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# Valorization of Phosphate Mine Waste Rocks as Materials for Road Construction

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**Abstract:** The road construction sector is a worldwide high consumer of natural aggregates. The use of unusual industrial by-products in road techniques can contribute to the conservation of non-renewable natural resources and the reduction of wastes produced by some industries. Phosphate waste rocks could be considered as potential alternative secondary raw materials in road construction. The use and valorization of these wastes is currently limited according to the Moroccan guide for road earthworks (GMTR). The guide has classified these materials as waste products, which consequently, cannot be used in road construction. However, phosphate waste rocks are sedimentary natural rocks which have not been subjected to any transformation other than mechanical fragmentation. The goal of this paper is to discuss key-properties of various phosphate mine waste rocks (PMWR) to be used as road materials. Samples were taken from different stockpiles in the phosphate mine site of Gantour in Morocco. The different waste rocks samples were characterized in terms of their physical, geotechnical, chemical, mineralogical and environmental properties using international testing norms. The obtained results showed that the studied PMWR presented satisfying characteristics; the specific (particle) density: $\rho s > 26$ kN/m$^3$, Los Angeles abrasion: $45\% < LA < 58\%$), methylene blue value MBV $< 1$ g/100g, organic matter: OM $< 1\%$ and plasticity index: PI $< 20\%$. All PMWR were confirmed as possessing the requested geotechnical properties to be used as materials for embankments. Moreover, leaching tests showed that none of them released any contaminants. In field application, these materials have been also successfully used in in situ experimental pilot testing. Therefore, the PMWR have to be classified in the category of natural aggregates that are similar to conventional materials.

**Keywords:** civil engineering; valorization; phosphate mine waste rocks; natural aggregates; road techniques wet process

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## 1. Introduction

Various stakeholders in the road construction sector have to deal with the increased demand of raw materials used in road infrastructures. The flexible pavement structure is generally composed of several layers of materials: embankment materials, capping layer, pavement aggregate (base and sub-base course) and surfacing (surface and binder course) course.

Recently, an increasing attention has been given to the potential use of alternative aggregates, particularly, in the road construction sector [1]. Several factors must be considered before using a waste or by-product in road engineering. The use of industrial wastes as secondary and alternative materials in the infrastructure sector depends on their availability, on the transport costs and their physical, geotechnical and chemical properties. The toxicity and solubility in water is a relevant factor to be considered with other factors [2]. The use of alternative materials in road construction provides several economic and ecological advantages. When waste materials with acceptable properties are available, it is possible to avoid the costs related to the extraction, and to minimize the transport distance, energy consumption and consequently the greenhouse gas emissions [3].

Many examples have been studied in detail in the literature. Fly ash and other agricultural wastes were used as soil admixture to improve the CBR values of soil in lower layers of road construction [4]. Incinerated bottom ashes were also investigated in road construction [5–7]. When stabilized with binder additives, these materials could be used successfully in embankment and pavement layers. Construction or demolition wastes have been used in the construction of embankments and pavement layers [2,8,9]. It was also demonstrated that steel-slag fly ash and phosphogypsum as a solidified material can be used as road materials with competitive characteristics [10–12]. It has been established that the use of fly ashes could improve the natural and mechanical characteristics of soils [13]. Dredged sediments mixed with binders (cement and/or lime) and other products (steel slag, fly ash) were compatible with the requested standards for their use as base or embankments course material [14–17]. According to the inventory carried out by OCDE, about twenty types of waste and by-products, to be used in road engineering, has been studied [1]. A classification according to the origin, the main characteristics, the current and the potential uses has been proposed.

In Morocco, phosphate mines produce millions of tons of phosphate mine waste rocks (PMWR) which are stockpiled on surface in waste rock piles covering large areas (several thousand hectares). These waste rocks represent mainly the intercalation layers (limestones, marls and flintstone) and the cover layer (topsoil, clays and marls) occurring within the phosphate sequence. During the extraction of phosphate ore, intercalation layers and the cover layer are blasted and stripped away. Due to their high calcite and dolomite content, the PMWR are inert geochemically. Hakkou, et al. [18] demonstrated that PMWR could be used as materials for the passive treatment of acid mine drainage [19,20], and had the appropriate properties for a store-and-release cover component in a semiarid climate [21]. Although they have characteristics similar to the natural aggregates used as building materials, PMWR are classified by the Moroccan Guide for Road Earthworks [22] in the organic soils and industrial by-products class and particularly in the phosphate wastes sub-class F3 and cannot thus, be used in road construction.

To our knowledge, very limited scientific research on the valorization of PMWR in road construction has been published. Ahmed and Abouzeid [23] investigated the use of phosphate waste rocks in road construction. The work consisted of geotechnical characterization, which led them to conclude an interesting potential of these by-products as road aggregates; similar to natural ones. The substitution of conventional aggregates by PMWR for the construction of road infrastructures might be considered as a promising and ecofriendly solution. A scientific approach of valorization of these mining wastes in road technique is, therefore, necessary to ensure the transition from a “waste” to “building materials”. The aim of this paper is to focus on PMWR and its use in the construction of road embankments and to discuss their status in Moroccan Guide for Road Earthworks. Laboratory tests were performed in order to determine the chemical, mineralogical characteristics, physical and geotechnical properties and the environmental behavior of the PMWR. In addition, in situ tests were realized in order to access the behavior of these materials during and after embankments construction using the wet process.

