Characterístics of phosphogypsum for utilisation in roadwork fills

Caractéristiques des phosphogypses utilisés dans les travaux d'emblayage des routes

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ABSTRACT

Phosphorus oxide for use as fertilizer is produced by using sulphuric acid to attack natural phosphates and this generates a byproduct essentially composed of hydrated calcium sulphate and known as phosphogypsum. The solubility of calcium sulphate, even in the natural rocks and soils containing gypsum, limits the utilisation of this material in slopes for linear works to a maximum calcium sulphate content of 20%. Other elements also appear in phosphogypsum that, in the event of solubilising, could contaminate the water in the surrounding area. In spite of all this, experience does exist with the use of phosphogypsum in experimental slopes designed to ascertain the possibility of storing this product in more areas capable of guaranteeing that the surroundings are not contaminated. This article reports on the characteristics of the Huelva phosphogypsum in terms of both composition and mechanical properties and the measures to be taken for its possible utilisation in roadwork fills.

RÉSUMÉ

La production d'oxyde de phosphore comme fertilisant est basée sur l'action de l'acide sulfurique sur les phosphates naturels, ce qui donne un sous-produit constitué fondamentalement de sulfate de calcium hydraté, connu sous le nom de phosphogypse. La solubilité du sulfate de calcium, même dans les sols et les roches naturels contenant du gypse, limite l'utilisation de ces matériaux dans les terre-pleins pour les travaux linéaires jusqu'à une teneur en sulfate de calcium de 20%. Dans le cas des phosphogypses, il apparaît en outre d'autres éléments qui, s'ils se solubilisent, pourraient contaminer les eaux voisines. Il existe malgré tout des expériences sur l'utilisation des phosphogypses dans des terre-pleins expérimentaux afin de savoir s'il est possible de stocker ce produit dans d'autres zones avec la garantie de ne pas contaminer l'environnement. Cet exposé décrit les caractéristiques des phosphogypses de Huelva, leur composition ainsi que leurs propriétés mécaniques, et les mesures à prendre dans le cas d'une possible utilisation dans les travaux d'emblayage des routes.

Keywords : phosphogypsum, fills, roadwork

1 INTRODUCTION

Phosphorus oxide for use as fertilizer is produced by using sulphuric acid to attack natural phosphates and this generates a byproduct essentially composed of hydrated calcium sulphate and known as phosphogypsum.

Phosphorus acid can be produced by three processes namely dehydrated, hemi-hydrated and hemi-dehydrated.

The dehydrated process involves a small capital investment and low production costs. It is the widest used internationally and the one applied in the Huelva plant. It generates 4.9 tons of phosphogypsum for every ton of phosphorus oxide produced. The phosphogypsum generated is the type containing the highest number of impurities compared to the other processes.

In 2007, phosphogypsum utilisation worldwide amounted to 35% in construction, 23% in cement production, 23% in agriculture, 5% in chemicals, 5% in oil extraction and 4% in ceramics.

This research work was designed to study the utilisation of phosphogypsum in roadwork fills, giving great importance to the characteristics of this material when compacted to the densities obtainable on site.

2 CHARACTERISTICS OF THE HUELVA PHOSPHOGYPSUM

The characteristics of the Huelva phosphogypsum were determined on a sample from the local phosphorus oxide plant.

2.1 *Grading and density of the solid*

Figure 1 gives the results of the grading analysis showing that 85% of the material passed through an 0.08 screen meaning it could be considered a gypspherous silt.

The phosphogypsum particles had a density of $\gamma = 2.36$ g/cm^3.

