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Phosphogypsum recycling: New horizons for a more sustainable road material application

Mustapha Amrani [a,b,*], Yassine Taha [c], Azzouz Kchikach [a,d], Mostafa Benzaazoua [d,e], Rachid Hakkou [b,c]

[a] L3G Laboratory. Equipe de Recherche « Génie Civil et Géo-Ingénierie », Univ. Cadi Ayyad (UCA), BP 549, Marrakech, 40000, Morocco
[b] LCME. Faculté des Sciences et Techniques. Univ. Cadi Ayyad, BP 549, Marrakech, 40000, Morocco
[c] Materials Science and Nano-engineering Department. Mohammed VI Polytechnic University. Lot 660.Hay Moulay Rachid, 43150, Ben Guerir, Morocco
[d] Geology and Sustainable Mining Department, Mohammed VI Polytechnic University, Ben Guerir, Morocco
[e] Institut de Recherche en Mines et en Environnement. Univ du Québec en Abitibi Témiscamingue, 445 Boul de l'Université, Rouyn-Noranda, QC, J9X 5E4, Canada

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ABSTRACT

It is aimed in this paper to suggest novel alternatives to recycle phosphogypsum (PG) in road applications. The performance of several formulations for the stabilization of raw PG with other selected materials was evaluated. The research began with a feasibility study of using raw phosphogypsum alone as embankment material, then focusing on the effect of stabilizers such as clayey soil (SM), fly ash (FA), lime (L), calcareous material (CM) and a special hydraulic road binder (HRB). The objective was to increase the strength up to a threshold favouring the use of the stabilized mix as a base material in road pavement, while ensuring a neutralization of acidity generated by PG. Environmental, mineralogical and geotechnical properties of raw materials were investigated. According to compressibility parameters, initial moisture content and compaction characteristics, this study confirms first that the use of PG alone should be limited to ordinary embankments of heights less than 4 m. Secondly, the mix PG:SM = 40:60 allows to use a significant amount of raw PG while maintaining good performances in terms of fractal behaviour of particles size distribution, immersion resistance, load bearing capacity and compaction characteristics. This mix has also successfully passed the compressibility risk under loading effect on the embankments. Finally, the compatibility of the mix (CM:PG:SM):HRB = (10:25:65):7 for the capping and pavement layers use was confirmed. TCLP method was carried out on the stabilized mix samples to verify the contamination risk.

1. Introduction

Phosphogypsum (PG) is the by-product of the wet chemical attack process of phosphate ores (apatite) by sulfuric acid [1,2]. It consists mainly of calcium sulfate dihydrate ($CaSO_4{\cdot}2H_2O$) in association with low amounts of silicon (Si), fluoride (F), phosphorus pentoxide (P_2O_5), organic matter, traces of rare earth elements (REE) and radioactive elements [3]. Several research works indicated that the annual worldwide PG production is around 280 million tons [4,5]. Only 15% of this volume is recycled in different fields such as soil treatment process, fertilizers and building materials [6]. The larger part of world produced PG quantities are evacuated into streams, thrown into the sea with no well-known effect, or stored in stockpiles above the ground level causing serious environmental contamination [7]. The significant increase of PG production and its environmental impact have prompted researchers to develop novel technical solutions that increase PG recycling and valorization. Recent works revealed the enormous opportunities related to the use of PG in different construction applications. The study conducted by Chang, Chin and Ho [8] showed that the recycling of PG as pavement material is a safety practice from the groundwater contamination and radiological impact point of view. The utilization of PG either solely or with an activator in road applications have been also investigated [9]. However, the laboratory tests reported by Moussa et al. [10] on the Tunisian PG showed that its use alone as embankment material seems possible but only in well-determined conditions of placement and with precautions to avoid soaking. It was also mentioned in other studies that from a geotechnical point of view, the realization of embankment with PG material is assimilated to the construction of a structure with a clean and very friable sand [11,12].

* Corresponding author. L3G Laboratory. Equipe de recherche « Génie civil et Géo-Ingénierie », Univ. Cadi Ayyad (UCA), BP 549, Marrakech, 40000, Morocco.
E-mail address: amrani.be.2p@gmail.com (M. Amrani).

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Acronyms and abbreviations

C_1B_5	Very silty sand and gravel materials
CBR (4i)	CBR after 4 days (±2 h) of soaking in water at 20 °C. in %
CBR	California Bearing Ratio
C_c	Compression index
CM	Calcareous material
C_p'	Effective cohesion for the peak criterion of the shear test.in kPa
C_v	Swelling index
D_{50}	Average particle diameter. The size for which the cumulative distribution is equal to 50w%. in mm
E	Elastic modulus. in MPa
FA	Fly Ash
HRB	Hydraulic Road Binder
IBI	Immediate Bearing Index. in %
IR	Immersion Resistance Index
L	Lime
MBV	Methylene blue value. in g/100g
PG	Phosphogypsum
Rt	Diametric compression strength. in MPa
SM	Soil Material
s'_p	Preconsolidation stress. in KPa
UCS	Unconfined Compressive Strength. in MPa
W_{opm}	Optimum moisture content of the modified Proctor test. in %
W_{opn}	Optimum moisture content of the standard Proctor test. in %
ρ_{dOPM}	Maximum dry unit weight of the modified Proctor test. in kN/m^3
ρ_{dOPN}	Maximum dry unit weight of the standard Proctor test. in kN/m^3
ϕ'_p	Internal friction angle for the peak criterion of shear the test. in degree

Road pavement construction requires materials with high mechanical performances and sophisticated design procedures related to quality criteria, optimization, strength and durability. Mixes of fly ash (FA) stabilized with lime and PG have been tested as pavement layers. The results of the experimental investigations concluded that the mix containing FA + 8% lime + 2% PG can be used in different geotechnical applications such as laying of backfill and the construction of rail and road embankments [2]. Gregory, Saylak and Ledbetter [13] observed that the use of the mix FA-PG-cement solidified material in road base could present a potential stabilization solution. The stabilizer amount, mix dry density, and phosphogypsum's pH have a major impact on strength development of the stabilized mixes.

The storage and management of PG are considered the main challenges facing the phosphoric acid production industries around the world. It requires the mobilization of significant resources and consumes considerable lands. Dozens of millions of tons of PG are produced annually in Morocco. Phosphogypsum has proven cementitious properties with the presence of a considerable content of Ca which is beneficial for generating the hydration elements. Moreover, the hydration reaction may be catalysed by the phosphate product in the PG [14]. It would be interesting to amplify this characteristic by blending it with other conventional stabilizers and chemical activators to neutralize its initial acidity and particularly to improve the mechanical characteristics of the designed mixes.

This paper investigates the compatibility of Moroccan PG with the conventional materials in road construction. In the first step, laboratory scale trials were carried-out to determine the main characteristics of the used materials; then analysing their reactivity with raw PG and evaluating their limit of application. The feasibility of using PG alone as embankment material was discussed. To reach more significant embankment heights, the use of phosphogypsum-soil solidified material was also examined. Then, the sustainability and strength of pavement design mixes were investigated by mixing raw PG in combination with soil material (SM), natural calcareous material (CM), fly ash (FA), lime and a special hydraulic road binder (HRB). To monitor the assessment criteria of each designed formulation, measurable geotechnical and mechanical parameters were applied. The contamination risk assessment of the optimal mix materials towards surrounding environment was controlled using Toxicity Characteristic Leaching Procedure (TCLP) tests. X-ray diffraction analysis was applied to determine the developed cementitious phases, to explain the solidification mechanism and highlight the chemical transformation observed in the retained stabilized mixes.