## 2. Materials and Methods

### 2.1. Materials Sampling

The studied materials were sampled from the mining site located in the central part of the sedimentary phosphate deposits in the Guantour region (Figure 1). The deposit is characterized by phosphate series of late Cretaceous–Eocene age consisting of alternating layers of phosphate separated by gangue silico-carbonate levels. The upper phosphate layer is overburdened by an alternation of different layers (topsoil, siliceous marl, clays, flintstone, calcareous marl and alluvium). The mine produces millions of tons per year of phosphate mine waste rocks (PMWR). The various rock lithologies are scattered in the mine site. Five different waste rocks piles referenced hereafter as I1, I2, I3, I4 and I5 were investigated. To ensure a representative sample, attention was given to the mode, history of storage, geological and petrographic lithology description to identify the parameters likely to impact the characteristics of the sampled materials. Given the heterogeneity of the waste rock piles, a rigorous technique was used to obtain the most representative sampling. The approach consisted of collecting 3 samples from the five stockpiled waste rocks of approximately equal size at different points, respectively at the base, at the middle and at the top of the pile depending on the actual segregation status (Figure 2). The field samples were collected, homogenized in the laboratory, and riffle-split into smaller sub-samples which were stored for further testing.

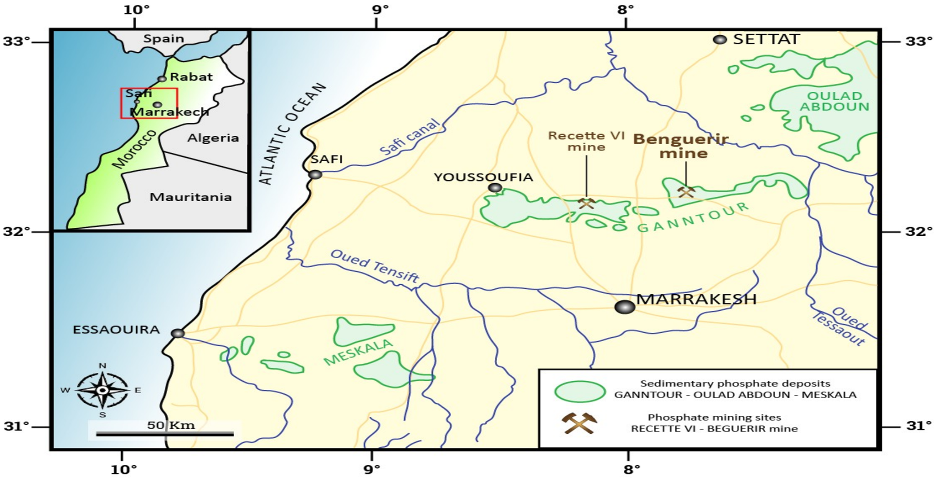

**Figure 1.** Geographical location of the studied mine site.

**Figure 2.** Phosphate mine waste rocks dumps.

### 2.2. Research Methodology

The GMTR guide does not include PMWR specifications and conditions of use, therefore, the geotechnical characterization in the laboratory has been completed by conducting in situ tests in a trail embankment and identifying other specific parameters (consolidation, shear strength, chemical and environmental properties) which are not provided by the NF P11-300 standard and which may affect their functional behavior. Figure 3 highlights a summary of the methodology used in this work.