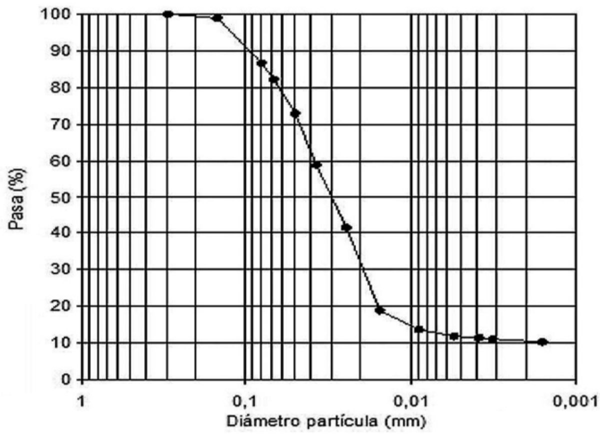

Figure 1 Grading analysis of the Huelva phosphogypsum

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2.2 *Chemical composition*

Table 1 gives the elements appearing in a higher proportion than 0.1%. The highest values among these corresponded to the gypsum $SO_4Ca\ 2H_2O$ components, which gave a 93.6% proportion of calcium sulphate in the phosphogypsum. The remainder essentially consisted of phosphates (PO^{3-}_4=3%).

Another interesting characteristic was the pH value of the phosphogypsum as the solubility of gypsum increases in an acid medium. In this case the value was pH = 3.7.

Table 1- Chemical Composition of the Huelva Phosphogypsum

Al_2O_3	CaO	Na^+	PO_4^{3-}	SiO_2	F	Cl	SO_3	H_2O
0.2	32	0.3	3.0	1	0.6	0.4	44.6	18

3 CHARACTERISTICS OF THE COMPACTED HUELVA PHOSPHOGYPSUM SAMPLES

The characteristics of the compacted phosphogypsum samples were determined using the Standard Proctor and the Modified Proctor Test energies.

3.1 *Maximum density and optimum moisture content corresponding to the Standard Proctor and Modified Proctor tests*

A Standard Proctor Test was run on this phosphogypsum sample (Fig. 2) achieving a maximum density of γ = 1.43 g/cm^3 for an optimum moisture content of w = 19.1%.

Comparing these results with those obtained in the seven Florida plant (Table 2), the Huelva phosphogypsum had intermediate values for maximum density and optimum moisture content.

With the energy of the Modified Proctor Test (Fig. 2), the maximum density obtained was γ = 1.56 g/cm^3 with an optimum moisture content of w = 16.9%,

Comparing these results with those obtained in the seven Florida plant (Table 2), the maximum density for the Huelva phosphogypsum was high, only exceeded by the phosphates of the Gardinier plant.

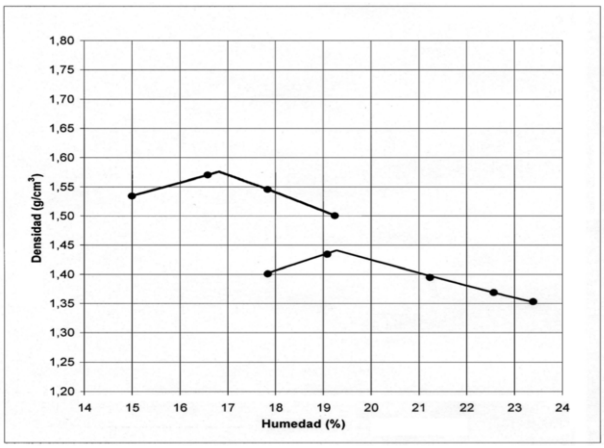

Figure 2. Result of the Standard Proctor and Modified Proctor tests run on the Huelva phosphogypsum

3.2 *Unconfined compressive strength*

The samples prepared with the SPT energy achieved an average unconfined compressive strength of qu = 0.147 MPa and those prepared with the MPT energy achieved an average unconfined compressive strength of qu = 0.340 MPa, both tested with the preparation moisture content.

3.3 *Triaxial compression. Angle of friction*

Four triaxial compression tests were run, two on consolidated and undrained samples and the other two on consolidated and drained samples. In each pair of tests, one was done on a sample compacted with the SPT energy and the other on a sample compacted with the MPT energy.

Figure 3 and Table 2 give the results of the tests. The angle of friction was between φ = 48º and φ = 54º, the higher value corresponding to the sample with the higher density γ = 1.60 g/cm^3. The cohesion also ranged between c = 0.040 MPa and C = 0.067 MPa.

Table 2 also gives the results of the tests run on the phosphogypsum from the seven Florida plant where the maximum friction angle value obtained was φ = 50º with zero cohesion.