2. Raw materials characteristics and experimental procedure

2.1. Raw materials

The physical, chemical and mineralogical properties of all raw materials used in this study are summarized in Tables 1 and 2.

- Phosphogypsum (PG)

Table 1
Chemical and mineralogical composition of raw materials.

	L	FA	PG	SM	CM	HRB
pH	12.42	11.4	2.2	6.89	12	10.5
Total organic carbon (wt%)	–	–	0.07	0.41	0.1	–
Chemical composition (wt%)						
CaO	89.12	4.31	31.16	10.8	44.1	45
CO_2	3	–				
SiO_2		51.29	1.03	60.1	4.4	15
SO_3	0.51		44.01			3
Al_2O_3	1.2	21.26	0.85	5.1	0.47	7
F			0.64	–		–
Fe_2O_3	0.46	6.15	0.012	0.3	0.1	4
MgO	2.77	2.61	–	5.4	9.9	1
Na_2O	0.42	0.15	0.21		0.03	0.5
K_2O	0.05	1.47	0.001	1	0.11	0.8
P_2O_5		0.78	0.96	2.5	1	–
MnO	0.17	0.09			–	–
Loss on ignition	2.3	7.89	21.1	12.9		20
Mineralogical composition (wt%)						
Quartz [SiO_2]		64	2.5	34	3	8
Cristobalite [SiO_2]				17.9		
Dolomite [$CaMg(CO_3)_2$]				23	75	
Calcite [$CaCO_3$]				4.1	17	26
Lime [CaO]	1					
Fluorapatite [$Ca_5(PO_4, CO_3)3F$]				6	2	
Albite [$NaAlSi_3O_8$]				5.3	3	
Anorthite [$CaAl_2Si_2O_8$]				1.5		
Illite [$(K,Al,Mg)_2(Si,Al)_4O_{10}[(OH)_2,(H_2O)]$]				8.2		
Gypsum [$CaSO_4\text{-}2(H_2O)$]			91			5
Bassanite [$CaSO_4.1/2(H_2O)$]			6.4			
Portlandite [$Ca(OH)_2$]	99					
Mullite [Al_5SiO_9]		36				4
Alite [$(CaO)_3(SiO)_2$]						34
Belite [$(CaO)_2(SiO)_2$]						13
Aluminate [$(CaO)_3\ Al_2O_3$]						3
Alumino-ferrite [$(CaO)_4\ Al_2O_3\ Fe_2O_3$]						7

Table 2
Geotechnical properties of raw studied materials.

Property	L	FA	PG	SM	CM	HRB
Atterberg limits (wt%)						
Liquid limit (LL)	–	–	57	43	37	–
Plastic limit (PL)	–	–	–	28	26	–
Plasticity index (PI)	–	–	<6	15	11	–
Grain size distribution (wt%)						
<50 mm				81.2		
<20 mm	100		100	70.4	100	
<2 mm	85	100	98.52	38	84.9	100
<80 μm	52	82	67.5	19.5	66.3	84.5
Sedimentometry analysis (values in wt%)						
<2 μm	5	3	4	7	5	2
2 μm–20 μm	30	60	12.5	3.6	42.5	70
20 μm–80 μm	17	19	51	8.9	18.8	12.5
The Methylene blue value (g/100g)	–	–	0.09	0.69	0,52	–
Specific gravity	2.38	2.37	2.41	2.64	2,57	2.98
Blaine specific surface area (m^2/g)	0.59	0.31	0.64	14.21	5.81	0.32

The PG sample was collected from the phosphoric acid production plant in Morocco. Its initial water content was 47 wt%. The maximum dry unit weight (ρ_{dOPM}) and the optimum moisture content (w_{OPM}) of modified Proctor test were respectively 14.8kN/$m^3$and 17 wt%. The specific surface area and specific gravity of the used PG were respectively 0.64 m^2/g and 2.41. PG sample is mainly composed of gypsum (91 wt%) and has a pH in the range 2–3. The radioactivity of PG was not evaluated in this study. However, many papers have already investigated the radioactive elements content of Moroccan PG [15,16]. A friability test was carried out on the 0.2–2 mm size gradation according to the NF–P18-576 [17] standard. The friability coefficient result of 68 reveals a low resistance to fragmentation.

- Soil material (SM)

An abundant soil material (SM) was collected nearby the phosphoric acid plant of Jorf Lasfer (El Jadida, Morocco). The main crystalline phases are silicates and aluminosilicates (65 wt%) and limestones (27 wt %). It had a specific gravity of 2.64 and a plasticity index (PI) of 15%. The fraction passing through 80 μm sieve was 22.3 wt%. The maximum dry density and optimum moisture content of modified Proctor test were respectively 18.8 kN/m^3 and 13.1 wt%. According to these properties, the SM sample was classified in the category of very silty sand and gravel materials C_1B_5 [18].

- Fly ash (FA) and Lime (L)

The studied coal fly ash was provided by the thermal power plant in Jorf Lasfar, Morocco. The collected sample presents a specific surface area of 0.31 m^2/g and a specific gravity of 2.37. With a low calcium content (4.31 wt%), FA sample is classified in the class F [19]. The utilization of another additive having better cementitious properties was tested. Dry lime (L) was acquired from local market with an effective CaO content of 89 wt%. Its specific surface area and specific gravity were respectively 0.59 m^2/g and 2.38.

- Natural calcareous material (CM)

The crushed natural calcareous material (CM) was obtained from a local quarry. It is mainly composed of limestone minerals and present a pH of 12. The plasticity index of CM was 11 wt%. It had a maximum particle size of 5 mm and the percentage retained on 80 μm sieve was 66.3 wt%.

- Hydraulic road binder (HRB)

HRB was provided by a local cement plantwith an effective clinker content of 55 wt% and 34 wt% of limestone. The fraction -80 μm represents 84.5 wt% of the total material weight. According to Ref. [20], the tested HRB corresponds to class 4 binders.

2.2. Experimental procedures

2.2.1. Mineralogical and geotechnical characterizations

The pH measurements were made after stirring for 5 min an amount of 10 g of dry samples in 100 ml of demineralised water using a direct reading pH meter. The water content of the samples was determined after steaming at adjustable temperatures from 50 °C to 105 °C [21]. Dimensional properties tests of mixes were investigated using granularity method by dry sieving after washing [22] while the fraction with a grain size less than 80 μm was obtained by sedimentation [23]. The maximum dry density and optimum moisture content of each mix were achieved using CBR mould following the Proctor test [24]. The methylene blue adsorption capacity (MBV) was measured on the 0/5 mm size fraction [25]. The mixes plasticity was carried out on 0/400 μm size fraction using the Atterberg limits tests [26,27]. The shear characteristics namely the effective cohesion C'_P and the effective internal friction angle ϕ_p' were determined from consolidated-drained shear strength test. The latter was carried-out on reconstituted sample compacted initially at the conditions of the modified Proctor test according to NF–P94-071-1 [28]. To simulate field compaction, a study of the fractal behaviour of the particles size distribution of the mixes was designed in the laboratory using automatic Proctor machine and standard mould at the conditions of the modified Proctor under three controlled stress strains (0.6 MPa, 1.5 MPa and 2.7MPa per layer). To assess the immediate compactness and water influence on load bearing capacity, the bearing ratio tests namely the CBR after 4 days (±few hours) of soaking in water CBR(4i) and immediate bearing index (IBI) were determined conforming to NF–P94-078 [29]. The IBI was measured on specimens compacted in the CBR mould at modified Proctor effort without any soaking in water or overloading.