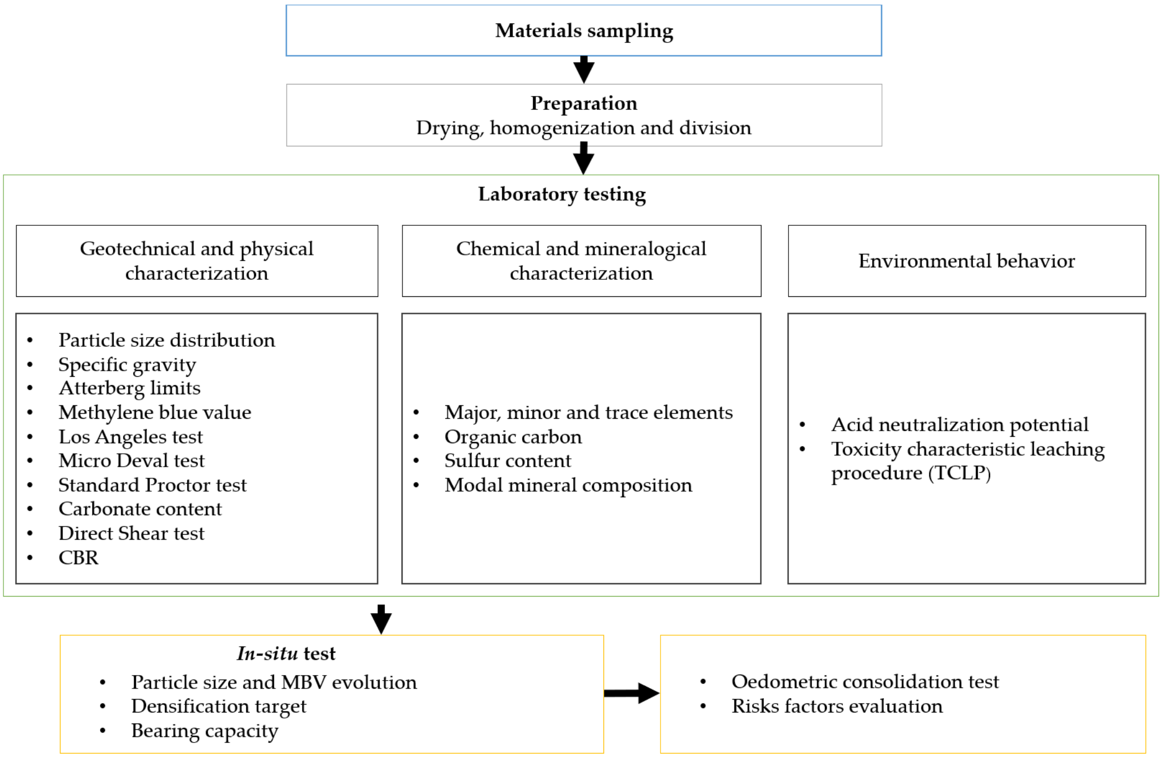

**Figure 3.** Summary of the followed methodology.

#### 2.2.1. Laboratory Tests

Laboratory tests have been conducted to determine the chemical characteristics, the environmental behavior, the mechanical and physical properties and the geotechnical properties. The laboratory tests were carried out in accordance with the relevant AFNOR standards [24]. The leaching behavior of waste rocks was assessed using the Toxicity Characteristic Leaching Procedure (TCLP) [25]. The leaching solution used has a pH of 4.93 ± 0.05. The obtained results are compared with US-EPA thresholds [26]. This test is used to verify a potential release of impurities and contaminants. The chemical composition of the solid samples was analyzed using an X-ray Fluorescence (Panalytical, Epsilon 4 Model, Malvern Panalytical, Malvern, UK) and liquid solutions were analyzed by inductively coupled plasma with atomic emission spectroscopy (ICP-AES) (Perkin Elmer Optima 3100 RL, PerkinElmer Waltham, MA, USA). The organic carbon content (Corg) was determined by dichromate oxidation in the presence of concentrated sulphuric acid according to ISO 14235 standard [27]. The crystalline phases were determined by the X-ray diffraction (Bruker AXS Advance D8. Bruker, Billerica, MA, USA), Cu K$\alpha$ radiation. The DiffracPlus EVA software (Bruker, Billerica, MA, USA) was used to identify mineral species and TOPAS software (Bruker, Billerica, MA, USA) implementing Rietveldt refinement to quantify the abundance of all identified minerals. Due to the presence of calcareous rocks, the carbonate content was determined on −400 µm of crushed samples in accordance with the NF P 94-048 standard [28].

The classification of the PMWR has been codified in relation to the nearest standardized materials, it will be therefore called to standard NF P 11-300 [24] for the geotechnical characterization. Prior to the characterization, the moisture content of the samples was measured [29]. The fraction 0/400 μm of the studied samples have been subjected to the plasticity test using the Atterberg limits method [30,31]. Also, dimensional properties tests were investigated using granularity method by dry sieving after washing [32]. The methylene blue test value (MBV) was measured on the 0/5-mm fraction taken from the 0/50-mm dry material according to the standard NF P 94-068 [33]. For compaction aptitude, the samples were compacted, in three layers, in a CBR standard mold using normal compaction [34]. When the proportion of elements greater than 20 mm exceeds 30% of the mass of the material, the Proctor test was performed on the 0/20-mm fraction, but its interpretation is then limited to the assessment of its moisture content $w_{\mathrm{opn}}$. In this case, the real dry density was measured in a full trial scale. To complete the knowledge of the petrographic features of the original rocks, evaluate the resistance of the material regarding the fragmentation, the wear and the particle size distribution evolution under the effect of mechanical solicitations, several tests were carried out using: NF P 94-064 standard for the density of a rock element by the hydrostatic weighing method. For better representativeness, the fraction 25/50 mm was chosen for the determination of Los Angeles abrasion value (LA) and Micro Deval value (MD) using NF P18-573 [35] and P18-572 [36] standards. To measure the sensitivity of these materials to fragmentation under the effect of mechanical stresses and climatic cycles, the fragmentation coefficient and degradability coefficient of samples were measured on the 40/80-mm fraction according to the standards NF-P94-066 [37] and NF-P94-067 [38], respectively. To determine the load bearing capacity of the materials after compaction and to assess their resistance to punching and heavy machines traffic, Californian bearing ratio (CBR) tests were carried out according to standard NF-P94-078 [39]. The shear strength parameters were investigated under the drained conditions, on the 0/20-mm fraction of the studied waste rock samples on the waste rocks samples [40].

In order to evaluate the vertical deformation (settlement) under the effect of the charges after saturation, an oedometric test was carried out on the 0/20-mm fraction of material I2 according to the standard XP-P94-090-1 [41] with a water content close to the optimum moisture content ($w_{\mathrm{opn}}$) and with a dry density equal to the reference dry density ($\gamma$dr) determined by the in situ tests.

### 2.2.2. In Situ Tests

In situ tests were performed with sample I2 which is the most abundant material among the PMWR. The objective was to determine the optimal conditions that offer the best results in terms of material compaction. These are related to: water content, thickness of compacted layer, speed and number of compactor passes. The ability of these materials to achieve the targeted compaction level for the embankments (q4 level) was also assessed. The dry densities of the materials were measured in situ using a membrane densitometer [42], while the measurement of the bearing capacity of the materials was evaluated through the determination of the module under static loading at the plate $EV_2$ (standard NF P 94-177-1).

A full-scale of trial embankment was constructed on 30 m length and 8 m width reinforced with around 1 m of embankment high. Three layers of 0.30-m thickness were constructed by wet method using a loading machine, a grader, a sprinkler vehicle and vibratory road roller (Figure 4). The trial test was carried out on a stable platform whose bearing capacity at the time of completion was greater than 50 MPa. The layers were compacted with a calibrated vibratory road roller with a single drum [43]. The speed was fixed at 4 km/h and the compaction energy of the machine was controlled. The representation of the three layers pile is shown in Figure 4. Many parameters were fixed during these tests: the speed of the compactor, the vibration amplitude of the compactor, the average water content range (between 0.9 and 1.1 of $w_{\mathrm{opn}}$) and the layer thickness.

