Comparative Study of Spanish Norms Used to Quantify Gypsum Content in Civil and Building Construction

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INTRODUCTION

The "Pliego de Prescripciones Técnicas Generales para Obras de Carreteras y Puentes (PG3)" is the Spanish's Government Technical Instruction that stablishes the properties that materials used in road and bridge construction must accomplish, and includes the corresponding standardized norms to test these properties.

During road construction works, the materials excavated from new cut slopes or nearby excavations are usually used as part of the road basement layers. Gypsum content in these materials is limited to <1% weight (wt) according to PG3, in order to avoid road subsidence or a decrease in the roadbed resistance due to dissolution processes (Vegas et al., 2008). In addition, recycling of construction and demolition wastes has been proposed as an alternative source for roadbed material in Spain, given its potential to preserve natural resources, control waste areas and contribute to a sustainable development (Agrela et al., 2011; Mas et al., 2012). Nevertheless, the presence of gypsum is common in these recycled aggregates (Martin-Morales et al., 2011). The amount of gypsum depends on both the waste origin and the process followed in the recycling plant, and usually ranges between 0 and 5% (Agrela et al., 2011 and references therein). If construction and demolition wastes have to be used as recycled aggregates, the presence of gypsum must also be avoided since it could cause concrete expansion (Mas et al., 2012).

AENOR is the Spanish Association for Standardisation and Certification, and publishes the following norms to regulate the determination of sulphate and/or the gypsum content in construction materials: Norm UNE 103 201, to determine the soluble sulphate content in a soil (May, 1996 & erratum April, 2003); Norm UNE-EN 1744-1, to determine the chemical properties of aggregates (March, 1999) and Norm UNE 83963, to determine the durability of concrete and aggressive soils and the amount of sulphate ion (April, 2008 & erratum December, 2011).

The "Centro de Estudios y Experimentación de Obras Públicas" (CEDEX), an official agency of the Spanish Construction Ministry, also published the Norm NLT-115-99, to determine the gypsum content in soils (CEDEX, 1999).

Objective

The objectives of this work are to compare the testing procedures and results obtained from the different existing norms and to verify the reliability of these standards related to the gypsum solubility.

METHODOLOGY

The methodology used to fulfil the previous objectives has consisted on: 1) a theoretical comparative study of the different norms; 2) determination of gypsum content of seven synthetic samples following the procedures described in the different norms; 3) comparison of experimental and theoretical results. Analytical tests have been performed in the GEOMAR laboratories (Barcelona).

RESULTS

Gypsum ($CaSO_4 \cdot 2H_2O$) is a quite soluble mineral whose dissolution follows r.1. Log K^0 value is -4.610 ± 0.260 (Giffaut et al., 2014). According to r.1, solubility of gypsum at 25 ºC and pH 7 is 1.5×10^{-2}mol/kgw (1.0×10^{-2} - 2.5×10^{-2}mol/kgw) or 2.6 g/L (4.2 – 1.7 g/L).

$$CaSO_4 \cdot 2H_2O = Ca^{2+} + SO_4^{2-} + 2\ H_2O \qquad r.1$$

The testing procedures proposed by the different norms basically consist in measuring the soluble sulphate concentration obtained after dissolving sulphate in acid water and weighting the amount of barium sulphate (barite) that precipitates after the addition of $BaCl_2$ to the solution. Therefore, if the only sulphate-bearing material is gypsum, the maximum sulphate content that could be measured will be gypsum solubility. This value, as seen in Fig. 1, is not the same for each norm, given that the S:L ratio used in each norm is not the same.

Table 1 shows the maximum percentage of gypsum that can be measured by each norm. As can be seen, norms UNE 103201 (considering note 2 of p.28) and NLT-115-99 are designed to measure percentages of gypsum as high as 100%; since the S:L ratio used is very low (0.002 g/ml). However, due to the high S:L ratio used in norms UNE 103 201 (if note 2 is not considered, S:L 0.02 g/ml), UNE-EN 1744-1:1998a,b (S:L 0.5 and 1 g/ml, respectively) and UNE 83963 (S:L 0.24 g/ml), these norms would not measure the correct amount of gypsum as the solution reaches saturation before dissolving all the gypsum. This would occur for gypsum contents higher than 1 %wt in case of norm EN 1744-1:1998, higher than 2% in case of norm UNE 83963 and higher than 20% in case of norm UNE 103 201 (if note 2 is not considered).

palabras clave: Yeso, Suelos Estabilizados, Normas UNE	*key words:* Gypsum, Spanish Standards UNE, Soil Stabilization.

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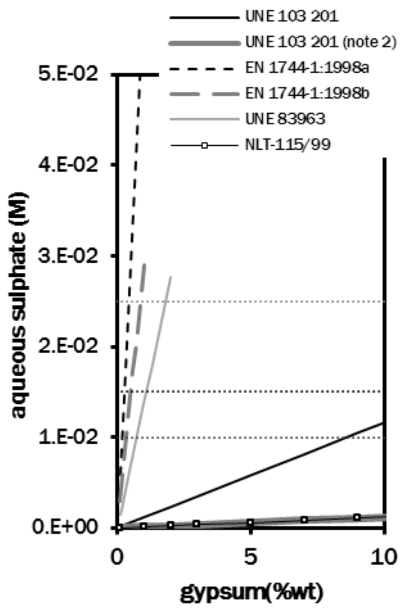

fig 1. Concentration of aqueous sulphate that would enter in solution for different gypsum contents if saturation is not considered. Dotted lines show the range of aqueous sulphate concentration in equilibrium with gypsum according to r.1.