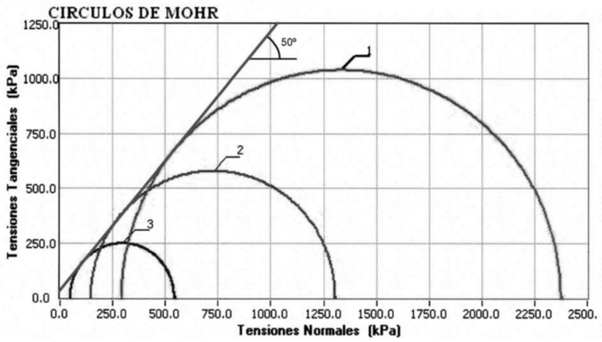

Figure 3. Consolidated and drained triaxial test (CD) (γ = 1.43 g/cm^3; w = 20.7%).

3.4 *Deformability*

An oedometer test was run on a sample with a density of γ = 1.570 g/cm^3 and moisture content of w = 13.5%, which produced the consolidation curves shown in Figure 4, indicating that for a pressure of σ' = 147.19 KPa scarcely any deferred settlement occurred but that from this pressure upwards, deferred settlement increased as also the time required to stabilize it. The oedometer curve (Fig. 5) shows the sharp rise in settlement when the load was increased to σ' = 294.30 KPa.

The deformation produced up to the point of obtaining a load of σ' = 294.30 KPa was ε = 4%.

3.5 *Permeabilty*

Permeability tests were run on samples prepared with the SPT and with the MPT density.

With the SPT density the permeability coefficient obtained was k = 4.8 . 10^{-5} cm/s, representing an intermediate value between those obtained by Ho & Zimpfer (1985) in the phosphogypsum from the seven Florida plant.

With the MPT density the permeability coefficient obtained was k = 2.3 . 10^{-5}, which was lower than the coefficient obtained with the samples compacted with the SPT energy.

3.6 *Collapsibility and expansivity*

The collapse of the Huelva phosphogypsum was obtained by running an oedometer test on a dry sample with a density of γ = 1.420 g/cm^3 and w = 0.7%. When a pressure of σ = 0.196 KPa was obtained in the oedometer, the sample was flooded with water saturated phosphogypsum. The collapse settlement obtained was ε = 1.4%, classifying this material as collapsible.

Table 2 - Proctor and Triaxial Tests on Phosphogypsum Samples from Florida, Ho & Zimpfer (1985), and from Huelva

	MP γ/w	SP γ/w	C MPa	Φ
W.R.GRACE	1.55 / 15.0	1.46 / 12.8	0	50.0º
AMAX	1.44 / 18.4	1.32 / 19.2	0	47.5º
OCCI. 1	1.47 / 17.3	1.46 / 18.9	0	45.0º
GARDINIER	1.61 / 14.1	1.52 / 16.8	0	49.0º
OCCI. 2	1.46 / 17.7	1.35 / 21.6	0	43.5º
CONSERV	1.52 / 15.0	1.44 / 19.1	0	47.5º
I.M.C.	1.55 / 16.4	1.47 / 18.4	0	46.5º
HUELVA CD		1.43 / 20.7	0.07	50º
HUELVA CD	1.60 / 14.6		0.05	54º
HUELVA CU		1.41/20.8	0.06	48º
HUELVA CU	1.57/16		0.04	48º