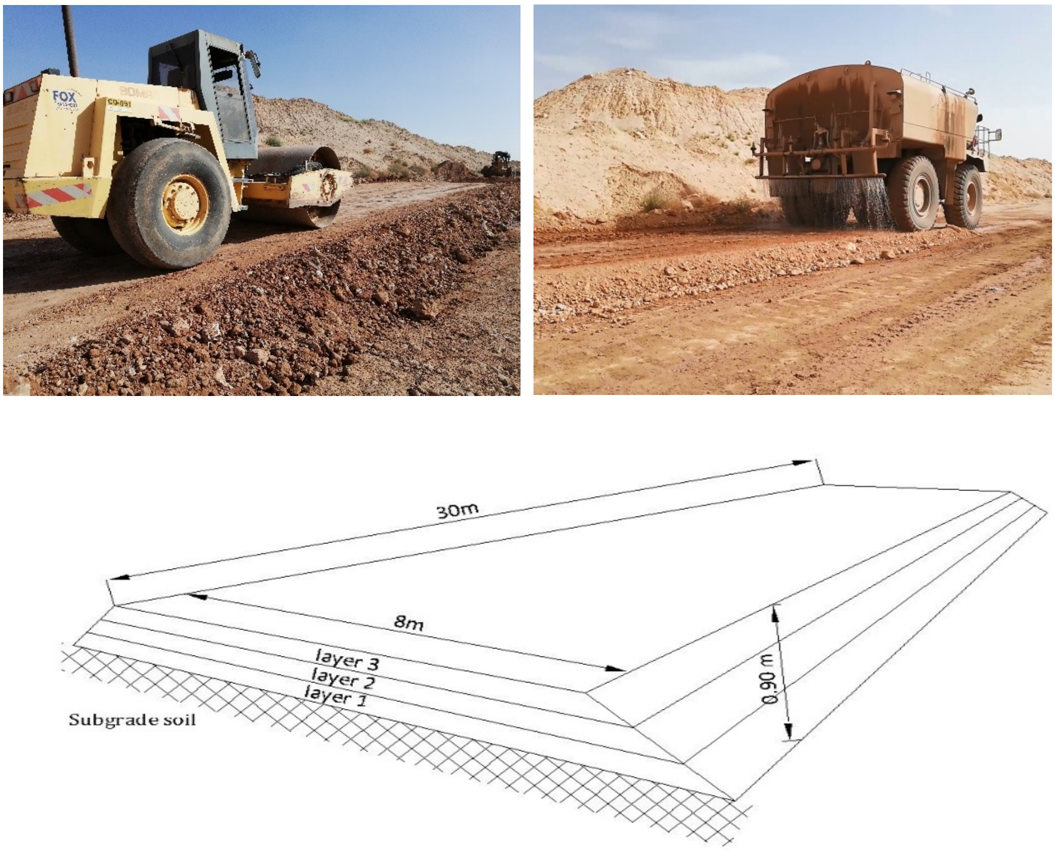

**Figure 4.** Photos and scheme illustrating the field trial embankment construction.

The shape of the granulometric curve obtained before compacting showed a continuity of the granularity, in addition, to have a better representativity, the water content was realized on the entire fraction of soil-rockfill mixture (0/80 mm).

Before compacting, the grain-size distribution curve, the MBV, the fine fraction (having a size less than 80 µm), the particles having a size less than 2 mm (determined on the 0/50-mm fraction) and the water content were measured. In addition to the same parameters followed before compacting, the layer thickness, the dry density and the bearing capacity for the three compacted layers with the different energy proposed (2, 4 and 8 of roller passes) were measured after compacting. The average of six samples will be selected for each monitored parameter before and after compaction.

The optimum dry density will be the one that corresponds to the maximum value of the six points of dry densities recorded on the compacted thickness and the control of the homogeneity of the distribution of compaction forces was verified by determining the maximum deviation from the mean value on the same compacted layer. In other words, the determination of the compaction energy which makes it possible to have the maximum dry density. It is this which will be taken as reference dry density ($\gamma$dr) for the calculation of the compaction rate and evaluation the ability of these materials to achieve the required compaction levels for embankments.

## 3. Results and Discussion

### *3.1. PMWR Characterization*

#### 3.1.1. Physical and Geotechnical Properties

Results of the geotechnical identification tests are summarized in Table 1. All the studied PMWR samples display approximately the same water content and very dry moisture content due to the arid climate of the region and the storage at the mine site. In addition, the tested materials show generally similar properties with a slight difference in grain size distribution results (Figure 5). The 0/20-mm fraction is important as it is considered in the Proctor and CBR tests. Only materials whose +20 mm particles weight proportion under 30% have been the subject of CBR and Proctor tests (it is the case only of I3 material). Also, the granulometry has a direct impact on plasticity of the studied materials. The materials with the highest content of fine fraction (I3, I4 and I5) showed the most plasticity features. The degree of plasticity that remains low for all these materials is particularly related to the mineralogical composition of the clays (illite). A difference was founded in mechanical properties of the studied PMWR samples. Unlike other materials with low values, I2 and I1 samples showed satisfactory values of LA, MD, degradability and fragmentability coefficients. The analysis of the results of the various physical tests indicates that the degradation of particles is related particularly to the presence rate of clay and flintstone in the studied samples. With a maximum particles diameter less than 150 mm, a non-zero cohesion (4–7 kPa), a plasticity index less than 16%, a specific (particle) density around 26 kN/m$^3$ and a percentage of fine elements less than 23%, the PMWR studied as shown in Table 1 could be classified in the category C1B5 ( friable soil); a gravelly coherent materials with a fine fraction [24] and the category of mixture of limestone and siliceous sandstone (for the case of the rocky origin). Thus, these materials can be used in the construction of road embankments. This is illustrated in the synoptic table of classification according to the nature of the materials proposed by the NF P11-300 standard (Figure 6).