To characterize the speed and amplitude of settlements after saturation and under axial stress over time, the oedometric tests were carried-out on cylinder moulds of 70 mm inner diameter and 20 mm height. For this test, The specimens were prepared with modified Proctor procedure under controlled loading level (from 5 to 800 KPa) according to the XP-P94-090-1 [30].

In order to evaluate the impact of aging time from the early days on the strength and durability results, the elastic modulus (E) and the diametric compression (Rt) strength tests were performed on specimens with a diameter of 50 mm and a height of 100 mm at 7, 28, 90 and 360 days curing period according to NF–P98-232-3 [31] (Fig. 1). For this test, the cylindrical specimens were prepared at the optimum of the modified Proctor characteristics. To define the structural class of the designed mixes, the maximum (E) and (Rt) values obtained at 90 and 360 days curing ages (aging times requested by the standards for classifying road material) were then reported in a specific abacus according to the capping layer technical guide [32] and the pavement layers standard NF–P98-113 [33]. To characterize the pozzolanic reaction development in the wetting and drying conditions and to evaluate the water immersion stability, the unconfined compressive strength (UCS) tests were conducted over each sample following the NF-EN-13286-41 [34] standard. For this test, the cylindrical specimens were compacted at the optimum of the modified Proctor characteristics before being stored at room temperature in the laboratory or immersed in the water at a temperature of 20 °C before testing at different curing times.

The chemical composition of the solid samples was analysed using an X-ray Fluorescence technique (Panalytical, Epsilon 4 Model) and the elemental analysis was investigated by inductively coupled plasma with atomic emission spectroscopy (ICP-AES) (Perkin Elmer Optima 3100 RL, PerkinElmer Waltham). X-ray diffraction analysis was used to verify the mineralogical evolution of the designed mixes. XRD patterns were

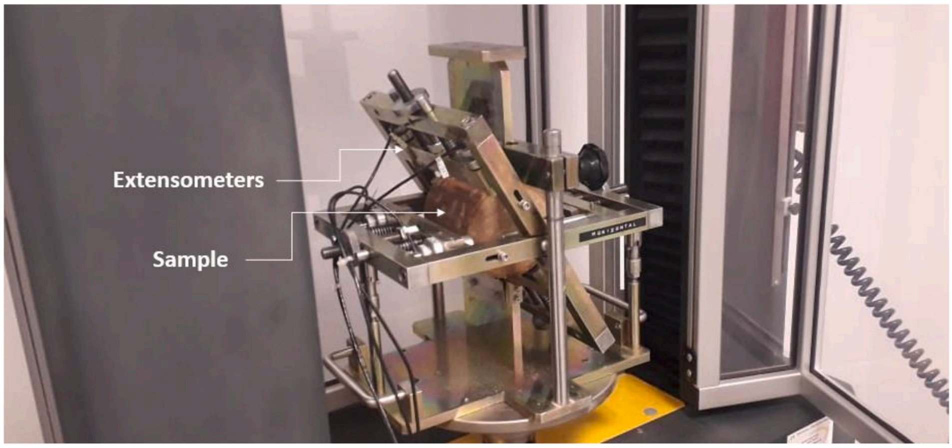

Fig. 1. Elastic modulus and diametric compression testing machine.

measured using Rietveld refinement method on dry samples prepared at the conditions of the modified Proctor and cured at 90 days aging for the capping layers and after curing period of 360 days for the pavement layers.

The environmental impact of pollutants from the material of the optimal mixes to work areas was investigated using TCLP approach. The leachate solutions were separated from the samples after CBR (4i) tests and filtered by 0.45 μm filter before tested following [35,36].

2.2.2. Testing protocol for the materials as embankment

Using material for embankment construction purposes requires investigating specific geotechnical criteria that are defined in local building rules. In Morocco, the granulometric evolution related to the fractal behaviour of grain size distribution under fragmentation, environmental impact, compaction characteristics, compressibility and sensitivity to water are the main criteria recommended for the conventional materials. All these parameters were assessed to determine the feasibility of using raw PG alone as structural embankment material. To improve its geotechnical properties for embankment use, laboratory mixes design of PG with the 0/20 mm size fraction of SM were performed. The parameters considered the most suitable for evaluating the effect of the addition of SM on the mixes from mechanical performances and geotechnical points of view are described in Table 3. These parameters allowed to provide the optimum dosage of SM to be adopted. Table 3 recapitulates the design criteria with related parameters.

2.2.3. Testing protocol for the materials use in capping and pavement layers

The specimens of solidified materials were prepared by blending the raw PG, 0/20 mm size fraction of SM with the other additives and eventually water together. Three families of formulations were tested. The first is mainly composed of PG, FA and L. To reduce the high natural moisture content and to neutralize the acidity of the raw PG, varying amounts of FA (30, 40 and 50 wt%) and L (3 and 5 wt%) were added. To improve especially finesse, plasticity and strength of the second mix, 60 wt% of SM and an amount of 3, 5 and 7 wt% of lime was added to the raw PG. To further improve the pH of the mixes, CM/PG ratio in the third mix was taken equal to 0.4 and 0.5, while the dosage of HRB has been kept fixed at 7 wt%.

To examine the suitability and ability of the mixes designed, the minimum requirements for capping layers materials as specified in French technical guide [32] were adopted. The recommended specifications are as follows:

Conditions of sensitivity to water:

- CBR (4i) $\geq$ 20%
- CBR(4i) $\geq$ IBI
- Immersion resistance index (IR) $\geq$ 0.8

Mechanical performances:

- The torque (Rt, E) determined at 90 days aging which defines the long-term structural properties must lead at least to a mechanical performance of zone 5 as specified in the French guide [32].

The criterion of the water sensibility was highlighted by the parameter of the resistance to immersion (IR) defined for capping layers by the report:

$IR = \frac{UCS\ (28+32i)}{UCS\ (60)}$, where:

- UCS (28 + 32i): UCS after 28 days in standard curing and soaked in water for 32 days at 20 °C.
- UCS (60): UCS after 60 days in standard curing conditions.

For the average daily traffic of heavy vehicles from T5 to T1 for the case of the sub-base layer and from T5 to T2 for the base layers [37], the utilization in pavement structure is conditioned mainly by the values of elastic modulus and diametric compressive strength measured at 360 curing days. These obtained values are reported in a specific graph to determine the corresponding performance class following NF P98-113 which must lead at least to S2 class [33]. The pavement use also requires the fulfilment of the immediate stability conditions [37]:

- IBI > 50 for the base layer
- IBI > 35 for the sub-base layer.

Table 4 summarizes the experimental plan for monitoring the proposed criteria.

2.2.4. Mixes design

To offer sustainable and economically competitive mixes, an

Table 3
Design criteria and quality monitoring parameters.

Criteria	Monitored parameter
Neutralization of acidity	pH
Fractal behaviour of grain size distribution under crushing process	Grain size distribution
Water erosion	Grain size distribution, MBV, PI, C_p'
water sensitivity	CBR (4i)
Compaction characteristics	(W_{OPM}.ρ_{dOPM})
Compressibility	C_c. C_s and s'p
Environmental stability	TCLP test of harmful elements

Table 4
Design criteria and quality monitoring parameters.

