(a)

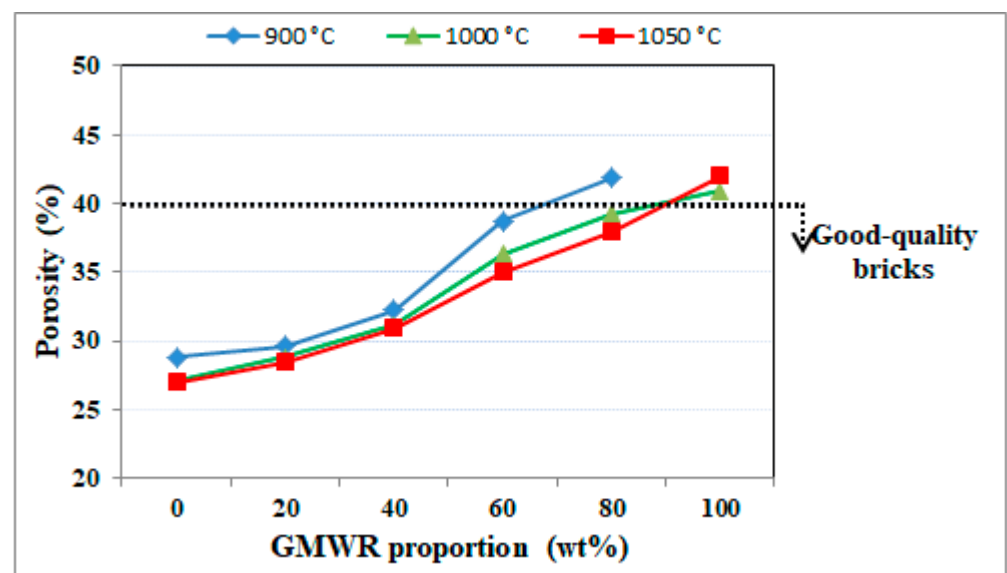


(b)

Figure 8. Cont.



(c)



(d)

**Figure 8.** The evolution of mechanical and physical properties of the fired bricks incorporating up to 100% GMWRs. (a) flexural strength; (b) bulk density; (c) water absorption; (d) porosity.

The bulk density results show a behavior similar to that of flexural strength, decreasing with GMWR addition and increasing slightly with the rising firing temperature. The decrease in bulk density with the incorporation of GMWRs at different firing temperatures can be explained by the increase in the volatile matter in the brick mixtures. This result is supported by the high porosity values of the elaborated bricks, which was the main contributor to the reduction in the strength of the bricks.

The results of water absorption and porosity are presented in Figure 8c,d. In general, the water absorption was related to the accessible open porosity of the fired bricks. A high number of open pores led to high rates of water absorption. The open porosity and water absorption rates increased significantly with the increase in GMWR addition and decreased slightly with the rising firing temperature. As an illustration, at 1050 °C, the porosity improved by about 31% from 27% to 39% when 80% of the NC was replaced with GMWRs; however, the increase in firing temperature from 900 to 1050 °C reduced the porosity of

bricks F0 and F80 from 28.82% and 41.84% to 26.98% and 37.93%, respectively, roughly 6% and 9% for the bricks, respectively. Moreover, the values of water absorption presented in Figure 8c confirmed the close relationship between porosity and water absorption. A similar trend was observed in these parameters. Therefore, at 1050 °C, the water absorption rates of elaborated bricks increased from 15.20% to 20.1% when 80% of the NC was substituted with GMWRs. The increase in the water absorption and porosity values of the bricks with the incorporation of a high percentage of GMWRs can be explained by the degradation of the carbonate minerals contained in the GMWRs. This decomposition caused the release of gases and subsequently greater porosity in the fired specimens. This observation was confirmed by the thermogravimetric analysis (LOI = 16.57 wt%).

Figure 9 highlights the inter-relation of different properties of bricks fired at 1050 °C. It is interesting to mention that a good relationship existed between water absorption and porosity Figure 9c ( $R^2 = 0.97$ ), open porosity and bulk density Figure 9a ( $R^2 = 0.97$ ), flexural strength and bulk density Figure 9d ( $R^2 = 0.94$ ), and flexural strength and open porosity Figure 9b ( $R^2 = 0.97$ ). These findings confirmed the preceding conclusions from the standardized tests, namely, the decline in flexural strength when open porosity increased and the increase in water absorption with the open porosity growth, which was related to the amount of volatile matter.