**Table 1.** Physical and geotechnical properties of collected materials.

| **Test** | | **I1** | **I2** | **I3** | **I4** | **I5** |
|---|---|---|---|---|---|---|
| Moisture Content | wt. % | 3.4 | 3.7 | 3.6 | 2.9 | 3.1 |
| Geotechnical properties—Natural parameters | | | | | | |
| Proctor Test | | | | | | |
| Optimum Moisture content ($w_{\text{opn}}$) | wt. % | 13.40 | 12.90 | 15.20 | 14.60 | 13.23 |
| Maximum dry density $\gamma_{\text{d max}}$ | kN/m$^3$ | * | * | 17.9 | * | * |
| Shear test | | | | | | |
| Friction angle (Ø′) | degrees | 30.00 | 32.40 | 27.00 | 27.5 | 27.00 |
| Cohesion (c′) | kPa | 4 | 5 | 6 | 7 | 7 |
| CBR | % | * | * | 13 | * | * |
| Atterberg limit | | | | | | |
| Liquid limit | wt. % | 39 | 37 | 41 | 44 | 45 |
| Plastic limit | wt. % | 26 | 25 | 26 | 29 | 30 |
| Plasticity index | wt. % | 13 | 12 | 14 | 15 | 15 |
| Methylene blue value | g/100g | 0.59 | 0.58 | 0.67 | 0.68 | 0.71 |
| Carbonate content | wt. % | 30 | 29 | 33 | 32 | 33 |
| Geotechnical Properties–Mechanical behavior | | | | | | |
| Specific (particle) densi | kN/m$^3$ | 2.61 | 2.65 | 2.56 | 2.6 | 2.58 |
| Los Angeles abrasion test 25/50 | wt. % | 48 | 46 | 66 | 67 | 53 |
| Mico Deval test 25/50 | wt.% | 55 | 50 | 68 | 70 | 54 |
| Degradability coefficient | wt.% | 10.10 | 9.10 | 13.80 | 14.60 | 12.70 |
| Fragmentability coefficient | wt.% | 8.90 | 7.50 | 10.10 | 11.40 | 10.50 |
| Material classification | - | $C_1B_5$ | $C_1B_5$ | $C_1B_5$ | $C_1B_5$ | $C_1B_5$ |

* Proportion of particles greater than 20 mm exceeds 30% of the mass of the material, in this case, the maximum dry density of the proctor and the value of CBR test are not significant.

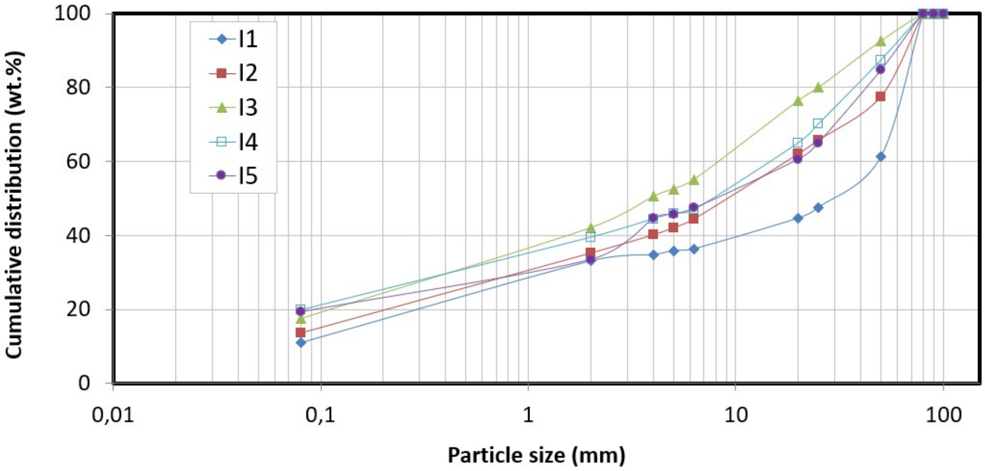

**Figure 5.** Particle size distribution of the five collected materials.

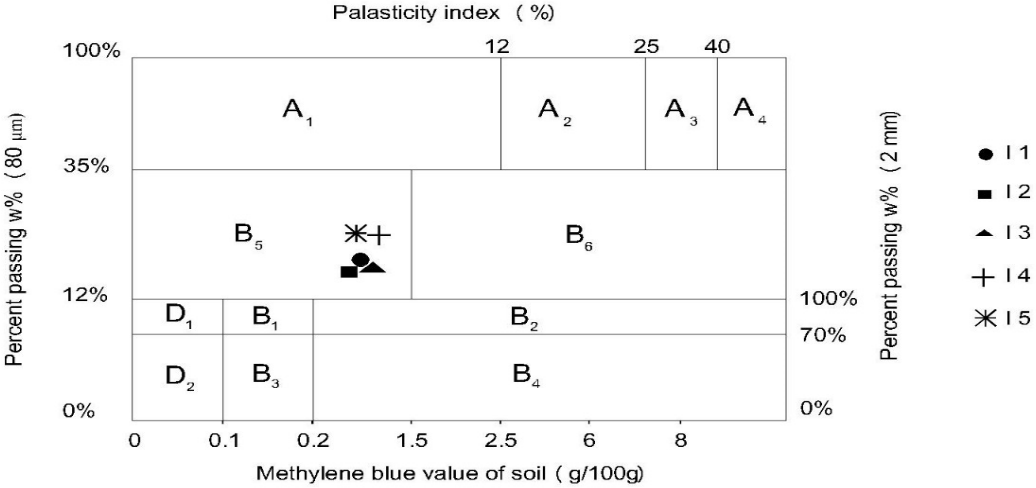

**Figure 6.** Classification of studied PMWR according to the soil classification table of the NF P11-300 standard (fraction 0/50 mm). A: fine soil; B: Sandy and gravely soil with fine particle and D: Soil insensitive to water.