Criteria	Monitored parameter	
	Capping layers	Pavement layers
Immediate stability	IBI	IBI
Water sensibility	IR, CBR(4i)	IR, CBR(4i)
Mechanical performances (long-term)	(E,Rt) at 90 days aging	(E,Rt) at 360 days aging
Neutralization of acidity	pH	pH

optimization approach was taken into consideration during the design of materials. The amount of raw PG has been maximised while HRB amount should not exceed the limit of 7 wt%. The mix composition of embankment and pavement road materials were listed in Table 5. Only optimal mixes were presented.

3. Results and discussions

3.1. Use of PG alone in embankments

The results exhibited in Fig. 2 indicate that raw PG presents an initial grain size curve continuously distributed with a uniformity coefficient value of 7.8 and an average particle diameter D_{50} of 57 μm. The raw PG had more than 85 wt% of passing through 250 μm mesh sieve and the total mass of 50/1000 μm size fraction represents about 51 wt% of the total sample. According to the study of Leguédois [38] and the above mentioned amounts of size fractions namely 0/250 μm and 50/1000 μm, raw PG is characterized by a very fine granular texture which promotes the water erosion of the embankment slide slopes.

The examination of the particle size curves under various stress strains are illustrated in Fig. 2. Results revealed a very sensitive evolution towards the fine fraction after fragmentation. The fine proportion was about 21.7 wt%, 34.70 wt% and 35.46 wt% under the stress of 0.6 MPa, 1.5 MPa and 2.7 MPa respectively. The evolution of grain fragmentation increases with increasing stress strain and the particle size distributions reached almost stable state when the stress strain exceeds the value of 1.5 MPa.

The fractal behaviour study showed that the significant degree of granulometric evolution under fragmentation make it inappropriate to envisage direct use of raw PG as embankment material without considerable prior compaction of at least 2.5 times of the standard Proctor energy.

Table 5
The mix composition of different proposed road technique materials.

Use	Mix	Mix proportion (wt%)
Embankment	E0: PG	PG = 100
	E1:PG-SM	PG:SM = 50:50
	E2:PG-SM	PG:SM = 40:60
	E3:PG-SM	PG:SM = 30:70
Capping and pavement layers	P1: PG-FA	PG: FA = 50:50
	P2: PG-FA	PG:FA = 60:40
	P3: PG-FA	PG: FA = 70:30
	P4: PG-FA-L	PG:SM:L = 40:60:3
	P5: PG-FA-L	PG:SM:L = 40:60:5
	P6: PG-SM	PG:SM = 40:60
	P7: PG-SM-L	PG:SM:L = 40:60:3
	P8:PG-SM-L	PG:SM:L = 40:60:5
	P9:PG-SM-L	PG:SM:L = 40:60:7
	P10:PG-SM-HRB	PG:SM:HRB=(25:75):7
	P11: CM-PG-SM-HRB	CM: PG:SM:HRB=(5:30:65):7
	P12: CM-PG-SM-HRB	CM: PG:SM:HRB=(10:25:65):7
	P13:CM-PG-SM-HRB	CM: PG:SM:HRB=(10:20:70):7

The observations of morphology of the raw PG samples obtained by scanning electron microscopy (SEM) were presented Fig. 3. The images obtained by SEM with various magnifications highlighted that the studied raw PG has a tabular crystalline morphology appear as platelet in parallelepiped shape randomly oriented with lengths ranging from a few micrometers to 400 μm and thicknesses less than 10 μm. The ratio thickness over the grain length remains extremely low, thus favouring easy crushing under various stresses.

A drained consolidated shear test with shear box apparatus was performed on compacted PG samples. As the effective vertical stress during sampling is less than 100 KPa (small stockpiles), the test specimens were consolidated at various confining pressures of 50, 100 and 200 KPa. The experimental tests allow to determine the following shear strength parameters: $C_p' = 2$ KPa and $\phi_p' = 35°$. The very low effective cohesion C'_P value indicates a pulverulent behaviour of the PG material that can be explained by the high sensitivity to water and the level of the applied pressure. One possible explanation for the large effective internal friction angle ϕ_p' could be the angular shape and the roughness surfaces of the particles of the PG material.

Load-carrying capacity using the California Bearing Ratio (CBR) tests were performed on raw PG samples moulded with variable water content. The results depicted in Fig. 4 shows that the maximum value of CBR (4i) does not coincide with the W_{OPM}. A remarkable drop in bearing capacity was also noted for moisture content values beyond the W_{OPM}. In fact, a decline of 65% in the CBR (4i) measurement was observed for only 4% moisture content variation. This demonstrates clearly the high sensitivity of raw PG to water variation. It can be concluded that for a maximum bearing capacity of implementation, the layers of PG embankment must be compacted at moisture contents in the range of 0.86–0.96 of W_{OPM}.

Fig. 4 illustrates also the Proctor compaction test results as obtained for the raw PG samples for both compaction stresses (standard and modified energies). It should be noticed that increasing the compaction energy from standard to modified Proctor increases significantly the maximum dry density from 13.4 kN/m^3 to 14.8 kN/m^3 (increase of 10.45%) and decreases the optimum moisture content from 18.3 wt% to 17 wt% (decrease of 7.1%). This can be explained by the fact that the increase in energy has been accompanied by a decrease in porosity, a rearrangement of PG particles and therefore a higher density. It can be concluded from the compaction characteristics, CBR ratios and grain size distribution after fragmentation that the raw PG presents a very low dry density the remarkable granulometric evolution of raw phosphogypsum does not allow to directly envisage its use in embankment without a true prior compaction energy equivalent to 2.5 times the effort of the standard Proctor test.

To define the compressibility and consolidation parameters of raw PG sample, an oedometric test was conducted. The raw PG compression characteristics are presented in Fig. 5. The settlement begins to appear just after a low loading strain and revealed an important accentuated slope to which correspond high values of the settlement declined by compression index C_c of 0.25. A significant secondary settlement has been observed which continues slowly even after 53 days loading. However, the shape curve indicates a very slight PG swelling index Cs with a value of 0.012 and a very low preconsolidation stress s'_p of 35 KPa which corresponds to a permeability coefficient of 1.02 10^{-7} m/s. The studied raw PG showed a strong compressive behaviour with slight potential swelling.

Taking into account the compaction characteristics results of the modified Proctor test which reveals a low dry density of 14.8 kN/m^3, the results of the compressibility study indicate a very low preconsolidation stress of 35 KPa, a significant compression index of 0.25 and a high initial water content (>1.2 W_{OPM}). Consequently, the compaction of PG is impossible for the direct use. Therefore, the application of raw PG in embankment requires the consideration of the stability measures. Considering that the regulation in Morocco often requires settlements less than 10 cm in 25 years on ordinary road embankments, the stability

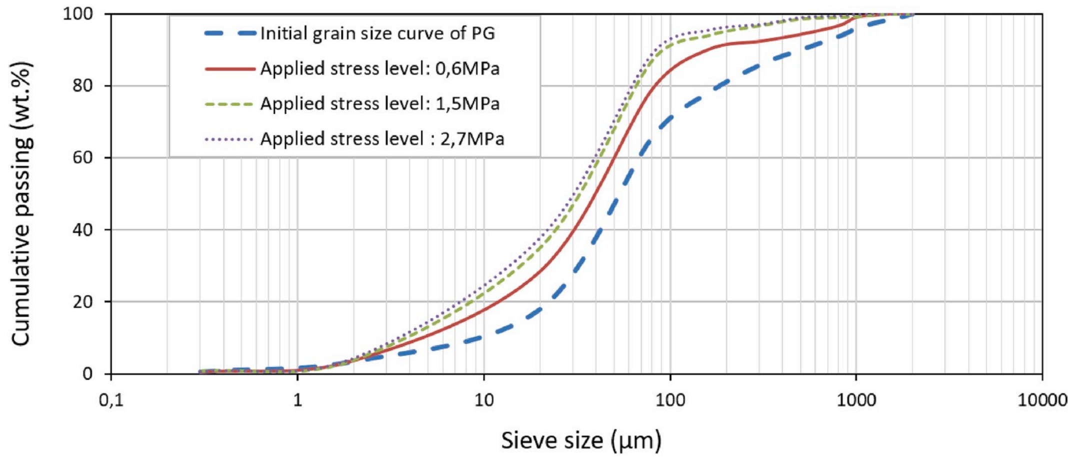

Fig. 2. Evolution of grading curves of raw PG under different stress levels.