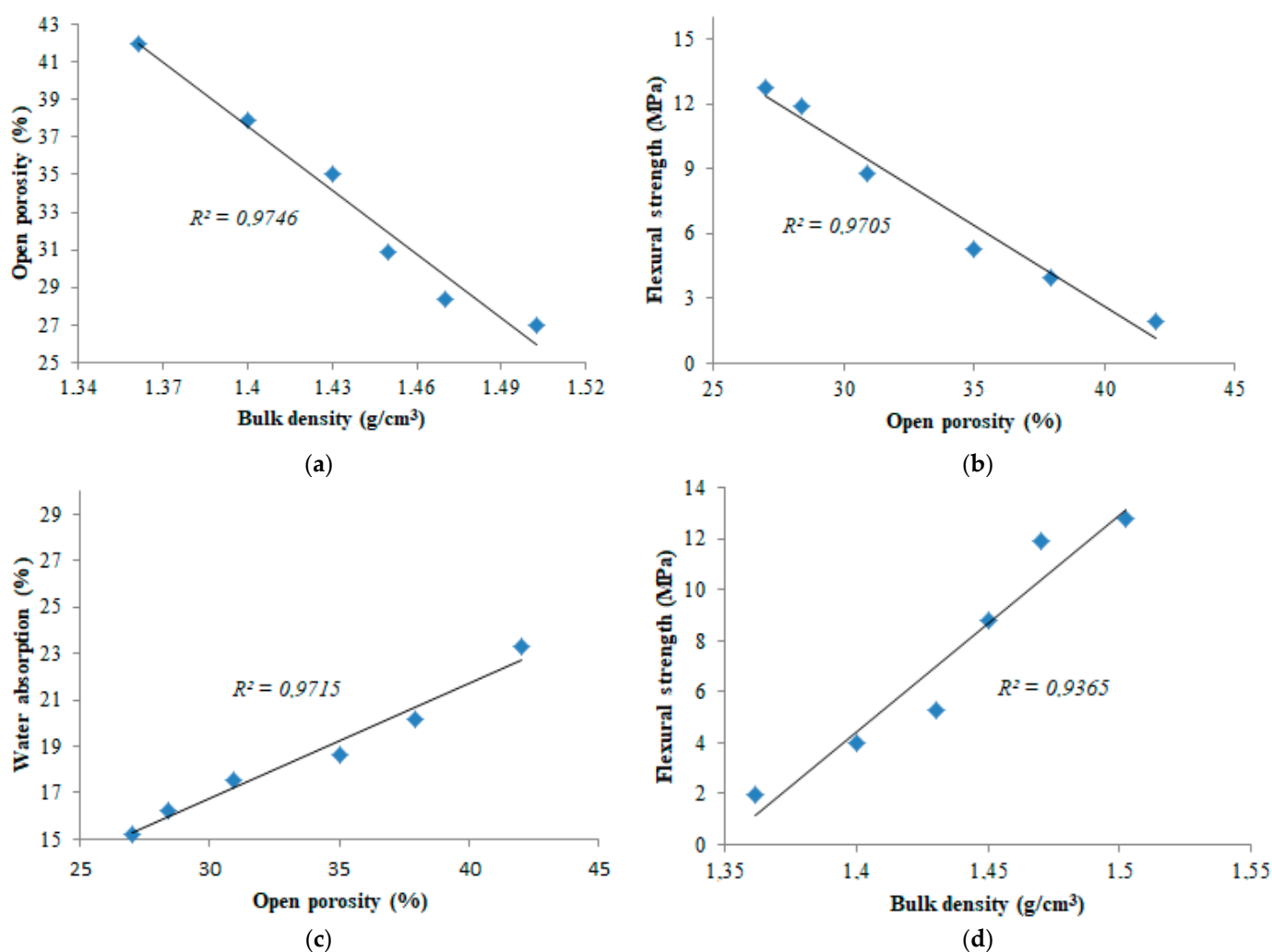


Figure 9. Correlation and linear regression of the different properties of bricks fired at 1050 °C.