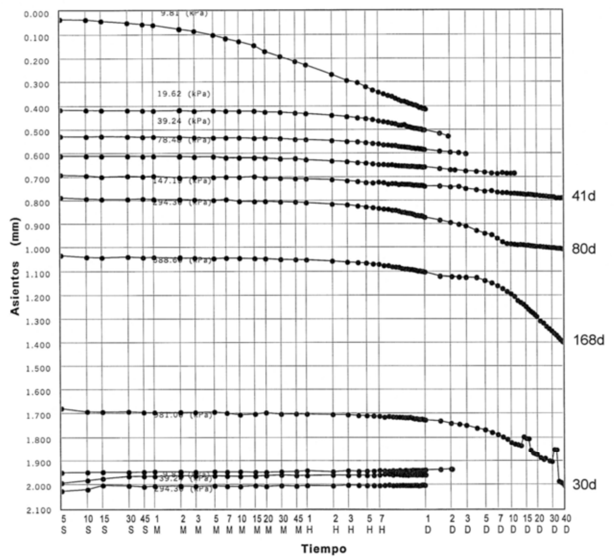

Figure 4. Consolidation of Huelva phosphogypsum samples

The swelling was determined on another sample, also dry (w = 0.7%) and with a density of γ = 1.42 g/cm^3, subjected to a pressure of σ = 98.1 KPa. It was flooded with water saturated phosphogypsum, producing a swelling of ε = 0.16% in the first five minutes. This gave way to a gradual settlement that was greater than 0.48%.

4 CLASSIFICATION OF PHOSPHOGYPSUM AS SLOPE MATERIAL

The phosphogypsum was to be classified in terms of suitability for slope material by comparing its characteristics with those specified for slope materials in the Spanish regulations.

The fines content of the phosphogypsum, as material passing an 0.063 screen, was greater than 80%, consisting of non plastic soils containing less than 2% organic matter but with over 20% gypsum and a swelling index of less than 1%.

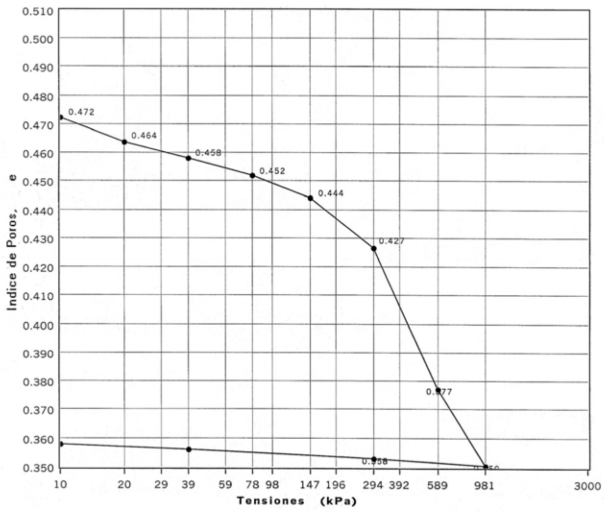

Figure.5. Oedometer curve of the Huelva phosphogypsum samples

When the soil contains over 20% gypsum it should not be used in any fill area. Its use is to be limited to cases where no other soils are available and always provided that this use is expressed and properly justified in the Design.

5 USE OF PHOSPHOGYPSUM IN SLOPE CORES

Phosphogypsum is a granular material with similar-sized grains to silts and that forms fills with a high sulphate content and substantial deformation in the long term as a result of creep, which increases as a function of the load involved.

If the use of phosphogypsum is to be permitted in the core of roadwork fills it must be capable of guaranteeing that it will not penetrate the water and that long-term deformation will not pose any problems.

Protection against water penetration can be achieved by executing the foundation, shoulders and crown of slopes with well-graded materials that on compacting are capable of achieving the desired impermeability, cores made of phosphogypsum needing to be sufficiently compacted to prevent water penetrating the interior, dissolving the sulphates or other minerals and giving rise to instability in the slopes proper in addition to polluting the water in the vicinity.

By way of added protection, the pH of fills made of phosphogypsum needs to be raised, normally by around values of 3.5, to put it over pH = 5, which maintains a low level of solubility in the dehydrated calcium sulphate comprising the majority of the phosphogypsum composition. Mixing the phosphogypsum with lime is the procedure recommended for raising the pH level.

5.1 *Behaviour of experimental slopes with phosphogypsum*

Fevre & Quibel (1998) and Quibel (1998) write up on the construction and behaviour of phosphogypsum slopes measuring 2 m, 3.5 m, 5 and 8 m high, in 1977, 1983 and 1990 respectively.

5.2 *Recommendations for the utilisation of the Huelva phosphogypsum in slopes*

In line with the Spanish regulations for roadwork slopes and the results of the lab tests on the Huelva phosphogypsum, and specifically the maximum density obtained in the Proctor tests, the angle of friction obtained through triaxial tests and the deformability obtained through oedometer tests, and bearing in mind the results of the experimental slopes executed in France, the following recommendations can be given for the use of phosphogypsum.