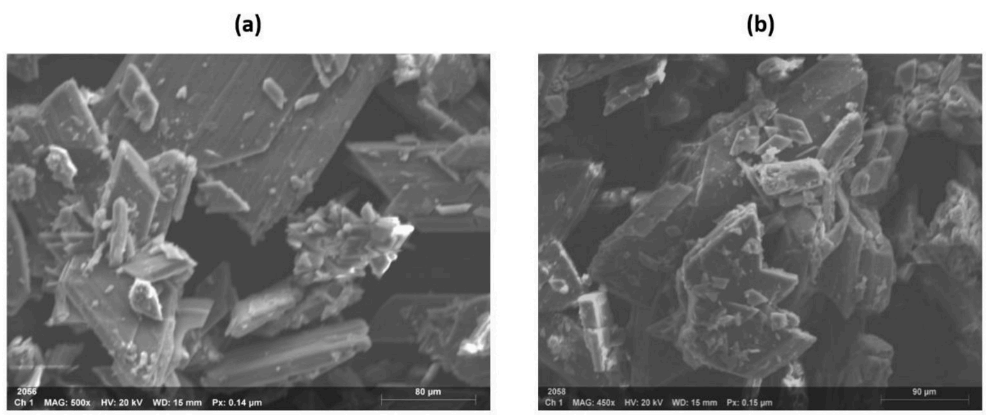

Fig. 3. SEM secondary electron images showing the morphology of the grains of raw studied phosphogypsum sample: 500x (a) and 450x (b).

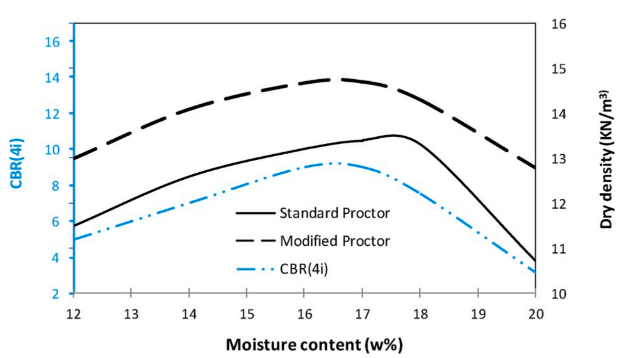

Fig. 4. CBR (4i) and dry density as a function of moisture content of raw PG material.

investigation was carried out by the calculation of the total predictable consolidation settlements by cumulating the elementary settlements determined for each fraction of 1 m of the embankment height under an operating load of 10 KPa. Based on the settlement results detailed in Table 6, it can be concluded that the employment of PG alone should be strictly limited to ordinary embankments (out of flood areas, engineering structures and top part of the earthworks) of heights less than 4 m. Moreover, to avoid the water erosion of the side slopes of the embankment, it is very advisable to provide containment with other selected materials.

The TCLP test was used to evaluate the leaching behaviour of raw PG. The obtained concentrations are compared with US-EPA thresholds

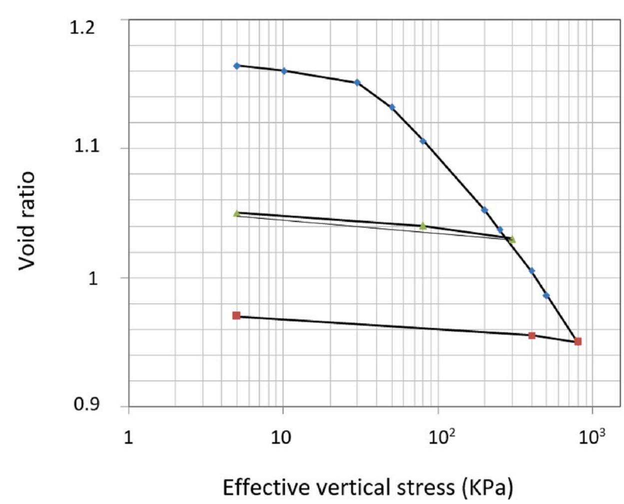

Fig. 5. Compressibility curve of compacted phosphogypsum.

Table 6
Predictable settlements in cm of PG embankment at different heights.

Embankment heights (m)	1	2	3	4	5
Settlements (cm)	1.4	4.8	7.2	9.2	11

Table 7
Results of the TCLP test of raw PG.

Mix	Zn	Se	Pb	Cu	Cr	Mo	Ni	Co	Cd	As	V
	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l
PG = 100	2.05	0.26	<0.60	0.19	0.56	0.18	0.3	1	<2	0.56	<1
Limits (US-EPA)	2	1	5	–	5				1	5	–

[36]. The results in terms of trace elements leaching are summarized in Table 7. Except Zinc which flirted with the requested threshold, the concentrations of the other elements were all in agreement with the standard.