### 3.2.2. Mineralogical Characterization of Fired Bricks

XRD analysis of the fine powders of the fired bricks was performed in order to investigate the mineralogical composition of the bricks prepared using GMWRs. Figure 10 illustrates the principal crystalline phases that were formed during the firing process. As can be seen, new crystalline phases were developed in the process of firing, with GMWR additions, such as akermanite, anorthite and muscovite. The formation of akermanite ( $\text{Ca}_2\text{Mg}[\text{Si}_2\text{O}_7]$ ) was the result of contact metamorphism of calcium, magnesium, silicon and oxygen. It was also observed that there was a decrease in quartz pic when increasing the sintering temperature (Figure 10b); this finding was confirmed by the increase in anorthite, akermanite, and albite pics.

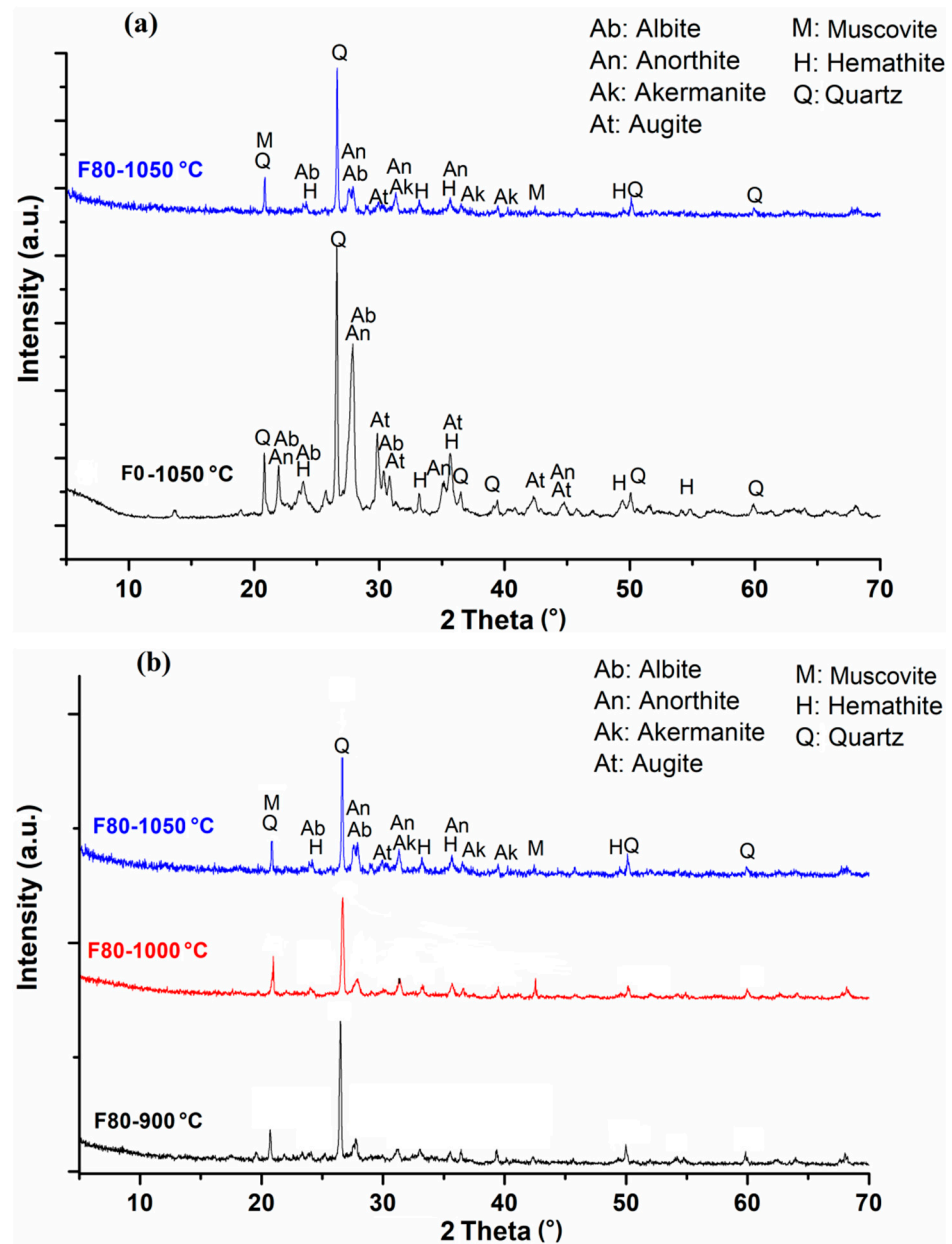


Figure 10. Principal crystalline phases in the (a) F0 and F80 mixtures fired at 1050 °C and in the (b) F80 mixture fired at different temperatures.