### 3.1.2. Chemical and Mineralogical Properties

The chemical and mineralogical composition of PMWR are presented in Table 2. All the samples contain mainly $SiO_2$ (41–57 wt. %), CaO (12–19 wt. %), MgO (4.9–9.1 wt. %), $P_2O_5$ (4.2–5.4 wt. %). Alkali and alkali earth oxides are present in low concentrations (less than 1 wt. %). The amounts of detected sulfur and organic carbon were generally below 0.5 wt. %. The PMWR could be classified easily as non-generating of acid mine drainage with a very neutralization potential, as already demonstrated [18]. In terms of trace element occurrence, only a very low concentration of Cr, Cd, Cu and Mo were detected. The relative abundance of minerals identified by XRD and quantified using a Rietveld refinement method is illustrated in Table 2. All materials contain a siliceous fraction represented by quartz and cristobalite, and carbonaceous fraction represented mainly by dolomite and little amount of calcite. Fluorapatite was also detected varying between 6 and 8.5wt. %. The mineralogical composition of the studied materials showed also plagioclase (albite and anorthite) and clays (illite which was observed only in samples I3, I4 and I5, that is why they showed the most plasticity features).

**Table 2.** Chemical and mineralogical composition of PMWR samples.

| **PMWR Sample** | | **I1** | **I2** | **I3** | **I4** | **I5** |
|---|---|---|---|---|---|---|
| Major elements (wt. %) | | | | | | |
| $SiO_2$ | | 41.30 | 50.10 | 55.50 | 53.90 | 56.40 |
| $Al_2O_3$ | | 0.40 | 0.44 | 4.10 | 3.80 | 3.10 |
| $Fe_2O_3$ | | - | - | 0.50 | 0.40 | 0.30 |
| CaO | | 18.70 | 16.20 | 12.10 | 12.90 | 12.50 |
| MgO | | 9.10 | 7.20 | 4.90 | 5.50 | 5.40 |
| K2O | | - | - | 1.60 | 1.31 | 1.00 |
| $P_2O_5$ | | 4.22 | 5.40 | 5.10 | 4.70 | 4.20 |
| LOI | | 24.20 | 18.30 | 15.50 | 17.40 | 16.90 |
| $C_{org}$ | | 0.21 | 0.18 | 0.31 | 0.44 | 0.38 |
| S | | 0.30 | 0.40 | 0.20 | 0.30 | 0.30 |
| Mineralogical composition (wt. %) | | | | | | |
| Quartz | $SiO_2$ | 41.21 | 49.80 | 32.00 | 30.10 | 33.60 |
| Cristobalite | $SiO_2$ | | | 17.40 | 18.40 | 18.40 |
| Dolomite | $(Ca,Mg)(CO_3)_2$ | 40.89 | 32.00 | 21.00 | 24.00 | 24.00 |
| Calcite | $CaCO_3$ | 6.04 | 5.00 | 3.90 | 3.50 | 3.70 |
| Fluorapatite | $Ca5(PO4)_3F$ | 6.42 | 6.20 | 8.30 | 8.10 | 7.90 |
| Albite | $NaAlSi_3O_8$ | 5.06 | 6.40 | 6.20 | 5.60 | 4.90 |
| Illite | $(K,H_3O)(Al,Mg)_2(Si,Al)_4O_{10}[(OH)_2,(H_2O)]$ | | | 11.00 | 9.20 | 6.50 |
| Anorthite | $CaAl_2Si_3O_8$ | 0.38 | 0.60 | 0.44 | 0.80 | 0.90 |

LOI: Loss on Ignition, $C_{org}$: Organic Carbon.

No montmorillonite has been detected, which eliminates the risk of swelling of the clayey fraction contained in these materials once used in road construction. The I2 sample exhibit low content of clays (illite) and high content of siliceous minerals (flintstone occurrence), which explain the best mechanical features: Los Angeles (LA = 46 wt. %), Micro Deval (MD = 50 wt. %), Degrability and Fragmentability coefficients respectively 9.1 and 7.5 as shown in Table 1.

### 3.1.3. Environmental Behavior of Materials

The results of trace elements leaching from PMWR using the TCLP test are summarized in Table 3. The mobility of heavy metals depends on several factors such as heavy metals bearing minerals and the pH of the leaching solution. All concentrations were in agreement with the limits for non-hazardous waste fixed by US-EPA regulation. Therefore, the studied PMWR cannot be listed as hazardous waste, in fact they should be considered as natural aggregates. The observed limited metal release is explained by the low initial content within PMWR and the relative high stability of the occurring inert minerals (silice and aluminosilicates) and high neutralizing capacity minerals such as dolomite and calcite. The fluoroapatite need strong acidity to be solubilized.