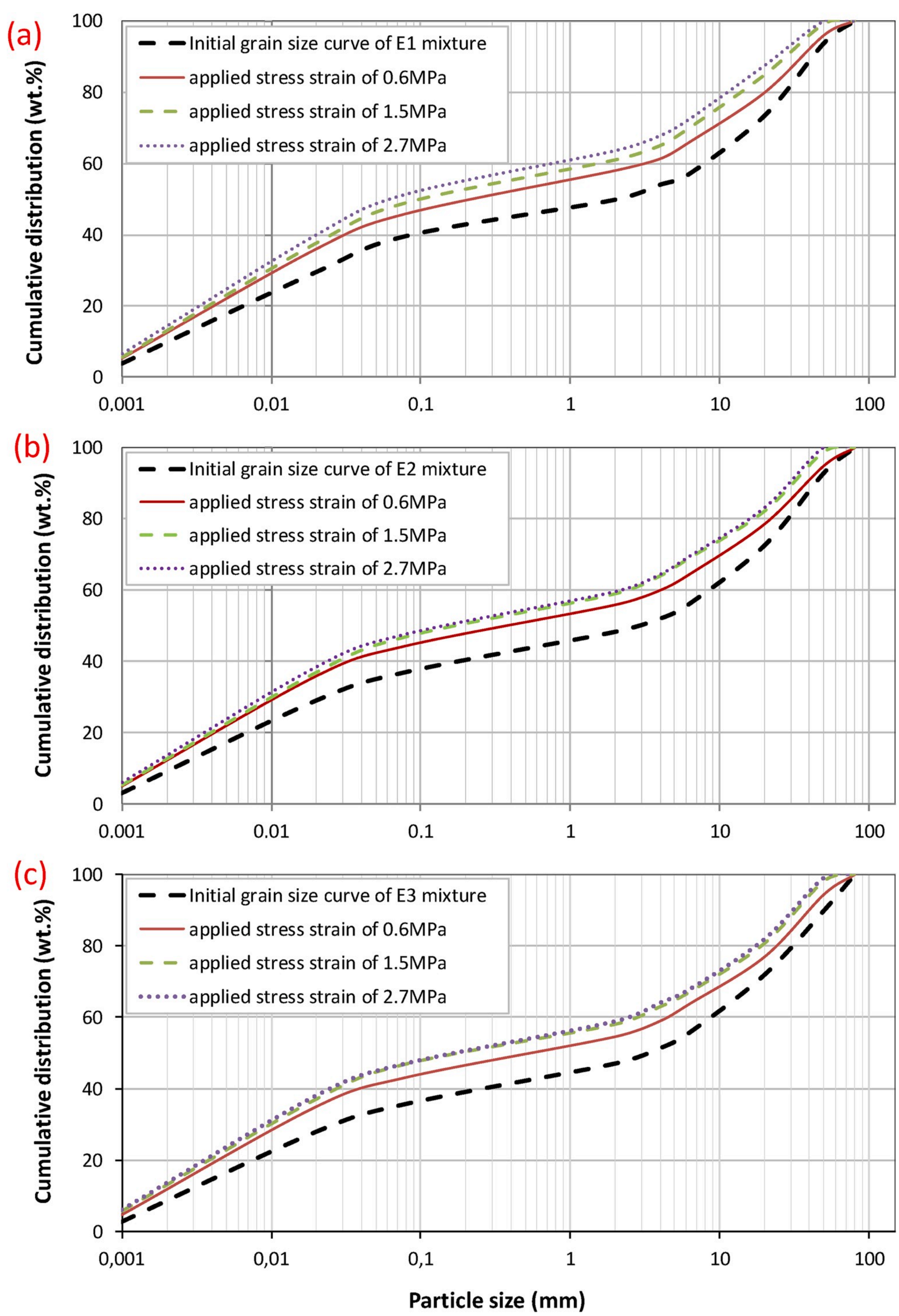

Fig. 6. Grain size evolution under fragmentation process of (a) E1 mix (b) E2 mix and (c) E3 mix.

3.2. Phosphogypsum-soil solidified material as embankment material

3.2.1. The effect of the soil material (SM) content on the fractal behaviour of particles size distribution

The impact of SM content on the mixes was evaluated in this section. The results of the evolution of grain size distribution of the designed mixes under stress conditions were given in Fig. 6. It can be observed that for the same energy level, the increase of the SM amount improves significantly the fineness of the PG texture. Under the effort level of 2.7 MPa, the D_{50} has been increased from 32 μm for PG mix to 60 μm (E1), 160 μm (E2) and 180 μm (E3). The curves of Fig. 5 depict the impact of stress strain on the fractal behaviour of the particle distribution. The granulometric evolution degree is more remarkable as the amount of raw PG is high. Fig. 6 also reveals that the grading curves shift downward and producing less fine particles as the SM content increases. The E1 mix which remains the most sensitive to fragmentation with a grain size evolution keeps still evolving even under the energy level of 1.5 MPa unlike other mixes whose reach almost a stable particle size distribution under the same degree of stress. Compared to the initial PG gradation curve, a very significant improvement in terms of granulometric evolution towards the fine fraction was observed for the three mixes. Moreover, under stress level of 2.7 MPa, the percentage of the particles size of less than 80 μm has narrowed from 89 wt% for raw PG to 51.4 wt%, 47.7 wt% and 46.9 wt% for E1, E2 and E3 mix respectively.

The two sizes fraction most affected by water erosion are significantly improved with the addition of SM to PG. In fact, for the case of E2 mix under stress strain of 2.7 MPa, the percentage was decreased from 96 wt% to 52 wt% which constitutes an enhance of 41.66% and from 30 wt% to 11 wt% which represents an improvement of 63.33% for 0/250 μm and 50/1000 μm size fraction respectively.

3.2.2. The effect of SM content on physicochemical properties of the designed mixes

The results of geotechnical characterization tests of the PG-SM mixes performed in this study are given in Table 8. The pH has been increased respectively from 2.2 of raw PG to 3.6, 4 and 4.4 for the E1, E2 and E3 mixes. The acidity was not the only characteristic improved by the addition of SM amount, but also all the monitoring parameters are positively influenced. The addition of SM to raw PG increases the maximum dry unit weight while decreasing the optimum moisture content by 10.1% and 13.33% respectively for E2 mix. The latter shows the maximum dry density results for both compaction energies of all mixes. Plasticity and cohesion, considered as the key parameters which reflect directly the resistance to water erosion, had been significantly improved with the addition of SM content. This has been highlighted by the results of effective cohesion, plasticity index and the methylene blue value of the mixes. The measurements of the load-bearing capacity of PG-SM mixes indicate that the maximum CBR (4i) value was observed as 16% for the E2 mix. Concerning the evolution of the CBR(4i), it was about 77.77%, 111%, and 100% for E1, E2, and E3 mix respectively. The mechanical response of the E2 mix is, therefore, less sensitive to water compared to the other mixes. This study also indicates that for E2 mix, the compressibility parameters have been significantly improved; indeed, the compression index C_c has been decreased from 0.25 to 0.158 while the preconsolidation stress s'_p has been increased from 35 MPa to 71 MPa which represents an improvement of 36.8% and 103% respectively. This can be explained by the important role of carbonate as a cementing and cohesive agent of mix. Based on the results analysis above, the E2 mix allows to use a significant amount of raw PG by maintaining the best performances in terms of evolution of granulometric distribution, immersion resistance, bearing capacity, compaction characteristics and compressibility parameters. The E2 mix can be considered as the most advantageous from a physicochemical and economical aspects.

The stability of the embankments that will be built with the materials of the E2 mix was investigated. The results make it possible to conclude that E2 mix can be used for construction of road embankment for heights less than 8 m without any significant risk of instability, obviously with design specifications to ensure sliding stability.

In addition to the study of the SM influence on the geotechnical properties of PG, the leaching behaviour of trace contaminants from PG-SM stabilized material was also investigated. The leachate quality results found in the optimal mix material are given in Table 9. It is obvious that the concentrations of the harmful elements are all negligible and far below the standard limits.

3.3. Use PG in layers of pavement structure

According to the procedure described above, the findings of the observed effects of addition of SM, FA, L, CM and HRB to the mixes on the behaviour in terms of initial acidity, durability, water sensitivity, bearing capacity and strength properties are summarized in Table 10.

Recognized by a strongly alkaline buffering effect (pH > 11.4), the addition of FA significantly reduced the PG acidity of the first family mix. However, their effect on the mechanical properties remains insignificant especially for the bearing ratio results. The unconfined compression strength increases very slightly with the increase of curing period.

From Table 10, It can be seen that the SM improved clearly both water sensitivity resistance and the mechanical performance of mixes of the second family. With the addition of 3 wt% of lime for the P2 mix, the load bearing capacity performances have undergone a very important improvement; 150%, 734.21% and 15.09% for CBR (4i), IBI and IR respectively. These last parameters are improved further by increasing the dosages of the lime. While the obtained results concerning diametric compression strength and elastic modulus measurements remained very low to the point that they cannot be detected by the testing machine (limits: 500 MPa for elastic modulus and 0.09 MPa for diametric compression strength). A possible explanation for this finding could be that L/PG incorporation ratio was very low to neutralize the initial acidity generated by the PG and therefore does not provide an appropriate environment conditions for cement hydration process.