### 3.2.3. Environmental Behavior of Fired Bricks

The toxicity characteristic leaching procedure (TCLP) test was carried out to assess the environmental behavior of the fired bricks. The TCLP test was performed following the protocol adopted by the US Environmental Protection Agency (EPA) [35]. The aim is to simulate the leaching of heavy metals from fired bricks at their end life (as demolition wastes). The principle is to compare the concentrations of certain contaminants in the leachate from the TCLP test, with the limit concentrations (for each element) defined by the same agency (EPA). It consists of extracting the contaminants present in the sample to be analyzed (mine waste or aggregates (obtained by crushing or grinding)) using an acetic acid solution whose pH was determined previously by a pre-test. The pH values tested using this test were 2.88 or 4.93. The solid:liquid ratio was 20:1. The samples were put in closed bottles and agitated for  $18 \pm 2$  h in a rotary agitator with a speed of 30 rpm. The leachates were then filtered ( $0.45 \mu\text{m}$ ), acidified and analyzed using ICP–AES.

In this study, 20 g of each sample was added to 400 mL of a leaching solution (pH of  $4.93 \pm 0.05$ ). Each mixture was then placed in a high-density polyethylene (HDPE) bottle, and the bottles were tumbled for 18 h at  $30 \pm 2$  rpm. Samples were filtered through a  $0.45 \mu\text{m}$  nylon filter and the leachates were analyzed by ICP–AES.

Table 3 summarizes the compared parameters between the obtained results of test and the US EPA thresholds. The results show that all concentrations of F80 were under the regulatory limits fixed by the US EPA. Therefore, the studied samples were considered non-hazardous waste. On the other hand, it was observed that the mobility of As in the F100 mixture (100% waste) exceeded the limit defined by the Agency, while this concentration was under the limit for the F80 mixture. This result confirmed that this waste cannot be used alone to produce high-quality fired bricks.

**Table 3.** Results of TCLP test of GMWR raw material and F0, F100 and F80 fired bricks at 1050 °C.

Sample	As	Ba	Cd	Cr	Cu	Mo	Pb	Zn
	$\mu\text{g}\cdot\text{L}^{-1}$	$\mu\text{g}\cdot\text{L}^{-1}$	$\mu\text{g}\cdot\text{L}^{-1}$	$\mu\text{g}\cdot\text{L}^{-1}$	$\mu\text{g}\cdot\text{L}^{-1}$	$\mu\text{g}\cdot\text{L}^{-1}$	$\mu\text{g}\cdot\text{L}^{-1}$	$\mu\text{g}\cdot\text{L}^{-1}$
GMWRs	86.74	2612.13	ND	43.14	212.62	ND	231.92	243.4
F0	3.66	114.14	0.22	10.56	10.7	ND	ND	280.48
F100	8014.33	15.94	0.28	291.87	1.66	25.33	ND	6.6
F80	2741.54	18.51	0.3	251.24	1.17	16.48	ND	8.04
Limits (US EPA)	5000	100,000	1000	5000	-	-	5000	2000

## 4. Conclusions

The objective of this experimental study was to assess the possibility to use GMWRs from the Agadir region, Morocco, as a secondary source and alternative raw material for manufacturing fired bricks. From the results obtained in this study, the following conclusions could be drawn:

- The chemical and mineralogical compositions of GMWRs show compatibility with NCs commonly used in the fabrication of fired bricks. The clay minerals (chlorite) contained in the GMWR favor their reuse in the bricks production.
- The obtained results show that the GMWR cannot be used alone to make good-quality fired bricks.
- The integration of GMWRs in the matrix of fired bricks as secondary raw material led to an increase in water absorption and porosity rates, which resulted in a decrease in flexural strength and bulk density.
- Laboratory bricks with up to 80% GMWRs and fired at 1000 °C and 1050 °C met the requirements of current construction standards. They met the mechanical requirements (ASTM-C674), which require a flexural strength higher than 2.5 MPa. They also

were in agreement with the ASTM C373 standards, which require a porosity rate of less than 40% and water absorption of less than 20%.

- The results of the TCLP leaching test showed that the mobility of heavy metals from bricks containing up to 80% GMWRs was below the limits set by the US EPA.

The use of solid mining wastes as aggregates and their incorporation in the field of building materials is a promising environmental solution, which aims not only to reduce their quantities but also to conserve the natural resources (clays, sands, aggregates, etc.) that are consumed abusively in current constructions.

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