- Phosphogypsum should only be used in the core of slopes. The side walls of the core can have a 1:1 inclination.
- The fill should be placed above the water level as a precaution against flooding.
- The slope foundation area must be high enough to prevent any possible water gathered at the foot of the slope from reaching the base of the phosphogypsum fill.
- A 30-cm thick granular layer of clean gravel must be placed between the phosphogypsum core and the fill foundation area, capable of stopping any possible rise in water by capillary action from the ground forming the base of the fill.
- The shoulders of the fill should have a minimum width of 2 m, be sufficiently waterproof, protected from surface erosion and with an inclination of 3H:2V.
- The phosphogypsum core should have a maximum height of 2 m.
- The height of the transition zone, the crown and the other pavement layers above the phosphogypsum core should not be higher than 2 m.
- The density obtained with the phosphogypsum fill should be over 95% of that obtained with the Modified Proctor Test.
- The moisture content at the time of installation should be between -2% and +1% of the optimum Modified Proctor. The most suitable construction period will be during the dry months, normally from May to December in Spain.
- In order to specify installation conditions, test layers should be carried out at the start of the phosphogypsum fill execution. An initial approximation could involve carrying out layers 0.25-m thick and compacted ten times.
- Owing to the deformability of the fill and the high sulphate content, phosphogypsum fills should not be built in the vicinity of masonry works, particularly in transition zones.
- The pH of the phosphogypsum at the time of installation should be equal to or greater than 5. To achieve this it should be stabilized with lime.

6 SUMMARY AND CONCLUSIONS

The Huelva phosphogypsum consists of gypsipherous silt in which 85% of the material passes through an 0.08 screen and the density of the solid is $\gamma = 2.36$ g/cm^3. The gypsum content is 93.6% and its pH = 3.7.

The maximum density achieved on compacting with the SPT energy is $\gamma = 1.43$ g/cm^3, corresponding to an optimum moisture content of w = 19.1%. and the maximum density obtained when the Huelva phosphogypsum is compacted with the MPT energy is $\gamma = 1.56$ g/cm^3, corresponding to a moisture content of w = 16.9%.

The unconfined compressive strength of the samples prepared with the SPT energy is qu = 0.147 MPa, and for the samples prepared with the MPT energy is qu = 0.340 MPa.

In consolidated and drained triaxial tests the angle of friction obtained was $\varphi = 50°$ with a cohesion of c = 0.067 MPa in a sample prepared with the SPT density while an angle of friction of $\varphi = 54°$ and a cohesion of c = 0.050 was obtained in a sample prepared with the MPT dnsity.

The compressibility of the Huelva phosphogypsum obtained in oedometer tests on compacted samples with a density of γ = 1.57 g/cm^3 is low for pressures up to $\sigma = 1.4719$ kg/cm^2 and scarcely any deferred settlement occurs, the deformability increasing from this pressure upwards.

The permeability of the phosphogypsum compacted with SPT or MPT energy is low, corresponding to a permeability of K = 4.8 . 10^{-5} sm/s for the former and K = 2.3 . 10^{-5} cm/s for the latter.

The General Technical Specifications for Road and Bridge Works in Spain (PG-3) states that when the potential fill material has a gypsum content higher than 20% it should not be used in any part of the fill unless no other materials exist. Consequently, phosphogypsum may only be utilised for environmental reasons or on account of force majeure.

The phosphogypsum may only be placed in the core of fills, with the foundation, crown and shoulders sufficiently compact to guarantee that water will not penetrate, with the core base lying above the level of any water flooding in the vicinity of the fill and installing a layer of gravel to cut off the rise of water by capillary action from the foundation.

Taking into account the specifications of the Spanish PG-3 for slopes and the results of the lab tests on the Huelva phosphogypsum, particularly the Proctor tests, the angle of friction obtained through triaxial tests and the deformability obtained through oedometer tests, and bearing in mind the results of the experimental slopes executed by the Laboratoire des Ponts et Chaussées, recommendations are given for the possible use of the Huelva phosphogypsum in fills.

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