Stronger trend of increase in terms of load bearing capacity and resistance to water was noted by changing lime with a specific road binder (HRB) but still with insignificants results regarding the mechanical performances (E,Rt) (the results always remain below the minimum detection threshold of the testing device).

The remarkable enhancement in stiffness was observed since the

Table 8
Physical properties of the designed mixes.

Mix	pH	Shear test		CBR (4i) (%)	Modified Proctor test		Standard Proctor test		MBV (g/100g)	Atterbreg limits		Oedometric test		
		C_p' (KPa)	ϕ_p' (°)		ρ_{dOPM}(kN/m^3)	W_{OPM} (%)	ρ_{dOPN}(kN/m^3)	W_{OPN} (%)		W_L (%)	PI (%)	C_c	C_s	s'_p (KPa)
SM	6.9	11	29	14	18.8	13.1	18.5	13.3	0.69	44	16	0.132	0.036	123
E0	2.2	2	35	9	14.8	17	13.4	18.3	0.09	57	<6	0.25	0.012	35
E1	3.6	5	33	16	15.9	15.3	15.2	16.2	0.32	49	9	0.17	0.017	62
E2	4	7	31	19	16.5	15	15.8	15.7	0.36	46	10	0.158	0.019	71
E3	4.4	7	30	18	16.4	15.1	15.7	15.7	0.39	43	11	0.153	0.024	70

Table 9
Results of TCLP test.

Mix	Zn	Se	Pb	Cu	Cr	Mo	Ni	Co	Cd	As	V
	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l
E2	1.05	0.23	<0.60	0.06	0.09	0.1	0.2	0	<2	<4	<1
Limits (US-EPA)	2	1	5	–	5	–	–	–	1	5	–

Table 10
The engineering properties of designed mixes.

Mix	pH	CBR ratio (%)		IR	E (MPa)				Rt (MPa)			
		IBI	CBR (4i)		E(7)	E(28)	E(90)	E(360)	Rt (7)	Rt(28)	Rt(90)	Rt(360)
E0	2.2	15	9	0.84	–	–	–	–	–	–	–	
P1	4.1	18	8	0.84	<500	<500	<500	<500	<0.09	<0.09	<0.09	<0.09
P2	4.8	21	10	1.00	<500	<500	<500	<500	<0.09	<0.09	<0.09	<0.09
P3	5.1	20	7	0.95	<500	<500	<500	<500	<0.09	<0.09	<0.09	<0.09
P4	5.7	39	29	1.05	<500	<500	<500	<500	<0.09	<0.09	<0.09	<0.09
P5	6.2	45	39	1.20	<500	<500	<500	<500	<0.09	<0.09	<0.09	<0.09
P6	4.3	38	16	1.06	<500	<500	<500	<500	<0.09	<0.09	<0.09	<0.09
P7	5	51	40	1.22	<500	<500	<500	<500	<0.09	<0.09	<0.09	<0.09
P8	6.1	59	53	1.31	<500	<500	<500	<500	<0.09	<0.09	<0.09	<0.09
P9	6.7	63	65	1.35	<500	<500	<500	<500	<0.09	<0.09	<0.09	<0.09
P10	6.8	71	78	1.47	<500	545	671	739	<0.09	<0.09	<0.09	0.1
P11	8.4	75	122	1.51	<500	675	723	812	<0.09	<0.09	0.11	0.15
P12	10.8	88	329	1.67	2864	6341	8390	9453	0.18	0.31	0.45	0.59
P13	10.4	82	301	1.65	1986	5997	7795	8972	0.16	0.22	0.37	0.44

early cure stage only after adding CM content and with incorporation ration CM/PG of at least 0.17.In fact, for the P12 mix, the addition of 10w% of CM content which constitutes a CM/PG ratio of 0.45, the diametric compression strength and elastic modulus were 0.18 MPa & 2864 MPa at 7 days curing time respectively; which augmented with the increase in curing period to 0.31 MPa & 6341 MPa, 0.45 MPa & 8390 MPa and 0.57 MPa & 9453 MPa when the curing time was raised to 28, 90 and 360 day respectively. Satisfactory but less important performances results compared to P12 mix were also obtained after the addition of the same content of CM but with a larger CM/PG ratio of 0.5. It can be easily concluded that the best mechanical performances have been obtained for incorporation ratio CM/PG ratios ranging from 0.17 to 0.45 which corresponds to P12 and P13 mixes (optimal mixes). The spectacular increase in stiffness performance can be attributed (first remark) to physical and chemical changes in alkaline conditions. The pH was enhanced from 6.8 for P10 mix to 8.4, 10.8 and 10.4 for P11, P12 and P13 respectively.

3.3.1. *Use of PG in capping layers*

From Fig. 7, the experimental results in terms of the (E,Rt) at 90 curing time indicate that the two mixes P12 and P13 are, respectively, classified in zone 3 and zone 4as specified in the French technical guide. Furthermore, concerning the minimum requirements of the water sensitivity and immediate stability as described in paragraph 2.2.3, the results analysis showed that they all are satisfactory, in terms of requirements. Mixes P12 and P13 can both be used successfully as material for the construction of capping layers.

3.3.2. *Use in pavement layers*

The obtained results from diametric compression strength and elastic

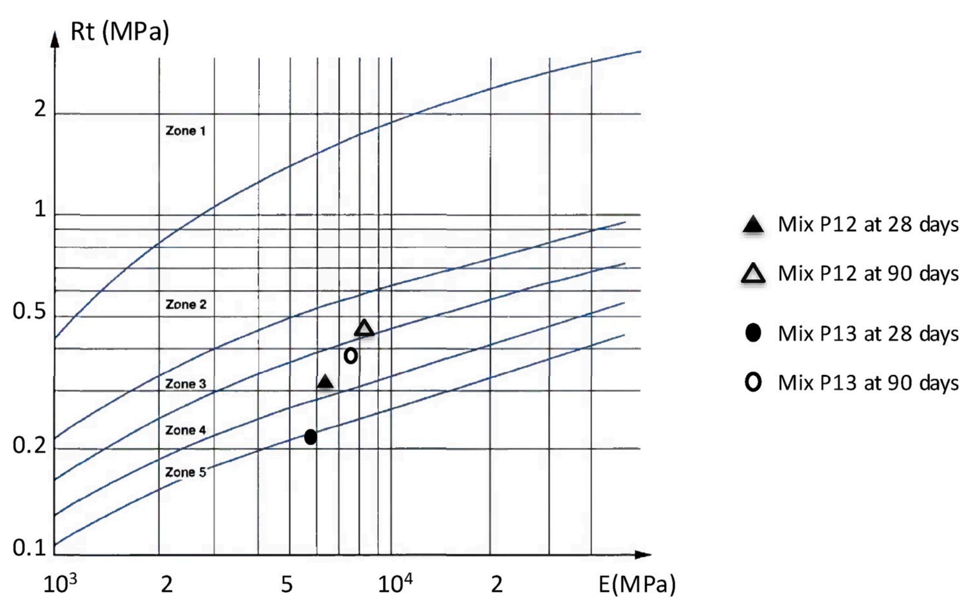

Fig. 7. Areas of classification of the designed mixes according to the torque (E, Rt) at 90 curing days French technical Guide.

modulus measurements after curing period of 90 and 360 days are presented in Fig. 8. According to the [33], P12 and P13 mixes are classified in the S2 class and S1 class respectively. It can be observed that only P12 mix can satisfy the minimum requirements of structural classification for the pavement use. Based on the results listed in Table 10, the P12 mix also verifies the adequacy of the immediate stability specifications for both foundation and base layers of the pavement structure.