**Table 3.** Results of the Toxicity Characteristic Leaching Procedure (TCLP) of PMWR.

| **Sample** | **Zn** | **Se** | **Pb** | **Cu** | **Cr** | **Cd** | **As** | **V** |
|---|---|---|---|---|---|---|---|---|
| | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **mg/L** | **mg/L** |
| I1 | 0.55 | <0.10 | <0.60 | <0.50 | <0.20 | <0.10 | <1 | <1 |
| I2 | 0.62 | <0.10 | <0.60 | <0.50 | <0.20 | <0.10 | <1 | <1 |
| I3 | 0.73 | <0.10 | <0.60 | <0.50 | <0.20 | <0.10 | <1 | <1 |
| I4 | 0.54 | <0.10 | <0.60 | <0.50 | <0.20 | <0.10 | <1 | <1 |
| I5 | 0.56 | <0.10 | <0.60 | <0.50 | <0.20 | <0.10 | <1 | <1 |
| Limits (US-EPA) | 2 | 1 | 5 | - | 5 | 1 | 5 | - |

## 3.2. *In situ Full Trial Tests*

The evolution criterion (in the granulometric approach) considered in this on-site study concerns fragmentation under the action of mechanical solicitation before and after compaction. This parameter was demonstrated during the essays in true size by the measurement of the particle size analysis and MBV before and after compacting by means of different compaction energies. The results show (Table 4) that the maximum particle size evolution of 18.39%( explained by the presence of friable limestone and marly rocks characterized by low mechanical resistance) was recorded with eight passes, almost identical evolution of 18.23% was recorded in the case of four passes which concerns the evolution towards the fine fraction, this explains that the production of the fine elements is stopped under the effect of compaction energy of four passes, it was also noted that MBV values increased slightly with increasing compaction energy; even with these evolutions, the material always keeps the same classification family after compaction ($C_1B_5$) according to the NF-P11-300 [24] standard.

**Table 4.** Granulometric and Methylene Blue value (MBV) evolution according to the compaction energy.

| State of Material | Before Compaction | After Compaction | | | | | |
|---|---|---|---|---|---|---|---|
| **Compaction Energy** | | **2 Passes** | **Evolution (%)** | **4 Passes** | **Evolution (%)** | **8 Passes** | **Evolution (%)** |
| <0.08 mm (wt. %) | 19.20 | 21.00 | 9. 38 | 22. 70 | 18.23 | 22. 73 | 18. 39 |
| <2 mm (wt. %) | 37.60 | 40.20 | 1.60 | 41.00 | 9.04 | 41.10 | 9.31 |
| <20 mm (wt. %) | 63.80 | 68.00 | 6.58 | 71.00 | 11. 29 | 71.20 | 11.60 |
| <50 mm (wt. %) | 89.20 | 95.00 | 6. 50 | 96.00 | 7. 62 | 96. 10 | 7.74 |
| <2mm (0/50 mm) (wt. %) | 42.15 | 42.32 | 0.39 | 42.71 | 1.32 | 42.77 | 1.46 |
| <0.08 mm (0/50 mm) (wt. %) | 21.52 | 22.11 | 2.70 | 23.65 | 9.85 | 23.65 | 9.89 |
| MBV (g/100g) | 0.57 | 0.61 | 7.02 | 0.66 | 15.79 | 0.67 | 17.54 |

The in situ tests have shown that with a very important compaction energy of 8 passes, the process of alteration of the blocks has been almost stopped (granulometric evolution less than 0.9% for fine elements and less than 3% for other diameters found by adding four additional passes of the compactor to get to 8 passes). The results of the dry density according to the compacting energy at the surface and at the bottom of the compacted layer have been summarized in the Table 5. Embankment dry density was examined as a function of roller passes.

**Table 5.** The compaction rate according to the compaction energy.

| **Layer** | | **First Layer** | | | **Second Layer** | | | **Third Layer** | | |
|---|---|---|---|---|---|---|---|---|---|---|
| **Compaction Energy** | | **2 Passes** | **4 Passes** | **8 Passes** | **2 Passes** | **4 Passes** | **8 Passes** | **2 Passes** | **4 Passes** | **8 Passes** |
| reference dry density (kN/m$^3$) | | 19.5 | | | | | | | | |
| Surface | dry density (kN/m$^3$) | 18.2 | 19.4 | 19.0 | 18.4 | 19.4 | 19.1 | 18.2 | 19.5 | 19.1 |
| | compaction rate (%) | 93 | 99 | 97 | 94 | 99 | 98 | 93 | 100 | 98 |
| Bottom | dry density (kN/m$^3$) | 17.4 | 18.2 | 18.1 | 17.5 | 18.0 | 18.0 | 17.3 | 18.3 | 17.8 |
| | compaction rate (%) | 90 | 96 | 95 | 91 | 96 | 94 | 90 | 95 | 96 |

The results showed that the maximum dry density corresponding to 19.5 kN/m$^3$ was recorded for the compaction energy of four passes, the reference dry density is therefore taken equal to 19.5kN/m$^3$.The calculation of the compaction rates (at the surface and at the bottom of the layer) makes it possible to show that the application of a compaction energy of two passes does not satisfy the required compaction levels for embankments contrary to four and eight passes (Table 5). After application of each compaction energy, the bearing capacity test was carried out just after the compaction of the third layer by ensuring the average water moisture of the material at the time and after compaction, this has been verified by sampling during the entire duration of the measurements by realizing a sounding through the three layers (Table 6).