In order to check these predictions, seven synthetic samples were prepared by adding a known value of pure gypsum, obtained from finely grinding a pure gypsum crystal, to a grinded sulfate-free matrix obtained from a siltstone of the OligoceneArtés Formation. The content of gypsum in these samples was 0.1, 0.3, 1.0, 9.0, 25.0, 50.0 and 100.0% wt.

The gypsum content of these samples was measured following the methodologies proposed in each norm, except the norm UNE 103 201 not considering note 2. Results are shown in Fig. 2. As expected, norms UNE 103 201 (considering note 2) and NLT-115-99 did not present any limitation when measuring gypsum contents from 0 to 100% wt. Nevertheless, norms UNE-EN 1744-1:1998 a,b and UNE 83963 did not satisfactory determine the gypsum content for those samples with gypsum contents higher than 1-2 % wt.

In these cases, these norms determined that the amount of gypsum was between 1-2 % wt, the amount of gypsum corresponding to the sulphate analysed in solution. However, this sulphate was the maximum sulphate able to enter to solution before the solution equilibrated with gypsum. The main factor controlling these differences was the S:L ratio derived from the methodology described in the norms.

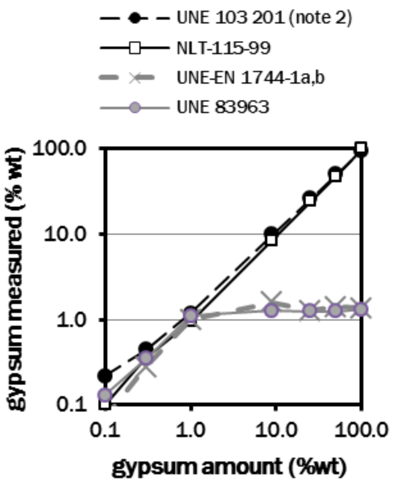

fig 2. Gypsum content (in %wt) determined according to the different norms, of the seven synthetic samples with known gypsum content.

CONCLUSION

Gypsum content has to be determined in materials used in both civil and building construction projects. An excessive amount of gypsum may cause decrease of resistance, subsidence and/or expansion problems. Gypsum content is quantified according to Standard Norms published by Governmental Agencies.

As it has been shown with the theoretical and experimental data presented above, norms using a too high S:L ratio would not provide real gypsum contents if solution is saturated with gypsum before dissolving all the gypsum content of the sample. This occurs at very low percentages of gypsum, between 1 and 2 %wt.

	S:L (g/L)	*maximum % gypsum measurable*
NLT-115-99	0.002	100
UNE 103 201 (note 2)	0.002	100
UNE 103 201	0.02	20
UNE 83963	0.24	1.8
UNE EN 1744-1:1998b	0.5	0.6
UNE EN 1744-1:1998a	1	0.3

Table 1.S:L ratio used in each norm and maximum content of gypsum (in %wt) that can be determined by each norm before solution reaches gypsum equilibrium.

Calculations presented here have been done assuming that gypsum is the only source of soluble sulphate in water. If there are other potential sources, soluble sulphate values measured by these norms, even by those with highest S:L ratio, may be different, depending on the solubility of these other phases.

Therefore, we strongly recommend not using the norms UNE-EN 1744-1:1998 and UNE 83963 to quantify the content of gypsum in soils. We also suggest the use of complementary techniques, such as DRX combined with thin section petrographic analyses.

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REFERENCES

*Agrela, F., Sánchez de Juan, M., Ayuso, J., Geraldes, V.L., Jiménez, J.R. (2011): Limiting properties in the characterisation of mixed recycled aggregatesfor use in the manufacture of concrete. Construction and buildingmaterials, **25**, 3950-3955.*

CEDEX (1999): Normas del laboratorio de transportes NLT (1999): NLT-115/99 Contenido de yeso en suelos. Centro de Estudios y Experimentación de Obras Públicas. Madrid.

*Giffaut, E., Grivé, M., Blanc, Ph., Vieillard, Ph., Colàs, E., Gailhanou, H., Gaboreau, S., Marty, N., Madé, B., Duro, L. (2014): Andrathermodynamic data base for performance assessment: ThermoChimie, Applied Geochemistry, **49**, 225-236.*

PG3. Pliego de prescripciones técnicas generales para obras de carreteras y puentes. Orden FOM/891/04.

*Martín-Morales, M., Zamorano, M., Ruiz-Moyano, A., Valverde-Espinosa, I. (2011): Characterization of recycled aggregates construction and demolition waste for concrete production following the Spanish Structural Concrete Code EHE-08. Construction and building materials, **25**, 742-748.*

*Mas, B., Cladera, A., Del Olmo, T., Pitarch, F. (2012): Influence of the amount of mixed recycled aggregates on the propertiesof concrete for non-structural use. Construction and Building Materials, **27**, 612-622.*

*Vegas, I., Ibañez, J.A., San José, J.T., Urzelai, A. (2008): Construction demolition wastes, Waelz slag and MSWI bottom ash: A comparative technical analysis as material for road construction. Waste Management, **28**, 565-574.*