3.3.3. The strength development mechanism of the PG:SM:CM:HRB mix

It was demonstrated by XRD (Table 11) that the addition of CM sample in the mix allowed the development of ettringite ($3CaO{\cdot}Al_2O_3{\cdot}3CaSO_4{\cdot}32H_2O$) phase during the early period of hydration reaction. Therefore, the strength behaviour of the CM-PG-SM-HRB solidified material is better compared to the other. The slight difference in structural classification between P12 and P13 given in Figs. 7 and 8 could be explained by the possible influence of moisture content and blending alkalinity conditions. The specimens corresponding to P12 and P13 were respectively prepared under a moisture content of 14.7 wt% and 14 wt% and an alkalinity of 10.8 and 10.4 respectively. With the addition of CM sample, the pH of the mix was increased to reach basic values, which will provide an appropriate environment for cement phases hydration. The CM has a slower neutralization kinetics than lime, which favours the robustness of the mix.

This calcium sulfo aluminate mineral is the result of the pozzolanic reaction between calcium, aluminium and sulfates species provided by the different raw materials following the reaction:

3CaO Al2O3 n H2O+3CaSO4 2H2O+(32-6-n) H2O → 3CaO Al2O3 3CaSO4 32H2O

The environmental impact of the used optimal mixes in the pavement layers was also assessed according to the TCLP test. The results of trace elements leaching found are given in Table 12. Except Zn which slightly exceeds the limits required by USEPA, the concentrations of the other elements were all in agreement with the normalized values.

4. Conclusions

In the present paper, the feasibility of using raw phosphogypsum alone in embankment was evaluated considering the criteria of competitiveness aspect compared to conventional materials. To improve the geotechnical properties of the raw PG, the stabilization with a predominantly silty gravel material was also studied. The use as capping layers and road pavement layers material requires appropriate mechanical performances which is why phosphogypsum has been stabilized with other additives having better cementitious characteristics. in the light of the experimental investigations results, the following conclusions can be drawn:

Table 11
Mineralogical compositions of cured samples P12 and P13.

Mineralogical composition (w%)	P12 Mix		P13 Mix	
	90 days	360 days	90 days	360 days
Gypsum	31.1	29.9	30.5	29.2
Calcite	29.4	30.1	31.6	30.4
Dolomite	17.6	17.2	16.4	16.1
Quartz	14.7	14.5	14.4	13.9
Fluorapatite	5.1	5.1	5.6	5.6
Ettringite	2.1	3.2	1.5	2.4

- Chemical and mineralogical analyses showed that the Moroccan phosphogypsum is characterized by a strong acidity composed mostly of gypsum.
- With a friability coefficient of 68, 51 wt% of the 50/1000 μm size fraction and more than 68% of passing through 250 μm opening size, the studied raw PG can be compared to an eroded and very friable fine sand. On the other hand, the significant degree of granulometric evolution under fragmentation conditions makes it inappropriate to envisage direct use of raw PG as embankment material without considerable prior compaction of at least 2.5 times of the standard Proctor energy. Therefore, the employment of PG alone should be strictly limited to ordinary embankments (out of flood areas, engineering structures and top part of the earthworks) of heights less than 4 m.
- The main parameters determining the geotechnical characteristics of the raw phosphogypsum namely: dry density of the modified proctor test, compression index, preconsolidation stress, CBR(4i), fractal behaviour under fragmentation conditions and resistance to water erosion have shown a significant improvement with the addition of SM content. with the following proportion PG: SM = 40:60, the height of the embankment can be doubled and reach 8 m.
- The utilization of the phosphogypsum is also possible in the construction of road pavements. This study shows that we can obtain the required mechanical performance in terms of (E, Rt) for the construction of capping layer, foundation and base layer by adopting the following formulation:(CM:PG:SM) HRB=(10:25:65):7. The improvement observed in terms of mechanical properties can be referred to the influence of moisture content and blending alkalinity

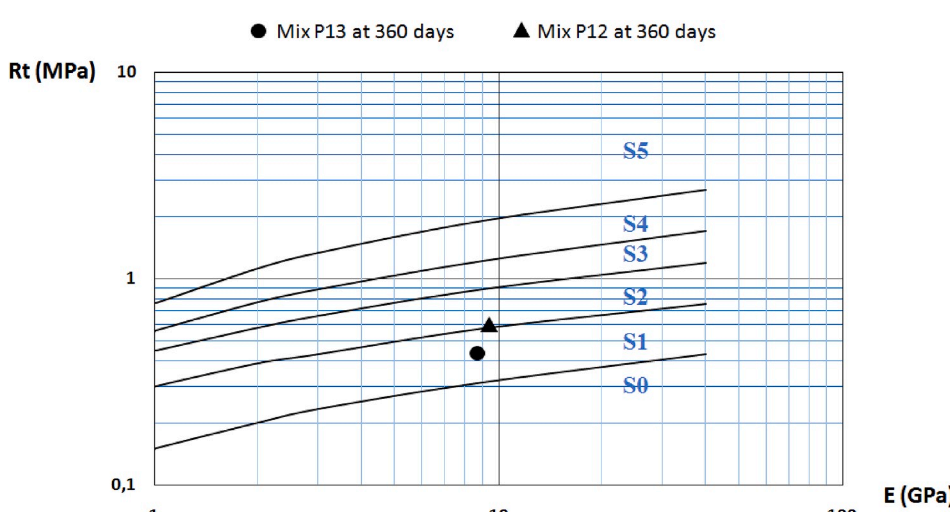

Fig. 8. Structural classification of mixes according to NF–P98-113 [33].

Table 12
TCLP leaching test results of P12 and P13 mixes.

Mix	Zn	Se	Pb	Cu	Cr	Mo	Ni	Co	Cd	As	V
	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l	mg/l
P12	3	<1	<1	<0.5	<0.2	<1	<0.5	<0.5	<1	<1	<1
P13	4	<1	<1	<0.5	<0.2	<1	<0.5	<0.5	‘1	<1	<1
Limits (US-EPA)	2	1	5	–	5	–	–	–	1	5	–

conditions which allow the formation of the ettringite phase that contribute to enhance the mix strength.

- The results of the environmental behavior of the optimal mixes indicate that except for the Zinc content, which slightly exceeds the limits required by USEPA, the concentrations of the other undesirable elements (leaching of heavy metals) were all in accordance with the standard specifications. Consequently, there is no potential risk of contamination from materials of the optimal mixes towards the surrounding environment
- Based on the results of the present study, large quantities of phosphogypsum material can be valorized in various layers of the road construction obviously inside the profitability perimeter. To test the behavior of the pavement and embankment layers that will be built according to the recommendations of this study, further full-scale field investigations will be carried out soon on an experimental road section.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRediT authorship contribution statement

Mustapha Amrani: Conceptualization, Validation, Writing - original draft. **Yassine Taha:** Investigation, Writing - original draft, Writing - review & editing. **Azzouz Kchikach:** Supervision, Methodology, Supervision, Writing - review & editing. **Mostafa Benzaazoua:** Supervision, Methodology, Supervision, Writing - review & editing. **Rachid Hakkou:** Supervision, Methodology, Supervision, Writing - review & editing.

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