**Table 6.** Results of lift tests.

| Compaction Energy | | | 2 Passes | 4 Passes | 8 Passes |
|---|---|---|---|---|---|
| Plate test (average of six points) | standard deviation | % | 2.23 | 2.52 | 2.68 |
| | $EV_1$ | MPa | 45.70 | 57.30 | 57.10 |
| | $EV_2$ | MPa | 79.80 | 91.20 | 89.40 |
| | K ($EV_2/EV_1$) | - | 1.75 | 1.59 | 1.57 |

With k ($EV_2$ / $EV_1$) < 2 (which makes it possible to appreciate the quality of the compaction) and an average $EV_2$ module > 80 MPa, material I2 has very satisfactory lift results. According to the LCPC-Setra guide [41], these materials, which are also sensitive to water, can therefore, be classified as Top part of the earthworks (PST3) from a class (AR2) formation if the constructive drainage arrangements make it possible to evacuate the water and prevent its infiltration.

The aforementioned criteria of grain size evolution, densities and bearing capacity justify the choice to be limited only to the compaction energy of four passes for the construction of embankments with PMWR. this retained energy, which remains more important than that required by the LCPC-Setra guide (limited to only three roller passes and for the same compaction parameters), allows to have a maximum fractionation especially for the marly rocks recognized by their evolving behavior and therefore avoid having two fractions with clearly differentiated granulometry (large particles and very fine fraction), this has been demonstrated by the realization of in situ trenches which show that the compacted material is coherent (the fines perfectly fill the voids between the blocks), resistant, and has a homogeneous appearance (a reduced standard deviation found during densities and lift measurements over the entire thickness of the compacted layer).

In view of the aforementioned results, the optimal compaction conditions which make it possible to obtain the compaction level required for embankments (by humidification), to ensure the minimum bearing capacity for the embankment materials and to avoid possible disorders due to the phenomenon of grain size evolution under the effect of mechanical stresses are the following (Table 7).

**Table 7.** Optimal compaction conditions of I2 material.

| Moisture Content (wt. %) | Compactor Class | Compactor Speed (km/h) | Compaction Energy | Thickness (m) |
|---|---|---|---|---|
| Average (0.9 à 1.1) $w_{opn}$ | V4 (vibratory compactor roller) | 4 | 4 passes | 0.30 |

### 3.3. Risks Factors Evaluation

The results of the oedometric test showed that the material I2 has An average oedometric modulus of 10,045 kPa, a low compressibility index of 0.125 and a very low swelling index of 0.04, considering that the contracting regulations in Morocco often require settlements of less than 10 cm in 25 years on ordinary road embankments, an evaluation study of embankment settlements (in case of construction with I2 materials) was carried out to determine the maximum height beyond which stability will be questioned, the results make it possible to conclude that this material can be used for embankments up to a height of 15 m respecting a minimum rate of compaction of 95% of $\gamma$dr and a water content close to $w_{opn}$ without any significant risk of instability. With an organic matter content well below 3% threshold required by the NF P11-300 standard, these releases are therefore far from the category of organic materials, with the availability of deposits, the passage of this materials from the status of waste to an alternative material can therefore be pronounced. This leads back to identifying and evaluating the possible risk factors resulting from this study (Table 8).

**Table 8.** Risk assessment.

| Characteristics of the Material | Risk | Proposed Remedies |
|---|---|---|
| Limited mechanical strength (presence of clay) | • Granulometric evolution towards the fine fraction (creation of fine elements under the effect of mechanical stresses which can influence the water resistance)<br>• settlement due to the collapse of fine fraction<br>• swelling | respect the optimum conditions of use (in situ tests) |
| Presence of rock of different petrographic origin (heterogeneity) | • Settlement for high embankments (>15m)<br>• (difference in drainage and water permeability in the embankment) | • Good identification of deposits<br>• Representative sampling<br>• Rigorous control of traceability and homogenization of storage process<br>• The use is limited to embankments with heights less than 15 m (otherwise, special construction arrangements for stability, embankment base, circulation and drainage of water, variation of permeability will be required) |

## 4. Conclusions

This study is the first of its kind consisting of a physicochemical, mineralogical, environmental and geotechnical characterization of phosphate mine waste rocks. The main conclusions from the laboratory and in situ trial tests to assess the potential use of these materials for road construction are the following:

- The mechanical behavior of these materials depends essentially on their flintstone and clay content.
- The chemical and mineralogical composition and leaching tests on PMWR suggests that they are chemically inert.
- The in situ full trial testing has defined the optimal compaction condition for the use of PMWR in ordinary embankment construction (used in a wet way). It consists of a compaction energy of four passes, a speed of a V4 vibratory roller compactor of 4 km/h and a thickness of the compacted layers of 30 cm.
- Embankments up to 15 m height can be built with PMWR without any significant physical instability risks. The respect of the constructive provisions is necessary.

Considering the results of the leaching tests, the organic content and geotechnical properties, the PMWR can be assimilated to the category of conventional natural aggregates. The use of these materials will have a very important impact on the preservation of the use of natural resources (avoiding the use of borrowing materials) and the recycling of PMWR.

Even with an important level of heterogeneity linked to several scales: the extraction mode, the storage method, and the petrography of the original rocks. It may be recommended that PMWRs be considered as alternative aggregates to be sorted according to a pre-defined zoning map in order to simplify their reuse in civil engineering. The Moroccan guide for road (GMTR) should be updated to allow PMWR to be classified as natural aggregates.

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## Acronyms and Abbreviations

| | |
|---|---|
| Corg | Organic carbon content |
| LA | Los Angeles abrasion value |
| MD | Micro Deval value |
| TCLP | Toxicity characteristic leaching procedure |
| XRD | X-ray diffraction |
| PMWR | Phosphate mine waste rocks |
| $w_{\mathrm{opn}}$ | Optimum water content of the standard Proctor test |
| CBR | California bearing ratio |
| MBV | Methylene blue value |
| ρs | specific (particle) density |
| $\gamma$dr | reference dry density |
| OCDE | Organisation de cooperation et de développement économiques |

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